

# How to Collect Reliable Soil-Gas Data for Risk-Based Applications—Specifically Vapor Intrusion

## Part 4 – Updates on Soil-Gas Collection and Analytical Procedures

by Blayne Hartman

Since I wrote Part 3 of this “Collecting Reliable Soil-Gas Data” series in LUSTLine #48, Nov 2004, the subject of vapor intrusion has continued to be a “box-office blockbuster” throughout the environmental community. The EPA-OSWER draft vapor-intrusion guidance is currently being rewritten and is expected to be released in the first half of 2007. The Interstate Technology and Regulatory Council (ITRC) has written a vapor-intrusion guideline document that is currently out for review and is due to be released in early 2007. ASTM International has entered into the fray and has convened a workgroup to amend the Phase I guidance to include assessment of the vapor-intrusion pathway and to write a standard on how to do it. Individual states continue to release their own policy/guidance on this pathway (e.g., CA, NY, NJ, MO). Many others are working on their own guidance. The concern about vapor intrusion extends to the United Kingdom (U.K.) and Australia. Since soil-gas data are preferred by the majority of agencies in evaluating the pathway, much debate has surfaced over some of the collection and analytical procedures. In this article, I present some of the results from recent studies that address some of the pertinent issues. I refer you to Parts 1 (LUSTLine #42) and 3 in this series for a good introduction to this article and for more information on some of the topics covered herein.

### The Two Most Common Errors

Vapors and vapor intrusion are unfamiliar territories for many practitioners in this field (regulators, stakeholders, consultants, subcontractors, attorneys). Here are two of the most common errors that people make with soil-gas programs or data.

- **Confusion with Units**

One common error that people make with soil-gas programs or data is thinking a ppbv is equivalent to a  $\mu\text{g}/\text{L}$  or a  $\mu\text{g}/\text{m}^3$ . The units are not equivalent, and the conversion depends on the molecular weight of the compound. Converting between units (e.g.,  $\mu\text{g}/\text{L}$  to  $\mu\text{g}/\text{m}^3$ , percent to ppmv) is also causing headaches. As I advised in Part 3, make your life simpler by:

- Instructing your lab in what units and detection levels you want the data reported.
- Going to [www.handpvm.com](http://www.handpvm.com) for a handy-dandy and easy-to-use unit conversion spreadsheet.

- **Required Soil-Gas Target Levels**

The other error I see too often is the regulator or consultant using incorrect soil-gas target levels. Residential values are erro-

neously applied at commercial sites, incorrect attenuation factors are being used to determine target values, or values determined from predictive models are incorrect. The soil-gas target level ultimately determines the required analytical method and the need for additional assessment. Determining the proper value is often an unfamiliar exercise for both regulator and consultant. So, consultants need to ensure that regulators are asking for the proper values, and regulators need to ensure that consultants are proposing the proper value.

### Sample-Collection Issues

#### Probe Installation Method

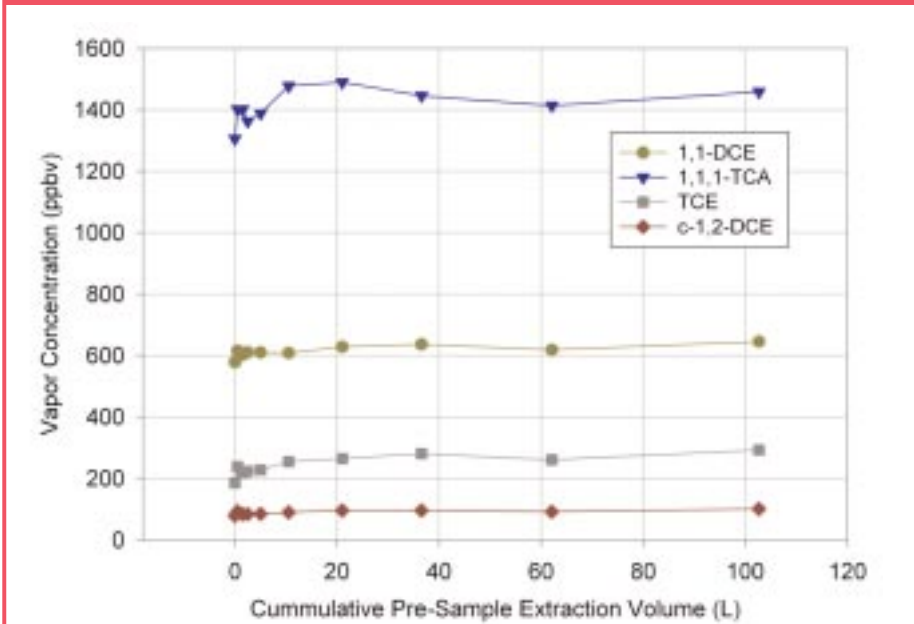
I have not seen a significant difference in results among samples collected either through the probe rod while the rod is still in place or through tubes that are buried in the ground after the rod is removed. This observation is based on data from hundreds of sites where we have made repeated measurements using different methods. While I have never compiled these data, a recent report by U.S. EPA (DiGiulio et al. 2006a) provides data that addresses this very topic. Although small differences were detected, the study showed that data collected using

hand-driven probes, direct-push rods, and buried tubes show good agreement, generally about the same as analytical precision.

The bottom line is that the probe installation technique does not matter so long as you do it right. So, the choice of which method to use should depend upon the site, access, and project goals. Typically, sampling through the probe rod is faster and less likely to disturb the insitu soil gas. For limited-access areas, a hand probe may be all that is applicable. For deeper depths, direct-push probes are more convenient. For repeated sampling, burial of small-diameter tubes offers advantages. If the probe-rod methods are used, samples should be collected through small-diameter inert tubing that runs down the probe rod so the sample does not contact the inside of the probe rod.

#### Extraction Volume

Three published studies are now out that compare soil-gas concentrations collected from volumes ranging from 0.5L to 100L (DiGiulio et al. 2006a; McAlary and Creamer, 2006; DiGiulio et al. 2006b). The results of these studies, done in relatively coarse-grained soils, show no significant difference in concentrations. (See Figure 1.) I have reviewed data from countless sites comparing on-site analysis from 50cc

**FIGURE 1 Soil Gas Concentration vs. Volume Extracted**

No significant effect is seen on volumes from 500 c (0.5L) to over 100L. Study conducted by EPA-ORD at an EPA test site. (DiGiulio et al. 2006a.)

syringe samples to off-site analysis done on samples collected in canisters (1L to 6L). I rarely see differences greater than 20 percent.

However, in finer-grained soils, large volumes are often not possible or difficult to collect. If larger sample volumes are attempted, the potential for leaks around fittings increases. I also have witnessed higher concentrations where large volumes are "forced" from tight soils, presumably due to contaminant desorption off the soils.

Finally, the larger the volume extracted, the greater the uncertainty of where the sample is located. The more complex the sampling system, the greater the chance of drawing air from the surface and the longer it takes to collect a sample. These factors increase the potential for sampling errors, nonrepresentative values, and increased costs. It is best to collect a sample volume that is no larger than that required by your lab and no larger than 1L.

#### Sample Flow Rate

Many state agencies have put a limit on sample flow rate (typically <200ml/min) because they are concerned that excessive flow might create turbulent flow at the probe tip and influence the soil-gas concentrations. DiGiulio et al. (2006b)

addressed this topic using airflow modeling and concluded that this general limit was reasonable. However, McAlary and Creamer (2006) actually measured soil-gas concentrations over different flow rates ranging from 100ml/min to 100L/min at a hydrocarbon contamination site. They saw no significant difference in measured concentration. This suggests that for relatively coarse-grained soils, flow rate does not appear to be an important variable on soil-gas concentrations. Allowing faster flow rates increases sample throughput and eliminates the cost and potential blanks of additional hardware (e.g., flow restrictors).

#### Tubing Type

Two studies have been done to evaluate different types of tubing. Air Toxics (Hayes et al. 2006) conducted tests of three tubing types (Teflon, nylon, PEEK) that showed little difference in the tubing type. Low-level blanks were detected in nylon, but the values were far below required soil-gas risk-based screening levels. An earlier study presented at a conference in 2004 (Ouellette, 2004) compared the adsorption of a hydrocarbon standard by five tubing types (Teflon, nylon, polyethylene, vinyl, and flexible tygon). Nylon and Teflon showed insignificant losses (<10%),

but the others showed higher losses, especially the flexible tubing, where losses were up to 80 percent.

Flexible tubing should be avoided. For rigid-wall tubing, in practice, the type of tubing is not nearly as important as where the tubing is stored and how it is handled. Any type of tubing will become contaminated and contribute to false positives if it is stored in the back of a truck unsealed or near the truck exhaust. My preference is 1/8" nylon tubing, which is easier to work with than the 1/4" tubing if soil-gas sampling is your only need. It has a smaller dead volume and is much less expensive than Teflon.

#### Tracer/Leak Compound

Most agencies are now requiring that a tracer compound be used to ensure there are no leaks around the installed probe and/or the soil-gas sampling train. There are methods using gases (e.g., helium, propane, SF<sub>6</sub>, freon) or liquids (e.g., freon, isopropanol, butane in shaving cream). Both types of tracers have pros and cons.

Gaseous tracers offer some advantages, but they are more of a pain logistically, especially if you are trying to cover leakage in the sampling train as well. Plus you need tanks, regulators, and other hardware. The entire process becomes much more cumbersome and time-consuming, resulting in higher sample-collection costs.

Helium offers a nice advantage in that it is readily measured on-site with a field meter, but due to its small molecular size, helium more readily permeates sampling materials than larger molecules typical of VOCs, so it may indicate a leak when there really isn't one.

Volatile liquid tracers offer logistical simplicity and accomplish the primary goal: detecting any leaks in the probe or sample train. The tracers are easily and quickly supplied at multiple locations (probe, sampling rod, and sampling train) simultaneously using paper towels or clean rags. This method is particularly better suited for sampling through the probe rod since it can be applied at the base and top of the rod.

This method is also qualitative since the concentration at the point of

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application is typically not measured (although it can be). One disadvantage is that small leaks (as low as 100µg/L) can cause a lab to raise its their detection levels depending on the tracer compound, especially if the toxic organic (TO) methods are being used. When on-site analysis is used, leaks can be found in real time and samples can be recollected as necessary.

With deeper soil-gas samples (>3 feet bgs), the likelihood of surface air being drawn down the length of the probe and into the sample is small, especially if small (<1L) volumes are collected. I suspect that most detected leaks are from poor sample collection procedures and poor tracer application procedures. To minimize these issues, collection personnel should keep sample volumes small and collection assemblies and protocols as simple as possible.

Finally, it is important to recognize that a small amount of tracer in a sample does not indicate a significant leak. If the concentration of the tracer indicates a leak of less than 10 percent, then the leak should be considered insignificant and the sample should be considered valid. For liquid tracers such as isopropanol, a 10 percent leak would give a value in the sample of ~10,000µg/L, assuming a starting concentration equivalent to the vapor pressure of isopropanol.

It is best not to limit your options to any one method. Let the choice be made by the soil-gas collector for a given site, based on his or her comfort with either method, the availability of on-site analysis, and the compounds of concern.

### Equilibration Time

In the process of burying sampling tubes in the ground, in situ soil gas is displaced and the tip is buried in a sand pack that contains atmospheric air. How long does it take for the sand pack to equilibrate with the surrounding soil gas? DiGiulio et al. (2006b) calculated and plotted equilibration times for different distances and soil water contents. For a 2" outer diameter borehole, the plot shows a required time of a few minutes to less than a few hours. A recent

test performed at a U.S. EPA test site in Indianapolis showed that the sand pack equilibrated within three hours for a 1.5" borehole. In practice, I advise clients to include the volume of the sand pack in their purging if samples are collected the same day as installation, but not to include the volume of the sand pack if samples are collected on a different day.

### Temporal Effects

This is a big issue for regulators and stakeholders. Do we need repeated sampling events, similar to monitoring wells? There have been a number of studies on this issue, and more are currently under way or planned. Dr. Thomas McHugh of Groundwater Services recently conducted a research program for the Department of Defense and saw variations of chlorinated hydrocarbon concentrations in shallow soil-gas samples of less than 30 percent over a 48-hour period (McHugh 2005).

Todd McAlary of Geosyntec has shown results from a site in the U.K. with hexachlorobenzene variations over seasons less than 40 percent (McAlary, 2002). Data from a site in Endicott, New York, presented by Dr. William Wertz of the New York Department of Environmental Conservation, show *maximum* variations of chlorinated hydrocarbon concentrations in shallow soil gas (~5 feet bgs) over a period of 16 months (8/04 to 12/05) of a factor of four, but typically less than a factor of two. The variation from the average or mean concentration is far lower.

At a site in Casper, Wyoming, Dr. Paul Johnson and others have monitored soil-gas concentrations of hydrocarbons using an auto-analyzer (Luo et al. 2006). Variations in soil gas at four feet or deeper were less than a factor of two over a period of 70 days from September to December. Variations in sub-slab samples were on the same order except near cracks. This study is still on going, and a companion study is planned for a hydrocarbon site in Ohio. I have seen little temporal variation in southern California based on repeated sampling at thousands of houses with methane in the shallow soils. Unfortunately, there is no compiled database.

U.S. EPA just awarded a contract to TetraTech to study temporal varia-

tions at a chlorinated hydrocarbon site. The testing is to be conducted this fall/winter and the results will be reported in 2007.

Looking at the big picture, the soil-gas concentration variations in these datasets, even for northern climates, are insignificant compared with the overprotectiveness of the risk-based screening levels. I suggest that if soil-gas values are a factor of five to ten times below the risk-based screening levels, there is no need to do repeated sampling unless a major change in conditions occurs at the site (e.g., elevated water table).

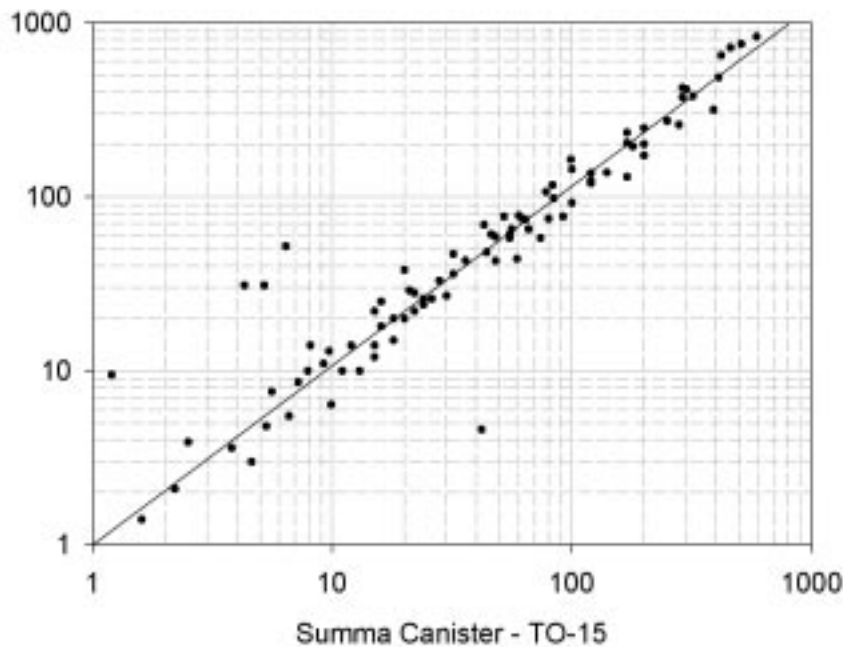
### Sub-slab vs. Exterior Soil-Gas Samples

In another hotly debated issue, some agencies are concerned that exterior soil-gas samples may not be reflective of soil-gas concentrations under the slab, and they are struggling to decide what to allow in their guidelines. Going inside structures, especially personal residences, can be a Prozac moment, so there is strong incentive to try to use exterior soil-gas concentrations to assess the vapor-intrusion risk. U.S. EPA-OSWER, based on limited data and some recent 3-D modeling, is feeling that deeper, exterior soil-gas data (10' to 15' below the receptor) may be more representative of concentrations under a slab than shallower soil-gas data. Currently, there is no comprehensive database to reach a definitive conclusion.

For petroleum hydrocarbons, a significant amount of bioattenuation can occur in the upper 10' (see Davis, 2006, LUSTLine #52 for a good summary), so collecting only deeper samples would not give an accurate picture of the vapor-intrusion risk. For chlorinated hydrocarbons, bioattenuation is not as prevalent, so collecting deeper samples may be justified if the contamination source is directly below. If the source is spatially away from the receptor, exterior soil-gas concentrations on the side of the receptor will likely be higher than concentrations below the receptor.

I recommend that for hydrocarbons, shallow (3' to 5' bgs) exterior soil-gas samples around the receptor should be representative of sub-slab samples if oxygen levels are high (>6%) and the contamination source

**FIGURE 2 A Comparison of On-Site Analysis of TCE by 8021 out of a Tedlar Bag vs. Off-site Analysis by TO-15 Out of a 6 Liter Summa Canister Collected by EPA-ORD at a Test Site**



*The correlation is excellent down to values as low as 2 ppbv. Units for both axes are ppbv. (DiGiulio et al. 2006a.)*

is not too high (<50,000 $\mu\text{g}/\text{L}$  in the soil vapor, based on 3-D modeling results by Lilian Abreau, 2006). For chlorinated compounds, if the source is below the structure, collect samples around the receptor as close to the source as possible. The sub-slab concentration can be no higher than the source concentration (e.g., immediately above the saturated zone if groundwater is the source). If the source is so deep that samples can not be readily or inexpensively collected, collect vertical profiles to at least the mid-depth as discussed in Part 3 of this series. If a consistent pattern is obtained around the structure you can likely safely extrapolate the data to below the structure. If not, it will be time to go inside.

## Sample Analysis Issues

### Use of Tedlar Bags for Soil-Gas Samples

A number of published studies (e.g., Denly & Wang, 1995) have been done over the years on the stability of compounds in tedlar bags. All of the studies I've seen, and some tests we've done, show the bags are reliable for the common VOCs for storage times of 24 to 36 hours (less than

15% over 24 hours), but the fall-off gets greater after 48 hours (30% to 40%). The other primary concern is blanks from the new bags. New tedlar bags can have low levels of VOCs in them. However, they are typically less than 10 $\mu\text{g}/\text{m}^3$ , which is also below most soil-gas risk-based screening levels (Hayes et al. 2006).

Sample collection in tedlars offers some sampling advantages. They are easier to handle and less expensive, spares are readily available, they eliminate the potential of blanks from dirty canisters, and there are a variety of simple ways to fill them. In tight soils or soils with high water contents, I prefer that samples be collected in tedlar bags to avoid the potential for leaks at fittings and water being sucked into the sampling system or canisters. If the samples cannot be analyzed within 24 or 48 hours, the sample can easily be transferred into a canister either in the field or when received by the laboratory.

I suggest that you consider using tedlar bags when collecting soil gas samples. Depending on the allowed storage time, either ship samples to the lab overnight for them to transfer or analyze, or transfer them into can-

isters in the field. For high-profile projects or projects in later stages, stick to the 24- to 36-hour storage time. For initial assessments or projects where a potential 40 percent error is not of concern, use the 48-hour storage time.

### TO-14/15 Analysis Method: The Gold or Plastic Standard?

If you're a regulator who believes the TO-15 method is the gold standard of VOC analytical methods, you're in for a shock when you read this section. Before I begin, it is important that you understand that my firm conducts both 8260 and TO-15 analyses, so I am not writing this because I have a conflict of interest or a bias toward either method. My purpose in writing this is to inform you of a number of QA/QC deficiencies in the standard method so you can ensure that the TO-15 data you are getting are of sufficient quality. This is of growing concern as the number of laboratories offering TO-15 is rapidly increasing to meet the vapor-intrusion market.

In truth, QA/QC criteria in the standard TO-14 and TO-15 methods are below the requirements of U.S. EPA SW-846 VOC methods (e.g., 8021 and 8260). A few states have realized this (e.g., NJ, NY), and their laboratory certification groups have stipulated additional QA/QC requirements for the TO methods (some EPA regions have done the same although they do not certify labs). Following are four of the most pertinent issues.

- **Lack of a Second-Source Standard.** The TO-15 method does not require a second-source standard. Second-source standards are required by the SW846 VOC methods and by most state standards (e.g., Cal-EPA/DTSC soil gas advisory) to be analyzed after the initial calibration to validate the calibration curve, and in some instances, daily with each batch as a laboratory control sample (LCS). Without a second-source standard there is no validation or check that the calibration standard is accurate.

The lack of this requirement for the TO method becomes even more shocking when you realize that the method allows use of the same standard for up to a year!

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Further, there are no criteria on the canister type for the standard. In other words, a lab is fully method compliant if it uses only one calibration standard stored in an aluminum cylinder for up to a year.

In contrast, most states only allow samples to be held in polished or glass-lined canisters for up to 14 days (Cal-EPA/DTSC only allows 3 days). You can see the obvious contradiction that the same hold-time criterion for samples is not applied to the single-calibration standard. So, how do you know your standard is still good after six months if you are not checking it?

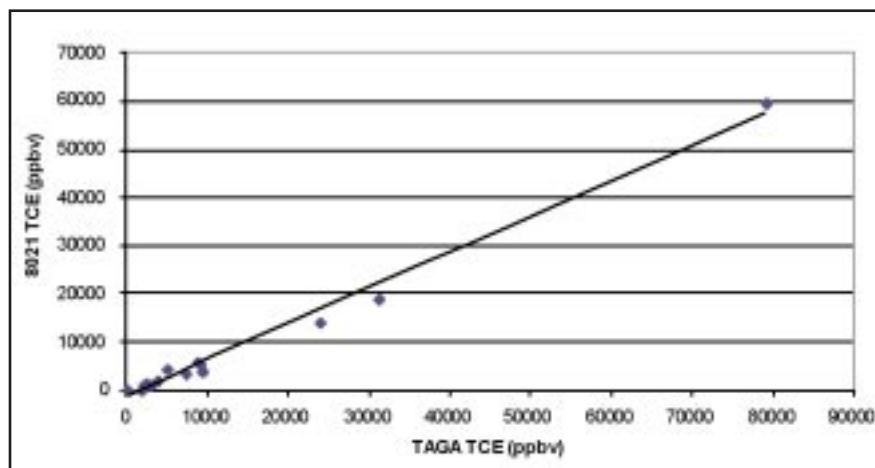
I suggest that states require a second-source standard analysis with the initial calibration and each analytical batch and require this analysis to be part of their data report. Further, if states are interested in naphthalene, they should require labs to have naphthalene standards, since they are not part of the standard TO-14 or TO-15 calibration mix (some labs report naphthalene based on the calibration of a similar compound).

- **Lack of Surrogates.** The standard TO-15 method does not require surrogates. The SW-846 VOC methods require surrogates within lab-derived recovery ranges. Surrogates are used to give an indication if a sample ran "properly." If the surrogates aren't measured within an acceptable range, say +/-30 percent, then the results for the other compounds are considered suspect. Without surrogates, you have no information as to whether sample recovery was 10, 50, 100, or 200 percent. Quality labs reanalyze samples if the surrogates do not report within the acceptable range.

It is best to require surrogates (at least three) in all samples. Require that surrogate recoveries be reported on each analysis with recovery acceptance limits similar to SW-846 methods.

**FIGURE 3**

### TAGA vs. 8021 TCE Concentrations



*A comparison of on-site analysis of TCE by method 8021 from 60cc syringe samples to on-site analysis by the EPA TAGA mobile laboratory from 1L tedlar bag samples collected at an EPA test site in Indianapolis in August 2006. The correlation is excellent ( $R^2 = 0.998$ ) and the values agree within analytical precision.*

- **Calibration Acceptance Windows.** The standard TO-15 method calibration standard recovery windows for some compounds are larger than the SW846 VOC methods and do not meet some state soil-gas guidelines (e.g., Cal-EPA/DTSC). The solution to this problem is to ensure that the laboratory's calibrations fulfill any state requirements and require that the continuing calibration analysis be included in the lab report.

- **Lack of Certification (Beware the "Wal-Mart" TO-15).** As the vapor-intrusion market increases, so too do laboratories offering TO-15, especially since the analyses command three to four times the price of soil and water VOC methods. The TO-15 QA/QC criteria are less stringent, and most states don't have a certification for the method, so it is very easy for a lab to quickly set up and offer TO-15 analyses. In other words, a lab can be running the method with no regulatory oversight/checks on whether the lab ever could or is currently running the method properly. I recently reviewed a lab report in which a lab posted its state certification number on its TO-15 lab report even though the state didn't certify the method!

**Strong Recommendation:** To ensure that you are getting a quality analysis that can withstand both the regulatory and legal challenge, use only a lab that can show it has upgraded the method QA/QC as described above and has a certification for the method from either NELAC or a state agency. Most high-quality laboratories have upgraded the published TO-15 method to correct the issues raised above.

#### **The Ongoing Debate on VOC Analysis by TO-14/15 vs. Methods 8260 vs. 8021**

This debate has been going on for many years now and much confusion and controversy still exist. There have been two recent presentations and published papers comparing method TO-15 to method 8260, one by Air Toxics (Hayes et al. 2005) and one by my company (Picker, 2005). Both studies reached the same conclusion: the methods match up quite well for the common VOCs. However, the jury is still out regarding naphthalene.

Figure 2 shows a comparison of on-site analysis of TCE by method 8021 out of a tedlar bag to off-site analysis by TO-15 out of a 6L Summa canister from a U.S. EPA test site (DiGiulio et al. 2006b). The correlation is excellent down to values as low as 2 ppbv.

Another comparison study was done this August at a U.S. EPA test

site. The values I measured on-site by method 8021 from 60cc syringe samples matched analysis by EPA's mobile Trace Atmospheric Gas Analyzer system from tedlar bags within analytical error. (See Figure 3.) We see similar agreement between on-site analysis using method 8260 from 60cc syringes and samples collected in 6L canisters and analyzed off-site by TO-15.

These studies prove that the soil and water VOC methods and the TO methods give equivalent results down to levels as low as 10µg/m<sup>3</sup>. The decision on what analytical method to use should be based primarily on the required detection level, expected contaminant level, project scope, and cost...in this order.

The TO methods and hardware (e.g., canisters, flow chokes, adsorbents) are designed for measuring low levels in ambient air. They are not designed for the high concentrations we commonly see in soil gas. Typical soil-gas concentrations at LUST, dry cleaner, and industrial-solvent sites are in the 100,000s to 1,000,000s of µg/m<sup>3</sup>. High concentration samples can lead to system carryover, large dilutions, and contaminated canisters, increasing the potential for false positives, raised reporting levels, and other logistical problems, such as canister management. Due to these potential problems, programs using off-site analysis should include canister trip-blank samples and sampling equipment blank samples.

In practice, a combination of these methods is the best approach. Most soil-gas risk-based screening levels can be reached with all of these methods. If expected values are high, then the 8021 and 8260 methods are more advantageous to use than the ultra sensitive TO methods. If expected values and risk-based screening levels are low, then the TO methods offer advantages. Further, the 8021 and 8260 methods can be run in the field, allowing real-time information. Refer to Part 3 for a summary table of the available methods.

### The Need for TO-15 SIM for Soil-Gas Samples

Too frequently, we get asked for TO-15 SIM analysis for soil-gas samples. TO-15 SIM (selected ion mode) is used to get to lower detection levels

(< 1µg/m<sup>3</sup>) than the typical TO-15 SCAN analysis (1 to 5µg/m<sup>3</sup>). But for almost all compounds at any collection depth, including sub-slab, soil-gas risk-based screening levels are higher than 5µg/m<sup>3</sup>. So TO-15 SIM is not necessary. Save your client (or the state reimbursement fund) the extra expense.

### Experience Goes a Long Way

A final topic of concern among the regulatory community is the spatial variability of soil gas, both around structures and under structures. In many cases, soil gas, like soil, is not homogenous. We have accepted this fact about soil data and have adjusted our site investigation methods, sampling plans, and interpretation accordingly. But for some reason, we are not yet comfortable with soil gas variability. Many of the interpretation problems I see people dealing with come from a lack of data. It's simply not possible to sort out the variations with a handful of analyses collected on a couple of occasions.

Since some variability is to be expected, you need enough data to give decent coverage near, around, and under the receptor. I encourage simpler collection systems that enable higher production per day (>20 samples per day) and the use of less expensive analytical methods (e.g., 8015, 8021, 8260) enabling more analyses for the same cost. I also encourage field analysis, when possible, as it allows for real-time decisions on additional sampling needed to sort out variations and recognition of inconsistent data and tracer leaks while you are still in the field.

The last important ingredient needed for high-quality, cost-effective, and efficient vapor-intrusion investigations is the experience of the consultant and the subcontractors. I advise consultants to use firms experienced in soil-gas collection and use labs experienced in soil-gas analysis. The stakes are simply too high with vapor intrusion to do anything else. ■

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