Long-term Integrating Samplers for Indoor Air and Subslab Soil Gas at VI Sites
Paper Number

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ABSTRACT:

Vapor intrusion (VI) site assessments are plagued by substantial spatial and temporal variability that makes exposure and risk assessment for volatile organic compound (VOC) exposure difficult. The most common approach to indoor air assessment in the industry is comparison of a limited number of one-day integrated SUMMA® canister data to chronic health benchmarks based on 1 to 30 years of exposure. In many cases, the pressure driven entry flow rate of VI is directly proportional to the differential pressure across the slab. Numerous studies of differential pressure across building slabs, including ours, show variance across several different time scales (transient, diurnal and seasonal. Thus, passive sorbent systems that provide a two-week integrated measurement of volatile organic compound (VOC) concentrations may be a superior VI assessment tool. Studies were recently conducted at two sites in which the performance of passive samplers was compared to SUMMA® canister measurements. Radiello samplers (both solvent extracted and thermally desorbed) as well as Chromosorb 106 passive tubes were tested. Radiello samplers showed good correlation to the conventional SUMMA® canister approach for chlorinated VOCs.

Subslab soil gas sampling methods for VOCs normally collect small volumes actively over 2 to 15 minutes (rarely 24-hour periods). The variance in differential pressure alters the flow direction across the slab, potentially dramatically changing the concentration in the immediate subslab region near cracks. Existing passive soil gas sampling methods are not considered to be quantitative because of the difficulty of establishing diffusion and thus uptake rates, when the sampler is in direct contact with soil. Proof of concept experiments have been conducted at two sites on an innovative subslab soil gas sampler. This sampler couples passive media that give good performance in indoor environments with a simple chamber refreshed by a continuous flow of subslab soil gas. The sampler integrates over both time and space. The innovative integrating subslab sampler agreed well with conventional measurements for Radon. The integrating sampler results were consistently somewhat lower than conventional measurements for VOCs.

INTRODUCTION

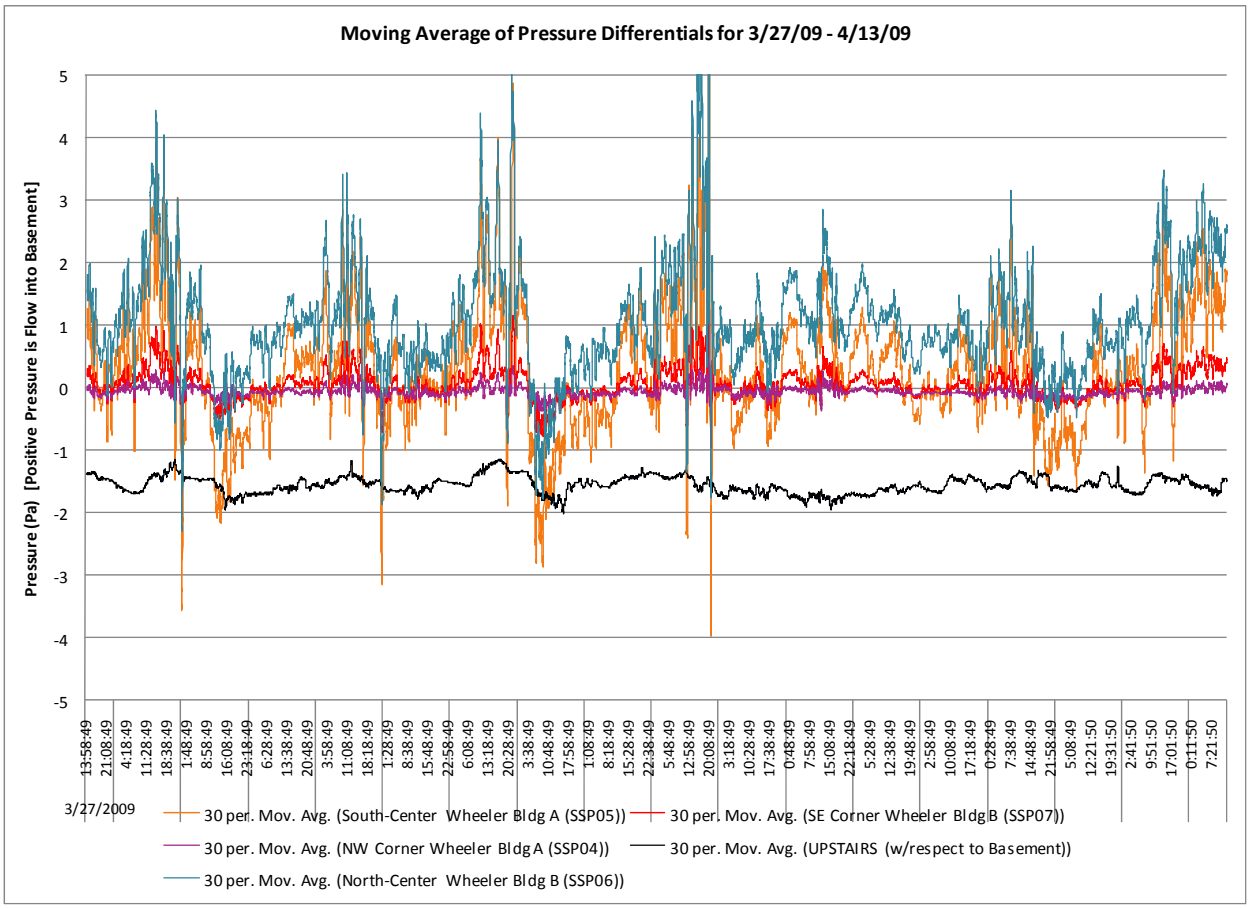
This paper discusses and demonstrates the application of long term passive samplers for both indoor air and soil gas. First we discuss temporal variability, current uses of passive samplers and current approaches to subslab sampling. We then present test methods and results at two sites, using passive sampling for indoor air as well as in an innovative integrating sampler for subslab soil gas.

Temporal Variability of Vapor Intrusion

Vapor Intrusion (VI), which is the migration of subsurface vapors, primarily volatile organic compounds (VOCs), to indoor air, has emerged as a priority contaminant pathway at hazardous waste sites nationwide. Current practice for evaluating the VI pathway consists of a combination of mathematical modeling and direct measurements in groundwater, external soil gas, subslab soil gas and indoor air. Modeling, though useful as a way to integrate measurements in multiple media, is not considered sufficiently accurate to predict risk on a building-specific basis due to subsurface and building uncertainties. No single line of evidence is considered definitive and all of the measurements are costly. Moreover, these measurements have temporal variability on various time scales, requiring repeated measurements to accurately assess the chronic risks of long term VOC exposure.

VI occurs due to the pressure and concentration differentials between indoor air and soil gas. Indoor environments are often negatively pressurized with respect to outdoor air and soil gas. This pressure difference allows subsurface vapors to migrate into indoor air. Vapor and liquid transport processes and their interactions with various geologic and physical site settings (including building construction and design) under given meteorological conditions, control migration through the VI pathway. Variations in building design, construction, use and maintenance, site-specific stratigraphy, subslab composition, temporal variation in atmospheric pressure, temperature, precipitation, infiltration, soil moisture, water table elevation and other factors combine to create a complex and dynamic system. ARCADIS and U.S. Environmental Protection Agency's (EPA) National Risk Management Research Laboratory (NRMRL) have acquired long-term pressure differential datasets at an Indianapolis study site at which both radon and VOCs are being measured in both subslab and indoor air. Figure 1 (approximately 17 days of pressure data) illustrates how the driving force for VI can be essentially absent one entire day, yet very strong the next day. We have not been able to identify a cause for these pressure variations in this building that would have allowed a practitioner to predict the most conservative day to sample.

Figure 1. Differential Pressure Variation over 17 Days in an Indianapolis Building



Passive/low-flow VOC Sampling

Passive sorbent-based methods are emerging technologies for VI assessment in the U.S. Current standard practice for indoor air VOC monitoring in the U.S. includes the use of negatively-pressurized, ultra-clean, stainless-steel canisters for sample collection. Practitioners frequently use 4- to 48-hour integrated samples with SUMMA® canisters in an attempt to average over an exposure period. This is the U.S. "gold standard" for indoor air analysis, but is expensive to implement. Professional experience shows that the flow controllers currently used in commercial practice are subject to substantial flow rate and final pressure errors when set for integration times in excess of 24 hours.

Active and passive sorbent sampling techniques are already in use in the U.S. for personal air monitoring for industrial workers. Samples are collected for up to 10 hours duration and are typically evaluated against occupational exposure levels [(as an example, see the National Institute for Occupational Safety and Health [NIOSH] Manual of Analytical Methods (http://www.cdc.gov/niosh/nmam/)]. However, the sampling and analysis detection limits of these methods are generally above EPA risk-based levels. Active sorbent methods (i.e., TO-17) have also been published by EPA for ambient air.[1] In those methods, air samples are normally

actively collected over one hour, with a sampling rate of 16.7 milliliters (mL) per minute (min) to 66.7 mL/min yielding total sample volumes between 1 and 4 liters (L).

One way to lower the detection limits and control day-to-day variability is to sample over a longer period of time. Recent studies have shown that it may be feasible to collect a continuous indoor air sample over several weeks. This approach would provide a lower detection limit, be cost-effective, and result in a time-integrated composite sample. Theoretical, laboratory and field tests of such an approach for occupational health and indoor air applications have been published.[2, 3] A previous test at Moffett Field, California,[4] showed that a short time badge-type passive diffusion sampler agreed well (3-37% Relative percent difference (RPD)) with SUMMA® canister measurements and provided better detection limits. Figure 2 and Table 1 compare the characteristics of some of the available passive samplers to more conventional techniques. The thermally desorbed models provide the lowest detection limits while the stronger sorbents of the solvent extracted system provide longer sampling times and greater dynamic range due to greater sorbent capacity and the ease of making multiple analytical dilutions.

Figure 2. Reporting Limit for Trichloroethylene (TCE) for Various Methods, In the Absence of Interferences, Based on Varying Sampling Durations

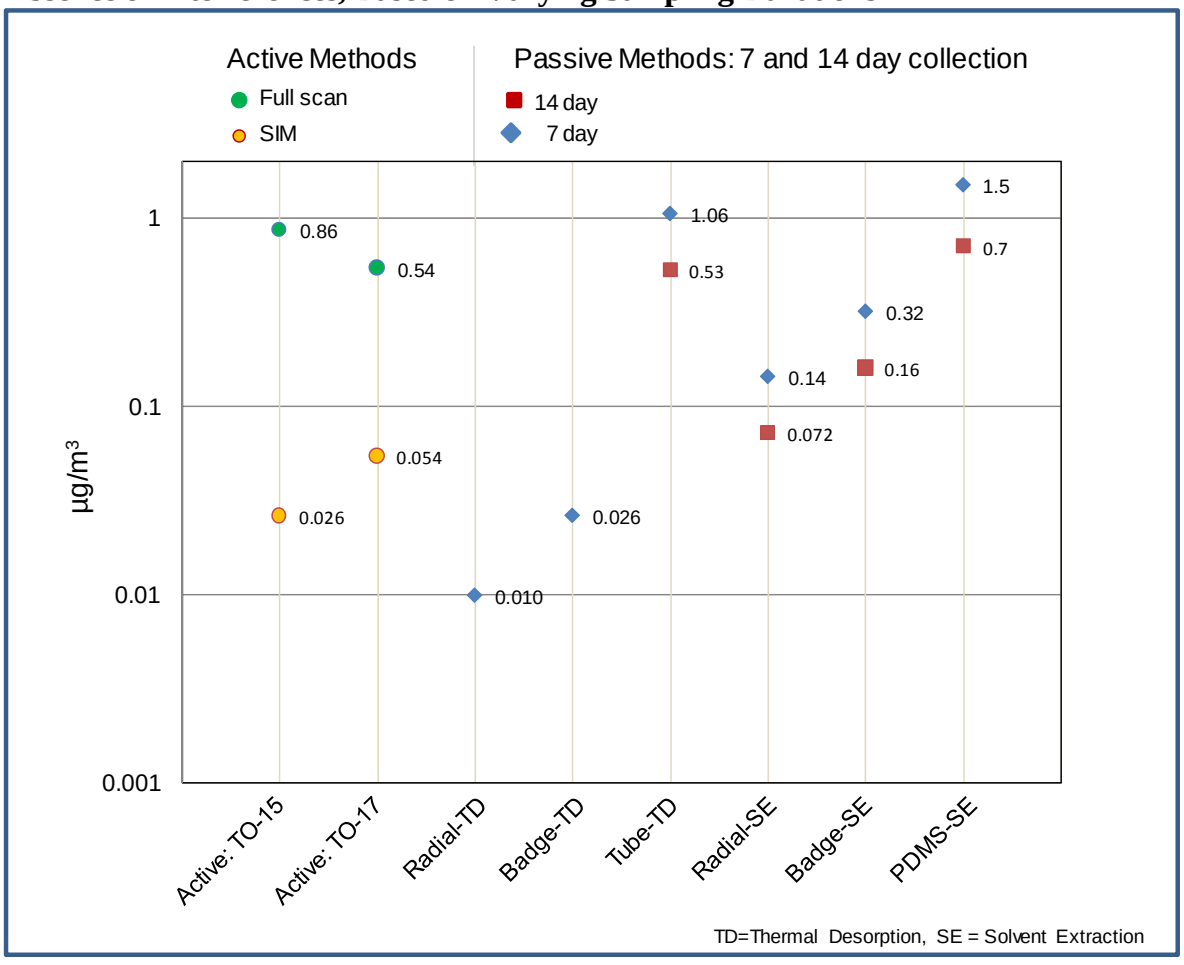


Table 1. VOC Indoor Air Sampling Method Options

	Whole Air	Sorbent-Active Sampling	Sorbent-Diffusive Sampling					
Collection Media	SUMMA® canister	Multi-bed automatic thermal desorption (ATD) tubes	Radial: charcoal (Radiello 130)	Radial: Carbograph 4 (Radiello 145)	Badge (SKC 575, 3M OVM3500): Charcoal type	Badge (SKC Ultra I, II, III AirLab™): Various thermally desorbable sorbents	Tube-style: various thermally desorbable sorbents	Permeable Diffusion Membrane Sampler (PDMS) Membrane (WMS™): Charcoal type
Method and Analysis	TO-15 GC/MS	TO-17 GC/MS	Solvent extraction GC/MS or GC/FID	TO-17 GC/MS	Solvent extraction GC/MS or GC/FID	TO-17 GC/MS	TO-17 GC/MS	Solvent extraction GC/MS
Sample Collection Period	24 hours to 7 days	8 – 24 hours	30 days	7 days	30 days	7 days	30 days	30 days
Estimated Sample Reporting Limits – ***Normalized to a 7-day period for diffusive samplers***	~0.05 (SIM mode) to 1 $\mu g/m^3$	~0.05 (SIM mode to 1 $\mu g/m^3$	~0.2 to 0.4 $\mu g/m^3$	~0.005 to 0.05 $\mu g/m^3$	~0.25 to 2 $\mu g/m^3$	~0.01 to 0.1 $\mu g/m^3$	~0.2 to 2 $\mu g/m^3$	~1 to 40 $\mu g/m^3$

European agencies have developed standard methods for passive sampling for VOCs that are applicable to the range of concentrations and durations that are relevant to environmental practitioners:

- Methods for the Determination of Hazardous Substances (MDHS) 88: "Volatile Organic Compounds in Air: Laboratory Method Using Diffusive Samplers, Solvent Desorption and Gas Chromatography," December 1997. Published by the Health and Safety Executive of the United Kingdom: http://www.hse.gov.uk/index.htm.
- Methods for the Determination of Hazardous Substances (MDHS) 80: "Volatile Organic Compounds in Air: Laboratory Method Using Diffusive Solid Sorbent Tubes, Thermal Desorption and Gas Chromatography," August 1995. Published by the Health and Safety Executive of the United Kingdom: http://www.hse.gov.uk/index.htm.

Subslab Sampling Current Practice

VI processes, whether for VOCs or radon, generally pose chronic rather than acute risks. Based on current conceptual models, VI is expected to be proportional to the concentration of the constituent of concern in the subslab space. Thus obtaining long-term integrated concentration estimates for the subslab space is desirable.

Although active soil gas sampling methods (including subslab soil gas sampling) are fairly well developed, these methods rarely integrate the measured concentration over a long period. Existing passive soil gas sampling methods are not considered quantitative because of the difficulty of establishing diffusion, and thus uptake rates, when the sampler is in direct contact with the soil (See ITRC[5], Appendix D5 for more discussion). Subslab soil gas sampling methods for VOCs normally collect samples over 15 min to 24 hour periods using flows of less than 200 mL/min (ITRC[5], Appendix D.4.6). The 200 mL/min criterion is designed to be conservative to ensure that the probe will not short-circuit in the full range of soil permeabilities expected. However, the report that is perhaps the most commonly cited reference for subslab soil sampling for VOCs collects the samples much more quickly, filling a 6 liter SUMMA® canister in only two minutes.[6] Instrumental methods for real-time monitoring of radon and VOC concentrations in air are available. However these methods are so costly and labor intensive that they are unlikely to be used to monitor subslab concentrations over long periods in routine site assessment practice.

METHODS

Field Testing – Moffett Field Site, Santa Clara County, CA

The Orion Park Housing units at Moffett Field overlay a TCE groundwater plume at the Naval Air Station, Moffett Field Superfund Site. Depths to groundwater are typically 30 ft or less. Concentrations of TCE in groundwater underlying the study area are believed to be in the range of 100-200 micrograms per liter (µg/L). The townhouse style units were slab on grade construction. Each townhouse unit was 1200 sq ft on two floors. The townhouses were grouped into buildings with eight to 10 townhouses per building arranged approximately in a single line with the longer walls of the townhouses abutting. The sampling reported here was conducted at Moffett Field in 2008. A population of up to 20 housing units of identical design, known to vary

substantially in indoor VOC concentrations based on previous data, was surveyed in Phase I using VOC indoor measurements with both passive methods and TO-15. The TO-15 measurements were made for 24- to 48-hour periods, on two occasions during the two-week passive monitoring period. Since the units were unoccupied and the climate is mild, concentrations were expected to be relatively steady over the period.

Field Testing – Southeast Neighborhood Development (SEND) Office Building and Wheeler Arts Center, Indianapolis IN

In 2009 intensive studies were conducted in a former industrial facility converted into residential and office space in Indiana (Lutes et al., 2010). These studies included measurements of subslab and indoor air concentrations of radon and VOCs, differential pressure and air exchange rates. The subject property was an industrial facility from 1911 until 1995, including the 100,000 square-foot Wheeler Arts building with a mix of slab-on-grade and basement construction. The property also includes the 1,300 square-foot slab-on-grade SEND office building (the former powerhouse for the industrial facility). Since SEND purchased the property in 1998 they have completed extensive renovations, converting it into 36 live-work lofts for low-income artists, galleries, office space for SEND, space leased to the University of Indianapolis, and a theater. For indoor air in some cases samples were obtained both with two-week integrated passive samplers and three 24-hour TO-15 SUMMA® canister samples taken during the same two week period.

Integrating Subslab Sampler

EPA Air Pollution Prevention and Control Division (APPCD) has proposed[7] a method to overcome problems related to temporal variability that combines:

- Active sampling of subslab soil gas into a chamber with
- The use of a passive sampler within that chamber, providing the potential to obtain an integrated concentration measurement over two weeks.

The novel Mark I apparatus (Figures 3 and 4) is fabricated from a modified 4 liter wide mouth sampling container, for example, the I-Chem tall wide-mouth 4-liter glass jar with Teflon®-lined polypropylene closure (http://www.ichembrand.com/) (Fisher part number 02-911-766). These containers are widely available on the market in pre-cleaned versions and are used normally for soil, sludge, and sediment sampling. The jar is modified with two Swagelok® stainless steel bulkhead fittings that allow Teflon® 1/4" tubing to carry air into and out of the jar while maintaining its leak-tight integrity The inlet to the jar is equipped with a gas diffuser (air stone) (Ace Glass part numbers 7197-02 or 7197-12) used with a 145 to 174 micron porosity frit 25-30 mm in diameter. If a flow is induced with a pump (for example at 1 L/min) the soilgas can be drawn continuously through the jar. One or more passive samplers are then placed within the jar in such a way as to allow air flow freely around them, as it does in normal ambient air applications.

Figure 3. Schematic Diagram of Integrating Subslab Sampler

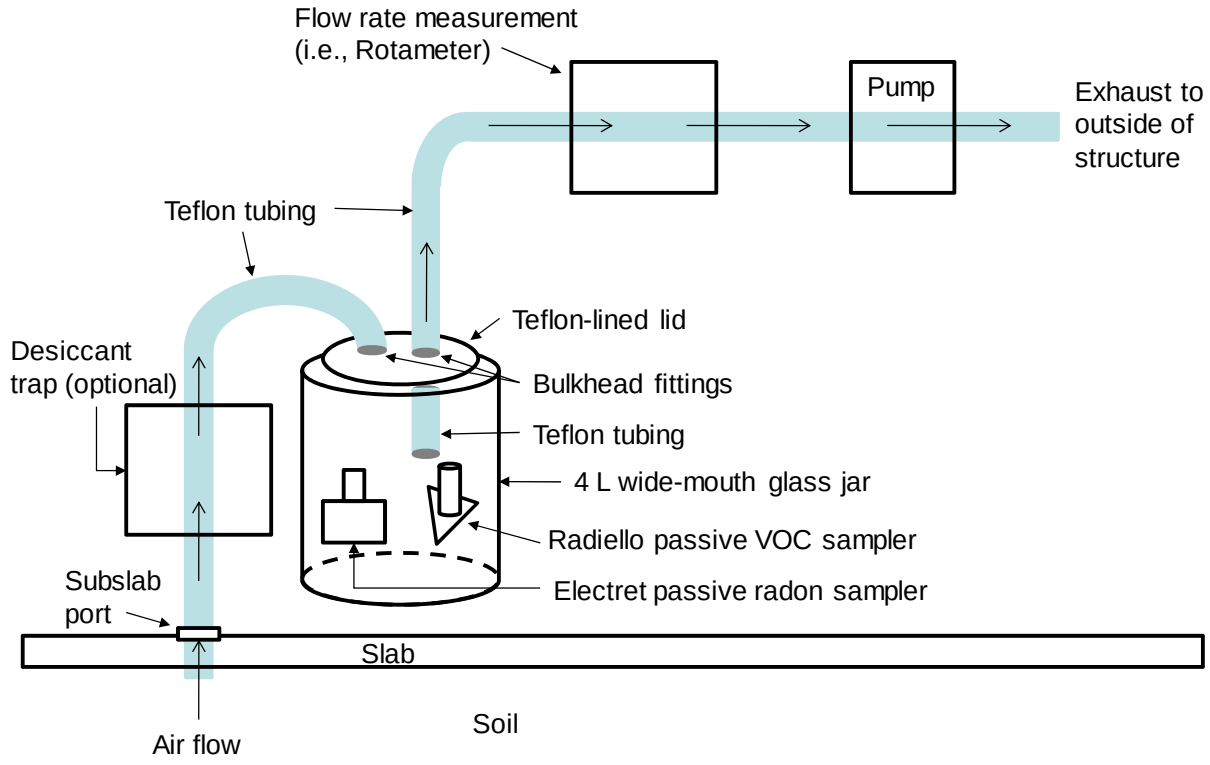
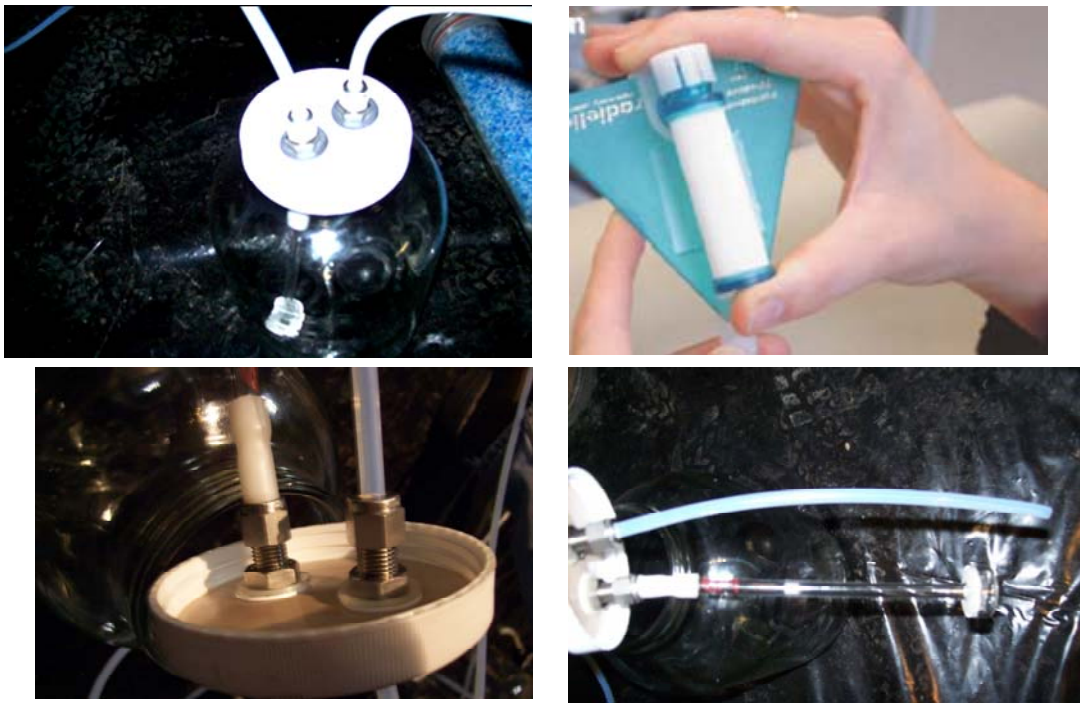
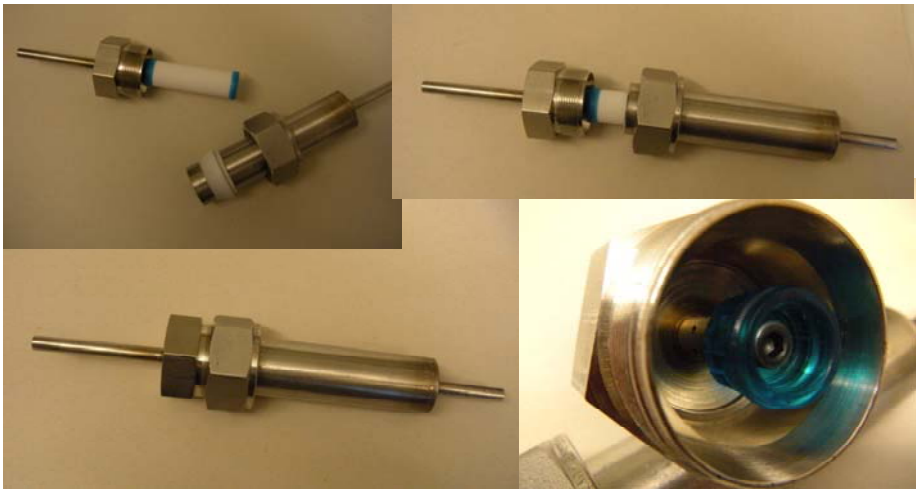


Figure 4. Integrating Subslab Sampler – Mark I - Photographs



A revised (Mark II) design is constructed from a 1" OD, 0.76" ID SS tube, 4" in length with a 1/4" OD tube butt-welded to the outlet end and a 1" SS Swagelok Cap acting as the opening/sealing mechanism on the inlet end. A 1/4" OD SS tube is welded in place and extends through the Swagelok Cap which provides a seating surface for the Radiello. The Radiello's plastic cap is sealed (mechanically fastened) to the end of the 1/4" tube, no epoxy or adhesive is used. Radially drilled holes in the 1/4" tube prior to the Radiello end cap force the incoming air around the media. To install and sample, the Radiello is screwed into its plastic cap located on the inlet portion of the sampler. The two sections are then assembled and the fitting tightened (Figure 5).

Figure 5. Alternate – Mark II Integrating Subslab Sampler Photographs



RESULTS AND SUMMARY

Indoor Air

During testing at Orion Park, Moffett Field CA by EPA, NRMRL APPCD, EPA Region IX and ARCADIS compared measurements of VOCs by Method TO-15 to three different sorbent systems (earlier results reported by Mosley[8]):

1. Radial: Activated Charcoal (with CS^2 extraction: GC/MS)
2. Radial: Carbograph 4 (TO-17: Thermal Desorption GC/MS)
3. Axial: Chromosorb 106 + cap (TO-17: TD GC/MS)

Testing was also performed at the Wheeler site in Indianapolis comparing SUMMA® canisters to Radiello solvent extracted samplers. Across the two sites the Radiello solvent extracted showed good agreement to TO-15 and precision at both sites for chlorinated aliphatic hydrocarbons such as PCE and TCE. Agreement was poor for polar compounds – ethanol, methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK) and acetone. Radiello thermal desorption correlated well with SUMMA® TO-15 for TCE but gave noticeably lower concentrations suggesting that two weeks is too long an integration time for these samplers. The agreement of the axial (tube) method was inferior (Figures 6 and 7).

Figure 6. Moffett Field Phase I: Comparison of Air Toxics TO-15 TCE vs. Passive Methods and Alternate TO-15 Lab

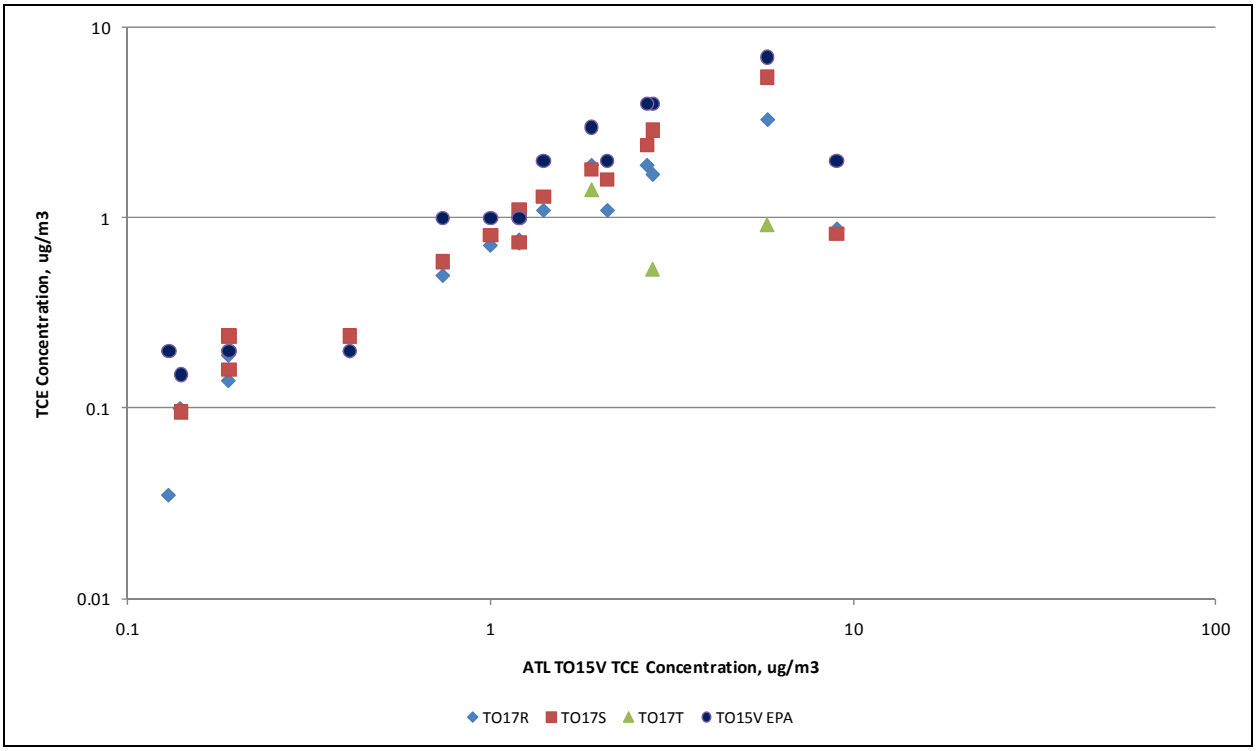
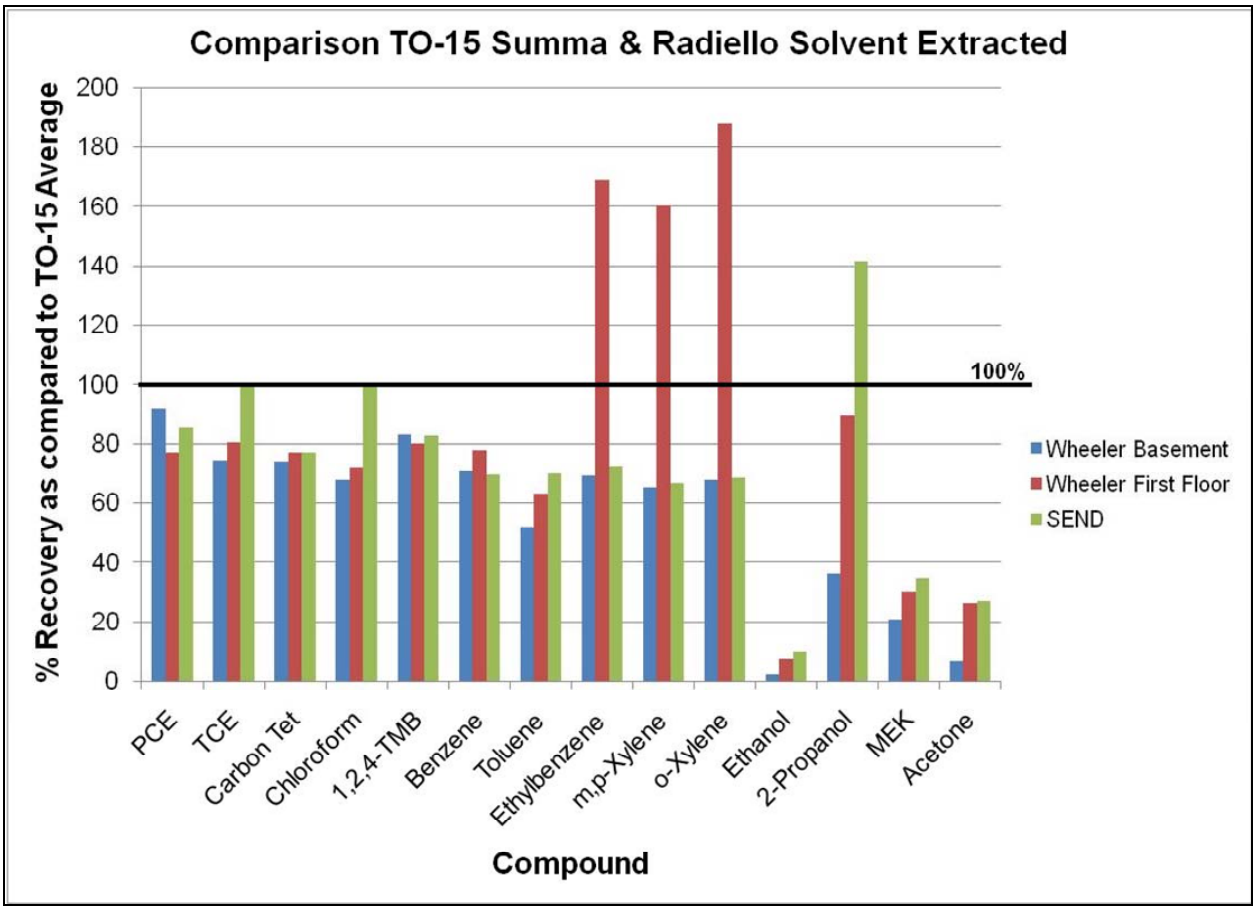


Figure 7. Comparison TO-15 SUMMA® & Radiello Solvent Extracted Passive Samplers in Indianapolis Building



Integrating Subslab Sampler

A very limited proof of concept experiment for the integrating sampler was conducted at Moffett Field, California, in 2008, with four subslab samplers operated at 200 mL/min each at four different ports in the same townhouse. During this experiment the concentration in subslab air was measured twice over a two week period using reference method TO-15 (SUMMA® canisters) at the same four subslab ports. This dataset suggests that there is a rough correlation between the two methods and thus the integrating subslab sampler should be tested further.

Multiple rounds of testing were conducted at the Indianapolis site with the Mark I integrating subslab sampler. Results are shown in Table 2 (radon) and Table 3 (TCE).

Table 2. Results of Multiple Rounds of Testing of Integrating Subslab Sampler - Radon

Site	Dates	Experimental Design Features	Radon Concentration, Standard Protocol (pCi/L)	Analytes	Radon Passive Result (% Recovery)
Moffett Field, CA	9/16/08-9/30/08	Four separate ports sampled with one electrets sampler each. Compared to Pylon.	286-460	Radon and VOCs in the same jar	65-109 (Average 89)
Indianapolis, IN	9/16/2009-9/20/2009	Duplicate electrets in four parallel jars on same port, compared to Alphaguard; pairs with and w/o desiccant	3263	Radon sampler only	92-167 (Average 117)
Indianapolis, IN	10/1/2009 – 10/5/2009	Four samplers on one port – 2 w/desiccant 2 w/o desiccant duplicate samples per jar	3259	Radon and VOCs samplers in same jar	88 to 110 (Average 95)

Table 3. Results of Multiple Rounds of Testing of Integrating Subslab Sampler - TCE

Site	Dates	Experimental Design Features	TCE TO-15 Concentration ($\mu g/m^3$)	Analytes	TCE Passive Result (% Recovery)
Moffett Field, CA	9/16/08-9/30/08	Four separate ports sampled with one integrating sampler each, each integrating sampler had one Radiello, compared to two short term SUMMA® samples.	11 -1622	Radon and VOCs in the same jar	53-87 average 67
Indianapolis, IN	10/1/2009 – 10/5/2009	Four integrating samplers – 2 w/desiccant and 2 w/o duplicate samples in each sampler; compared to five sequential SUMMA® samples.	5483	Radon and VOCs samplers in same jar	41 to 57
Indianapolis, IN	11/10/09-11/13/09	Four integrating samplers in parallel, one Radiello per sampler; compared to three sequential SUMMA® samples.	3250	VOC only	63 to 80
Air Toxics Ltd. Laboratory	1/17/2010	Two Radiello samplers in one integrating sampler jar, test run for only 2 hours, compared to TO-15 Tedlar bags	4650	VOC only	95 to 120 average 107

The key results are:

- The integrating subslab sampler provides good agreement with "Gold Standard" methods – periodic Pylon samples or continuous Alphaguard monitoring over a range of radon concentrations.
- Agreement is not as good for VOCs. Agreement was especially poor in the presence of radon samplers in the same jar. In a separate static chamber test conducted at Air Toxics Ltd in Nov 2009 we showed that the electrets used for radon testing adsorb VOCs. This was attributed to the carbon included in the electret housing to improve the housing's electrical properties.
- The Radiello-measured VOCs from the integrating subslab sampler are generally

somewhat lower than the gold standard (Table 3). The direction of this effect is consistent with what might be expected in terms of varying uptake rates with increasing duration/concentration as discussed below.

- We have also considered in some depth the possibility that the low recoveries from the Radiellos are attributable to low face velocity due to quiescent air in the jars. The Radiello manual says the method has been tested between 0.1 and 10 meters/second (m/sec). Assuming we are achieving an even velocity distribution across the full width of the jar we have calculated the velocity being provided by the flow at approximately 0.001 m/sec. Therefore EPA has designed the "Mark II Integrating Sampler" discussed above to allow the Radiello to be used in a higher face velocity environment. Alternately the Mark I integrating sampler could be modified with a small electric fan to provide circulation.

The manufacturer of the Radiello has stated that for VOCs the "*sampling rate is invariant with humidity in the range 15-90% and with wind speed between 0.1 and 10 m·s-1*." http://www.sigmaaldrich.com/analytical-chromatography/air-monitoring/radiello/learning-center/literature-and-downloads.html. Radiello bases this claim on studies by Lugg.[9] The manufacturer of the E-PERM electrets for radon claims "*E-PERM measurements exhibit no significant error due to variations in temperature, humidity, air velocity, or any other site conditions except elevation.*" Subslab soil gas is expected to often be highly humid, but testing of the integrating subslab sampler with desiccant in line did not provide any clear benefits.

The effective Radiello uptake rate for contaminants of interest ranges from 40 to 80 mL/min, for example, 69 mL/min for TCE. Thus, assuming that the integrating subslab sampler chamber is completely mixed, it is desirable to refresh the air in the chamber at a rate of 800 mL/min or greater to minimize the bias attributable to depletion of the VOC concentration in the chamber by the sampler. A mathematical correction was performed for the depletion due to the uptake rate in this study, so this effect does not explain the somewhat low recoveries.

For a given sampling duration, passive sampler sensitivity is a function of the sampler's uptake rate and the analytical technique. The uptake rate is primarily a function of sampler geometry - axial, badge-style, or radial as well as the aspects of the design that control diffusion. Accuracy of the sampler is largely determined by the variability of uptake rate during deployment. For indoor air environments in which VOC concentrations are expected to be low, a decrease in the effective uptake rate can occur due to back-diffusion or from the analyte having a residual vapor pressure once it reaches the sorbent surface. This non-constant uptake rate can result in a low bias in the VOC measurement. For benzene, toluene, ethylbenzene, and xylene (BTEX) compounds, for which this effect has been best studied, the effect for medium-strong sorbents such as Carbograph TD-1, Carbopack B or Chromosorb 106 is about 10 to 15% as the sampling time is extended from one to four weeks.[10] Back-diffusion effects are especially important when the "*sampler is first exposed to a high concentration and then to a much lower or even zero concentration.*"[11]

We examined the potential significance of a recovery in the 60-80% range as seems typical in our experiments for the integrating subslab sampler, in comparison to other relevant sources of measurement error:

- Schuver and Mosley reviewed the literature on radon sampling intervals and said "*There are many known but hard-to-predict variables that influence….variability in indoor air…In general, the range of temporal variability decreases when using longer duration samples….The vast majority of the short term (> 2 day) data lie within a factor of three (3x) of the long term (e.g., annual) average concentration.*"[12]
- In two recent TO-15/8260 interlaboratory comparisons administered by the firm Environmental Resource Associates (ERA)[13], the acceptance range for tetrachloroethylene results were:
    - 4.31-22.3 ppbv (July -Sept 2009 study)
    - 31.6-74.1 ug/L (October – November 2007 study)
- In a 2007 TO-14/TO-15 study, conducted by Scott Specialty Gasses[14], the reported values for toluene reported by 12 laboratories varied from 3.1-18.6 ppb.

Thus, we recommend that designers of future studies consider both sampling error and measurement error in selecting methods. The moderate low biases observed in this study for chlorinated aliphatic hydrocarbons using passive samplers may often be outweighed by the benefits of integrating over a longer sampling time.

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KEY WORDS

Vapor intrusion, VI, Passive sampling, Subslab sampling, Long-term sampling