## Soil-Gas Surveying for Subsurface Gasoline Contamination Using Total Organic Vapor Detection Instruments

Part II. Field Experimentation

by Gary A. Robbins, Brendan G. Deyo, Mark R. Temple, James D. Stuart, and Michael J. Lacy

## Abstract

In Part I of this study (Robbins et al. 1990), laboratory experiments demonstrated that the responses of total organic vapor detection instruments commonly employed in soil-gas surveying were influenced by sampling procedure and air composition. A theoretical framework was presented that permitted quantifying response effects and that suggested that serial dilutions of sampling bags could circumvent adverse effects. This was confirmed by laboratory experiments. In this paper, field studies performed at a service station site are presented to illustrate instrument responses under actual field conditions and to demonstrate the use of the serial dilution technique. Field measurements confirmed that instrument responses were influenced by the relative humidity, low oxygen content, high carbon dioxide level and relative abundance of detectable constituents of the soil gas. The distribution of organic vapors as determined by sampling soil-gas probes directly or undiluted Tedlar bags did not delineate the area of known contamination. Also, poor correlations were obtained among field instrument readings and laboratory measurements. Application of the serial dilution technique resulted in improved correlations among field and laboratory soil-gas measurements as well as the delineation of the area of known gasoline leakage. Response factor calculations performed using field data, and theoretical and laboratory relations derived in Part I, closely approximated observed instrument responses. The field study demonstrated that the casual use of total organic vapor detection instruments in soil-gas surveying can result in misleading information on the presence, location, and extent of subsurface contamination. As demonstrated in this study, the application of these instruments can be significantly improved through performing serial dilutions.

## Introduction

A soil-gas study was conducted at a service station that had a subsurface storage tank leak of gasoline. Soilgas measurements at the site provided an opportunity to evaluate the response of total organic vapor detection instruments under actual field conditions and to field test the serial dilution technique for circumventing adverse instrument responses. Flame ionization detector (FID), photoionization detector (PID) and explosimeter detector (ED) instruments used in testing were a Foxboro Century Systems Organic Vapor Analyzer 128 GC (in the total mode), an HNU Systems Model ISPI 101, and a GasTech Natural Gas Indicator Model NP-204, respectively. The FID, PID, and ED were calibrated using 78 or 100 Vppm (parts per million on a volume basis) methane in dry air standards, 100 Vppm isobutylene in dry zir standard, and a 2.5 percent methane in dry air standard, respectively. Relative humidity, carbon dioxide and oxygen were measured using a Fisher Scientific fast response digital thermohygrometer, a GasTech portable carbon dioxide detector model RI-411, and a GC Industries oxygen gauge model GC 501, respectively. The relative humidity meter was factory calibrated. The oxygen and carbon

dioxide detectors were calibrated to ambient air samples (20.9 percent oxygen and 0.05 percent carbon dioxide).

#### Field Measurements

Site Conditions

Figure 1 shows the layout of the service station, located on the campus of the University of Connecticut in Storrs, Connecticut. Approximately five years ago, a 5000-gallon, steel underground storage tank containing super-unleaded gasoline was found to be leaking. The tank was replaced by another tank (SU on Figure 1) with minimal subsurface remediation performed. The replacement tank was surrounded with a backfill of sand. The other tanks on-site were surrounded with a backfill of reworked native soil, which consisted of glacial till composed of silty, gravelly sand. The till extends from a depth of a few centimeters below the asphalt to 2.7 to 3.3m below ground surface and overlies crystalline metamorphic bedrock. The tills have hydraulic conductivity values between 1 x 10<sup>-5</sup> to 1 x 10<sup>-4</sup> cm/s, as determined by slug tests performed on monitoring wells. Over the last two years, the depth to ground water at the site has ranged from about 1.8 to 2.7m and the direction of ground water flow has been to the west.

Soil-Gas Survey

The site was surveyed with the FID and ED at the probe locations shown on Figure 1. Probes consisting of 0.635cm O.D. and 0.318cm I.D. stainless steel tubing were placed to depths of about 45 to 60cm below ground level by first drilling a pilot hole to a depth of about 20cm, then driving a solid rod the remaining depth. This rod was extracted and the probes were then driven into the pilot hole made by the solid rod. To prevent clogging, a wire was inserted in the probes prior to their placement in the ground and the probes were driven to approximately 5cm short of the bottom of the pilot holes.

After probe placement and removal of the wire, the probes were sampled directly with the ED and FID. The probes were then sampled by attaching the sampling system shown in Figure 2. The system consisted of a stainless steel in-line particulate filter (Fisherbrand, Cat. No. 09-753-13A, Fisher Scientific, New York, New York), two flow gauges (0 to 3124 mL/min Teflon® Flowmeter, and 0 to 333 mL/min. Teston Flowmeter, respeclively, Cole-Palmer Inc., Chicago, Illinois), a pressure gauge (0 to 15 inches of water, vacuum/pressure magnehelic gauge, Cat. No. 2330, Dwyer Inc., Michigan City, Indiana), a needle valve (Whitey, Model SS-1RM4. Hartford Valve and Fitting Inc., Hartford, Connecticut). and a diaphragm pump (Model TD- 4N, Brailsford and Co. Inc., Rye, New York). Components were connected using 0.635cm O.D., 0.318cm I.D. stainless steel tubing and Swagelok fittings. Soil-air samples were collected in 3-liter Tedlar bags (equipped with septum and hose/ valve fittings, SKC Inc., Eighty Four, Pennsylvania) attached to the pump output and then analyzed with the FID and ED.

Following the initial sampling, Tedlar bags were serial-diluted using ambient air. The ambient air generally exhibited no detectable background vapors but on occasion concentrations up to 2 Vppm were measured. The serial dilution was accomplished in the following manner: After a sample was collected in a Teolar bag, the sampling system was disconnected from the probe and purged. The Tedlar bag was then attached to the system inlet (the particulate filter shown on Figure 2) and a volume of gas was removed by pumping the system at a set flow rate for a desired period of time. Pumping times and flow rates were generally adjusted to remove between 1/3 and 1/2 of the initial gas sample volume in the bag (about 2 liters). The system was then purged a second time. The bag was then attached to the exhaust Port of the pump and a volume of ambient air was pumped into the bag that approximately equaled the volume of gas sample removed. The bag was then resampled and the procedure repeated. Following soilgas analyses, the sample bag and the system were Purged. The system was purged by pumping ambient zir through it. Purging was checked by sampling the system with the FID. The system was found to readily Purge to a non-detectable level with the exception of the Tygon tuhing, which had to be occasionally replaced. The sample has was purged by repeatedly filling the Deg with embient air and evacuating the ambient air

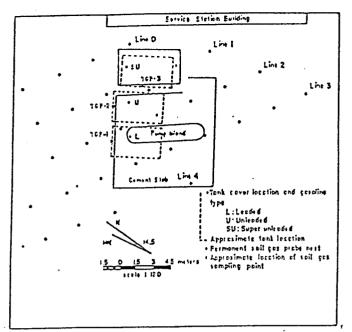


Figure 1. Plan map of service station site.

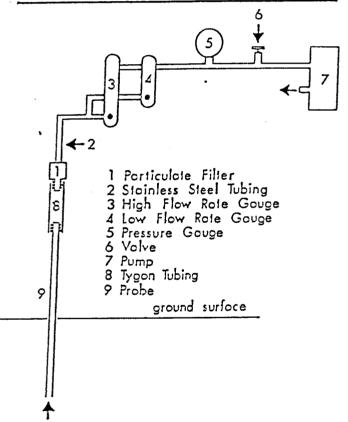


Figure 2. Soil-gas sampling system for collecting Tedlar bag samples and for performing serial dilutions.

with the pump. Based on FID readings, the bag readily purged to a non-detectable level.

#### Probe Nesis

Three nests of probes were installed at the site (labeled TGP-1,-2, and -3 on Figure 1). The nests consisted of 0.635cm O.D., 0.318cm I.D. stainless steel probes. These were placed by augering a shallow hole and then driving them to the desired depths. Prior to placement, wires were inserted in the probes to prevent clogging. After placement the wires were removed.

Probes were completed just short of the surface with Swngelok fittings and secured in flush-to-grade manholes. Nests TGP-1 and TGP-2 consisted of two probes each at 61 and 109cm below ground surface. Nest TGP-3 consisted of four probes at 61, 122, 185, and 246cm below ground surface. Thermocouples were also emplaced along side and at the depths of each probe.

Probe nests were sampled by connecting a stainless steel sampling system to a probe using a Swagelok fitting. The sampling system consisted of a series of valves connected by stainless steel tubing. A probe was purged with a vacuum handpump and was then connected to an evacuated 800mL stainless steel canister. Canister samples were analyzed by laboratory gas chromatography. Following the collection of a canister sample, the sampling system shown in Figure 2 was connected to a probe and a Tedlar bag sample was collected for field analysis. The bag was then serially diluted using ambient air. To obtain more accurate regulation of extracted gas sample volumes and inserted ambient air volumes, the procedure used in the survey was modified in the following manner. After collecting a Teolar bag sample, a desired volume of gas was transferred to another evacuated bag. This was accomplished by attaching the sample bag to the system inlet and the second bag to the pump output port. The second bag was then sampled with the ED, FID, PID, and carbon dioxide detector. The original bag was then diluted by refilling it with a volume of ambient air equal to that transferred. This procedure was then repeated. The serial dilutions were performed until two to three successive instrument readings diminished by consistent fractional increments indicating the dilution curve was in the log-linear range.

After Tedlar bag sample collection, soil-air oxygen and relative humidity were measured. These measurements were performed by connecting instrument sensors in series to the exhaust port of the sampling system's pump. Soil air was then pumped from a probe through the system and measurements were taken when instrument readings stabilized.

## Results and Discussion

Figure 3 illustrates the direct-probe FID soil-gas survey results. Data were contoured by linear extrapolation and interpolation using only those data representing quantifiable concentration readings. As illustrated on Figure 3, the quantifiable concentration readings did not coincide with the area of the known source of gasoline leakage (the area of tank labeled SU). In the vicinity of the tanks, the instrument readings exhibited an initial rise then a flame-out. Although this indicated the presence of detectable vapors, it did not permit concentration readings over a significant portion of the site. The ED on the high scale exhibited no discernible response. This indicated that vapor levels were too low for the instrument to resolve. The ED on the high scale reads from 0 to 100 percent combustible gas and has a practical meter resolution of 2 percent (20,000 Vppm). On the low scale, in the areas where the FID slame was extinguished, the ED meter readings initially rose then fell off. In consideration of the laboratory results in Part I,

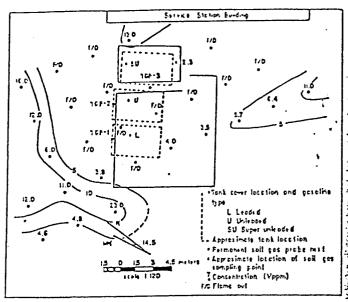


Figure 3. FID soil-gas survey results using direct-line sampling.

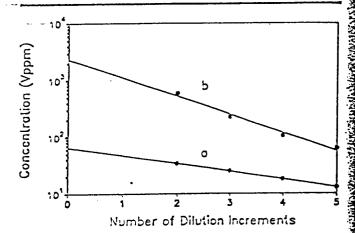


Figure 4. FID serial dilution curves. Curve a illustrates circumventing low oxygen conditions when the initial concentration is within the instrument's detection range; curve b illustrates circumventing low oxygen conditions and extrapolating above the instrument's detection range.

these readings, along with the FID response, implied that either soil-air oxygen levels were too low to support combustion or that the soil had low air permeability. If a soil has a low air permeability due to its lithology or a high moisture content, an appreciable vacuum can be generated in a soil-gas probe during direct-probe sampling. The vacuum can diminish instrument response or extinguish the FID flame. Outside of the flame-out areas, the ED exhibited no discernible response, implying that concentration levels were too low to resolve. On the low scale the ED reads from 0-to 5 percent combustible gas and has a meter resolution of about 0.1 percent (1000 Vppm).

Initial Tedlar bag concentration measurements using the FID and ED on the low scale from probes in the flame-out area exhibited similiar responses to those noted previously. This would indirectly indicate that low soil-air oxygen levels were responsible for the observed FID and ED responses in direct-probe sampling rather than conditions of low soil-air permeability. Again, the ED on the high scale provided little quantifiable infor-

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mation. Figure 4 illustrates typical serial dilution curves for the FID. The log-linear response of the curves indicates relatively constant response conditions and well-regulated volume control during the dilutions. The curves were extrapolated to the concentration intercept to obtain a pre-dilution concentration reading of total organic vapors in the sample bags. Curve a on Figure 4 illustrates circumvention of low oxygen conditions to obtain an initial bag concentration. Curve b illustrates not only circumvention of low oxygen conditions but also conditions where initial vapor concentrations may exceed the meter range of an instrument.

Figure 5 is a map of FID soil-gas concentrations determined by serial dilution extrapolations and Tedlar bag measurements. The data were contoured by linear extrapolation and interpolation. The high soil-gas concentration zone lies just downgradient from the former leaking tank. A ground water grab sample at the probe location of the highest soil-gas concentration confirmed the presence of contamination. The water sample exhibited a sheen and had a total BTEX level of 6100 µg/L.

Table 1 summarizes probe nest field data from one round of sampling. Soil-zir oxygen was significantly depressed below that of ambient air (21 percent), while soil-zir carbon dioxide levels were greatly elevated above that of ambient air (0.05 percent). These observations confirm the cause of the FID flame outs and the ED response fall-offs observed in conducting the soilgas survey and in taking initial Tedlar bag measurements of probe nest samples. In the laboratory experiments, the FID was found to flame out below oxygen levels of

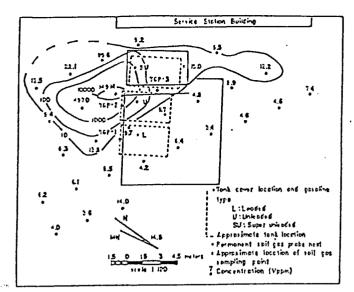


Figure 5. FID soil-gas survey results based on serial dilution measurements.

about 10 to 13 percent. The high carbon dioxide levels in association with subsurface gasoline leakage observed in this study would also corraborate similiar observations made by Marrin (1987). In that study, involving a soil-gas survey above a benzene/chlorobenzene ground water plume, Marrin observed that soil-air carbon dioxide levels above the plume were up to nine times that of ambient air. Here, carbon dioxide levels were found to be even higher and were up to 275 times that of ambient air.

As noted on Table 1, soil-gas relative humidity values were found to be high, even at relatively shallow depths. On the date of sampling the air, relative humidity was

## TABLE 1 Probe Nest Field Measurements

Date: 5/9/89								
Parameter	Probe							
	TGP-1	TGP-1	TGP-2	TGP-2	TGP-3	TGP-3		
Depth (cm)	61	109	61	109	61	122		
% O <sub>2</sub>	8.4	6.2	0.6	0.5	1.3	0.4		
% CO <sub>2</sub>	11.1	12.7	12.7	13.4	13.8	12.4		
T°C	16.0	15.0	16.3	15.3	16.2	15.1		
% Rel. Humid.	78	82	68	83	88	87		
ED In. (%)	-		RF	RF	0.2	RF		
ED Ex. (%)		<del></del>	1.6	2.6	0.2	1.7		
FID In. (Vppm)	F/O	F/O	F/O	F/O	F/O	F/O		
FID Ex. (Vppm)	_		4470	6992	450	3704		
PID In. (Vppm)	_		48	.21	24	32		
PID Ex. (Vppm)			1075	1925	84	610		

<sup>%</sup> tel. humid, values are corrected for difference between sensor temperature and probe temperature.

= not detected.

ED In., FID In., and PID In. = initial hag concumration.

ED Ex., FID Ex., and PID Ex. = extrapolated concentration.

RF = meter reading initially rose then fell off.

F/O = name nut.

TABLE 2
Probe Nest Laboratory Canister Data

Date: 5/9/89									
	Probe								
Parameter	TGP-1	TGP-1	TGP-2	TGP-2	TGP-3	TĠP-3			
Depth (cm)	61	109	. 61	109	61	122 ·			
Tot. Conc. (Vppm)	·		812	3261	12	2939			
Constitutent			, % by vo	lume					
n-butane	-	_	96.7	40.9		39.5			
2-m-butane		<del></del>	_	52.6	100	57.8			
2-m-2-butene		<u> </u>		6.8	_	_			
2,3-d-m-butane	<del>-</del>	_		2.4	_	1.2			
n-hexane	******			2.2					
2-m-2-pentene		•				_			
benzene		_		-		f			
sooctane		_		0.5	*****	1.5			
oluene	, <del></del>	-	3.3		*****	_			
o-xylene	_			_	. <del>-</del>				

- = not detected.

at 37 percent and the water table was at a depth of 1.8m. Given the accuracy of the thermo-hygrometer (about 1.5 percent of reading), errors in correcting the relative humidity data to account for temperature differences between the position of the sensor and depth of a probe, and errors in thermocouple calibrations, it is likely that these values underestimate the soil air relative humidity.

Table 2 summarizes probe nest constituent (percent by volume) and total concentration data from the laboratory analyses of the canisters. The constituents listed are only those that were quantified. In addition to these constituents, appreciable amounts of isobutane were present, as well as methane in some canisters. Although the soil gas was dominated by alkane components as might be expected, levels of aromatic compounds were either less than that reported for fresh gasoline vapors (Wadden et al. 1986), or absent. This may be due to biodegradation of aromatic constituents and differences in soil-air diffusional characteristics of aromatic and alkane constituents (Thompson and Marrin 1987, Robbins 1987, and Silka 1988).

Initial sample bag concentration measurements by the ED on the low scale and the FID were not quantifiable due to low oxygen levels. Although initial PID readings were obtained, they correlated poorly with the total canister concentrations. Figure 6 illustrates typical ED, FID, and PID serial dilution curves. The ED and FID curves indicate constant response factor conditions and, as would be expected, the curves are parallel.

The PID curve in Figure 6 illustrates variable

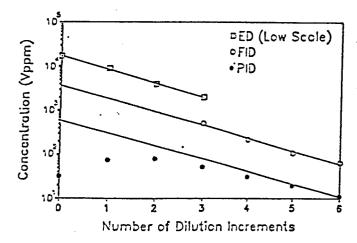


Figure 6. Probe nest TGP-3 (122cm) serial dilution curves illustrating constant response factor conditions for the FID and ED, and variable response factor conditions for the PID.

response factor conditions with dilution. Here, the PID concentration initially rose with dilution then declined. This is a consequence of the PID response factor initially increasing in an amount greater than the concentration in the bag was decreasing during dilution. As constant response factor conditions were approached with dilution, the slope of the PID curve approached that of the others. A subsequent serial dilution test at the site was performed with another PID instrument (OVM 580A, Thermo Environmental Instruments Inc.) and exhibited a similar type of response suggesting that this type of response may be generic to PID instruments.

## TABLE 3 Experimentally Derived PID Response Factor Relations

Carbon Dioxide:  $\log (R_i) = -5.7 \times 10^{-3} \% \text{CO}_2 - 0.049$ Relative Humidity:  $\log (R_i) = -6.3 \times 10^{-3} \% \text{RH} - 0.017$ Alkane Quenching:  $\log (R_i) = -1.6 \times 10^{-2} \text{C}_{\text{bu}} \div 0.055$ Non-linearity:  $\log (R_i) = -2.6 \times 10^{-2} \text{C}_{\text{be}} \div 0.013$ 

R<sub>i</sub> = response factor = ratio of measured concentration influenced by response conditions to that not influenced.

%CO<sub>2</sub> = % carbon dioxide: %RH = % relative humidity: Chu = butane concentration (Vppm): Che = benzene concentration (Vppm).

TABLE 4
Comparison of Calculated and Observed PID Composite Response Factors

Probe	TGP-2	TGP-2	TGP-3	TGP-3	
Depth (cm)	61	109	61	122	
Response Condition		Response Factor <sup>1</sup>			
Carbon dioxide	0.76	0.75	0.75	0.76	
Relative humidity	0.36	0.29	0.27	0.27	
Alkane quenching	0.84	0.33	1.13	0.37	
Non-linearity	0.54	0.33	0.98	0.72	
Calc. Composite Response Factor <sup>2</sup>	0.12	0.02	0.22	0.06	
Observed Composite Response Factor <sup>3</sup>	0.04	0.01	0.29	0.05	

<sup>1</sup> Carbon dioxide and relative humidity response factors were computed using regression relations in Table 3 and earbon dioxide and relative humidity values from Table 1. The response factor for alkane quenching is based on the butane response regression in Table 3 and the total canister concentration listed in Table 2. Response factors for non-linearity are based on the benzene response regression in Table 3 and extrapolated PID concentration values in Table 1.

The calculated composite response factor is taken as the product of the individual response factors (see theory section in Part 1).

The observed composite response factor equals the ratio of PID In. to PID Ex. values listed in Table 1,

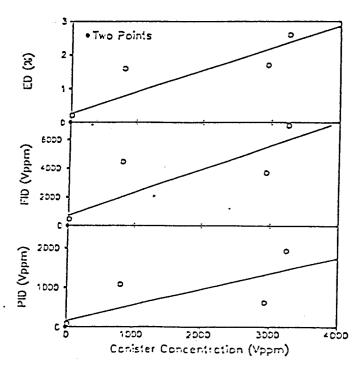


Figure 7. Correlation of ED, FID, and PID extrapolated serial dilution concentrations with canister concentrations.

In the laboratory experiments in Part I, the PID response to a detectable vapor constituent was shown to be diminished in the presence of carbon dioxide. alkanes, and relative humidity. Also, the PID was found to exhibit non-linear response when detectable vapor constituents exceeded about 125 Vppm. To evaluate whether the initial PID readings could be explained by these conditions, individual and composite response factors were calculated using the response factor relations derived in Part I and the data in Tables 1 and 2. Table 3 lists the laboratory-derived response relations. Table 4 summarizes the results of the calculations. The calculated PID composite response factors were found to closely match the observed response factors. This would confirm that the initial PID response is being influenced by the observed soil-zir conditions. It would also explain the non-linear form of the PID serial dilution curves.

# Soil-gas measurements, no matter how quantitative, do not permit quantifying soil or ground water contamination.

FID, ED, and PID intercept concentrations extrapolaied from the serial dilution curves are plotted against canister concentrations in Figure 7. Linear regression curves are also shown. The intercept concentrations for all three instruments had similiar levels of correlation with the canister data ( $r_{ED} = 0.907$ ,  $r_{FID} = 0.857$ ,  $r_{PID}$ = 0.783). Scatter in the data may be attributed to measurement errors, variation in the relative abundance of constituents in the soil gas as revealed by the canister data, and the fact that the total canister concentration does not represent all the constituents that may be present and detectable by the total organic vapor instruments. As further evidence of comparable correlation, the FID and PID data were regressed. As indicated in Figure 8, extrapolated intercept concentrations of both instruments were highly correlatable. This would imply that both instruments would provide the same level of information for delineating relative variations in subsurface vapor levels, providing that serial dilutions are performed.

## Conclusions

The laboratory and field tests conducted in this study would indicate that commonly employed total organic vapor detectors used in soil-gas surveying for subsurface gasoline contamination give results that depend on sampling technique, soil-air permeability, and soil-air composition. Thus, casual use of these instruments for indications of relative subsurface contamination levels can be misleading and non-definitive (e.g., when the FID

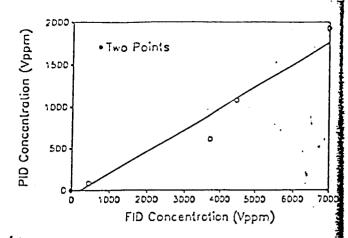


Figure 8. Correlation of FID and PID extrapolated serial dilution concentrations [ $C_{\text{PID}} = 0.26C_{\text{FID}} \cdot 60$ ; r = 0.979].

flame is extinguished or when PID readings are quenched). Both laboratory and field tests would indicate that the serial dilution technique can overcome deleterious response conditions and provide more meaningful information. Even then, it should be kept in mind that the instruments are non-constituent specific, and that their response depends on relative constituent abundances. As exemplified by the probe nest data in this study, relative constituent abundances can vary substantially over short distances and depths. Hence, the instruments are inherently limited as to the information they can provide. Importantly, it should be realized that soil-gas measurements, no matter how quantitative, do not permit quantifying soil or ground water contamination. Instead, they provide general and relative indications of subsurface contamination that require confirmation. In this regard, with proper use, consideration for factors affecting response, and cautious interpretation, total organic vapor detectors can be valuable exploratory tools.

## Notice

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