

Evaluating Leaks in a Soil Gas Sample Train

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ABSTRACT

The current advisory for active soil gas investigations in California¹ requires leak tests to be conducted at every soil gas probe while taking a sample. A leak may allow ambient air into the sample train thus diluting the soil gas and underestimating the results. Also, the leak check compound itself may have impurities thereby contaminating the sample. The advisory suggests leak check compounds and lists potential leak points in the sample train that should be investigated. If a leak has been detected but unable to be resolved, the soil gas probe is decommissioned, and a new soil gas probe is installed, or consultation with agency staff required. Since the leak check results may draw soil gas data into question, the presence of any leak check compound in the sample is a concern. This paper investigates the response of several leak check compounds at determined leak rates into the soil gas sampling train. The diffusion of leak check compounds through the walls of commonly used tubing for soil gas investigation is also evaluated. Finally, considerations for performing leak checks and interpreting leak check data are presented.

INTRODUCTION

In order to insure the highest quality data for soil gas measurements, leak checks are often performed by exposing the potential leak sites to a known compound during sample collection. The compound is then analyzed for in the soil gas sample to determine if the sample was compromised by ambient air intrusion. A wide array of leak check compounds are used in active soil gas investigations. The presence of a leak check compound in soil gas data often raises more questions for the data user than answers on how to interpret the results. Understanding the relationship between a leak and concentration of a leak check compound, the role of diffusion of leak check compounds through sample train tubing, and potential for interference by the check compound with VOC measurements is important in answering these data usability questions.

APPROACH

Definition of a Leak

Leaks which may occur at fittings and connections of a sampling train exist on a continuum from negligible to severe. To evaluate the presence and/or extent of the leak, check compounds are used to test the potential leak points during sample collection. When the sample is analyzed by the laboratory, the check compound is measured and reported with the soil gas results. The question for the data user is how to translate a leak check concentration to a determination of data usability. Different interpretations regarding the definition of leak are suggested by various agencies. For example, the state of California "Advisory-Active Soil Gas Investigations" recommends a detection limit of 10 µg /L or less for a leak check compound.¹ A detection limit of 0.1 µg /L for the leak check compound has also been suggested in subsurface vapor sampling guidelines.²

To model the impact of differing leak rates on concentrations of the leak check compound in the sample, experimental calculations were performed using the following two equations. Examples are presented using varied leak rates and several common leak check compounds. For these calculations, the sample flow rate was fixed at 100 mL/min through the entire sampling period.

Equation 1.

$$\text{Concentration (ppmv)} = \frac{\text{Leak Volume (mL)}}{\text{Sample Volume (mL)}} * (\text{Purity of check standard}) * 1,000,000$$

Where:

$$\text{Leak Volume (mL)} = \text{Leak Rate (mL/min)} * \text{Sample Time (min)}$$

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Equation 2.

$$\text{Concentration (}\mu\text{g/L)} = \frac{\text{Conc. ppmv} * (\text{MW})}{24.45}$$

Where:

24.45 is the molar volume of any gas at normalized pressure and temperature, derived from the ideal gas law (PV=nRT) and MW = Molecular Weight of compound.

Table 1. Leak rate relationship.

Check Compound	Leak Rate (mL/min)	% Leak Volume* Introduced	Calc. Conc. (µg/L)	Calc. Conc.* (ppmv)
Isobutane or Butane**	0.5	0.5	11,000	4500
	0.05	0.05	1100	450
	0.0005	0.0005	11	4.5
2-Propanol	0.5	0.5	12000	5000
	0.05	0.05	1200	500
	0.0005	0.0005	12	5.0
1,1,1,2-Tetrafluoroethane** (Freon134a) (Office Depot Duster)	0.5	0.5	19,000	4500
	0.05	0.05	1900	450
	0.0005	0.0005	19	4.5
Sulfur Hexafluoride	0.5	0.5	30000	5000
	0.05	0.05	3000	500
	0.0005	0.0005	30	5.0

*Sample flow rate assumed to be 100 mL/min.

** Purity assumed to be 90% for these compounds.

Diffusion of Leak Check Compounds

When a leak check compound is detected in a soil gas sample, it is often assumed that a leak in the train must be the cause. However, compounds can also diffuse through the sample collection tubing. Diffusion of some common leak check compounds through the walls of different types of tubing used in soil gas sampling was investigated.

Experimental Conditions

A five-foot section of tubing was attached to an evacuated 1 L Summa canister. The other end was attached to a cylinder of humidified zero air with a flow meter set at 90ml/min. Using a vacuum check, the system was demonstrated to be leak free prior to the test. A two-foot mid-section of the tubing was sealed into a 5.0 L Tedlar bag. The bag was filled with leak check compound and allowed to stand for 10 min. Next, zero air was passed through the tubing into the evacuated 1.0L canister at a flow rate of 90 ml/min. for 8.0 minutes to yield a volume of 720ml. The canisters were pressurized with nitrogen to 15 psig and analyzed by GC/MS or GC/ECD. Three types of tubing were evaluated: Teflon®, PEEK, and Nylaflo®.

The presence of leak check compounds at varying concentrations was observed in most of the tests. The presence of these compounds may be due to diffusion. During different periods of these tests, cross contamination could not be ruled out from one test to the next. This was due in part to the highly concentrated nature of the sources used to obtain the leak check compounds. This same difficulty may also cause contamination during

field sampling and extreme care must be used in the handling of neat materials during an air sampling event.

Analytical Considerations

The choice of a leak check compound should be made carefully to insure that both sampling and analytical data quality objectives are met. Some initial questions should be asked before choosing a leak check compound.

- 1) Is there a potential that the leak check compound is present at the contaminated site? It may be necessary to collect a test sample to characterize the soil gas in order to select a non-interfering leak check compound.
- 2) What reporting limits are needed? The samples may be diluted due to compounds present in the sample, but may also need to be diluted because of the leak check compound. All compounds in Table 3, with the exception of sulfur hexafluoride, exhibit similar volatility to many of the compounds of concern for soil gas analysis. As such, presence of these compounds at high levels may necessitate dilution to limit any negative effect on the instrumentation. This may compromise reporting limit objectives for other VOCs of concern. For several of the check compounds, even small leaks <10 µg/L can be problematic when sub-ppbv reporting limits are required.
- 3) Does the source of leak check compound have the required purity? Some of the “over the counter” leak checks contain other VOCs that may be compounds of concern for soil gas analysis.

A summary of some common leak check compounds and their attributes are presented in Table 3.

Table 3. Leak Check Compound Comparison

Leak Check Compound	Reporting Limit	Detector	Availability	Purity
Isobutane (Shaving Cream)	0.5ppbv	GC/MS	Readily Available	88%
2-Propanol (Rubbing Alcohol)	2.0ppbv	GC/MS	Readily Available	70-99%
1,1,1,2-Tetrafluoroethane (Freon134a) (Office Depot Duster)	0.5ppbv	GC/MS	Readily Available	Variable
Butane (Butane Lighter)	0.5ppbv	GC/MS	Readily Available	Variable

Sulfur Hexafluoride	0.5ppbv	GC/ECD	Special Order	99.8%
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CONCLUSION

The presence of a leak in a soil gas sampling train can compromise the sample either through dilution or contamination. Because of this, many regulatory agencies are requiring leak tests. Careful thought should be used when choosing a leak check compound, whether it is site specific, detection limit or cost driven.

REFERENCES

1. *Advisory – Active Soil Gas Investigations*, Department of Toxic Substances Control: Glendale, CA and California Regional Water Quality Board Los Angeles Region: Los Angeles, CA, January 2003.
2. *Subsurface Vapor Sampling Using a Geoprobe and Summa Canisters (GPP Guidelines)* DRAFT, San Mateo County Environmental Health Services Agency – Ground Water Protection Program , June 2004.