Introducing...

Advanced Tools for Sampling and Analysis

insight

Microseeps, Inc.
From Insight Comes Solutions

(800) 659-2887
www.microseeps.com

220 William Pitt Way
Pittsburgh, PA 15238

Look for Mike O' Seeps as he unveils the latest advances at Microseeps!

Microseeps, Inc.

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Microseeps, Inc.

(800) 659-2887
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Microseeps is a specialty analytical laboratory that employs unique technologies to improve the understanding of complex environmental and geotechnical issues. Since 1984, Microseeps has helped to lead the industry in better understanding the fate and transport of chemicals in the subsurface through innovations in analytical and sampling technology.

While Microseeps provides the services common to traditional environmental laboratories, we go beyond to offer performance-based analyses and sampling services which facilitate a comprehensive geochemical understanding of the subsurface environment.

Microseeps' Services

Specialty Analyses
- Dissolved Gases including Hydrogen
- Anions / Cations
- Volatile Fatty Acids
- Total Inorganic Carbon
- Fuel Oxygenates
- Vapor Analysis
- Compound Specific Isotopic Analysis

Traditional Laboratory Analyses
- Volatiles, Semi-Volatiles, Metals, Wet Chem

Consulting Services
- Geochemical Consulting
- Report Generation

We have established ourselves as an environmental services company which facilitates environmental solutions through the application of superior geochemistry, analyses, field services, and geochemical consulting. Along the way, we have achieved significant results.

- Microseeps built a series of sampling and analytical tools to better quantify the effects of natural attenuation.
- Microseeps developed the ability to identify the redox process in the lab and developed a sampling device to ensure the sample was representative of the aquifer.
- Microseeps worked with industry and USEPA to develop a method to accurately measure all the components of reformulated gasoline.
- Microseeps is the first commercial US Lab to develop and offer Compound Specific Isotopic Analysis (CSIA)

Specialty Analyses

Microseeps, Inc.

Microseeps offers unique analytical services to support specific environmental applications

Microseeps offers a series of specialty analyses to quantify the presence of specific suites of analytes. Each analytical series is designed to accurately and reliably evaluate subsurface processes.

Analyses Supporting Monitored Natural Attenuation and Enhanced Biodegradation

Monitored Natural Attenuation (MNA) is the monitoring of the decrease of contaminant concentration resulting over time from the processes of dispersion, sorption, volatilization and biodegradation.

- Microseeps combines exceptional analytical services and insight into the data to understand your MNA projects
- In particular, our dissolved gas analyses and our anion/cation analyses will help delineate the redox zones and thus the achievable rates of various transformations of the dominant TEAPs (terminal electron accepting processes).

The *Insight* Report

The *Insight* report is a series of graphical representations that plot analytical results for each sample point. This report provides insight into the capacity of the groundwater to degrade organic and inorganic constituents. The report is ideal for use in support of sites that are using MNA, stimulant injection or any other remediation technology. Parameters can include:

- Dissolved Gases including Hydrogen
- Anions/Cations
- Volatile Fatty Acids
- SW-846 Analyses

Demonstrating Microbial Activity

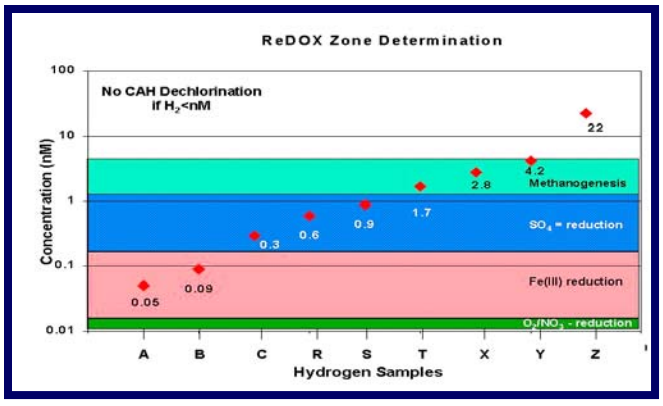
With the proliferation of enhanced biodegradation as a remediation technology, Microseeps recognized the need to provide analytical methods to demonstrate microbial activity.

- Microseeps developed a new methodology for analysis of the volatile fatty acids that uses ion chromatography instead of gas chromatography. This methodology has significantly lowered detection levels and is ideal for remediation approaches using the addition of organic substrates for stimulation of anaerobic degradation.
- If TIC is measured before and after a stimulated degradation process is implemented, the difference in TIC is the amount of organic carbon converted to inorganic carbon during the stimulated degradative process. Microseeps has developed a method for analyzing Total Inorganic Carbon that takes the guess work out of calculating TIC across a site.

Determination of Fuel Oxygenates

Most gasoline plumes today also contain fuel oxygenates. There are many hydrocarbon/fuel oxygenate plumes where the hydrocarbon portion of the plume is relatively short and the fuel oxygenate portion of the plume is much larger.

- Microseeps recognized the need for an analytical method that quantifies analytes commonly found in reformulated gasoline. Microseeps' RFG method reliably measures the analytes and reduces uncertainties that result from traditional methods of preservation and preparation of samples.



Compound Specific Isotope Analysis

Supporting Monitored Natural Attenuation

Microseeps Is the first commercial laboratory in North America to provide CSIA.

Compound Specific Isotope Analysis (CSIA) is an evolving technique which generates isotopic characterization of individual compounds. The isotopic data can be used to more definitively characterize processes in groundwater which degrade contaminants of concern such as BTEX, MTBE and CVOC's.

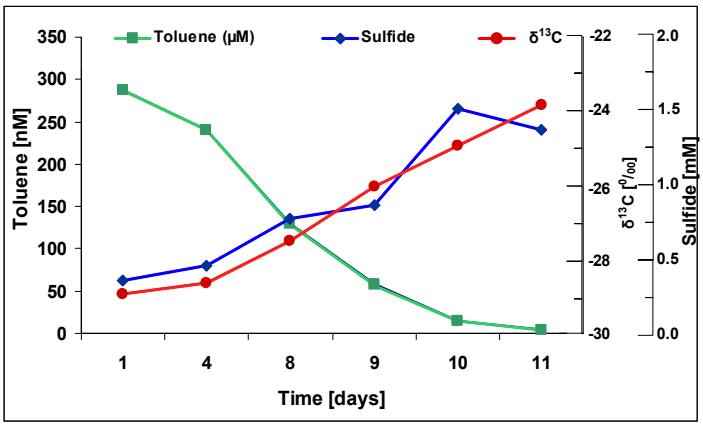
Mike O'Seeps spots nature's clue.

Nature has provided internal evidence in the form of the isotopic ratio. Biodegradation causes a progressive alteration in the isotopic ratio so CSIA can be used to quantitatively assess how much and at what rate remediation has occurred in field samples.

The data that is generated by CSIA can be used to:

- unambiguously determine that biodegradation is occurring
- identify the mechanisms of degradation
- determine the rate and extent of degradation in some contaminants of concern

Many processes which affect contaminants in groundwater such as dilution, sorption and volatilization have either very small or no isotopic effects, however processes like biodegradation and abiotic degradation are associated with significant isotopic effects.



The study of isotopes in groundwater plumes of fuel oxygenates like MTBE has provided unequivocal proof of its degradation, revealed the mechanism of its degradation and provided an in-situ measurement of the rate of degradation.

Applications of CSIA

- In-situ Chemical Oxidation (ISCO) - demonstrate that contaminants degraded, not just moved.
- cis-DCE Stall - demonstrate that there really is degradation of cis-DCE.
- MTBE— Conclusive evidence of the presence or absence of anaerobic degradation.
- Abiotic Degradation— Currently, CSIA is the only way to demonstrate that abiotic degradation is occurring.

These are examples of Isotopic studies that are possible with CSIA that can be a powerful tools in evaluating the progress of in-situ degradation.

Microseeps currently offers analysis of stable carbon isotopes in groundwater samples for MTBE TBA, chlorinated solvents and BTEX. Method development for stable hydrogen isotopes on the same suite of analytes is in process. Finally, we will develop our systems' capability for stable carbon and hydrogen isotopes on vapor samples, either indoor air from summa canisters or soil gas in a variety of sample containers.

This figure (from Schmidt, et al., 2004) illustrates the anaerobic degradation of toluene under sulfate reducing conditions. Notice that as the concentration of toluene remaining in solution decreases, the measure of its isotopic composition, $\delta^{13}C$, increases.

Dissolved Gas Analysis

Supporting Monitored Natural Attenuation

Be sure to learn the proper sampling methods for Dissolved Gas Analysis

Both abiotic and biologically facilitated reactions of contaminants with components of groundwater often produce species that exist in groundwater as dissolved gases. Typical dissolved gas species are carbon dioxide, methane, ethene, ethane, hydrogen, and acetylene.

The identification and quantification of these dissolved gasses is often useful in:

1) identifying the redox process that is predominant in a given volume of groundwater, thus determining the achievable rates of various transformations
2) verifying that degradation has taken place
3) identifying the path that degradation has taken

Microseeps classifies three suites of dissolved gases:

- the permanent gases including methane, carbon dioxide, oxygen and nitrogen;
- the light hydrocarbons including methane, ethane, ethene, propane, propene, i-butane, n-butane and acetylene;
- dissolved hydrogen

Dissolved Gas Detection Limits

Hydrogen	**0.12nM**
Methane (FID)	**0.024 ug/L**
Ethane and Ethene	**0.008ug/L**
Carbon Dioxide	**0.39 mg/L**
Oxygen	**0.11mg/L**
Nitrogen	**0.097mg/L**
Methane (TCD)	**0.07 mg/L**

Microseeps is the only certified laboratory that offers dissolved hydrogen analyses. Microseeps' analytical methods for dissolved gases are fully documented and have been reviewed by several state agencies and the USEPA.

- Each method includes all QA/QC that would be expected from promulgated USEPA methods.
- **Microseeps' detection levels are the lowest in the industry**.
- Microseeps' gas chromatograph is a customized instrument built in-house for this procedure. The instrument includes three detectors arranged so all three suites of analytes may be determined from the same sample.

  This eliminates potential variables between separate samples due to sampling error. This feature is extremly important in the analysis of light hydrocarbons and permanent gases.
- Microseeps built a unique flame ionization detector for the analysis of light hydrocarbons that can achieve MDL's for ethane and ethene of 5 ng/l. This is three orders of magnitude lower than can be achieved on commercial FID's.

  At many sites levels of ethene or ethane are below the usual 5 – 10 ug/l detection limits of FID's used by other laboratories, thus their data for these samples is reported as all < 5.0 ug/l. This may suggest that vinyl chloride is not degrading when in fact it may be. Such data may lead to erroneous conclusions, faulty remedial decisions, and unnecessary costs.

RSK 175 - LowLevel

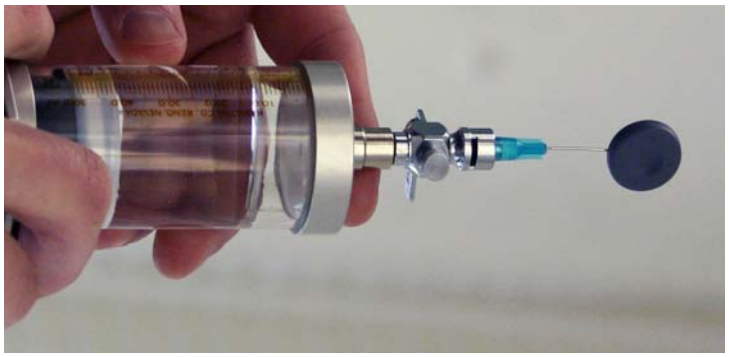
Supporting Monitored Natural Attenuation

It is often stipulated that a particular method be used to measure dissolved methane, ethane and ethene (MEE). The technical reason for that measurement is straight-forward: the methane is a by-product of the reactions that facilitate reductive dechlorination, and the ethane and ethene are the end-products of dechlorination. The choice of method is considerably less straight-forward.

RSK 175 describes a procedure, particular to the R.S.Kerr USEPA laboratories in Ada, OK. It is not a promulgated method. Indeed, the work-group that develops methods for the EPA is tasked with developing methods to measure hazardous contaminants, and MEE is not hazardous. However, the scientists at Ada developed RSK 175 as a high quality, defendable and documented way to measure MEE and have been gracious enough to share it with many practitioners.

Microseeps scientists also wanted to measure MEE but realized that it was important to look at concentrations below those measurable with RSK 175. AM20Gax was developed to allow Microseeps to measure very low MEE concentrations, but because it was not strictly an RSK 175 method, many practitioners could not use it.

Microseeps, the best source for RSK 175



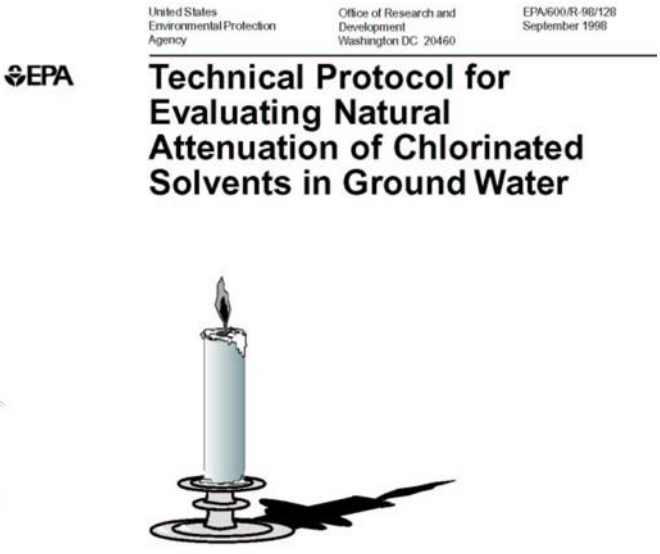
Microseep's RSK 175 is a combination of the technology behind the high sensitivity of AM20Gax and the quality control program of RSK 175..

Microseep's RSK 175 gives the superior sensitivity AND the more widely recognized quality assurance of RSK 175.

The reporting limits of AM20Gax are still slightly lower. For that reason Microseeps routinely recommends AM20Gax for MEE. However, if RSK 175 is required, we can supply it with superior detection limits whenever it is requested.

RSK175 Reporting Limits

Methane	**0.1ug/L**
Ethane	**0.1ug/L**
Ethene	**0.1ug/L**


United States Environmental Protection Agency | Office of Research and Development Washington DC 20460 | EPA/600/R-98/128 September 1998

EPA

Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Ground Water

Volatile Fatty Acids

Demonstrating Microbial Activity

Microseeps developed a new method for analyzing volatile fatty acids

With the proliferation of enhanced biodegradation as a remediation technology, Microseeps recognized the need to provide analytical support to clients using organic substrates to stimulate anaerobic degradation.

Some of these substrates are just lactic acid which is subsequently fermented by the in-situ microbial consortium into pyruvic, butyric, propionic and acetic acids. Thus, the presence of this suite of acids after addition of only lactic acid is evidence of the activity of the microbial process. This rather unique suite of so called volatile fatty acids is also a convenient tracer to monitor where the treated water flows.

The older method for determination of these acids was a gas chromatographic (GC) method with a detection level of about 15 mg/l for lactic acid, 5 mg/l for pyruvic, and about 1 mg/l for the rest. Given these detection levels, the suite of acids could not be traced down the groundwater flowpath very far before the concentration was below the detection level. Reports would often show reductive dechlorination in areas where VFA's were not detected, giving the impression that the engineered stimulation was not responsible for reductive dechlorination. Therefore, it became important to be able to look at very low concentrations of volatile fatty acids so that the migration of the acids could be used as a tracer.

Microseeps developed a new methodology for analysis of the volatile fatty acids which uses ion chromatography (IC) instead of gas chromatography (GC). This method has achieved significantly lower detection levels in the range of a few tens of ug/l.

In addition, Microseeps has extended the method to include the 5 and 6 carbon acids to accommodate clients who are using vegetable oils and other organic substrates instead of lactic acid.

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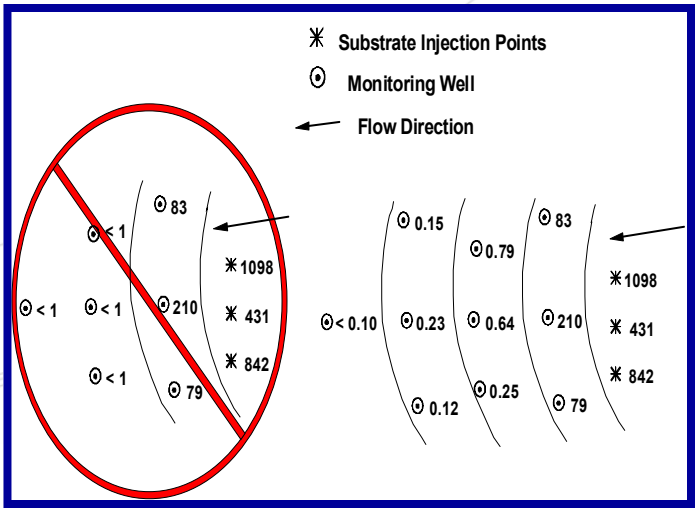

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Compound Name	Acetic Acid	Propionic Acid	Butyric Acid	Pyruvic Acid	Lactic Acid	n-Pentanoic Acid	i-Pentanoic Acid	n-Hexanoic Acid	i-Hexanoic Acid
Low Level PQL (ppm)	0.07	0.07	0.07	0.07	0.10	0.07	0.07	0.10	0.10

Total Inorganic Carbon

Demonstrating Microbial Activity

Microseeps has developed a method for analyzing Total Inorganic Carbon that takes the guess work out of calculating TIC across a site.

Carbon dioxide is one of three forms of inorganic carbon that exist in groundwater. The other two are carbonate, and bicarbonate. These three forms of inorganic carbon exist in groundwater in a pH dependent equilibrium.

Thus if carbonate from soluble carbonate minerals in the soil matrix dissolves into the groundwater, there will be concentrations of all three forms of inorganic carbon at equilibrium. Since soluble carbonate minerals are common, there is almost always some background level of TIC, even in uncontaminated aquifers. In contaminated aquifers, there is almost always oxidation, either aerobic or anaerobic, of organic material. Thus there is almost always the generation of inorganic carbon in the form of carbon dioxide in contaminant plumes.

Therefore if one were to measure TIC across a contaminant plume, one would find background concentrations over uncontaminated portions of the aquifer, and anomalous concentrations of TIC over contaminated portions of the aquifer.

Microseeps has a reporting limit of 1 mg $CaCO_3$/l for TIC

TIC can be determined from a groundwater sample if the sample is acidified before determining the dissolved carbon dioxide. Acidification causes the equilibrium to shift such that most of the inorganic carbon is in the form of carbon dioxide. Thus a dissolved carbon dioxide measurement of an acidified water sample gives an excellent measure of TIC.

Many may feel that TIC is unnecessary, measuring alkalinity is sufficient. That is not true because Inorganic Carbon is present as both alkalinity and as carbon dioxide, and the ratio between the two can vary widely across a site depending upon pH and dissolved solids. The crucial pH is near neutral, and the dissolved solids will increase dramatically as biological activity causes mineral dissolution. Thus there can be wide swings across a site that alkalinity alone or carbon dioxide alone do not point out. Granted, by doing both tests, accounting for the pH and the TDS, and making lots of assumptions, TIC could be calculated. However, the method Microseeps has developed eliminates all of that and provides TIC in one measurement, avoiding multiple analyses and arduous interpretation.

Why Measure TIC?

- TIC is the final product of ALL fuel Oxidation.
- TIC analysis provides the most comprehensive look at inorganic carbon across a site.
- Mapping TIC across a site reveals the background TIC that results from aquifer sediments.
- Mapping TIC across a site also reveals the elevated TIC resulting from bioxidation of contamination from MTBE and other fuel oxygenates.
- TIC combines bicarbonate, carbonate, carbonic acid and dissolved carbon dioxide into one analysis.

Anions and Cations

Supporting Monitored Natural Attenuation

Microseeps' ion chromatographic methods are superior in reliability and detection limits

There are a group of ionic species in groundwater whose concentration is also useful in characterizing chemical and biological degradative processes which are active in a contaminant plume.

These ionic species may be naturally occurring, such as

- nitrate
- ferric iron
- sulfate

Or they may be products of the degradative process itself, such as:

- chloride
- nitrite
- ferrous iron
- Manganese (II)

For several of these species there are field kits and other field analytical tools which may be utilized depending on the needs of the client.

For species like ferrous iron and nitrite which are in-situ reduction products of the active biological processes in the plume, there was concern that these species reoxidized during transport to the laboratory. For that reason, field data was preferable to laboratory data.

Microseeps has overcome this problem by the utilization of glass vials with septa which precludes the diffusion of oxygen into the sample, thus the reduced state of these species may be preserved for long periods of time and laboratory analyses may be equally as reliable as the field methodologies.

Even in the laboratory, there are choices in methodologies that may be used to determine the concentration of each of these species. Microseeps has developed ion chromatographic methods which monitor each ion independently and in general are superior in terms of reliability and detection limits.

Bioavailable Ferric Iron Analysis—BAFeIII

Supporting Monitored Natural Attenuation

Bioavailable ferric iron is one indicator that natural attenuation can occur

Ferric iron is used by iron-reducing bacteria under anaerobic conditions to degrade benzene, vinyl chloride, and various other organic compounds. In the process, ferric iron (FeIII) is reduced to ferrous iron (FeII).

Successful natural attenuation requires an adequate quantity of bioavailable ferric iron. However, predicting the amount of bioavailable ferric iron present is difficult.

To be bioavailable, ferric iron must be capable of being reduced by microorganisms that oxidize another chemical species and derive energy from the electron transfer. The amount of bioavailable ferric iron is affected by many factors, such as surface area, groundwater pH and specific conductivity, concentrations of divalent cations, and concentrations of electron shuttles.

Bioavailable Ferric Iron (BAFeIII) Assay measures the amount of ferric iron in soil or sediment that can be reduced to ferrous iron (FeII) by iron-reducing bacteria. In the process, the test also identifies the amount of bioavailable manganese.

The BAFeIII test is used in conjunction with other analyses to evaluate site suitability for natural attenuation; and for monitoring effectiveness and efficiency of ongoing natural attenuation.

Some of the reasons to use BAFeIII include:

- To prove that iron added in an "iron wall" treatment can also increase the bioremediation capacity of an aquifer by measuring the quantity of iron that can be used in bioremediation.
- To measure how much carbon substrate (e.g. HRC®, ethanol or molasses) would be consumed in "burning through" the bioavailable ferric iron and establishing methanogenesis.
- To assess the assimilative capacity of an aquifer into which a petroleum product has been released.

Aqueous and Mineral Intrinsic Bioremediation Assessment

Supporting Monitored Natural Attenuation

Microseeps' has developed a suite of analytical methods in support of AMIBA

Over the last decade, Microseeps has developed the means to determine that intrinsic biodegradation is an active process at sites by studying their aqueous geochemistry. Efforts to calculate a mass balance or to determine the efficiency of these in-situ processes, however, have been difficult at best.

There is growing evidence that these efforts may be helped by consideration of the minerals which accumulate as a result of the in-situ redox processes associated with each plume, particularly the reduced iron and sulfur minerals. AFCEE has suggested a detailed set of analyses as a part of their Aqueous and Mineral Intrinsic Bioremediation Assessment (AMIBA) protocol to characterize both the aqueous and mineral phase to evaluate intrinsic bioremediation processes.

In support of such AMIBA studies, Microseeps has developed and is performing a suite of analytical methods for clients who are seeking a more definitive evaluation of their sites.

AMIBA samples are solid samples collected from beneath the water level and stored frozen under anoxic conditions. This process is not technically intensive and every effort has been made to make it practical for the average sampling team. To help our clients take advantage of the power offered by AMIBA, Microseeps has written descriptive sampling procedures that cover a range of sample collection methods and contain flow-charts to help sampling technicians rapidly and easily determine which procedure they are to use.

Weak Acid Soluble Iron (WASFe) – The measurement of surface bound iron by WASFe. It is useful for assessing if iron reduction has begun and may also be useful for assessing the potential for abiotic degradation.

Acid Volatile Sulfide (AVS) – The measurement of FeS produced by the products of iron reduction (Fe^{+2}) and sulfate reduction (S^{-2}). This measurement helps prove that sulfate reduction is an active part of the remediation, even if sulfide is not observed in the ground water. AVS measurement is sometimes referred to as ferrous sulfide measurement.

Strong Acid Soluble Iron (SASFe) – The measurement of the capacity of material to support iron reduction. This is a particularly useful measurement when a carbon substrate such as EOS or HRC is to be added. It can be determined how much iron must be overcome to get to methanogenesis or sulfate reduction.

Chromium Extractable Sulfide (CrES) – This is the measurement of slightly aged and oxidized products of sulfate reduction, such as FeS_2 and elemental sulfur. This helps assess the historic contributions of sulfate reduction. CrES measurement is sometimes referred to as ferrous disulfide measurement.

Vapor Analysis — Method—AM 4.02

Without The Use of Tedlar Bags

Soil vapors from vapor intrusion pathways are an excellent opportunity to use SM 10 and AM 4.02

Microseeps recognized the need for a sampling method specifically for collecting vapor samples from soil gas surveys and from soil vapor extraction (SVE) systems.

Currently there are two methods that are normally referenced when SVE sampling or soil gas screening samples are required. They are Method 18 – Tedlar Bags and Method TO 14 or 15 - - Summa canisters. These are methods that were adapted for these situations and are not practical or economically feasible.

The Advantages of Using Glass Vials

- **Holding Time** – Samples collected in bags must be analyzed within 48 hours. The holding time for glass vials is the industry standard, 14 days.
- **Shipping** – Tedlar bags must be shipped overnight, limiting sampling to Monday through Thursday unless special arrangements are made for Saturday analysis. With glass vials, overnight shipping is unnecessary.
- **Dependability of Analyses** – When a sample is taken using a bag, it is adsorbed on the sampling port and also the inner wall of the bag. With vials there is still adsorption, but during the analytical process the vial is heated to approximately 75 degrees centigrade. This is more than enough to desorb all constituents and make them available for the analytical process.
- **Sample Collection** – Using a disposable syringe you merely secure the sample from the sample port and transfer it to the 22cc glass vials. We supply all of the necessary equipment with the vials. None of this equipment is reused, therefore there is no costly clean up.
- **Cost** – Two glass vials per sample are sent out in each kit and a duplicate sample is taken at each sample point. If there is any reason to believe that the sample result is not correct the duplicate sample can be analyzed. It is cost prohibitive to take a duplicate sample with either a Tedlar Bag or a Summa Canister.

Neither of these methods was developed specifically for soil vapor sampling. Therefore, Microseeps' developed Method SM 10 specifically for these purposes.

Microseeps' method SM 10 was designed to capture a discreet vapor sample and to provide the highest degree of confidence that the analytical result will give an accurate representation of the efficiency of the system.

Analytical method AM 4.02 utilizes glass vials as the sample receptacle as opposed to the commonly used Tedlar bags or Summa canisters.

Glass Vial / VOC Recovery Study Using AM 4.02

Microseeps' research showed no significant VOC loss using glass vials

Compound Name	Day 1	Day 4	Day 7	% Recovery After 7 Days
Vinyl Chloride	1027.8	1002.3	978.5	95.2
Bromomethane/ Chloroethane	28.05	28.20	27.83	99.2
1,1,Dichloroethylene	11.07	11.18	10.99	99.3
Trans-1,2 Dichloroethylene	11.02	10.97	10.6	96.2
1,1 Dichloroethane	11.00	11.11	10.96	99.3
Chloroform	9.07	9.11	8.91	98.3
1,1,1 Trichloroethane	8.13	8.23	8.08	99.4
Carbon Tetrachloride	8.81	6.83	8.77	99.4
1,2 Dichloroethane	10.92	10.66	10.55	97.0
Trichloroethylene	8.02	6.15	7.81	97.4
1,1,2 Trichloroethane	7.92	7.72	7.68	96.4
Tetrachloroethylene	6.01	5.59	5.36	89.5

Insight Reports are more than just the numbers!

Reporting Questions?

Call
800-659-2887
Mon. - Fri.
9 -5 EST

For some of the work that environmental professionals do, the number is the story....is it above or below the MCL? Is it within the limits of quality and reliability? Is it higher or lower than it was before?

For most of Microseeps work, however, the number is only part of the story. Particularly when designing and implementing an in-situ degradation process it is understanding the meaning of the numbers that can make the difference in the effectiveness of the entire project.

At Microseeps, we not only have the means to generate the numbers that will support a complex work plan, we have the insight and understanding to help you interpret the numbers and the ability to present a report that demonstrates and makes clear the results which your client and the regulators need to know and understand when deciding how to proceed or when to stop.

Microseeps reports do not replace the standard reports that you are used to seeing. In fact for our specialty analyses you will receive a report that is just numbers, results, QA/QC, etc. However, Microseeps will provide a report that can be used as an addendum to your report that provides insight and tells you what the numbers mean. That understanding will be useful in the decision making process.

These are not standard reports. The scope and fee for developing insight reports should be discussed and agreed upon in advance. However, if it becomes apparent that a special report will be helpful, it is still possible to create the reports as part of a post project review.

For more information on how Microseeps can be of help in the reporting process or for a sample report please contact your sales person or customer service at Microseeps.