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Write in 199
Don’t make waves

Analysis of water samples for total petroleum hydrocarbons

By James E. Bruya and Andrew John Friedman

The problem with measuring petroleum products in water samples is that the individual compounds that make up these products are generally water insoluble. Each product is a complex mixture of individual chemical constituents that constantly changes from batch to batch and from site to site.

They tend to float on top of water (though occasionally they sink) or they adsorb onto any solid surfaces present within a water column. Usually only a small fraction of a petroleum product is truly water soluble. This means that petroleum hydrocarbons will often be present as a sheen on top of a water phase or they will be adsorbed onto any suspended particulates present within a water column. The material actually present in the water phase may be the water soluble portion of the petroleum product or can be oxidized hydrocarbons either from naturally occurring sources or from the degradation of the petroleum product itself.

Because of the possibility that the petroleum contamination can be associated with any or all of these multiple phases, standard analytical methods require extraction of both the water and the container in which it was sent. This means that the laboratory analyzes everything in the sample container.

It is important for anyone using an analytical laboratory to be able to review the analytical data. The current system functions such that the standard lab does not receive the information necessary for them to determine if the data are being used correctly.

There are several considerations to review analytical data to assure that the information received addresses the issues of concern at the site.

What happens when a petroleum product hits a water layer?

The old adage, “oil and water don’t mix,” applies well to petroleum products and groundwater. Petroleum products are composed primarily of hydrocarbons (alkanes, alkenes, aromatics) with low levels of oxygen, nitrogen and sulfur-containing compounds (hetero-atom-containing compounds). Except for the low molecular weight alkanes (pentanes...
and hexanes) these compounds are insoluble in water. The alkenes are slightly more water soluble than the alkanes and the aromatics are even more water soluble.

Benzene, toluene, ethylbenzene and the three xylenes (o-xylene, m-xylene and p-xylene) are the most notorious and most water soluble of the aromatic hydrocarbons.

The vast number of hetero-atom-containing compounds present in petroleum products, especially in degraded petroleum products, can be much more water soluble than their hydrocarbon counterparts. The identification and quantitation of these hetero-atom-containing hydrocarbons can be virtually impossible using standard analytical techniques. These compounds are usually present at low levels, they are chemically labile, their presence can vary from sample to sample and from site to site and it is difficult to obtain appropriate analytical standards.

Figure 1, left, shows a typical 8015-type gas chromatographic (GC) trace of a crude oil pumped from an oil field in the Cook Inlet of Alaska. The dominant feature to notice is the pattern of 20 evenly spaced peaks. These are the n-alkanes and are the dominant compounds present in virgin crude oils. Many of the petroleum products made from crude oil will also show this dominance of the n-alkanes. The branched and cyclic alkanes and alkenes, as well as the aromatics, alkylated aromatics and hetero-atom-containing compounds make up the rest of the small peaks.

When crude oil comes into contact with water, only a small portion of the

*Continues on page 46→*
Where to draw the line

Hazardous? Non-hazardous? Which regulations apply to what?

By Kevin M. Bonzo

While service stations, utilities, industrial facilities and others strain to weather the burden of underground storage tank (UST) corrective actions, consultants, contractors and waste handling facilities continue to harvest a bonanza of remedial work. The remediation business continues to emerge as a significant growth industry, fueled by a profusion of environmental regulation.

Although intimidating, the regulations generated under the rulemaking authority of the Environmental Protection Agency (EPA) are the backbone of the industry and offer opportunities for those businesses poised to take advantage of their effects. Therefore, a working knowledge of the regulations is tantamount to success regardless of which side of the cleanup you’re on.

The corrective action mandates of RCRA (Resource Conservation and Recovery Act) Subtitle I, are responsible for a great deal of the recent attention given to the management of hydrocarbon contaminated soils (HCS). When the option of choice is simply “hog and haul” to the nearest landfill, a minimum amount of regulatory expertise is required for waste characterization. However, waste soil management decisions become increasingly complex in cleanups at large industrial facilities where a frightening assortment of contaminants, manufacturing processes, use of product/waste storage units and historical site operations must all be considered before sending in the excavators. Complicating this process is the bewildering medley of state and federal regulations.

Individual states have taken a variety of approaches to the regulation of non-hazardous solid wastes, including the management and disposal of HCS. Some states have enacted stringent rules which require handling of certain HCS as hazardous waste. Interestingly, the disposal of contaminated waste soils may either be exempt from regulation, regulated as a solid waste or fall under hazardous waste regulations. The hazardous category may be
further divided, depending on whether the hazardous waste soil may be directly land disposed or requires treatment prior to disposal.

Some states permit various methods of treatment which effectively change the regulatory status of contaminated soils. Treatment of HCS to specified “clean standards” is permitted in some states so that the treated soil is no longer considered to fall under the solid waste regulations. Similarly, on-site stabilization treatment of hazardous waste soils may be approved through an “agency-sanctioned” RCRA closure or corrective action.

Regardless of what state you are in, remember to “read before you dig.” Consider the following steps:

Sources of information

All the participants in a cleanup project must first understand it is impossible to ask or answer too many questions about the site. The first thing everyone wants to know is who or what is driving the cleanup in the first place.

Although more and more businesses are recognizing the need to voluntarily clean up their back yards, still, more often than not, remediation activity is fueled by some regulatory agency pressures or other legal means. Regardless of how the regulatory agencies compel industries to undertake remedial work, one certainty is that a paper trail or administrative record of official agency activities has been compiled along the way and should be available for review.

These documents often describe the agency’s concerns, summarize historical compliance problems which resulted in the formal action and provide technical insight into the nature of the contamination—all of which is valuable information to proceed with the cleanup.

If more than one firm is involved in the project, it is beneficial to everyone if they share information—site characterizations, closure plans, remedial investigations, feasibility studies or other documents that may bear on the ultimate success of the project. Such documents are typically submitted to regulatory agencies for review and comment—and often include special terms attached to the final report as a condition of approval.

For example, an approved closure plan may require contaminated soils to be managed as hazardous waste if certain metals are present at statistically significant levels above background or if organic constituents are detected above the analytical detection limit.

If the cleanup is a voluntary undertaking with little or no agency oversight, responsibility falls on the owner/operator to collect and provide as much information as possible about the site. The disposal firm must ask all the right questions, conduct an on-site audit or field verification and document everything.

Source of contamination

Whereas any one of a million organic or inorganic contaminants may be present in soil generated through a remedial action, the contaminant and

Continues on page 10→

What is it, and where did it come from?

Someday you may be asked to clean up someone else’s spill.

Often, in the mad rush to satisfy state or federal regulators, characterization and identification analyses are overlooked. After the regulators are gone, questions come up like: “Was it all ours?” or “Just how old was it?”

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Draw the line, from page 9

its concentration in the waste soil is not nearly as important as how and from where did the contaminant originate. Management of waste soils under RCRA is a risky undertaking when disposal decisions are based on contaminant concentration alone. Consider not only the source material, but the source unit (tank? pipeline?) as well.

Analytically “clean” soils may be listed hazardous wastes. Soils which exceed the limit for benzene may either be a regulated hazardous waste—or not—depending on the regulatory status of the tank which leaked. Don’t let yourself get caught up in the “numbers game” before considering all regulations, policies and agency rule interpretations which ultimately govern disposal.

Evaluate how the storage of the material and the source unit is regulated. RCRA Subtitle I (40 CFR 280) establishes the technical standards and corrective action requirements for owners and operators of USTs. Pay particular attention to what a UST isn’t, such as a “tank used for storing heating oil for consumptive use on the premises where stored” and other exclusions to these rules.

A tank excluded by the UST rules is not afforded the special exemption incorporated into the TCLP (Toxicity Characteristic Leaching Procedure) rules (see 55 FR 11798, March 29, 1990). A last minute drafting change specifically exempted petroleum-contaminated media and debris that fail the test for the toxicity characteristic of 40 CFR 261.24 and are subject to the corrective action regulations under part 280 of this chapter. This exclusion was clarified in a final rule correction (55 FR 26986, June 29, 1990) which noted that the exclusion applies only to petroleum contaminated media and debris which exhibit toxicity characteristics for any of the new organic constituents and are subject to corrective action under part 40 CFR 280.

Essentially, contaminated soil generated from corrective action measures involving a Subtitle I-regulated tank would not be considered a hazardous waste under federal rules even if the TCLP extract for benzene exceeded the regulatory threshold of .5 mg/l. On the other hand, that same soil would be a hazardous waste if lead content exceeded the regulatory threshold of 5 mg/l.

Although this exclusion was made in recognition of a potential national capacity shortage for disposal of HCS as hazardous waste, the peculiar aspect of this exclusion is why the EPA would require such a generator to conduct a TCLP analysis for organics in the first place!

RCRA Subtitle C (40 CFR 260 et seq.) establishes the hazardous waste rules. Soil remediation involving hazardous wastes requires unique regulatory oversight for management of residuals. An evaluation of potential contaminant sources must be conducted before a waste soil management program can be developed.

Contaminants may have originated from listed hazardous wastes, including those commercial chemical products found in 40 CFR 261.33. Careful attention to section (d) of this reference reveals that contaminated soils resulting from the cleanup of a spill of any commercial chemical product or manufacturing chemical intermediate having the generic name listed in paragraph (e) or (f) of this section would be a hazardous waste.

For example, remediation of soil contaminated by a release involving technical grade benzene (U019) would generate waste residuals retaining this hazardous waste number. However, this assumes that the waste soil contains the hazardous waste as described in EPA’s “contained-in” policy.

The basis of this policy recognizes that environmental media, such as soil and groundwater are not normally considered as waste materials. As such, the mixture rule (see 40 CFR 261.3) does not apply to mixtures of listed hazardous wastes and environmental media.
Soils that contain the waste in question (benzene, in this example) must be managed as hazardous waste—although the concentration level is somewhat open to debate.

As a rule of thumb, concentrations in excess of the method detection limit, for non-naturally occurring organic constituents, may be sufficient to require such material to be managed as hazardous. Decisions on such technical matters are seldom consistently applied by different district or regional offices of regulatory agencies.

What about PCBs?

Management of soils contaminated with polychlorinated biphenyls (PCBs) can be a complicated undertaking. Classification of such soils for disposal must not be confused with the cleanup standards required by the PCB spill cleanup policy found at 40 CFR 761, Subpart G.

To begin the analysis, the site’s operating history must be thoroughly evaluated, keeping in mind critical regulatory dates on which various TSCA (Toxic Substances Control Act) regulations were promulgated. The disposal of soils contaminated by pre-February 17, 1978 spills of untested mineral oils (which presumably contained PCBs) may be permitted in a sanitary landfill without consideration of the PCB “dilution” rule and providing the resultant PCB concentration in the soil is less than 50 mg/kg as required by 40 CFR 761.60 (a) (4).

However, beginning in 1978 (with the promulgation of TSCA regulations) the prohibition on dilution is operative and the concentration of PCBs in the spiked material must be evaluated.

As defined under TSCA (see 40 CFR 761.123) the term, “spill” requires that the concentration of PCBs spilled is determined by the PCB concentration in the material spilled—as opposed to the concentration of PCBs in the material onto which the PCBs were spilled.

This definition further states that where a spill of untested mineral oil occurs, the oil is presumed to contain greater than 50 ppm (parts per million) but less than 500 ppm PCBs and is subject to the relevant requirements of the spill cleanup policy. Similarly, the waste contaminated soil would also be subject to the disposal requirements for greater than 50 ppm contaminated soil regardless of the actual PCB concentrations.

Further complicating any analysis of historical PCB contamination are recent administrative decisions by the U.S. EPA regarding two cases. Standard Scrap Metal Co. and City of Detroit Public Lighting Dept., whereby the prefatory note to 40 CFR 761.60 (which contains the exemption for pre-February 17, 1978 disposal of PCBs) was closely examined. Briefly, these rulings indicate that the scope of the exemption applies only to PCBs that were placed in a “disposal site” rather than “spilled” prior to February 17, 1978. The offshoot of these rulings may affect the regulatory classification of pre-1978 spill cleanup residuals and application of the dilution rule. Stay tuned.

In the spring of 1991, TSCA enforcers took on three big league PCB players, namely, General Motors Corp., Waste Management Inc. and Browning-Ferris Industries. The complaint, filed by EPA in March 1991, alleges that the two waste disposal facilities accepted PCB-contaminated sludges which should have been incinerated in a TSCA-regulated facility. The waste sludges were supposedly contaminated by PCBs in hydraulic oils used in the Messena, N.Y. GM plant. Hydraulic oils containing greater than 500 ppm PCBs are required to be incinerated. So too, are sludges and soils contaminated by such oils regardless of the resultant concentration after dilution. In their defense, a spokesperson for Waste Management, Inc., was quoted in the Wall Street Journal saying, “We can’t be policemen to the world.” Unfortunately, this is exactly what

Continues on page 51

**PAVING THE WAY TO SOIL REMEDIATION**

**THE PROBLEM**

The former Johnson Steel and Wire Company site in Worcester, Massachusetts, presented a challenge to both the Massachusetts Department of Environmental Protection (DEP) and Intratrans Container, Inc., who wanted to develop the site as a major intermodal rail terminal in Worcester. The soil was contaminated with both petroleum products (fuel oil, and high levels of lead). American Reclamation Corporation (AmRec) was contacted and asked to solve the problem.

**THE SOLUTION**

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Looking for pesticides

Advances in gas chromatography equipment and software characterize chlorinated pesticides in soil


The introduction of portable gas chromatography (GC) devices with higher range temperature controls now means that volatile as well as semi-volatile compounds can be analyzed with the same instrument. This means high molecular weight pesticides and polychlorinated biphenyls (PCBs) can now be analyzed.

Over the past decade, the main application of portable GCs has been the detection of volatile hydrocarbons. The growth in popularity of GC use has been enhanced by the flexibility, improved accuracy and lower cost afforded by on-site measurements. Until recently, portable GCs with photoionization detectors (PID) have either had no temperature control or limited ability to control temperatures up to 50°C.

While techniques such as direct injection and headspace analysis are common for volatile hydrocarbons, different techniques are required for non-volatile hydrocarbons. The most suitable technique for quantitative removal of organochlorinated pesticides from soil or water is probably solvent extraction.

The use of pesticides is an integral part of agriculture in developed countries to destroy or control weeds, fungi, insects and other pests. The widespread use of agricultural pesticides has created additional problems due to runoff and subsequent contamination of substantial quantities of surface water as well as groundwater tables.

In addition, there are numerous sites where chlorinated pesticides have been stored or used—such as areas surrounding airplane hangars where these pesticides are loaded for aerial spraying. One problem with the chlorinated pesticides is that they do not easily break down and thus, remain in the environment for a long time.

The Spittler extraction procedure is a simple field method which appears to be applicable for a variety of pesticides in a number of different matrices, including soil and water.

It involves taking an 800 mg soil sample or a 10 milliliter (ml) water sample, adding one cubic centimeter (cc) of a 1:4 water methanol mixture and adding one ml of hexane. Shake for 30 seconds, let stand for 30 seconds (if the mixture emulsifies, then centrifuge the sample). Inject the top layer (hexane) into the GC. This method was originally developed for the analysis of PCBs. As a result, tests were performed on the extraction efficiency of DDT and some of its isomers to determine the viability. Tests were run at the one to 10 parts per million (ppm) levels since the action level for dichloro diphenyl trichloroethane (DDT) is presently two ppm. The efficiencies of extraction from soil spiked with DDT were 95-99 percent.

Samples were analyzed on site with the portable GC but grab samples collected for laboratory analysis were stored in glass jars and analyzed within a few weeks of the time of collection. All soil samples were dried prior to weighing.

Field analyses were performed using an HNU Systems model 311 GC equipped with a PID. Following the extraction, a one µL sample of the hexane layer was injected into a capillary injector and separated on a 15 meter by 0.32 millimeter capillary column which was main-

J.N. Driscoll is founder and president of HNU Systems of Newton, Mass. M. Whalen is application chemist, C.D. Wood, Ph.D., is manager of research and development and M. Duffy is product manager of HNU Systems. C.A. Cihak is a chemist for the Army Corps of Engineers.
ttained at 180°C. The carrier flow rate was 15 ml per minute of ultra high purity nitrogen. The GC was calibrated with a DDT standard. A dichloro diphenyl dichloroethane (DDD) standard was also run to verify that no peak overlap occurred with DDT.

A quality control (QC) protocol was maintained for the analytical results obtained in the field which consisted of analysis of a standard to determine percent recovery and analyzing duplicates on sample extracts to verify analyst reproducibility. The instrument was recalibrated at the beginning of each morning and afternoon shift and at any change in condition.

Laboratory analyses for a number of field samples were performed with a GC equipped with dual electron capture detectors (ECDs) and a retention index monitoring (RIM) system. The RIM system is a tool for the automatic interpretation and identification of complex mixtures based on a unique pattern recognition algorithm for search of index peaks. Compound identification is based on two columns of different polarity.

Separations were accomplished on a pair of 0.32 mm by 25 meter (per identification) fused silica capillary columns. The oven temperature for these analyses was 150°C. Both column inlets were installed into a single column injector.

Identities were made with Micman identification software which compares the results on both columns to a pre-established library and then lists results only when the compound is found on both columns. Identifications were based on absolute retention time.

Continues on page 14→

Fig 2: DDT in soil analysis—field vs. lab results shows correlation coefficient of 0.998.

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Looking for pesticides,
from page 13

Sampling was accomplished using stainless steel coring tools to depths ranging from zero to 19 inches. Surface samples were collected with stainless steel spatulas. Soils recovered from the coring device were placed in a stainless steel bowl and mixed thoroughly prior to analysis. All equipment was decontaminated between sampling depths and locations.

In the absence of any regulatory cleanup standard, the Environmental Protection Agency's (EPA's) proposed corrective action level of two ppm was used as the guideline.

The soil was manually excavated and contained in 55-gallon steel drums. Upon completion of the first excavation lift, the bottom of the pit was sampled. Excavation proceeded to areas with concentrations of DDT at levels exceeding two ppm. This process was repeated until all concentrations in the pit were less than two ppm. The soil was disposed in a permitted hazardous waste landfill.

A typical chromatogram of a field sample measured as 10 ppm DDT is shown in Figure 1, page 13. A number of field samples were returned to the U.S. Army Corps of Engineers, Missouri River Division laboratory, for verification of results for DDT using SW-486 method 8080.

The agreement between the field data and the laboratory results was excellent with a correlation coefficient (r²) of 0.998 (see Figure 2, page 13). The regression equation which describes the relationship between the two variables is: y=0.85x + 0.295. A three-dimensional plot of the results is shown in Figure 3, above left. The X/Y axis is the plane of the hazardous waste site while the Z axis is the concentration found—expressed in ppm.

The three-dimensional image is an easy way to visualize the spatial profile of pollutants at the site.

The present method of testing for DDT, which has a detection limit of about 0.2 ppm, is satisfactory for the current action level of two ppm. The method could be improved by changing the extraction solvent from hexane (IP=10.18) to pentane (IP=10.35) which has a considerably lower response to the PID. Tests have shown that DDT is still extracted with this solvent with an efficiency exceeding 95 percent. Since pentane has a lower response on the PID (10.2 eV) than hexane, it is possible to use range one, which is ten times more sensitive than range two, as required with the stronger response of hexane. The chromatogram in Figure 4, (above), in range one demonstrates the improved results obtained.
with pentane. The detection limit with pentane extraction can be reduced to < 0.05 ppm.

The chromatogram in Figure 1, page 13, with the PID indicated that the major peak was DDT, although a number of minor peaks were noted.

The chromatogram in Figure 5, (right), demonstrates both the sensitivity of the ECD for organochlorinated compounds as well as the unique dual column sensitivities of the Micman software. In Figure 6 (below), other chlorinated isomers of DDT were identified along with low levels of malathion—which was not supposed to be present at this site. Note that the peaks for DDD and dichloro diphenyl ethane (DDE) elute before DDT in Figure 2, page 13, but the lower sensitivity of the PID on range two makes these peaks appear quite small. The peaks with a longer retention time than DDT do not appear to be pesticides since there was no response with the ECD. They are hydrocarbon impurities in the solvent. The detection limit for DDT with the ECD was less than one ppb (part per billion).

This particular hazardous waste site had been visited twice with samples sent for laboratory analysis. On the third visit with the on site analysis, additional areas of contamination were observed that had been undetected previously, in spite of 44 sample analyses. This type of discovery is not unusual and serves to highlight the benefits of using on site analysis equipment.

The combination of the Spittler extraction technique with a portable GC equipped with a PID results in good field analysis for DDT in soil. These data were in high correlation with laboratory data. The method is useful to one-quarter of the two ppm action level with hexane as the extracting solvent and to 1/40th of the action level with pentane. And, this method demonstrates that volatile and semi-volatile compounds can be run on the same GC with a PID.

The original method was described for the analysis of PCBs. Since PCBs also respond with excellent sensitivity, these species could also be determined with a GC. An obvious advantage of using the PID for analysis of PCBs is that the response does not vary with the degree of chlorination like the ECD, thus it could provide a better quantitative method for the various PCB isomers.

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Reduce sampling errors

Careful extraction method can improve accuracy of soil analyses by as much as an order of magnitude

By Ellen G. Cool

Most remediation events begin with a sampling program to delineate the nature and extent of the affected area. Most investigators realize that the greatest potential for sampling errors occurs in the field, in the actual collection process. Attention to detail and quality control is essential to obtain accurate characterization of a site.

Subsurface samples may be collected from a backhoe bucket used to excavate a test pit or from a split-spoon sampler driven by a drill rig. A portion of the soil retrieved by one of these methods is generally transferred into a clean glass jar or vial, packed with ice and shipped (with appropriate documentation) to a laboratory for analysis of the chemical parameters of concern.

Laboratory personnel perform the analysis by placing a portion of the sample in a fluid which chemically extracts the contaminants from the soil. If an organic chemical such as a chlorinated solvent or a fuel-related compound is the contaminant of concern, the extracting solution is generally methanol. Freon may be used for analysis of total petroleum hydrocarbons.

The extract is analyzed and the concentration in the extract is converted to a concentration of contaminant in the extracted soil on a weight per weight basis.

Soil investigation programs are generally designed to identify the source of the release, to determine whether remediation is warranted and to determine the most feasible and cost effective remediation method. Specifically, engineers and scientists rely on the results to address some of the following questions:

- What is the overall mass and concentration of the contaminants?
- What are the relative proportions of contaminants present at the site? Can the ratios of contaminants be used to gain insights regarding the source of the release or the age of the release? For example, weathered fuel may be distinguished from a more recent spill by the decrease in the proportion of the lighter volatile hydrocarbons.
- Is it cost-effective to excavate the contaminated soil or feasible to remediate it in situ?
- Can the soil be remediated by vapor extraction?
- What health and safety precautions may be necessary if the soil were excavated or if other intrusive remedial construction were performed?
- Could excavated soils be accepted by an off-site disposal contractor such as an incinerator, sanitary landfill or hazardous waste landfill?
- How leachable are the soil contaminants and do they threaten groundwater beneath the site?
- What is the risk to human health and the environment posed by the in situ contamination?

If a remedial system is already in place and operational, soil samples may be periodically collected and analyzed to monitor the progress of the cleanup.

Quality assurance and quality control (QA/QC) procedures issued by the U.S. Environmental Protection Agency (EPA) and other agencies govern the methods and protocols used in most chemical testing laboratories. Most labs adhere to these procedures and have a commendable record of reporting accurate results. But, the lab can only report on the samples they are given, so the integrity of the sample is crucial.

As soon as the sample is removed from the subsurface and exposed to the atmosphere, volatile compounds begin to be released into the air. As the field sampler selects a portion of
the material retrieved in the backhoe bucket or split spoon, the soil is further disturbed and still more volatile compounds are lost from the sample.

If the investigation protocol requires that the sample be placed in a 40 ml Teflon-capped vial, the sampler usually has to crumble or deform the sample even further to fit it through the small neck of the vial. In order to most easily fill the vial, the field investigator may also prejudicially select a portion of soil which is free of pebbles, cohesive material or other particles which do not readily fit through the opening.

And, once in the vial, volatile organic compounds originally dissolved in the soil moisture or adsorbed to the soil particles volatilize into the pore spaces until equilibrium between the soil and the air in the vial is reestablished. The net result is that the soil which ends up in the capped and labeled vial that is shipped for chemical analysis may be quite different from the parent soil remaining in the ground.

As an example of how standard tests of soil contamination may be misleading, an industrial site in eastern Massachusetts offers a case in point. Soil and groundwater contaminated with chlorinated solvents was recognized as emanating from the location of a former vapor degreaser. Initial borings through the floor slab and a number of groundwater monitoring wells identified the primary contaminant as tetrachloroethylene (PCE) with trichloroethylene (TCE) and breakdown products of these compounds as subsidiary contaminants.

The lateral extent of contaminated soil in the vicinity of the former degreaser pit was evaluated by a soil gas survey. During this procedure, small diameter probes were driven through holes drilled through the floor slab and the vapor extracted from these probes was analyzed on site using a portable gas chromatograph. The highest concentrations of total volatile organic compounds were measured within a radius of approximately 50 feet from the presumed source. This area was interpreted to represent the area of contaminated soil which was the chief source of the groundwater contamination across the site. A limited number of soil samples collected by conventional split spoon recovery were also analyzed. PCE and TCE were confirmed as the primary contaminants at the vapor degreaser location. Groundwater beneath the contaminated soil was measured to be approximately 10 to 12 feet beneath the floor slab.

Due to the large presumed volume of contaminated soil, excavation was originally ruled out as a feasible remedial alternative. Instead, the original plan to clean up this PCE- and TCE-contaminated soil called for installation of a vacuum extraction system.

As the design for the vacuum extraction was refined, it became apparent that more detailed information regarding the distribution of the contaminants in the soil was necessary. In order to estimate the time required to reach the remediation goal, an accurate measurement of the in situ contaminant concentrations was essential.

In addition, because the total cost of the remedial program was predicted to be principally a function of the amount of

Continues on page 18
Reduce sampling errors, from page 17

carbon used to remove the contaminants from the effluent gas, the total mass of contaminants in the soil became a critical parameter. Therefore, a pilot study for the vacuum extraction system was developed which included intensive and comprehensive sampling of the sub-slab soils.

Prior to installation of the pilot vacuum extraction probes, continuous split spoon soil samples from both the vadose zone and the upper part of the saturated zone were retrieved at each probe location.

Recognizing that loss of PCE, TCE and related compounds during the field sampling could seriously compromise the results of the study, special field procedures were implemented to minimize volatile loss. Large mouth (250 ml) glass vials with Teflon septae in the caps were used to collect all samples. These vials were pre-numbered, labeled and filled with approximately 100 ml of reagent grade methanol. The vials were weighed before and after the methanol was added so the precise amount of methanol could be measured and recorded.

As soon as the split spoons were retrieved and opened, field personnel removed a portion of the soil with a stainless steel spatula, placed it intact into the vial and capped the container. Extreme care was taken not to splash any of the methanol and to submerge the sample completely in the liquid. By using the wide mouth vial, the sample was minimally disturbed. The effect of placing the sample into the methanol was to short-circuit the analytical protocol and preserve the integrity of the sample. Instead of performing the solvent extraction in the laboratory, it was done in the field. When the samples were received by the lab, methanol was removed by syringe through the Teflon septum and injected directly into the gas chromatograph, with no exposure to the air.

In several locations, a “conventional” sample was collected adjacent to the first sample for comparison with the field extraction method. When the analytical results were compared, it was apparent that the concentrations of the samples preserved in the methanol were approximately an order of magnitude higher than the unpreserved samples. For example, a pair of samples collected from eight to 10 feet below the slab was found to contain 790,000 ppb (parts per billion) of PCE in the unpreserved sample, but 6,400,000 ppb in the adjacent preserved sample.

Overall, the results of the pilot test and the soil sampling program showed that not only were the contaminant concentrations locally higher than anticipated, but the volume of contaminated soil was also much less than expected. The soil borings advanced beyond a distance of approximately 15 feet from the source encountered only clean soil above the water table. The earlier soil gas survey had reported “hits” in these areas. The methanol preservation method added confidence to lab reports that contaminants were not detected in these peripheral areas. Therefore, the previous soil gas survey results beyond the 15-foot radius of the source were interpreted to reflect contamination derived from the groundwater plume and not from the overlying soil.

The implications of these results were highly significant. At this site, excavation of the contaminated soil became a more tractable, cost-effective and speedier remedial alternative. Consequently, the remedial response plan which had previously been submitted to the state regulators was revised. The new plan specified excavation of approximately 300 cubic yards of soil and construction of a waste soil pile to be treated by soil venting. Predicted cleanup time was significantly reduced.

The data from this investigation were also used in another facet at the site. In one of the monitoring wells located near the former vapor degreaser, a dense, non-aqueous product liquid (DNAPL), consisting of PCE with approximately 15 percent TCE, was detected. The presence of DNAPL at other locations within the contaminated area had been suspected, but not confirmed.

During the pilot study, the porosity, moisture content and bulk density of the soil was measured at several locations, in addition to the concentration of PCE and TCE. These physical properties were used in conjunction with the distribution coefficients for PCE and TCE to calculate the masses of these compounds within each of the soil phases: adsorbed on soil particles, in soil vapor and in the pore liquid.

Although DNAPL was not visually apparent in some of the samples, the amount of PCE calculated to be present in the pore liquid exceeded the solubility of PCE in water. Therefore, PCE NAPL was inferred to be present in specific samples. These results were critical to the design of the groundwater pump and treat system which was installed at the site.

The soil sampling techniques used at this industrial site have wide application to site investigations in general. At this particular site, field preservation of soil samples with methanol permitted a much more accurate and representative evaluation of the nature and extent of PCE and TCE contamination in soil. The field data reliably support the revised remedial design. Had accurate measurements of contaminant concentrations in soil not been available, costly errors or miscalculations may have occurred.

Lessons to be learned from this study are clear. If the total concentration of organic contaminants in soil or the distribution of the contaminants are critical data, then investigators should seriously consider using the field preservation technique. For example, soil cleanup targets typically are based on the potential of the contaminant in the soil to leach into the groundwater and generate aqueous concentrations in excess of the regulated maximum contaminant levels (MCLs). If the actual contaminant concentrations in soils are underestimated (potentially by up to an order of magnitude), then the feasibility of attaining the desired cleanup target may be misconstrued. Similarly, accurate and reliable volatile organic concentrations are needed to effectively design a vacuum extraction system to evaluate the biotreatability of a batch of contaminated soil and to evaluate other remedial alternatives.

When total concentrations of volatile organic compounds are critical to an engineering or a human health risk evaluation, the accuracy of the field sampling should be the best available technology.

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Write in 154
Gambling with financial assurance

There are at least two good reasons not to take chances

By Tom Gallagher, J.D.

How many good reasons do you need to get financial coverage for underground tanks? Well, it’s the law according to the Environmental Protection Agency and that’s the first good reason.

The EPA final rules regarding financial responsibility for regulated petroleum underground storage tanks (USTs) require owners and operators to acquire an allowable financial assurance mechanism to cover clean-up costs and third-party damages resulting from a release of petroleum. The eight financial assurance mechanisms allowed by EPA are self-insurance, insurance or group risk retention coverage, a guarantee with a standby trust fund, a surety with a standby trust fund, a letter of credit with a standby trust fund, an EPA-approved state-required mechanism, an EPA-approved state fund and a trust fund.

Failure to obtain an allowable mechanism by the deadline date subjects both owner and operator to a civil penalty of up to $10,000 per day for each UST not covered.

But, avoiding penalties and expenses of a catastrophic cleanup are not the only incentives for an owner or operator to find an allowable financial assurance mechanism. Under certain circumstances, obtaining such coverage could enable the owner or operator to survive in business—and that’s a second good reason.

Owners who are gambling with the “wait-and-see-what-happens” approach (“Maybe they won’t find me...Maybe my tanks won’t leak...Maybe no one will notice...”) to financial coverage need to understand the second, less obvious good reason to step forward and sign up for coverage.

In 1986, Congress passed the Superfund Amendment and Reauthorization Act (SARA). A part of SARA created a Leaking Underground Storage Tank (LUST) Trust Fund to provide money to states to finance cleanups of releases when the owner or operator of the UST could not be found, or was unable or unwilling to pay. If LUST Trust Fund

Continues on page 22

Tom Gallagher, J.D. is senior program manager for BH&E, Inc., an environmental management, science and engineering firm in Cincinnati, Ohio.
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Other good reason, from page 20

money is spent on a release, subsection (h) (6) of Section 9003 of RCRA (Resource Conservation and Recovery Act) states that the owner and operator of the UST are strictly liable to EPA or the state for the amount spent. EPA’s interpretation of this subsection can be found in EPA’s Office of Solid Waste and Emergency Response, Directive Number 9610.10, issued October 7, 1988. On page 13 of that directive, EPA says. “Although Section 9003 of RCRA generally allows consideration of whether pursuit of full cost recovery will significantly impair an RP’s (responsible person’s) ability to continue in business, States are precluded by statute from considering this factor if the RP has not complied with financial responsibility requirements in effect at the time.” (Italics added.) So if you fail to show good faith intentions by obtaining an allowable mechanism and if your pockets are empty, don’t expect sympathy and understanding.

The per-occurrence amount of coverage required is $1,000,000 for owners or operators of tanks at petroleum marketing facilities or tanks that handle an average of more than 10,000 gallons per month. Owners or operators of USTs not located at petroleum marketing facilities or which handle an average of 10,000 gallons or less must have coverage of at least $500,000 per release occurrence. Owners or operators of one to 100 regulated petroleum USTs must have an annual aggregate amount of coverage equal to $1,000,000 while those having more than 100 regulated USTs must have an annual aggregate amount of coverage equal to $2,000,000.

EPA employed a phased compliance approach to these financial responsibility rules. Presently, under the federal rules, all owners and operators must have an allowable financial assurance mechanism except for petroleum marketing firms with one to 12 petroleum USTs at more than one facility or fewer than 100 USTs at a single facility—and non-marketers with a net worth of less than $20,000,000.

On August 14, 1991, EPA proposed that the compliance deadline for these presently exempted owners and operators be extended to December 31, 1992.

Various states have also implemented UST programs and have imposed financial responsibility requirements. In order to become an EPA-approved program and thus operate in lieu of the federal program within their jurisdiction, states must have UST rules and regulations which are at least as stringent as the federal rules. State program compliance deadlines vary widely.

As an example, Ohio has one of the 26 EPA-approved state funds. In Ohio, all owners or operators of regulated petroleum USTs are required to have paid into the state fund. The Ohio fund has a deductible amount for which the owner or operator must obtain a second allowable financial assurance mecha-
nism to assure that money will be available to pay the deductible in the event of a petroleum release.

For owners of six or fewer petroleum USTs and the operators of such USTs and for political subdivisions, the deadline to obtain an allowable mechanism to cover the deductible is July 1, 1992. All other owners and operators of regulated USTs in Ohio must presently have such coverage.

If the cost of cleaning up a release from a regulated UST exceeds $1,000,000 and LUST Trust Fund money is used to complete the cleanup, EPA will consider the ability of the owner and operator to stay in business when it considers the amount of money it will try to recoup—only if the owner or operator is in compliance with the financial responsibility requirements. Such a consideration could well allow an owner to stay in business even if total clean-up costs exceed $1,000,000 and there is absolutely no possible way the owner or operator could pay the costs not covered by their allowable financial assurance mechanism. However, if neither the owner nor operator is in compliance with the financial responsibility requirements, current EPA policy calls for full reimbursement of LUST Trust Fund expenditures regardless of how it affects the owner’s or operator’s ability to stay in business.

EPA’s or a state’s approach to cost recovery negotiations can be crucial to the ability of an owner or operator to stay in business. Whether or not the owner or operator is in compliance with financial responsibility requirements can likewise be a crucial factor in whether or not the owner or operator has a chance to stay in business.

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Write in 192
January-February 1992 Soils 23
Proper installation critical to avert future problems

Give tanks a ‘proper burial’

By Mose Chandler

The Environmental Protection Agency estimates it will cost the nation in the neighborhood of $70 billion to comply with their underground storage tank rules. This estimate did not include cleanup costs or long-term testing and record keeping. Over 75 percent of the $70 billion is for new equipment.

While tank, pump and associated suppliers are beginning to feel the upsurge in sales associated with companies’ compliance efforts, the majority of the petroleum marketers have yet to make any drastic moves to change out tanks. Add to this number the ranks of state, municipal and federal tank owners who are just now looking at “Musts for USTs” (EPA’s 1988 introductory summary of the “new” regulations) and you’ve got a large cross section of the two million (or more) registered tanks that will be removed and replaced over the next decade.

All U.S. tank manufacturers produce products today with 30 year warranties. Ensuring that such a warranty is valid and potentially transferable is generally a function of whether the installation instructions were followed by the contractor. Whether the tank is fiberglass or steel, cathodically protected, single or double wall, proper installation plays the greatest role in your environmental compliance future. The single greatest cause of tank failure in the first five years after placement is improper installation.

Mose Chandler is vice-president of Carter Equipment Co., a petroleum distributor in Kansas City, Mo.

More than two million registered tanks will have to be replaced over the next decade to comply with EPA regulatory mandates.

It is not the purpose of this article to debate the respective advantages of fiberglass or steel, nor is it to promote one manufacturer’s tank over another’s. The fact is, with the quality of products available today, any tank is a good buy if properly installed.

Tank backfill material

As with any structure, buried tanks require a good foundation. Covering a tank with backfill creates added weight, which pushes down on the tank top and creates an outward force at the tank side. As the tank is loaded with product, there is also an internal pressure pushing down on the tank bottom and out at the sides. Proper backfill provides foundation support at both bottom and sides to counteract these pressures and transfers the tank load to the surrounding soil.

Without proper support, any tank—whether steel or fiberglass—may be damaged; structural failure or weld breaks can result. Or, the protective coating may be damaged sufficiently for corrosion to develop. Improper bottom support causes the tank bottom to flatten, creating excessive pressures at the corners, which could damage the tank walls or its protective coating.

Industry groups such as the Petroleum Equipment Institute and American Petroleum Institute recommend clean, compacted backfill material. Most commonly used are pea gravel, crushed stone or sand.

Backfilling with pea gravel or crushed stone is relatively foolproof and time efficient. Clean gravel is self-compacting and has generally been washed free of corrosive materials. This eliminates the need for jobsite labor to compact the backfill. The flow characteristics of gravel make it easier to push under the tank for proper bottom support.

Sand must always be compacted. This compaction requirement makes the quality and effectiveness of the tank backfill support more labor intensive and less predictable than gravel. If sand backfill is used, an independent soil technician/engineer must verify that the sand has been compacted to a minimum 90 percent standard proctor density. While the material cost of sand may be less than gravel, the labor to properly compact the sand adds around $6 per ton to the cost.

Native soils should never be used to backfill under and around tanks or horizontal piping. Dirt, clay and other native soils cannot be reliably compacted. If tanks are being installed in a sandy area, it is necessary to hire a soils engineer to verify that the excavated sand meets the backfill material specification. Backfilling a new installation with previously used backfill

automatically voids all manufacturer’s warranties.

Tank anchoring
There are four primary methods of anchoring tanks in the course of an installation:
1) deep bury method
2) deadman anchors
3) anchor pad
4) mid anchoring

Tanks require anchoring to compensate for natural buoyancy. Tank buoyancy forces occur when a high groundwater condition develops and the tanks are partially or totally empty. Under these circumstances, if the offsetting ballast weight of backfill material on top of the tank (or attached to it with a mechanical anchoring configuration) is less than the buoyancy forces on the tank, the tank will float. The tank industry standard for an anchoring safety factor is 1.2:1 for a worst case condition (groundwater to grade and tank empty). This means that there should be at least 20 percent more ballast weight on the tank (or attached to it) than the maximum uplift forces.

Many people believe that because a tank is made of fiberglass it automatically requires anchoring. However, the maximum tank buoyancy forces greatly overshadow tank weight, whether fiberglass or steel. To calculate tank buoyancy, a good rule of thumb is to multiply the tank volume by 8.33. Therefore, a 10,000 gallon tank will develop 83,300 pounds of uplift when it is fully immersed in a high groundwater condition. If tank weight were the only offsetting ballast, the weight of the steel tank

Continues on page 26→

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A 10,000 gallon tank will develop 83,300 pounds of uplift when it is fully immersed in high groundwater.

"Proper burial," from page 25

(8,000 pounds) would compensate for less than 10 percent of these forces—while the weight of a fiberglass tank (2,600 pounds) would offset a little over three percent. The remaining uplift forces must be offset by tank overburden (deep bury) or a mechanical anchoring system (mid anchors, deadman anchors or anchor pad).

Depending of the burial depth (from tank top to finished grade) backfill material on top of the tank can offset all or a portion of the buoyancy loads. Dry gravel or sand both provide similar anchoring deadweight—about 95 pounds per cubic foot. However, if the backfill material over the tank is submerged in water, the net ballast weight of the backfill is decreased to about 60 pounds per cubic foot by the buoyant force of the water. Using a thicker concrete traffic pad at grade is typically not a cost effective method to increase the ballast weight over the tank. While concrete submerged in water weighs 50 percent more than gravel, it costs about ten times more. So, it’s usually less expensive to bury the tank deeper and use the backfill material for ballast.

Ignoring tank weight for a moment, the tank internal diameter is a key variable in determining the added safety factor provided by the backfill over the tank. As a general rule of thumb for multiple tanks installed in the same excavation, when the tank burial depth exceeds 60 percent of the tank internal diameter, the backfill material directly over the tank weighs 20 percent more than the maximum buoyancy force (safety factor equals 1.2:1). The rule of thumb for a single tank in an excavation is that the burial depth must exceed 50 percent of the tank diameter.

At shallower burial depths where high groundwater conditions may occur, a mechanical anchoring system is needed to achieve a 1.2:1 safety factor for the worst case condition. It is the responsibility of the tank owner or his technical representative to determine if mechanical anchoring is required at a specific jobsite. All potential sources of water (underground water table, rainwater runoff, etc.) should be considered. If water can accumulate over the tank top, most tank manufacturers recommend the tanks be mechanically anchored unless the tank burial depth provides sufficient ballast.

The need for a mechanical anchoring system is more likely for tanks with larger internal diameters because the buoyancy forces are more concentrated in a smaller area. Although lighter than a steel tank, a 10,000 gallon capacity fiberglass tank gains additional safety factors because the tank diameter is less than a steel tank. Fiberglass tanks have an internal diameter of 92 inches, while most steel tanks are 96 inches in diameter—some are 100 inches in diameter.

So, when the effects of both tank weight and tank internal diameter are considered, anchoring safety factors are comparable for fiberglass tanks (92 inch) and steel tanks (96 inch). Steel tanks larger than 100 inches in diameter have lower anchoring safety factors and therefore are more likely to require mechanical anchoring systems. This is true for all methods to anchor underground tanks.
TANK INSTALLATION SUMMARY—FIBERGLASS VERSUS STEEL

<table>
<thead>
<tr>
<th>TANK HANDLING CHARACTERISTICS</th>
<th>FIBERGLASS</th>
<th>STEEL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of 10,000 gallon tank</td>
<td>2,600 lbs.</td>
<td>8,000 lbs</td>
</tr>
<tr>
<td>Coating repair for superficial chips/scratches</td>
<td>None</td>
<td>Required</td>
</tr>
<tr>
<td>Pipe Stiffness rating</td>
<td>7</td>
<td>2</td>
</tr>
</tbody>
</table>

**EXCAVATION LOGISTICS**
- Excavation to be free of hard or sharp objects: YES
- One foot sand/gravel bottom requirement: YES
- Bottom quadrant tank support requirement: YES
- Backfill requirement with concrete slab (min.): 12 in.
- Sidewall and tank to tank clearance (min.): 18 in.
- Remove dirt clods and foreign materials before placing tanks: YES

**PRE INSTALLATION TESTING REQUIREMENTS**
- Permanent plugs in all unused openings NO
- Maximum torque rating of fittings (in foot pounds): 2000
- Test with 5 psi air pressure aboveground soap test: YES
- Do NOT leave tanks unattended while under pressure: YES

**INSTALLATION REQUIREMENTS**
- Dielectric bushings: NO
- Check soil for anode compatibility: Not Applicable
- Anode installation: Not Applicable
- Remove plastic covering: Not Applicable
- Verify connection after placing tank: Not Applicable
- Connect wire to test station: Not Applicable
- Continuity testing to all piping/equipment: Not Applicable
- Install grade manhole for test station: Not Applicable
- Electrically isolate anchoring straps: NO
- Verify operation of cathodic protection system: NO
- Test cathodic protection & maintain records: NO
- Complete installation checklist to validate warranty: YES

**Installation specifications**
Fiberglass and steel tanks require pretty much the same basic good sense installation practices. The primary difference in installing a steel versus a fiberglass tank comes in ensuring that a steel tank is electrically isolated and that the cathodic protection system is operational. This requires the use of dielectric bushings at tank and line connecting points. It also requires that the steel tank contractor knows something about soil resistance factors and corrosion potentials. In the short term, steel tanks are more forgiving of an improper installation. Longer term, even a cathodically protected steel tank improperly installed will be susceptible to premature failure. Protecting your tank warranty can be accomplished by carefully selecting an installation contractor. Look for a more involved posture from local codes administration groups, fire marshals, tank (above and below ground) regulatory agencies and trade associations.

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Write in 159

January-February 1992 Soils 27
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The system is activated at our factory and goes right to work. HydroGuard\textsuperscript{TM} checks the tank’s condition during transportation...
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If you're looking for a new tank that will protect the environment from pollution and your business from liability risks, choose the HydroGuard™ System. Don't bury a mistake.
Clearing your headspace

Pack up your samples in a plastic freezer bag

By Jack Murphy

Lab In A Bag is a portable field screening device that provides field interface between contaminated samples and a vapor analyzer for rapid and cost-effective estimates of contamination. Both site assessment and remediation activities involve chemical analysis of contaminants. These analyses may be conducted in situ, on site or in the laboratory.

Laboratory measurements are generally accepted as the most reliable estimates of contaminants. However, the sample may deteriorate between the time of collection and the time of analysis due to volatilization, adsorption or biological transformation so that laboratory results may not accurately reflect the character of the sample as collected at the site.

In situ measurements offer the least disturbance of the material being analyzed but also afford the least control of analyzing conditions. Few truly in situ analytical methods are currently available and generally accepted, particularly for contamination by organic substances. Numerical data from in situ measurements suffers from lack of control of conditions so that only semiquantitative results are available. In situ methods can be useful to determine whether further action or analysis is required.

In on site analysis, a sample is taken and immediately analyzed with a portable measuring device at the site. Because the sample is measured on the spot, sample deterioration is minimized. And, because the sample is in hand, analytical conditions can be more carefully controlled. Lab In A Bag, developed by In-Situ, Inc., of Laramie, Wyo., in cooperation with the EPA (Environmental Protection...
Agency) is an on site analytical tool. It was commercialized under the authority of the Federal Technology Transfer Act of 1986 using research sponsored by the Las Vegas Laboratory of the EPA and the Office of Underground Storage Tanks.

Lab In A Bag actually uses one-quart size zipper closure food storage bags to measure volatile organic compounds (VOCs).

The principle is that the bags constitute a sealed container which provides a "headspace" over the sample. Several versions of the headspace method have been used, but in each, some kind of equilibrium distribution of VOCs between sample and headspace is approached during an equilibrium period. The equilibrium may be hastened by agitation or developed slowly. Soil samples may be allowed to equilibrate directly or water may be added to disaggregate the soil and facilitate displacement of VOCs.

The headspace is then drawn off and analyzed with a total organic vapor detector (TOVD), a device which responds to numerous compounds and gives a single readout characteristic of the mixture as a whole. No product discrimination or molecular identification is attainable with TOVDs.

The headspace may be created in a rigid container or in a flexible bag. Rigid containers can cause problems when the headspace is sampled by the TOVD—if the rigid container is sealed, a back pressure is generated which retards flow to the TOVD and can alter the readout. If the container is open to the atmosphere, ambient air may intermingle with the sample headspace, diluting the sample. To overcome these problems of the rigid container, the headspace can be developed in a sealed, collapsible bag.

The headspace method must provide reproducible sample size and headspace volume, constant headspace pressure during sampling by TOVD and sealed integrity from outside influence—during both equilibration time and TOVD sampling time. Use of the polyethylene bags meets all these requirements.

Researchers worked with one-quart zipper closure bags to measure headspace volatiles and obtained satisfactory results for benzene, gasoline and chlorinated hydrocarbons in water and soil samples. G.A.

"Standardized measurements are necessary to determine whether significant contamination is present during a site assessment or remediation..."

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Robbins, R.D. Bristol and V.D. Roe published results of their research in *Ground Water Monitoring Review*, vol. 9, no. 4 in the fall of 1989. They compared field data to laboratory measurements and obtained excellent correlations.

Headspace methods yield relative semiquantitative values directly and can also be standardized by comparison to prepared standards. Relative measurements give numerical values on a relative scale, permitting comparison of relative values of contaminant levels between samples. This kind of quantification would be useful, for example, to plot contours of contamination at a given site to help locate contaminant sources or migration pathways. For relative measurements, no standard samples are run through the headspace method for calibration.

Standardized measurements require that the headspace procedure be calibrated in some way against accepted standards. After calibrating the TOVD to a particular standard gas such as methane or isobutylene, the TOVD response is recorded for a series of aqueous solutions of appropriate volatile liquid standards run through the headspace method. The values measured for the samples are then reported in the units appropriate for the volatile liquid standards. Standardized measurements are necessary to determine whether significant contamination is present during a site assessment or remediation—and when reporting...
levels to a client or regulatory agency.

A variety of standards and corresponding units may be used. For example, one may report the amount of gasoline related constituents in water samples as:

- ppm (parts per million) benzene or hexane (etc.) equivalent, because the bag was calibrated with benzene or hexane (etc.)
- ppm of fresh unleaded 85-octane winter-gasoline equivalent—because that standard was used to calibrate the method.

While it is best to choose as standards substances that are identical or similar to field samples, it may be that no standard is really similar to the environmental product measured. Even so, the TOVD response can be reported as equivalent to the response obtained by a certain level of the chosen standard and treated the same way as an environmental sample. This is a standardized measurement.

It is possible to create a bag sampling system with a collection of loose parts. But for optimum reproducibility and to minimize accidental fouling of the TOVD, In-Situ’s Lab In A Bag provides:

- a sturdy base to secure the bag
- a battery-operated pump to inflate the bag in-line, with a pressure switch to shut off the pump automatically at full bag inflation
- a battery-operated magnetic stirrer for sample agitation during the equilibration period
- a timer with audible and visual alarm for automatic cut-off to indicate the end of the equilibration period
- valves to direct flow of air to bag and to connect bag to TOVD for headspace measurement
- rechargeable battery to provide full operation of instrument functions for a full field day
- auxiliary equipment kit for measuring water and soil samples and for preparation of standards.

The focus of Lab In A Bag is the bag. A hole is cut in the wall of the bag so it can be attached to the instrument and sealed against air loss or infiltration. The valves are used to isolate the bag from the TOVD and connect the bag to the pump. The sample and a stir bar are inserted through the zipper opening and sealed in.

The pump introduces ambient air into the bag until the internal pressure reaches the small value of six inches of water (0.22 psi or 0.015 atm) and opens a pressure sensitive switch which shuts off the pump.

The stirrer agitates the sample for a predetermined time (one to 11 minutes) to effect equilibration of volatiles between the sample solution and the headspace. At the end of the set time, the stirrer stops automatically and alarms prompt the operator to turn a valve connecting the bag to the TOVD for headspace measurement. After the TOVD is disconnected, the pump purges the tubing before a new sample is introduced.

Again, the bags are the one-quart size, zipper closure bags which can be inflated to a capacity of 1.36 liters, which provides adequate headspace volume for most TOVDs to complete a measurement before depleting the headspace.

It is important to understand that bag inflation is not like the inflation of a balloon. In a balloon, most volume increase results from stretching the balloon wall—increasing the pressure in the balloon as the volume increases. In bag inflation, volume increase simply involves opening the bag without wall expansion. The small amount of pressure required to activate the switch (0.015 atm) involves only 1.5 percent of the total volume of the inflated bag. As a result, day-to-day changes in ambient pressure and temperature have little effect on total volume.

On site analysis can often provide the most useful method when fast results are important.

Write in 506 for more information
Simplified method cuts sampling costs

Single-stage extraction represents small margin of error

By Donald S. Lavery and Edmund C. Manke, Jr.

How fast is fast enough to analyze soil samples that may be contaminated with petroleum hydrocarbons? Investigators can’t get answers and information about a site too soon. Immediate availability of data is only possible when the analysis can be done in the field as samples are taken. What is the quickest way to get results? Current procedures aren’t fast enough.

Modification of laboratory methods either to provide faster analysis or to accommodate the non-laboratory environment—or both—is necessary. The “standard” method to analyze total petroleum hydrocarbons (TPH) in soil is commonly Environmental Protection Agency (EPA) Method 418.1. In fact, EPA 418.1 (Petroleum Hydrocarbons, Total Recoverable, Spectrophotometric, Infrared) is a water analysis method that is usually modified for soil analysis by using a Soxhlet extraction followed by the sample cleanup and infrared analysis.

It is possible to use a method that minimizes sample handling, can significantly reduce Freon usage and extends the infrared method to obtain more information about the sample than is provided by any method that uses single wavelength infrared detection.

This simplified method uses disposable components to do a single-stage Freon-113 extraction, a solid-phase extraction for sample cleanup and either a single, referenced infrared absorbance or dual referenced infrared absorbance measurement for detection.

A single-stage extraction saves time and minimizes Freon usage. By comparison, a classical extraction method attempts to remove all the analyte from a sample matrix, quantitatively dilute the extract and measure the concentration of the final solution. Typically, the original sample is extracted at least three times (or, in the case of a Soxhlet extraction, many times). At each stage of the extraction, the analyte is distributed between the original sample and the extraction solvent. After separation of sample and extract, analyte remains in the sample matrix, both bound due to the partition between phases and dissolved in the residual solvent that is not completely removed in the separation step. A single-stage extraction with a measured volume of solvent will not recover the analyte remaining in the sample due to partition, but it does correctly measure the concentration of the extract in equilibrium with the sample so the dissolved analyte left behind does not represent a loss. The single-stage extraction thus fails to recover only that fraction of the analyte left in the soil due to the partition and, provided the partition strongly favors the solvent phase, as would be reasonably expected in the case of Freon-113 and soil, this should represent a small error.

Since sampling variability and sample degradation during

Dr. Donald S. Lavery is laboratory director and Edmund C. Manke, Jr., is applications chemist for General Analysis Corp. of South Norwalk, Conn.

Simplified method uses disposable components to perform a single-stage Freon-113 extraction and infrared absorbance measurement for detection.
transportation and storage tend to dominate the variation in environmental analyses, the sample partition loss of a single-stage extraction should be unimportant. Significant variation in results is by no means restricted to TPH analyses. For example, Paul R. Locinto (in LC-GC, 1991) cites acceptable recovery ranges for 11 priority pollutants from soil of which 26-90 percent (phenol), 38-107 percent (trichlorobenzene) and 35-142 percent (pyrene) are typical. Viewed in this light, measured recoveries by the simplified method are quite good. In addition, the improvement in sampling strategy and the avoidance of sample loss provided by analyzing immediately may well yield a better overall result than conventional sampling with delayed laboratory analysis.

There are two approaches to the infrared detection part of the method. The simpler system is an infrared filter photometer using an interference filter centered at 3.4µm—the wavelength normally used for this analysis. However, a simultaneous measurement is also made at a reference wavelength (2.5µm) where hydrocarbons are transparent. The measurement at the primary wavelength is continuously ratioed to the reference measurement.

The second detection method uses two measurement wavelengths, each of which is continuously referenced to the 2.5µm reading. The two-wavelength system measures both the conventional 3.4µm absorbance due to aliphatic hydrocarbons and also the absorbance at 3.3µm which is characteristic of aromatics.

These two classes of hydrocarbons absorb both in different regions of the infrared spectrum and with very different intensities. The single (3.4µm) measurement is, for all practical purposes, insensitive to aromatic compounds and relies on the calibration standard to have about the same aromatic content as the sample. The two-wavelength approach separately measures and scales the absorption characteristics of the two classes of petroleum hydrocarbons which gives a better measure of the

*Continues on page 36→

**Figure 2: TPH Analyzer Response to Weathered Diesel Fuel and Weathered Gasoline.**

---

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Write in 162 January-February 1992 Soils 35
Simplified method, from page 35

overall contamination picture.

In many cases, the apparent defect in the single wavelength method is not as serious as it might appear. Many, perhaps the majority, of petroleum contaminated sites involve a fuel. Compared with the other uncertainties in environmental sampling and analysis, the variation in aromatic content of petroleum fuels is not large (say, 0 to 40 percent). A method intended to define the extent and overall level of contamination of the site will not be too far off the mark if it assumes an undetected aromatic content of 35 percent. This is just what the synthetic oil standards specified in EPA standards do. On the other hand, a more complex site could involve multiple sources and non-fuel products so the aromatic content of samples might vary from 1 to 100 percent. In these cases, direct detection of aromatics would provide invaluable information. An infrared analyzer that uses a reference wavelength has photometric advantages. The reference cancels short term source and electronic fluctuations (noise) as well as longer term source and optical component changes (drift). However, there may be a special advantage for TPH analysis. N. Thomey reported positive interferences with infrared TPH analyses in clay and limestone samples. It seems likely that this is due to fine particulates that cause broad-band scattering. A single wavelength analyzer cannot distinguish scattering from a hydrocarbon absorption. But a multiple wavelength system would be similarly affected at all wavelengths so the effect would, to a large extent, cancel.

The equipment required for the simplified extraction method is shown in the photograph on page 34 and includes:

- solvent dispenser capable of precisely dispensing 20 ml of Freon-113
- battery-powered top loading scale
- 40 ml EPA VOC sample vials, caps and septa
- spatula
- disposable sample reservoirs with integral filter frit
- clean-up cartridges
- pressure seal assembly and 20 ml gas-tight syringe
- infrasil cuvettes, one cm
- infrared filter photometer
- Freon-113 spectrophotometric grade or better
- silica gel 60-200 mesh, chromatographic grade
- appropriate calibration standards

---

**Figure 3: EVAPORATION LOSS FROM PETROLEUM PRODUCTS AFTER 24-HOUR EXPOSURE**

<table>
<thead>
<tr>
<th></th>
<th>INITIAL (g)</th>
<th>FINAL (g)</th>
<th>% LOSS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gasoline</td>
<td>34.50</td>
<td>4.97</td>
<td>85.6%</td>
</tr>
<tr>
<td>Diesel Fuel</td>
<td>40.37</td>
<td>39.20</td>
<td>2.9%</td>
</tr>
<tr>
<td>Kerosene</td>
<td>31.09</td>
<td>28.47</td>
<td>8.4%</td>
</tr>
</tbody>
</table>

---

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Write in 174

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Prior to analysis the analyzer was calibrated by preparation of standards in a concentration range of 1-500 mg/L (milligrams per liter). Five standards were prepared at 100 mg/L increments using hexadecane and benzene. The aliphatic channel was calibrated with hexadecane (C16) and the aromatic channel was calibrated with benzene. Each channel of the dual channel analyzer was "linearized" via a built-in adjustable linearizing circuit to compensate for any non-linearity within the calibration range. In addition, the response of each channel was corrected for interference from absorption on the opposing channel by a matrix circuit built into the analyzer to provide more accurate discrimination of the true aliphatic and aromatic contribution of the samples. Results are reported as hexadecane or benzene equivalents. Figure 2 (page 35) shows the response of an analyzer calibrated with benzene and hexadecane to evaporated (weathered) gasoline and diesel fuel.

Calibration can also be done using actual samples (gasoline, diesel, jet fuel, kerosene) or synthetic reference oil standards. Standards may be chosen based on the type of contamination found on a given site, but care must be taken to prepare a standard which emulates the characteristics of the condition of the site. For instance, for an older site known to be contaminated with gasoline, a gasoline standard may not be appropriate since weathering and biodegradation may have significantly altered the chemical composition of the contaminating gasoline. Figure 3 (page 36) shows the effect of evaporation of gasoline, diesel fuel and kerosene in just 24 hours. For older sites, a standard reference oil may be more suitable than a fresh sample of the original contaminant.

Method 418.1 calls for use of a reference oil prepared with 37.5 percent hexadecane, 37.5 percent isooctane and 25 percent chlorobenzene by weight. The use of chlorobenzene as an aromatic standard seems a poor choice since the weight contributed by the chlorine is significant but contributes nothing to the aromatic absorbance. The result is a disproportionate reduction in the aromatic contribution to the standard. In addition, the chlorobenzene absorption is both unusually weak and significantly displaced in wavelength from the bands that are characteristic of the aromatic compounds actually found in petroleum. (See Figure 4, above).

The analysis method is as follows:
1) Weigh about 20 grams of soil into a 40 ml VOC vial and record the weight to the nearest 0.1 gram.
2) Add 60-200 mesh chromatographic grade silica gel (up to 5 grams) to the sample, after shaking, is dry and free-flowing.
3) Dispense precisely 20 ml of Freon-113 into the vial and cap it. If necessary, wipe the top edge of the vial and the septum clean in order to allow a leak-tight seal.
4) Shake the sample vigorously for five minutes, let stand one minute and decant the liquid into a sample reservoir (with filter cartridge attached) leaving as much of the soil as possible in the extraction vial.

Continues on page 38→
5) Close the sample reservoir with the pressure seal, attach the pressurizing syringe and pressurize the reservoir so the extract is forced through the filter cartridge dropwise into a one cm quartz cuvette.
6) Discard the first 0.5 to one ml (1/6 to 1/3 of the cuvette volume) and collect the next 2.5 to 3 ml.
7) Fill a cuvette by processing Freon from the same batch as that used for the extraction through a cleanup cartridge. Place it in the sample holder of the infrared analyzer and set the zero control(s) so the display(s) read zero.
8) Place the sample cuvette in the analyzer and read the extract concentration in mg/L.
9) Calculate the soil TPH concentration in ppm (parts per million) according to:

\[
\text{soil concentration} = \frac{\text{solvent volume} \times \text{extract concentration}}{\text{sample weight}}
\]

The sample cleanup technique was tested by processing standard solutions of partly evaporated gasoline and diesel fuel through the cleanup step. The results are shown in Figure 5, above. Clearly, the cleanup leaves the petroleum components virtually

---

### Figure 5: RESPONSE OF STANDARD SOLUTIONS PROCESSED THROUGH SAMPLE TREATMENT CARTRIDGE

<table>
<thead>
<tr>
<th>CONCENTRATION (mg/L)</th>
<th>AROMATIC RESPONSE</th>
<th>ALIPHATIC RESPONSE</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>EXPECTED MEASURED</td>
<td>EXPECTED MEASURED</td>
</tr>
<tr>
<td></td>
<td>MEAN  ō</td>
<td>MEAN  ō</td>
</tr>
<tr>
<td>GASOLINE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>100.4</td>
<td>27  17  1</td>
<td>52  51  2</td>
</tr>
<tr>
<td>200.8</td>
<td>54  46  1</td>
<td>104 100 1</td>
</tr>
<tr>
<td>301.2</td>
<td>80  72  2</td>
<td>156 156 2</td>
</tr>
<tr>
<td>401.6</td>
<td>107 94  1</td>
<td>208 208 2</td>
</tr>
<tr>
<td>502.0</td>
<td>134 120 0</td>
<td>260 271 4</td>
</tr>
<tr>
<td>DIESEL</td>
<td></td>
<td></td>
</tr>
<tr>
<td>105.0</td>
<td>7    3  3</td>
<td>80   72  1</td>
</tr>
<tr>
<td>210.0</td>
<td>14   6  4</td>
<td>161  151 2</td>
</tr>
<tr>
<td>315.0</td>
<td>21   8  1</td>
<td>241  239 1</td>
</tr>
<tr>
<td>420.0</td>
<td>28   17  1</td>
<td>322  332 1</td>
</tr>
<tr>
<td>525.0</td>
<td>35   31  1</td>
<td>402  422 1</td>
</tr>
</tbody>
</table>

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FIGURE 6: Effect on Standard Solutions of Silica Gel Used to Dry Soil Samples

<table>
<thead>
<tr>
<th>SILICA GEL ADDED</th>
<th>0g</th>
<th>1g</th>
<th>3g</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACTUAL CONCENTRATION (mg/L)</td>
<td>MEASURED CONCENTRATIONS (mg/L)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzene</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>118.7</td>
<td>123</td>
<td>125</td>
<td>108</td>
</tr>
<tr>
<td>296.8</td>
<td>313</td>
<td>299</td>
<td>295</td>
</tr>
<tr>
<td>474.9</td>
<td>475</td>
<td>464</td>
<td>457</td>
</tr>
<tr>
<td>Hexadecane</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>119.5</td>
<td>118</td>
<td>125</td>
<td>126</td>
</tr>
<tr>
<td>298.8</td>
<td>297</td>
<td>308</td>
<td>316</td>
</tr>
<tr>
<td>478.1</td>
<td>467</td>
<td>474</td>
<td>494</td>
</tr>
</tbody>
</table>

unchanged. By comparison, vegetable oil in a similar solution was 95 percent retained by the cartridge. Figure 6, above, shows that the silica gel used to dry samples has very little effect on standard solutions.

The performance of the simplified method was examined by testing the recoveries obtained from two different soil types, sandy clay and high humus topsoil. Test samples were made up in the lab by obtaining clean samples of both types and contaminating them with known concentrations of petroleum products. Diesel fuel, unleaded regular gasoline and kerosene were all evaluated. All petroleum samples used as standards were evaporated by placing 35 to 40 grams of each in a beaker and allowing it to evaporate for 24 hours. Those losses were outlined in Figure 3 on page 36. The evaporated samples were used to prepare stock solutions which were diluted to prepare standards at the desired concentration levels. Each standard was run through the extraction system, measured on the two channel photometer to determine aromatic and aliphatic response. Those results were shown in Figure 5. Soil samples were prepared by dissolving each sample type in Freon-113 and mixing the solutions with soil. After shaking, the Freon-113 was allowed to evaporate. The soils were extracted using the outlined method and the recoveries calculated.

Many factors affect the recovery of hydrocarbons from soil, including soil type, porosity, moisture, pH and particle packing. This experiment examined the effect of water on the recovery of diesel fuel from the sandy clay and the high humus topsoil. Wet samples were prepared in duplicate by adding water to 20 grams of dry soil. The volume of water added was from 0 to 5 ml. Five ml of water was sufficient to produce some standing water in the sample vials. Reference samples at each water increment were extracted with Freon-113 following the described method. Attempts were made to dry samples using both sodium sulfate and silica gel. Silica gel proved to be more effective. The results for untreated samples were compared with those for samples which were treated with sufficient silica gel to cause the sample

"The performance of the simplified method was examined by testing the recoveries obtained from two different soil types, sandy clay and high humus topsoil."

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to appear dry. The results (Figure 7, left) show significantly greater recoveries for treated samples. Results are reported on a wet basis since this is the only option for field analysis. The amount of silica gel added was varied to facilitate total sample drying, but for the largest amounts of silica gel, very little Freon-113 was available for measurement. It is recommended that for these samples, additional Freon-113 be added and that the additional volume be included in the solvent volume used in the final calculation.

At least two mechanisms appear to affect the extraction. Wet sandy clay samples congregated into large balls when shaken with Freon-113. In these cases, the volume of free solvent was much greater than for dry samples. This suggests that the Freon could not penetrate the wet agglomerates which prevented contact with and extraction of the hydrocarbons. In addition, water in porous soils at levels that do not cause serious agglomeration may still fill the pores and exclude Freon, reducing the contact between the hydrocarbon contaminants and the solvent. This would explain why wet samples that were dried rather quickly with silica gel showed lower recoveries than those with lower initial moisture content.

The simplicity of this extraction method and its success in dealing with very difficult samples promises to allow field analyses to be used much more frequently than they have in the past. This should lead to reduced costs for sampling and remediation programs by providing immediate information to equipment operators—allowing sampling plans to be optimized and reducing Freon-113 usage.

References:


D. DeAngelis, Manual of Sampling and Analytical Methods for Petroleum Hydrocarbons in Groundwater and Soil, American Petroleum Institute, Publication No. 4449.


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Austin has piping solutions

Austin Engineering, Rolling Meadows, Ill., says they have the solution to difficulties related to assembling double-walled UST piping systems.

The Flex Protector® IV product was designed to serve as the necessary flexible component in rigid secondary containment systems for piping, Bill Babbin, president says.

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What’s New

Thermotech plans seminar on thermal processes
Thermotech Systems Corp., Orlando, Fla., announces their biannual seminar on soil remediation focusing on thermal processes, technical operations, regulations, testing and asphalt plant conversions.

Business considerations, including current industry status, market interest for portable and stationary plants, economics, competitive pressures and legal issues will also be covered.

Dates of the meeting are January 29-31, 1992 in Orlando at the Sheraton Orlando North. Fee for the seminar is $450 per attendee. The cost includes meals and refreshments. Call Rick Graddy, 407-290-6000 for more information.

State-by-state summary correction
The listing for the state of Indiana is:

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<td>20ppm</td>
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</table>

Source: Lynette Fogel, Department of Environmental Management, 317-240-6217

SPECIAL CALL FOR PAPERS
for an Association of American Railroads sponsored session on DIESEL CONTAMINATED SOILS

DEADLINE FOR SUBMISSION IS APRIL 1, 1992.

at the Seventh Annual Conference on HYDROCARBON CONTAMINATED SOILS ANALYSIS, FATE, ENVIRONMENTAL & PUBLIC HEALTH EFFECTS, AND REMEDIATION University of Massachusetts at Amherst September 21-24, 1992

This session is designed for the railroad industry and will serve as a platform for research on diesel remediation technologies. The presentations will be produced as a special publication.

For a paper to be considered, please submit a ONE-PAGE ABSTRACT containing: presentation title; 300 word narrative; and for each author, name, degree, title, affiliation and complete address and phone number.

FOR FURTHER INFORMATION and abstract submission please contact:
Paul T. Kostecki, Ph.D.
Environmental Health & Sciences
N344 Morrill
University of Massachusetts
Amherst, MA 01003
Phone: (413)545-2934
FAX: (413)545-4692

Write in 147
Write in 188

42 January-February 1992 Soils
What's New

Insul-Lite Tank

Tank insulates in case of fire
We-Mac Manufacturing Co., North Kansas City, Mo, offers the Insul-Lite, a double wall aboveground tank with a two hour fire rating without the added weight of concrete. Using U.L. listed Thermo-Ceramics, Insul-Lite simultaneously insulates the product tank reducing evaporation and virtually eliminating condensation in the interior, the manufacturer says. Insul-Lite double wall tank systems come with overfill protection and spill containment.

Write in 515 on inquiry card.

Brass Soil Tubes

I-Chem offers sampling tubes
I-Chem, Hayward, Calif., introduces ready-to-use Brass Soil Sampling Tubes as part of their new Field Team™ line of products.

The Brass Soil Tubes are six inches long and come with end-caps and labels inside each case. They are packaged to accommodate large or small projects, I-Chem says.

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Write in 201
January-February 1992 Soils 43
It’s just a phase you’re going through

What services to expect in each phase of risk assessment

By Lyle A. White

Tank owners and environmental consultants need to know their way through the rapidly growing risk assessment maze.

In the process of getting tanks and facilities into compliance with government regulations, it is easy to lose sight of the bigger picture—property value. Tanks and tank facilities are real property and environmental exposure to real property must be considered a top priority.

As tank owners weigh their options to stay put, grow, buy, sell or make any change, aspects of environmental risk assessment must be clearly understood.

Environmental risk assessment is an objective review of a property to determine any actual and/or potential sources of environmental contamination.

Assessment is the immediately necessary first step in appraising property value. It is necessary when real assets are bought and sold, when collateral is reassessed or when there is a change in instrument by the lender. At some point, tank owners become sellers—and it is essential to know what you have because any serious buyer insists on knowing what he or she is getting.

Risk assessment is divided into three distinct phases. Breaking the assessment into phases takes into account the uncertainties, the unknowns and the unseens associated with properties.

Phase one is the identification and discovery process. Phase two is known as the characterization process—investigation to obtain quantitative evidence of the presence of hazardous substances and prescribing corrective action. Phase three is the remediation process—actually solving or containing the problem.

The generally accepted criteria for each phase of an environmental assessment are:

**Phase one—Identification and Discovery**

- Records and Regulation Review
  - Title search to reveal chain of ownership
  - Federal, state and local regulatory review
  - State hazardous waste lists
  - CERCLA (Comprehensive Environmental Response, Compensation and Liability Act) and FINDS (Facility Index System) hazardous waste permit holders
  - Tank registration status and inventory records

- Site Condition Assessment
  - Land and/or facility use
  - Site topography and drainage
  - Site resources and water wells
  - Evidence of hazardous materials
  - Site geology
  - Site hydrogeology
  - Physical inspection of site, may include limited sampling and analysis

The Phase one report summarizes findings of the evaluation. It should pinpoint potential problems, define client risks and liabilities and recommend corrective action options.

**Phase two—Characterization Process**

Extensive site investigation should gather quantitative evidence.

- Subsurface investigation
- Geophysical surveys
- Vapor probe surveys
- Groundwater monitoring
- Tank integrity tests

---

*Lyle A. White is vice-president of environmental services for EnviroQuest Technologies of Kansas City, Mo.*
The Phase two report delineates the extent of the problem, the risk involved and the cost guidelines for remediation. It also should analyze alternatives and rank them in terms of risk and liability.

**Phase three—Remediation/Corrective Action**
- Remedial action design
- Remedial action implementation
- On-going monitoring and maintenance

Industry surveys indicate the cost of Phase one assessments range from $2,000 to $15,000. The lower end is for raw land and residential investigations and the high end is for more complex industrial situations. Commercial properties can fall anywhere in between.

Since Phase two may include air, water and soil sampling as well as other specialized tests, the cost can range from $10,000 to $100,000—depending on size and complexity. The average falls between $25,000 and $30,000.

Phase three costs can vary greatly, with many cleanups easily exceeding $100,000 in cost.

Often, buyers and lenders will insist on a Phase one and two assessment as a prerequisite to considering purchase—even when there is no suspicious evidence to indicate the need.

In one case, the owner of a full service gas station located at a high traffic intersection in an older part of town wanted to sell. The owner had done all the homework in terms of the current mandates for underground storage tank compliance. His tanks were tight, had leak detection and corrosion protection. In addition, he kept meticulous product inventory records. Still, the potential buyer and his lender insisted on a risk assessment.

“We want to know what we’re getting. We don’t want to buy a problem,” they said.

“I am in compliance,” protested the owner.

“We know. But there could be other problems you don’t know about.”

So, even though the seller was on the top rung of the regulatory ladder, his exposure to risk was not over. The buyer offered to bring in a firm or he would accept an audit from a reputable firm of the seller’s choice. But no matter how much the seller protested, the serious buyer would not go near the dotted line without an assessment in hand.

Anyone in the position of having to procure a risk assessment needs to know what is included (and what is not) in each phase. The true value of environmental assessment is more closely associated with the service provided than with the costs incurred. One cannot research and comparison shop environmental firms without an understanding of the phases.

Buying or selling, satisfying lender requirements, supporting insurance purchases and complying with regulations—all may well require an environmental risk assessment as a property “fact of life.” And, although each assessment is site specific, following the guidelines to each phase can assure the parties involved that they are getting valuable information. Just remember the golden rule: do your homework and know your phases.
Don’t make waves, from page 7

crude oil dissolves. The water soluble phase tends to contain the small hydrocarbons (pentanes and hexanes), small aromatic hydrocarbons (benzene, toluene, ethylbenzene and the xylenes), as well as the hetero-atom-containing hydrocarbons. Figure 2, (page 6) shows the water soluble portion of a crude oil. The regular pattern of the n-alkanes is no longer present. As expected, the compounds seen are the low boiling alkanes and benzene, C1-benzene (toluene), C2-benzences (ethylbenzene and the xylenes), C3-benzenes and naphthalenes. Hetero-atom hydrocarbons are also present, however the abundance of these compounds and their low concentrations make their identification and detection very difficult. These latter compounds often appear as closely spaced peaks, sometimes forming a broad hump when present at high concentrations.

Even the most advanced gas chromatographic columns cannot separate these complex mixtures into their individual components. Analysis by gas chromatography/mass spectrometry (GC/MS) can lead to confusing data because the ions from several co-eluting compounds are matched for the best fit to a single analyte.

Other petroleum products also show large differences between the whole petroleum product and its water soluble fraction. Figure 3 (page 7) shows a GC trace of a typical 1991 automobile gasoline. The gasoline is comprised of the highly volatile portion of the crude oil. This material is blended with other petroleum intermediates, such as reformate (usually containing high levels of benzene, toluene, ethylbenzene and the xylenes), to make a final, highly sophisticated fuel needed to power cars.

Figure 4 (page 7) shows the water soluble portion of the same gasoline. As expected, the small hydrocarbon compounds and benzenes dominate the GC trace. The relative amount of each compound decreases as their water solubility decreases. Benzene is more water soluble than C1-benzene (toluene) which is more soluble than the C2-benzenes (ethylbenzene and the three xylenes) which are more water soluble than the C3-benzenes and naphthalene.

It is interesting to note that the water soluble portion of crude oil is fairly similar to the water soluble portion of gasoline, even though the starting materials are quite different.

Diesel is prepared from a different portion or cut of crude oil than is gasoline. Figure 5 (above) shows a typical GC trace of diesel fuel #2. The regular pattern of the n-alkanes can be seen in this chromatograph. The compounds present are higher boiling than with gasoline and, as a whole, diesel is less water soluble than gasoline. Figure 6 (above) shows the water soluble portion of the same diesel. As with the crude oil, the dominant n-alkanes are absent from the water soluble portion. The material that is present in the water is dominated by the benzene and toluene peaks, even though very little of these compounds is present in original diesel. Also present are C1, C2 and C3-benzenes, naphthalene, alkylated naphthalenes and some of the phenanthrenes and anthracenes.

What happens in the real world?

Consider what happens to a water sample submitted for a “Total Petroleum Hydrocarbons as Diesel” analysis. The water sample and its container are extracted into an organic solvent. (Quality assurance samples must also be processed.) The solvent extract is then injected into a GC and a GC trace, such as seen in Figure 7, above, is generated. The area under the peaks is calculated and this number is compared to standards and a concentration reported.

The result from this test may satisfy a regulatory requirement, but it may not adequately assess the environmental situation present at the site. The GC trace reveals the presence of the n-alkanes (X). Again, the n-alkanes are insoluble in water and were absent in the chromatogram of the water.
溶于水的石油烃的分布。它们的存在表明这种污染是由于在水中形成的油膜，或者由于吸附到固体材料上的油膜。如果需要测量潜在的外渗迁移，可以通过运动监测地下水，这在一定程度上是正确的。但是，要精确测量水溶性石油烃的量，就需要清除油膜和悬浮的微粒。这可以由合适的实验室取样技术或在实验室中使用适当的化学家完成。

图8所示的是水的第二GC图。不是n-烷烃的常规模式，而是水溶性石油烃的模式。图中有一个宽的峰，顶部有尖峰。实验室可以计算出这个峰的面积，或者他们可以计算出峰顶部的区域。这将使数百种化合物得以分类，其中许多化合物可能包括异原子化合物。它们可能是从燃料的降解而来的，或者是生物材料，可能是从水中摄取的。

如果必须将生物材料与饱和烃的混合物区分开来，则需要进行典型的样品加工步骤，这些步骤可以帮助做出正确的判断。

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HERE'S HOW IT WORKS:

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- HROBETZ HOT AIR PROCESS, P. 52

**COMMENTS:**

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**SOUND OFF!** COMMENTS, COMPLAINTS, COMPLIMENTS, CRITICISMS, ARTICLE IDEAS, SUGGESTIONS, FEEDBACK, WHAT YOU LIKE, WHAT YOU DISLIKE, WOULD LIKE MORE...? LESS...?
The O/C Tanks Hydroguard™ system was engineered to combine secondary containment with rustproof construction, built-in leak monitoring and precision testing capability, says the company. Secondary containment is provided by rigid double walls. Fiberglass material is inherently rustproof. Leak protection is accomplished by salt water—factory installed between the double walls. A sensor monitors the liquid level in the reservoir, which is surface-mounted to the tank top. The salt water, under hydrostatic pressure, has a stable level if both the inner and outer tank are tight. If an inner wall breach occurs, monitor fluid drains into the primary tank, causing the reservoir to drain. If an outer wall breach occurs, monitor fluid drains into the soil causing the reservoir to drain. If the groundwater table rises over the tank top, the reservoir will overfill with groundwater and activate the high level alarm on the sensor. An optional sensor will activate an alarm if the reservoir drains or overfills. A turbine enclosure limits ingress of water, protects the submersible pump from corrosive soil conditions and allows access to the pump. An electronically monitored collar provides secondary containment of possible piping leaks. It can detect the accumulation of leaked product. The systems are available in nominal capacities ranging from 600 to 20,000 gallons. Tanks have three or four 4-inch fittings (depending on tank size) to accommodate large capacity overspill containers or separate fill and vapor recovery systems. A fitting near the center of the tank can accommodate electronic inventory gauges. Four flow channels run the length of the tank to assure free flow of liquid between surfaces of both shell walls and end caps. The system has been independently tested and statistically validated to meet EPA and National Fire Protection Association criteria as a precision tank test. Write in 509 for more information.
AMS says their new gas vapor probe permits rapid testing, without interruption to service, for early detection of small quantity leakage around underground storage tanks. 4130 chrome molybdenum or stainless steel drive extensions are used to place the vapor probe tip, then removed to leave the probe imbedded at the desired location and sampling depth. A Teflon vapor tube runs from the tip to the surface. The outside diameter of the tip is larger than the diameter of the drive tubes to allow for insertion of the probe and so the drive extensions can be removed by hand in most applications. Optional mesh SST screen is available for the dedicated tips. A retrieval jack is available to assist in the removal of the extensions if needed. The vapor inlet holes are recessed in the tip. Just above the vapor inlet holes is a machined flange. This design creates a dead air space, says the company. A Teflon umbrella can be attached to the probe just above the flange. The umbrella is folded and inserted into the drive extension along with the tip. After the tip has been inserted to the desired depth, the removal of the drive extensions causes the Teflon umbrella to unfold. This prevents any dirt from falling in and around the vapor tip and prevents plugging of the vapor inlet holes. The Teflon tube is attached to the tip by pushing the tube over a barbed tang so the tubing cannot be pulled off. The Teflon vapor tube is threaded through the drive extensions and into the drive head. The drive head is attached to the up-and-down hammer attachment or a rotary hammer drill. The company says the tip can be used not only in soils, but also for liquid sampling in high water table areas.

Write in 510 for more information
disposal facilities must do in order to protect their long-term liability and viability.

Practical guidelines for field work
Whereas soil samples are often collected from the bottom of an excavation to determine whether all the contamination has been removed, waste disposal firms are only interested in regulatory analysis and analytical characterization of the excavated soils.

Because samples used to evaluate the effectiveness of the cleanup are often analyzed on a per weight basis rather than based on an extraction analysis, such as the TCLP method, their usefulness in waste characterization is somewhat limited...beside the fact that the bottom of the hole is not the waste under consideration for disposal!
Total constituent analyses are useful when making decisions involving soils contaminated by listed hazardous wastes and for determining the effectiveness of the remediation.

Soils determined to be hazardous—whether by listing or by hazardous characteristics—must be handled accordingly. These standards require notification of hazardous waste activity to the EPA, impose storage time limitations, as well as numerous recordkeeping, reporting management and administrative requirements. Therefore, before excavation, be sure all arrangements for disposal have already been made. Storage of hazardous waste soils in excess of 90 days could result in enforcement of standards applicable to treatment, storage disposal facilities (TSDF). The remediation of spills involving listed chemical products can turn a small quantity generator into an illegal TSDF overnight.

Remediation of sites where hazardous and non-hazardous waste soils are excavated will require close scrutiny by the disposal facility. To an owner or backhoe operator, dirt is dirt. As such, these projects require oversight by a representative of the disposal facility who is familiar with the cleanup plan and knows how to supervise the off-site management of residuals.

Sampling and analysis plans for contaminated soils should be developed under the guidance outlined in EPA publication SW-864. This document describes procedures for obtaining representative samples of tanks, impoundments, waste piles, etc. For contaminated soils which contain metals at or near regulatory thresholds, consider application of the statistical methods outlined in SW-846 as prerequisite for disposal approval.

The last word
There is no such thing as the “last word” on the disposal of contaminated soils. With the anticipated reauthorization of RCRA and focus on non-hazardous industrial wastes, the regulatory climate will continue to change. All the players in a cleanup must be aware of where the regulatory lines are drawn, which apply to what and how to stay out of trouble.

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See HNU at the Hydrocarbon Contaminated Soils Conference in Long Beach, Calif. March 9-12. Write in 131
The Hrubout® process is a portable system for in situ treatments that injects pressurized, super-heated air, via perforated piping, into and directly below the contaminated soil zone, says the company. As soil water is evaporated, soil pores are less constricted, soil permeability increases and greater air flow carries higher temperatures. Lighter hydrocarbons are volatilized and move to the surface which is sealed with an impermeable cover. Most volatiles will go out with the soil water, but the heavier constituents of the hydrocarbon chain will not. Under vacuum, the volatile gases are directed into an incinerator for a destruction rate of not less than 99.5 percent. Heavier hydrocarbon contaminants which will not volatilize are oxidized in-situ with higher temperatures. Some fractions of petroleum fuel such as residual heating oils and lubricating oil will not evaporate. As the temperature increases, they decompose to carbonaceous constituents and to carbon itself. They are oxidized at about 800°F. The incinerator is built to California specifications. Vapor destruction is at 1500°F with a .6 second retention time for a 99.9 percent volatile destruction rate. The auxiliary air blower dilutes incoming vapors if they approach lower explosive levels. The unit has automatic shutdown for high and low gas pressure, flame failure and high temperature. Heated air is provided by an adiabatic burner rated at 3.2 mm btu. Air is compressed by twin air blowers powered by a 150 hp electric motor. Burners for both incineration and injection are fueled by either propane or natural gas. The unit can also be used for ex-situ treatments involving either containerized soil or horizontal pipes laid on the surface, with the soil piled on top and sealed. Cost for in situ treatment is about $45-90 per cubic yard. Ex-situ costs range from $40 to 50 per cubic yard. The unit is trailer mounted, 38 feet long, 8 feet wide with a stack height of 13 feet and weighs 10 tons.

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