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PRESIDENT'S FAREWELL MESSAGE BY SUSAN GARCIA

I have had a wonderful time these past two years serving you as GRA Statewide President. How fortunate I have been to have worked with an excellent team of Officers, Directors, and Volunteers. Through our combined efforts, GRA has advanced forward as an advocate of groundwater issues. Within the past few years, we have worked jointly with a variety of organizations in achieving common educational goals, such as the Water Education Foundation, the California Department of Water Resources, Cal/EPAÕs Department of Toxic Substances Control and Regional Water Quality Control Boards, and the United States Environmental Protection Agency (USEPA). In addition to these entities, we have cosponsored a variety of courses with the University of Davis Continuing Education Department and have participated in local groundwater festivals. I thank these organizations, and others that I have not mentioned, for providing GRA the opportunity to work with you.

During 1996 and 1997, we have accomplished our objective to provide low cost, high quality technical programs for our membership. Seminars that we have sponsored that come readily to mind are the Rapid Site Characterization Seminar held in November 1996 and the more recent Innovative Monitoring and Remediation Applications Seminar held in September 1997. Both these seminars were well received by attendees. In addition to the seminars, our annual meeting technical programs have been quite informative and well received. I thank all the speakers, instructors and volunteers that have worked so hard to make our seminars and annual meetings such successes. In particular, I would like to thank Vicki Kretsinger of Luhdorf and Scalamini and Brian Lewis of the DTSC for their tremendous efforts to assure success for these events. We look forward to continue providing you with such fine technical programs.

Although I've briefly discussed some positive accomplishments for GRA, not everything has been rosy these past two years. We have experienced some growing pains as an organization, that have been compounded by the changing environment of our maturing industry. During these past few years, we have seen many organizations downsize, merge, and/or close shop. This has resulted in many individuals being preoccupied with seeking a new livelihood, accomplishing the work of many individuals, and/or leaving the industry for more lucrative territories. These external pressures have resulted in our membership growth leveling out, a reduction in new volunteers, a decrease in dinner meeting attendance within some Branches, a loss of some of our past sponsors, and the periodic inability of some of our volunteers to meet their organizational commitments. These items appear to be a reflection of the change occurring within our industry and should not be viewed solely as a reflection of GRA. They just mean that as an organization, we must work harder to assure we continue to grow and provide quality programs. I apologize to our membership for any inconveniences that have been experienced during our growth.

Acknowledgment of Volunteers

I contacted the various GRA Branches and have asked them to please identify individuals that should be recognized for all their volunteer services for GRA. These individuals have been dedicated toward assuring the continued success of GRA. Please recognize the following individuals:

- Sacramento Branch: David Von Aspern, Tom Mohr and Tim Parker
- San Francisco Branch: David Abbott, Jim Jacobs, Mary Keane, and Marcus Niebank
- Central Coast Branch: Tim Thompson, Deanna Cummings, and Kevin Kneese
- Southern California Branch: Jim Carter, Carmen Guzman and Lou Reimer
- South San Joaquin Valley Branch: Gary Corbell
- North-Central California Branch: Tom Venus

My apologies to any individuals that I may have overlooked. I thank you for your assistance.

On a statewide level, I would like to thank Bart Simmons of the DTSC for his Chemist Corner contributions to HydroVisions and all other contributors to the newsletter as well as our Editor, Floyd Flood. Also on a Statewide level, I would like to thank Carl Hauge of the California Department of Water Resources for all his assistance in our technical programs and as a mentor for GRA. Thank you everyone.

Newly Elected Statewide Officers

On November 10, 1997, we elected our new slate of Statewide officers. Join me in welcoming our new officers:

- President, Brian Lewis
- Vice President, Tim Parker
- Secretary, Paul Dorey
- Treasurer, David von Aspern

Best wishes for your 1998 term.

California Water Issues

In the last HydroVisions (Fall 1997, see Web page for edition), I asked GRA members to please provide us with their thoughts on what they believed were the most pressing water issues facing California today and in the future. Comments were to be printed in HydroVisions and later forwarded to the Honorable Cruz M. Bustamante, Speaker of the California Assembly. We have extended the time period for this request until February 15, 1998, so that we can include more of your comments. Please send your comments to GRA or e-mail them to editor@grac.org. I encourage you to provide us with your comments so that we can include your opinion.

Susan has served GRA well in her two years as President. Our by-laws have term limits. At our annual meeting, Susan was given an award for her contributions. We look forward to Susan's continued involvement with GRA.

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GRA 1997 Seminar: Innovative Soil Gas Monitoring and Remediation Applications

The GRA along with the Cal/EPA/Department of Toxic Substances Control cosponsored two, one day seminars on Innovative Soil Gas Monitoring and Remediation Applications this past September as part of their 1997 Seminar Series. Course instructors for the seminar were Dr. Donn L. Marrin, Consulting and Research Scientist; Dr. Harold W. Bentley, President of Hydro Geo Chem Inc.; and Mr. Craig Cooper, Superfund Project Manager for United States Environmental Protection Agency (USEPA). Topics covered by the course instructors included the following:

- Overview of Soil Gas Sampling and Analysis
- Applicability of Soil Gas vs. Soil Analyses
- Estimating Site Volatile Organic Compound (VOC) Mass Distribution
- Cleanup Objectives and Site Closure Criteria
- Monitoring Intrinsic Biodegradation and Vapor Flux for Site Closure
- Soil Gas Techniques to Support RI Process
- Monitoring of In-Situ Remediation

Specific points presented during the seminar consisted of the following items. The instructors encouraged the use of soil gas sampling alone or in combination with soil matrix sampling. Soil gas sampling for VOCs at depths 5 feet or less was not recommended, because the majority of the VOCs would have volatilized. Deeper sample depths were recommended. For coarse-grained soils, soil gas samples were preferred over soil matrix samples. The rationale provided was that contaminants did not readily sorb to the coarse grained soil matrix and an area would be construed as not impacted if solely based on soil matrix samples. Soil gas samples would be able to detect these contaminants in coarse grained samples and would permit the investigator to better assess if contaminants had reached groundwater. Soil gas techniques are an under used tool that should be considered beyond the site characterization phase of a field program. Soil gas techniques can be used for monitoring the effectiveness of remediation and assess natural biodegradation at a site. Soil gas monitoring techniques should be considered when developing operation and maintenance programs for sites being remediated. These are just some of the items discussed during the seminar.

We still have some extra Seminar Manuals available. Individuals interested in a copy can purchase it for \$40 from GRA. Please send your requests, along with your name, mailing address and payment to:

GRA Soil Gas Seminar Manual P.O. Box 1446 Sacramento, CA 95812

Please make checks payable to Groundwater Resources Association. Orders will not be processed without payment. Funds acquired from the sale of these manuals will be used to help offset costs for future seminars so that we may provide you with high quality seminars at the most economical prices.

GRA greatly appreciates the presentations provided by Dr. Marrin, Dr. Bentley, and Mr. Cooper. We thank them for making time in their busy schedules to prepare course handouts and give their presentations. We thank McClellan Air Force Base Environmental Management Directorate for letting us use their facilities in northern California. We thank InterPhase Environmental, Inc. for handling registration and for sponsoring travel and lodging for seminar instructors. Lastly, we thank those individuals behind the scenes who undertook the necessary effort to assure these seminars were a success. Keep your eyes open for our 1998 Seminar Series. We are always receptive for suggestions on future seminars.

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GRA Publishes Handbook on California Groundwater Management

The handbook on California Groundwater Management contains general information about the law and hydrology of groundwater basins as they pertain to groundwater management. The authors have tried to meet the needs of the greatest number of people and consequently the handbook is designed to be read and used by people of many levels of understanding, background and knowledge. Topics covered include groundwater hydrology, groundwater quality, wellhead protection, developing groundwater management plans, legal and institutional framework including groundwater law. A glossary of groundwater terminology and an extensive list of cited references are included.

To request a copy of California Groundwater Management, contact Harrison Phipps, GRA executive director phone/fax (530) 758-3656, address 601 Villanova Dr., Davis CA 95616-1827, e-mail <u>execdir@grac.org</u>. Authors of the handbook are Steve Bachman, Carl Hauge, Kevin Kneese and Anthony Saracino. The handbook was supported by a grant from the US EPA

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Letter to the Editor WSPA responds to article

The Western States Petroleum Association (WSPA) appreciates GRAÕs interest in stimulating further scientific debate on the role of purging in groundwater sampling Technical publications such as yours play a critical role in the continuing education of the scientific community. In that capacity, you are responsible to the scientific community, the regulatory community and the public at large to report new information in an objective manner. Unfortunately, your article, ÒRegulatory Board Duped by Flawed StudyÓ (Summer, 1997 edition of HydroVisions), disregards this responsibility by presenting a strikingly one-sided treatment of WSPAÕs research (ÒThe California Groundwater Purging Study for Petroleum HydrocarbonsÓ). Certainly, GRA is doing a disservice to its readership by perpetuating a number of misconceptions and misinterpretations about the intent and recommendations of WSPAÕs study. Although we have previously attempted to address many of the specific concerns raised in your article, for the benefit of your readers and in the interest of motivating a more balanced and productive scientific debate on this issue, this letter and the following attachments are intended to revisit these concerns in a more definitive fashion.

The decision to evaluate bailer and vacuum truck purging methods continues to be a focal point for criticism of the study design and findings by some reviewers. In previous correspondence with Dr. Michael Barcelona (University of Michigan), WSPA emphasized that the widespread use of bailers and vacuum trucks necessitated their inclusion in the study to ensure that study findings were applicable in terms of the current regulatory environment. WSPA also acknowledged without question that low-flow purging through dedicated pumps in depth-discrete wells can yield samples which more closely resemble formation water than samples taken by bailer or vacuum truck purging methods. An important point that was overlooked in your article is the fact that 47% of the samples in WSPAÕs data set were obtained from submersible pumps. Not surprisingly, there is less variability in this data than in the bailer and vacuum truck data. However, a threshold question which was a primary focus of WSPAÕs research and which continues to be obscured by this debate, was whether any differences observed in constituent concentrations before and after purging would be significant enough to affect a regulatory decision (i.e. a petition for site closure). Our analysis suggests that this is generally not the case, even where bailers are used for purging. Certainly, the incremental sampling prevision attributed to dedicated pumps does not appear to provide adequate justification for maintaining purging during routine groundwater monitoring.

Perhaps the most unfortunate aspect of your article is the conclusion that regulatory agencies choosing to implement some form of direct (no-purge) sampling have rushed to judgement based on ÒflawedÓ science. This conclusion is misdirected and highly speculative. Rather, it is our understanding that these agencies have carefully considered the same core questions that motivated WSPAÕs study and have come to the conclusion that the study findings do provide a reasonable basis for discontinuing purging during routine groundwater monitoring where sites meet the conditions specified in the report. In recognition of the uncertainty in the data set, some agencies have conditioned their policies to require responsible parties to reinstate purging for a single sampling event to validate direct sampling data before a final regulatory decision is made. This approach appropriately balances emerging scientific information with resource protection and economic concerns and in so doing seems far more rational than making a wholesale and potentially unnecessary commitment to an alternative purging technology.

The following attachments speak to specific technical issues raised in your article. WSPA hopes that this correspondence will help resolve much of the confusion surrounding application of the study findings. We urge GRA to print this letter and the attachments in their entirety in a subsequent edition of HydroVisions. In addition, we encourage your readers to draw their own conclusions based on a full reading of WSPAÕs report, available on internet at http://www.secor.com/purge.htm. If you have any questions, please contact Paul Horton of Secor at (510) 686-9780, Ralph Moran of ARCO at (213) 486-1257 or me at (916) 498-7753.

Sincerely, Jeff Sickenger Environmental Issues Coordinator

Attachments cc: Dr. Michael Barcelona - (U of M, Ann Arbor) James Giannopoulos - SWRCB Allan Patton - SWRCB

Attachment 1

Comment:

Volatile compounds may be lost from ÒstagnantÓ or poorly purged wells.

Response:

There is myriad research supporting the position that water within the screened portion of monitoring wells is not stagnant, even in low permeability formations. In addition, findings from WSPAÕs study show that samples taken before purging have a tendency toward slightly higher hydrocarbon concentrations which suggests that concern is generally not significant from a regulatory policy standpoint. With respect to the small percentage of sites where the pre-purge sample exhibited lower concentrations than the post purge sample, adherence to the implementation conditions specified in WSPAÕs report should be sufficient.

Comment:

Wells screened across the water table are not useful to determine chemical and hydrogeologic conditions in the subsurface.

Response:

This comment disregards the context of the study which is critical to the validity of the study findings. The vast majority of monitoring wells at California UST sites are screened across the water table.

Comment:

Comments of "experienced" critical reviewers were ignored.

Response:

This statement is simple not true. The study incorporated dozens of comments from dozens of reviewers, including Dr. Barcelona. In fact, WSPA responded directly to all of Dr. Barcelona's comments. The fact that many of Dr. Barcelona's comments did not affect the scope of the study does not mean that they were ignored.

GRA has made available to its members copies of Dr. Barcelona comments. If you would like a complete set of his comments and annotations to WSPA's study, submit \$5.00 to cover postage and reproduction costs to: GRA, P.O. Box 1446, Sacramento, CA, 95812.

My apologies for using the word "Duped." A less demeaning word could have been used. Floyd Flood

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Job Announcement Hazardous Substances Engineering Geologist (Engineering Geologist Series)

The Department of Toxic Substances Control (DTSC) has two job openings in Sacramento and one position in Long Beach for a Hazardous Substance Engineering Geologist. The Sacramento positions provide statewide technical support and the Long Beach position (which is pending personnel approval) provides technical support to Southern California. These positions assist the permitting, site mitigation, and compliance divisions. The incumbents undertake engineering geological studies of operating and closing hazardous waste sites. They provide geological expertise for use in the characterization of soils and groundwater; evaluation of geological hazards effecting hazardous waste sites, environmental monitoring programs, containment and abatement of releases, and development of design criteria for hazardous waste disposal sites.

Submit resumes and a state application to: Karla Gillespie Department of Toxic Substances Control P.O. Box 806 Sacramento, CA 95812-0806

Final Filing Dates: Post marked by Jan. 30, 1998 for the Sacramento position. To be determined for Long Beach position. Please indicate the location (Sacramento or Long Beach) you are applying for on your application.

(To be eligible for these positions, you must be on a state hiring list for Engineering Geologist or a current state employee.)

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Sponsor Acknowledgment 1997

GRA operations are funded through membership dues and donations made by members and their affiliated companies. We would like to recognize those that have contributed to GRA's future in 1997:

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Future Role of Groundwater in California BY HARRISON PHIPPS

Groundwater is seen by many as playing a big part in the solution to meet CaliforniaÕs growing water needs. In addition to population growth, there are currently a number of programs proposed in California that would result in significant increases in the use of groundwater.

The U.S. Bureau of Reclamation has just released the Central Valley Project Improvement Act (CVPIA) draft EIR. The intent of the CVPIA is to balance limited water resources among many different users including agricultural, municipal and fish and wildlife. All the alternatives in the EIR would reduce the average water deliveries to CVP contractors.

USBR intends to purchase surface water from willing sellers to meet the increased water needs for instream flows and refuge water supplies. As surface water is sold, land must either be retired from irrigated agricultural production or water must be pumped from wells. The EIR estimates groundwater depths will increase by varying amounts as a result of the project.

The CALFED Bay-Delta program was created to develop a plan to restore the Sacramento-San Joaquin Delta. CALFED has developed a number of alternatives to achieve its goals. Whichever alternative CALFED chooses, it will include the Ecosystem Restoration Program Plan (ERPP) also developed by CALFED. The goal of the ERPP is to restore habitat and fisheries in streams and rivers that are tributary to the Delta. Most of the watersheds identified for restoration in the ERPP would need to decrease surface water diversions in order to meet instream flow needs. In some cases the surface water reallocated to instream flow is expected to be replaced with groundwater.

In 1995 the State Water Resources Control Board (SWRCB) adopted a water quality plan for the Delta. The Plan identifies municipal and industrial, agricultural and fish and wildlife beneficial uses and specifies objectives to protect those uses. The California Department of Water Resources (DWR) and USBR have agreed to implement the objectives until SWRCB adopts a water right decision to reallocate shares of the responsibility to other parties. The SWRCB has just released its EIR that describes how it intends to reallocate water rights for diversions that affect the Delta. According to the EIR, implementation of the plan would significantly affect water supplies which would result in increased groundwater use.

The Department of Water Resources developed its Supplemental Water Purchase Program (SWPP) to help meet the water needs of the State Water Contractors. One part of the SWPP was a groundwater substitution program in which DWR would purchase surface water from willing sellers who would then switch to using groundwater. After much heated debate and opposition from northern California water interests, DWR dropped the groundwater substitution part of its program.

In addition to the above programs, the California Water Plan predicts that in the near future during normal rainfall years, the state will be short several million acre-feet of water per year due to population growth.

It is not clear how much surface water will be reallocated but it is certain that groundwater use will increase significantly as a result of proposed programs and imminent population growth.

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Investigative Analyses for Evaluating a Petroleum Released Beyond the Basics

BY JOE WIEGEL, COLUMBIA ANALYTICAL SERVICES

Introduction

This document consolidates our 3-part series as published in three consecutive issues of the *Lab Link*, published by Columbia Analytical Services, Inc. This series discusses investigative techniques, collectively referred to as forensic analyses, used to evaluate a petroleum release. The introduction provides some general information on methods and their applications. Part 2 covers the use of the techniques described for approximating the age of a petroleum release, along with difficulties encountered in attempting to age a hydrocarbon product after it has been released to the environment.

Part 1: Summary of Forensic Techniques

The term ÒforensicÓ generally implies some judicial or legal application. Forensic medicine, for example, describes a science that deals with the application of medical fact to legal problems. This conjures up memories of Quincy MD racing to beat the legal clock and save the innocent from wrongly serving time for a crime not committed. Or how about the O. J. Simpson trial: endless hours of boring scientific detail on DNA blood typing and disastrous foibles on the part of the LAPD medical examination.

The crux of any forensic investigation must be based on scientific evidence. However, the interpretation of the results is almost always argumentative. The following is a summary of analytical techniques that may be used in building an argument. Techniques range from making discrete, precise measurements to complex and sometimes speculative models. Each has its own place in the investigation. When used in combination, it may be possible to develop a well formulated argument in support of a theory regarding the legal aspects of a petroleum release.

Common forensic techniques are described in broad strokes. They have been discussed as categories of similar analyses. More specific details associated with any single technique are available from an analytical lab. Table 1 (see page 9) summarizes analytical techniques, provides method references, and lists some of the common applications of each analysis.

Costs associated with these investigations can range from a few hundred to a few thousand dollars per sample. Costs tend to go up with the need to establish a more precise explanation of contamination at the site. Therefore, prior to submitting samples for analysis, it is recommended that some time be spent defining the scope of the investigation, performing a cost benefit analysis, and evaluating whether the site offers adequate opportunity for a reasonably successful investigation. A tiered approach is advisable, otherwise, there is the potential for generating costly, but non-usable information.

Chemical Analyses

- **High resolution composition analyses:** These tests includes: Fuel Fingerprint, PIANO (Paraffins, Isoparaffins, Aromatics, Napthenes, and Olefins), Simulated Distillation, GC/MS techniques for Alkylbenzene ratios, PAHs/PAH Homologs and Biomarkers. These tests are designed to provide detailed information regarding the hydrocarbon content of the sample. They are useful in product type identification and in estimating the amount of environmental weathering. The analyses typically involve GC/FID or GC/MS instrumentation. The procedures can be performed on product, soil, or water samples. Methods are well established and documented as USEPA or ASTM protocols, although some modifications are required.
- **Clean-ups of organic extracts:** Typical techniques involve the use of Silica Gel, Alumina, Gel Permeation Chromatography, and/or Hydrolysis with Sulfuric Acid. There are many cases where, based on results of the fingerprint analysis, evidence indicates that the petroleum content is biased due to interfering co-extractable organics. This is frequently observed at or around wood processing sites, in tidal areas, or in locations where natural processes result in an organic enriched matrix. Contribution from the non-petroleum organics, results in a high bias in TPH measurements. These clean-ups provide a quick and relatively inexpensive way of establishing the interfering effect on the petroleum hydrocarbon result. A GC/FID or GC/MS analysis is usually performed on

both the uncleaned and cleaned extracts, with a comparison of the results providing evidence of the interfering component. Different clean-up fractions may be analyzed for specific hydrocarbon content (e.g., aliphatic, aromatic, and polar hydrocarbons) which may provide further insight into the nature of the interfering compounds relative to the petroleum content.

- **Hydrocarbon Segregation:** Fluorescent Indicator Analysis is used to determine total saturates, olefins, and aromatic content of the petroleum distillate. It provides information relevant to comparing a free product to ASTM fuel specifications.
- **Dye Additives:** UV-Visible Absorption Spectrometry, and Thin Layer Chromatography techniques may be useful in developing a relationship between dispensed petroleum products and free products released in the environment. Different dye additives are used by various manufacturers to distinguish between products. Color can occasionally be used to establish source relationship. However, these dyes are typically unstable in the environment and will undergo a color change as they degrade. Therefore, application is limited to relatively fresh releases, and ideally would involve a comparison between suspected sources and the free product. The technique may be useful in helping to establish if a free product is a mixture of different grades (e.g., different dye additives in a free product may indicate the presence of both premium and regular gasoline at the site).
- **Metals:** Information on total lead, total organic lead, individual organic lead species, manganese, barium, boron, phosphorous, nickel, vanadium, zinc (wear metals in oil) can be used to establish a link to specific events or practices. Often, organic complexes of these metals are added to the product during formulation to enhance operating performance. Additives have changed over the years as the result of both economic and social factors.

For example, gasoline containing organic lead was first marketed in 1923. The regulation of lead in gasoline has resulted in a well documented chronology of allowable lead concentrations. Furthermore, the chemical constituency of the lead additive has changed over time. Original lead additives were exclusively tetraethyl lead. The formulation changed to a mixture of tetramethyl lead, tetraethyl lead, and their cross products (in various combinations) from about 1960 through 1980. After 1980, tetraethyl lead was again the predominant organic lead species in gasoline. As a result, it is sometimes possible to establish a reasonably good estimate of the age of a gasoline release by examining the total lead levels in contaminated soil and groundwater, and individual organic lead species in the free product.

Various metals have been added to gasoline, diesel and jet fuels for reasons ranging from anti-knock improvement (lead and manganese), combustion chamber deposit control (phosphorous, boron, nickel and zinc), and smoke control (barium).

• **Halogenated Organics:** Ethylene dichloride and ethylene dibromide data can be used to establish a link to specific events or practices. Analysis is performed using standard USEPA protocols, or quantitation may be made using alternative techniques.

Ethylene dichloride (EDC) and ethylene dibromide (EDB) were added to leaded gasoline as part of the antiknock mixture (motor mix) to prevent a build-up of lead oxides in the combustion chamber. Motor mix formulations have been designed so that during the combustion process, lead is scavenged by chloride and bromide ions to form more volatile and less corrosive compounds than lead oxides. As a result, EDC and EDB are referred to as lead scavengers. The proportion of EDC and EDB in the total lead additive mix has changed over the years. The mixtures have been formulated so that an excess of lead scavenger is present to assure that all lead is complexed to the corresponding lead halide during combustion. In more recent formulations, combined EDC and EDB levels accounted for up to 36% of the total motor mix added to leaded gasolines.

• **Fuel Oxygenates:** A variety of alcohols and ethers are associated with gasoline production and formulation practices. They are added to improve octane rating, extend the fuel supply, and reduce vehicle emissions. The primary compounds of interest are methyl *tert*-butyl ether (MTBE) and ethanol, although other ethers and alcohols are used in oxygenate blends.

The use of alcohol as an anti-icing agent dates to as early as 1950. In the late 1970s, up to 10% ethanol mixtures gained favor as a way to extend the fuel supply during the fuel embargo. MTBE has been used on the east coast since the early 1980s, and on the west coast since the late 1980s. Oxygenates are extremely soluble in water and they tend to move freely with groundwater flow. Therefore, the presence of fuel oxygenates can provide information on gasoline formulation, a time frame for evaluating the release, and a mechanism to evaluate dispersion rates relative to other hydrocarbon components of the fuel.

- **Sulfur and Nitrogen:** The presence and amount of sulfur and nitrogen in a petroleum product can sometimes be linked to specific time events or the addition of fuel additives. Sulfur content has been regulated in most diesel fuels to below 500 ppm by weight. Nitrogen compounds are added to gasoline, diesel and jet fuels as detergents, gum inhibitors (antioxidants), corrosion inhibitors, and cetane enhancers. However, the use of this information for timing a release is limited. Sulfur is a common byproduct of anaerobic biological processes and sulfur content of a fuel in the environment can change dramatically depending on biological activity. Most detergents and other nitrogen-containing additives are unstable and easily biodegradable. Furthermore, although a specific marketer may claim to have a unique detergent, in reality the additive may be used by several marketers and referred to by different names. This further reduces the usefulness of nitrogen indicators for source differentiation.
- **Stable Isotope Analysis (13C/12C, 2H/1H, 207Pb/206Pb):** The measurement of stable isotope concentrations using mass spectrometry can provide precise information regarding the parent product. Stable isotope ratios of carbon and hydrogen have been used to differentiate sources of a petroleum release and may have application to a wide variety of petroleum products. Stable isotope ratios of lead have been used to differentiate sources and to estimate the age of a release. Its primary application is with leaded gasoline.

Other Analysis Tools

• **Compound ratio analysis:** Establishing ratios of specific compounds or classes of compounds in a hydrocarbon product or extract can be used to evaluate weathering, to discriminate between different sources, and in some cases to estimate the age of the release. For example, the concentration ratio of benzene + toluene to ethylbenzene + xylenes can be somewhat useful in estimating how long a gasoline product has been exposed to the environment. Another example involves the ratio of major C-10+ hydrocarbons (molecules containing 10 or more carbon atoms) to n-propylbenzene concentrations. Prior to 1985 the ratio exceeded 1. After 1985 the ratio was equal to 1. This allows an estimate of whether the gasoline is of a pre or post 1985 vintage. Other examples involve the use of stable isotope ratios. Global Geo-chemistry, Inc. (Canoga Park, California) has successfully used stable isotope ratios of carbon and hydrogen to establish source relationships between dispensed fuel and free product at a release site. CHEMPET Research Corp. (Moorepark, California) has used a model based on stable isotope ratios of lead to estimate the age of a gasoline release.

Compound ratio analysis can be useful in helping to establish liability for a release. Furthermore, plots of these ratios in the form of star diagrams and bar charts can be very instructive in presenting the data to non-technical people.

• **Modeling:** There are a variety of models that have been used in evaluating petroleum releases to help predict or explain site chemistry. Models can be used to estimate the age of a release, to test a time of release hypotheses, to predict or evaluate weathering effects over time, and to predict dispersion rates of various hydrocarbons relative to each other. Models can be based on a variety of parameters. They frequently involve chemical or physical properties of individual compounds, site geology and hydrology, and a knowledge of refining practices and fuel marketing trends.

Part 2: Aging a Hydrocarbon Release

Typically, the most pressing reason to establish the approximate age of a fuel release to the environment is to help determine liability for the event. Investigations of these events are frequently subject to controversy due to a lack of good records. It is sometimes possible to develop a reasonable approximation of the age of a fuel release by understanding refining practices, site geology, hydrology, and chemistry.

Costs can range from a few hundred dollars for a fingerprint of a fuel product to tens of thousands of dollars for a fullblown investigation involving several analytical techniques, extensive modeling, and expert witness support. Before starting an investigation, a cost-benefit analysis may prove to be one of the best uses of time and money spent on the project.

All of the commonly used aging techniques are based on developing an understanding of the fuel composition at the site in relation to time. Several approaches have been devised to gather and evaluate this information. Linking specific components in the fuel to refining practices, which have changed dramatically over time, is probably one of the most common techniques.

The presence of organic lead at a gasoline contaminated site indicates that the release occurred prior to the phase out of lead additives (late 1980s in most states, although leaded gasoline was used in sparsely populated areas into the 1990s). Allowable lead concentration has changed significantly over time. Between 1950 and 1980, concentrations of lead in gasoline averaged over 2 grams per gallon. This average concentration decreased steadily throughout the 1980s to about 0.5 gram per gallon by 1985, and to 0.1 gram per gallon by the late 1980s.1

Fuel oxygenates offer another indicator of a fuelÕs age. Oxygenates such as methyl tertiary butyl ether (MTBE) and alcohols began to be used with gasoline in the early to mid 1980s to meet regulatory standards imposed on the refining industry. Gasohol (gasoline with 10 percent alcohol) was prevalent during the 1972 fuel embargo in an effort to stretch the fuel supply. The presence of particular oxygenates in a free product gasoline and in contaminated soil and groundwater can be used as a time marker to estimate the release in combination with site history and other records.

Establishing the presence or absence of specific marker compounds provides a gross approximation of the time of release. The next tier of investigative techniques focuses on narrowing the window of approximation. Typically, these techniques involve more detailed analysis and may attempt to link chemical composition to established weathering characteristics.

With automotive gasoline, it is helpful to understand the historical trends associated with the lead additive mixture (motor mix). Motor mix formulations have changed considerably over time. The typical motor mix for leaded gasoline contained a mixture of antiknock agents (organic lead and organic manganese compounds), lead scavengers (ethylene dibromide (EDB) and ethylene dichloride (EDC), and small amounts of other ingredients (dyes, antioxidants, and stabilizers).

Formulations have changed considerably over time. Tetraethyllead (TEL) was the primary antiknock agent used in motor mixes prior to 1960. Throughout the 1960s and 1970s, other anti-knock compounds gained acceptance. Tetramethyllead (TML), cross products of TEL and TML, and methylcyclo-pentadienyl manganese tricarbonyl (MMT) were used in as many as nine different formulası. After 1980, tetraethyl lead again became the dominant antiknock agent used in various motor mix formulas.

Changes have also occurred with respect to the lead scavenger (EDB and EDC) formulations in motor mix. In order to scavenge all of the lead oxide formed during fuel combustion, an excess of EDB and EDC was needed in the motor mix. A typical motor mix in the 1980s contained approximately 62% lead, 18% EDB, 18% EDC, and 2% inactive ingredient2. Economic factors in combination with combustion chamber chemistry changed the ratios of EDB and EDC over time. Since EDC is the less expensive of the two scavenger compounds, there was a tendency to increase its use over the more expensive EDB. However, since hydrochloric acid is an unwanted byproduct of EDC combustion, EDB continued to be used in motor mixes to prevent excessive exhaust valve wear. Over time, these factors influenced the ratios of the two scavenger compounds added to different motor mix formulations.

Lead isotope analysis is another aging technique for leaded gasoline releases. This technique relies heavily on analytical testing in combination with historical trends and is based on a model that predicts the age of the organic lead added to the fuel. When tested on well documented fuel releases, the technique successfully predicted fuel ages within one to five years3. This level of accuracy is not typically achievable by other aging techniques.

The degree of product weathering and changes in specific hydrocarbon classes provides another approach to aging a fuel release. Again, techniques vary in accuracy and sophistication. In general, light petroleum distillates, such as gasoline, weather by pathways of evaporation and water washing. This weathering is most evident by the preferential loss of C1 to C5 hydrocarbons (due mostly to volatilization) and the reduction of benzene and toluene relative to ethylbenzene and xylenes.

Under certain conditions it is possible to approximate the age of a release based on the concentration ratios of these aromatic hydrocarbon groupings. For example, in silty soil saturated with gasoline the concentration ratio of benzene + toluene to ethylbenzene + xylenes will demonstrate about a 50% reduction over a 5 year period. Similarly, in groundwater, this ratio can be a useful gauge as to the freshness of the release. A concentration ratio of greater than 5, generally indicates a very recent spill. Over time, this ratio of benzene + toluene relative to ethylbenzene + xylenes will decrease exponentially due to greater solubility and subsequent transport away from the contamination2.

Middle distillate and heavy residual fuel oils are weathered primarily by biodegradation processes. Since the n-alkane fraction represents the most easily biodegraded portion of heavier fuels, loss of these components is the most obvious result of the weathering process. The ability to estimate the age of a release involving middle distillate and residual fuel oils can be highly dependent on site specific parameters. However, one of the most useful techniques involves a ratio of

n-C17 to pristane (the adjacent branched chain hydrocarbon). This ratio tends to decrease in a linear fashion with time as a result of the preferential removal of the n-alkane relative to the branched hydrocarbon chain. Other ratios that can be useful in estimating the degree of weathering include the n-C18 to phytane ratio and pristane to phytane ratio.

In spite of the level of sophistication used to analyze some petroleum releases, site characteristics frequently make it impossible to pinpoint a time of release. Extreme conditions include saturated soil zones, floating product on the water table, and tightly packed, fine grain clay soils. In addition to these extreme conditions, site chemistry can be misinterpreted as a result of multiple and undocumented releases, chronic releases over extended periods of time resulting in considerable product mixing, and releases involving multiple grades and/or fuel products. To maximize the chances for a successful investigation, it is important to include a good review of the site history, preliminary investigations, a detailed study of how site geology and hydrology might be expected to influence the chemistry of the released fuel, and perhaps most importantly, the formation of specific goals for the investigation. Developing a systematic approach to the problem, complete with clearly defined decision points, is the best way to ensure a reasonable use of time and money on an investigation of this nature.

Sources:

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- 2. Kaplan, I.R., Galperin, Y., ÒHow to Recognize a Hydrocarbon Fuel in the Environment and Estimate Its Age of ReleaseÓ, reprinted from ÒGroundwater and Soil Contamination: Technical Preparation and Litigation ManagementÓ, John Wiley & Sons, Inc., 1996.
- 3. Hurst, R.W., Davis, T.E., Chinn, B.D., OThe Lead Fingerprint of Gasoline ContaminationÓ, Environmental Science & Technology/News, Vol. 30, No. 7, 1996.
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Part 3: Differentiation of Sources of Hydrocarbons at Contaminated Sites

In this article we address two scenarios of hydrocarbon contamination. The first involves determining the primary source when a variety of sources may be responsible for the contamination. In this case, the age of the release is often critical. Since the previous issue dealt extensively with this topic, the current discussion is limited to using analytical techniques specifically geared towards differentiating between hydrocarbon sources. In some cases, the same techniques used to evaluate age of release are used in distinguishing the source of the product. These cases typically involve gasoline sites.

A second scenario involves differentiating between petroleum and non-petroleum sources of hydrocarbon contamination. Samples in this category are usually high in organic content. For example, the presence of wood debris will result in false positives for diesel range organics when analyzed by a typical TPH-D method. Middle distillate and residual range hydrocarbons make up the majority of these investigations.

In either scenario, a quick and inexpensive fingerprint or hydrocarbon scan using EPA Method 8015 Modified provides a good starting point. When interpreted by an experienced chromatographer the fingerprint can be used to evaluate choices for further analysis, and can help define the scope of the analytical plan. In some cases, the fingerprint analysis may be sufficient to identify the responsible party. More difficult cases involve distinguishing between sources of the same type of petroleum product (e.g., two different brands of gasoline).

Scenario 1

The two primary techniques used in differentiating between fuel products are identification of specific chemical indicator compounds present in one source but absent in the other, and performing component ratio analyses on the contaminant and the various potential sources.

The most common indicator compounds include fuel oxygenates, lead, and lead scavengers. These chemicals are all associated with gasoline releases.

Other chemical indicators are less useful, mainly due to their unstable nature in the environment. Dye additives can be useful in differentiating between brands and grades of fresh gasoline dispensed from the pump. However, once exposed to the environment, the dyes break down and begin to change color. These alterations limit the usefulness of dye chemicals in forensic studies to recent releases.

Component ratio analyses can be useful in differentiating between sources and in helping to identify the fuel type in a highly weathered sample. The application can be applied to both volatile and middle distillate fuels. A PIANO analysis (Paraffins, Isoparaffins, Aromatics, Naphthenes, and Olefins) is particularly useful for volatile fuel contamination. By comparing relative percentages of these classes of chemicals in environmental samples and dispensed fuels, it is sometimes possible to identify the source of contamination.

A similar technique is to compare relative concentrations of individual aromatic compounds (BTEX). When plotted in the form of star diagrams these comparisons provide a good visual representation of the data. Comparing ratios of specific compounds allows interpretation on biodegration and water washing compositional changes.

Another analysis that is used in differentiating sources of a fuel release involves ratios of stable isotopes of carbon and hydrogen. Because isotopic ratios do not change significantly as a sample undergoes weathering, it is possible to compare isotopic ratios in a sample to those of a dispensed fuel. If ratios in the contaminated sample strongly resemble those in the dispensed product, a case can be made that the source has been identified. Stable isotopes of lead can be used in a similar fashion to help differentiate between possible sources of leaded gasoline (although this differentiation is more dependent on time of release than comparisons to a dispensed fuel product).

Scenario 2

Several techniques can help determine the significance of non-petroleum hydrocarbon contamination at a site. For middle distillate and residual ranges, extract cleanup options are available for the removal of polar hydrocarbons that can cause high biases to a TPH-D analysis. Typically, the most useful application is to analyze the extract before and after cleanup, then compare the analytical results.

Significant reductions in TPH values following cleanup indicate positive bias due to interfering co-extractable organics. These cleanups, though not entirely effective in removing interferences, can be a relatively inexpensive way of evaluating high biases. Common cleanups include sulfuric acid digestion, saponification with sodium hydroxide, and separation using common adsorbents such as silica gel or alumina. New TPH methods developed in Washington and Oregon (i.e., NWTPH Methods) include options for these cleanup techniques, and the data is being accepted for regulatory review.

Though sometimes useful, cleanup techniques are not entirely successful at removing biogenic interference as shown in Table 2. Even after sulfuric acid and silica gel cleanup, pure woodwaste exhibits a response between 300 and 1900 ppm. These clean-up steps only removed 74 to 86% of the biogenic interferences.

Another relatively inexpensive technique for evaluating high biases at sites that contain wood debris is to compare analytical results obtained from standard TPH-D analyses to a Tannin and Lignin analysis. Samples that are mostly contaminated with petroleum hydrocarbon do not show good correlation with the tannin and lignin concentration. Samples that are primarily contaminated with wood debris tend to show good correlation between the two tests. To further characterize these sites, it is possible to oxidize the lignin in the sample and analyze for specific indicator chemicals by GC/MS SIM. The lignin degradation products can also be analyzed directly from water samples. These results can be used to evaluate the type of wood debris present at the site.

GC/MS techniques offer great flexibility in identifying petroleum and non-petroleum hydrocarbon compounds that can be used as specific markers in evaluating a site. Analyses for petroleum related alkylated benzenes, polycyclic aromatic hydrocarbons (PAHs) and their alkylated homologs, common wood related terpenes, and biomarkers such as terpanes, sterols, and steranes each have a place in fully characterizing contaminated samples. These analyses can provide detailed information regarding the molecular species currently inhabiting a contaminated site. Some of these compounds, such as the PAHs, are common to middle distillate and residual fuels.

Based on ratios of individual PAH compounds, the sampleÕs homolog fingerprint, and known concentrations of PAHs in fresh petroleum product, it is possible to estimate the amount of fuel responsible for the total hydrocarbon contamination in a sample. Wood waste marker compounds can be used to establish a definitive link to non-petroleum hydrocarbon contamination. Bio-markers provide information regarding the amount of weathering a fuel has undergone in the environment. All of this information can be useful in establishing links to the suspected sources of contamination at a site.

How a Lab Supports Forensic Studies

We appreciate the favorable feedback and support our readers have provided throughout the series. A question repeatedly asked is *ÒWhat support can a lab provide during a forensic investigation?*Ó

An analytical chemistry laboratory can perform the analytical procedures described throughout this series. A lab can coordinate and manage outside testing required. In addition, much of the work associated with a forensic study involves interpreting the analytical data, presenting the results in a format that strengthens the impact of the findings, and expert witness testimony.

Labs are best utilized when it also assists with the up-front preparation of a cost effective analytical plan. A lab can perform the initial investigative analytical support, evaluate options for further testing, and be utilized as a resource for technical litigation support and expert testimony, if necessary. A labs electronic information capabilities allow the data to be presented in a variety of formats to meet your specific application. This can be especially important for litigation purposes.

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GRA thanks the Vendor's for their participation in the 1997 Annual Meeting and 21st Biennial Groundwater Conference

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The Methods They Are'a Changing

BY BART SIMMONS, PH.D.

Better, Cheaper, Faster

In June, US EPA adopted Update III to SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. The EPA Office of Solid Waste, which maintains SW-846, has consistently pointed out that, with a few specific exceptions like the Toxicity Characteristic Leaching Procedure (TCLP), use of SW-846 methods is not required, and that any reliable method may be used for most testing. Nevertheless, states and other federal agencies have come to rely on SW-846 methods for a variety of testing. Update III includes 40 revised methods, 8 revised chapters, and 62 new methods. In addition, Update III deletes 16 methods, such as 8010, 8020, 8080, and 8240, which are packed-column gas chromatography methods, that have been replaced by capillary column methods. SW-846, although originally written for testing under the Resource Conservation and Recovery Act (RCRA), is widely used for a variety of environmental measurements, including groundwater monitoring. Update III includes several changes to the collection and analysis of environmental samples, including a variety of immunoassays (4000 series), and a closed-system purge and trap technique for volatile organics in soil (5035).

The transition to the new methods will have an impact on the whole environmental testing community. The elimination of some EPA methods poses a problem, since some methods are listed in Title 22 hazardous waste regulations, and because some permits and sampling and analysis plans have listed EPA methods which now technically no longer exist. Some of the calibration criteria have changed, which requires labs to change their standard operating procedures. The closed-system purge-and-trap system will require new sampling procedures for field staff. US EPA has encouraged some flexibility in the conversion to Update III, and it will certainly take some time for the conversion to Update III to be completed. In particular, some regulations will need updating, and some permits will need formal permit modifications to incorporate the new methods. Although the EPA Rule was effective immediately, it will take some time for conversion to the new methods.

Enter the Performance-Based Measurement System

The other solution to the methods problem is to eliminate the reliance on specific test methods. On October 6, US EPA published in the Federal Register a Notice of Intent to establish a Performance-Based Measurement System (PBMS) for all environmental media to the extent feasible. Although the exact details of a PBMS remain to be worked out, it would eliminate some of the prescriptive methods with a system which gives labs flexibility on methods so long as they meet criteria which are established by federal (and, presumably, state) programs. PBMS is also being considered for incorporation in the National Environmental Laboratory Accreditation Conference (NELAC) standards, which will be used for the new National Environmental Laboratory Accreditation Program (NELAP).

The debate over PBMS versus prescriptive methods has been going for some time, and the resolution seems to be that in some cases, e.g., the TCLP, a prescriptive method is necessary, but for many needs a PBMS should work more efficiently than the current system. EPA plans to implement a PBMS as early as next fall, so we are likely headed for some significant changes in the way environmental testing is done. Elimination of the reliance on specific test methods could force regulatory agencies, consultants, and laboratories to think more about exactly what data quality objectives are needed for a specific project. The outcome is likely to be a more flexible and rational system for environmental measurements.

Bart Simmons, Ph.D., is Chief of the Hazardous Material Laboratory, Department of Toxic Substances Control, Berkeley, CA.

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Legislative Corner

BY HARRISON PHIPPS

The Governor has signed into law several bills relating to MTBE:

AB 521 appropriates \$500,000 for a study of the risks associated with MTBE,

AB 592 requires the State Department of health Services to establish drinking water standards for the gasoline additive,

SB 1189 addresses pipelines and storage of MTBE near drinking water supplies,

AB 1491 restricts depositing petroleum into an underground storage tank unless it meets described requirements.

Another groundwater related bill that was passed, SB 660 addresses hazardous waste management and creates a new account \tilde{N} the Toxic Substances Control Account \tilde{N} to fund toxic waste cleanup, emergency response activities, and scientific and risk assessment programs.

SB 1307 makes changes to CaliforniaÕs Safe Drinking Water Act to conform to federal guidelines; provides the framework for the state to take advantage of \$1 billion in federal capitalization grant money for water system infrastructure improvements; and Requires DHS to develop a program to protect sources of drinking water.

In all there were 321 water-related bills considered, 101 dealt with flooding and 31 directly adressed groundwater.

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1998 Board of Directors' Meeting Date

All Members Welcomed

April 4, 1998 Contact: Brian Lewis Saturday (916) 323-3632

August 22, 1998 Contact: Brian Lewis Saturday (916) 323-3632

November 11, 1998 Contact: Brian Lewis Wednesday (916) 323-3632

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GRA Offers Lapel Pins

If you would like to buy a lapel pin, attend your nearest branch meeting or order a pin now. Pins cost \$7.00 at a branch meeting or \$8.00 thru the mail. Send your checks to: GRA, P.O. Box 1446, Sacramento, CA 95812

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Environmental Quality Team Cites Problems, Makes Recommendations

BY ANDREW D. EATON, MONTGOMERY WATSON LABORATORES/ACT/LABS, AND BRIAN LEWIS, DTSC, MEMBERS OF THE ENVIRONMENTAL QUALITY TEAM

The Environmental Quality Team (EQT), sponsored by Cal/EPA with members from the various boards and departments within Cal/EPA, as well as from other state labs and actLabs (Association of California Testing Laboratories), has finalized its report. DTSC has created a follow-up workgroup for implementing the recommendations.

The team's charter was to develop mechanisms to improve the quality of environmental data in California. The team was formed, in part, in response to significant problems that developed as a result of the laboratory fraud committed by Eureka labs on several military sites.

The report provides background on the problems that have been documented concerning the quality of environmental data used for decision making. These problems include poor field sampling procedures, poor lab procedures, production of fraudulent results by labs taking shortcuts, and sample integrity.

Recommendations

Many of the report's recommendations are relatively easy to implement, while others may require a significant commitment of resources. The EQT concluded that all of the recommendations were justified by gains to be realized in the quality of data available to the state. Among the recommendations for Cal/EPA Boards, Offices and Departments:

- Establish Quality Management Plans (QMP).
- Ensure Quality Plans are prepared in accordance with U.S. EPA recommendations. Of particular interest to laboratories, this recommendation also includes requirements that labs maintain magnetic tape data, that Quality Assurance Project Plans (QAPPs) specify how to deal with Onontarget analysts, Ó and QAPPs include reasonable data quality objectives.
- Use and monitor performance on double blind samples whenever feasible.
- Establish a frequency for lab and field audits of contractors.
- Ensure that all subcontractor labs performing sampling on a project are identified by name and identified as to the specific tests performed so that performance can be monitored.
- For environmental data management, adopt the use of standardized content for hardcopy deliverables, specify requirements for electronic deliverables, ensure that 10% of data on projects is validated by entities independent of all involved parties, and adopt EPA functional guidelines for review of data as a default guideline to evaluate data quality.
- Maintain and update standards of quality by adopting NELAC standards.
- Establish consistent laboratory and field audit protocols.
- Require that state laboratories, which support Cal/EPA programs, both within and outside Cal/EPA, be accredited by NELAP, when accreditation is available.

There is also a plan to follow up on the recommendations over the next few years to evaluate the long-term effectiveness of the selected recommendations by monitoring a number of parameters.

Overall, the team concluded that implementation of the recommendations should result in significant improvements in the quality of data produced for Cal/EPA programs. For further information on the report, or to obtain a copy, contact Fred Seto, Cal/EPA/DTSC-HML at (510) 540-3388. You can also download the report from GRA's web page: http://www.grac.org.

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