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Field Analytical Technology Verification: The ETV Site Characterization Program

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ABSTRACT

Innovative field characterization and monitoring technologies are often slow to be adopted by the environmental engineering/consulting community because of concerns that their performance has not been proven by an independent testing body, and/or they have not received the EPA's "blessing" on a regional or national level. The purpose of the EPA Environmental Technology Verification (ETV) Site Characterization Pilot, a joint effort between EPA and DOE, is to accelerate the acceptance of technologies that reduce the cost and increase the speed of environmental clean-up and monitoring. Technology verifications that have been completed or are underway include: *in situ* technologies for the characterization of sub-surface hydrocarbon plumes, field-portable GC/MS systems, field-portable X-ray fluorescence analyzers, soil sampling technologies, field-portable PCB analyzers, analyzers for VOC analysis at the wellhead, and decision support software systems to aid site sample collection and contaminant plume definition. The verification process follows a somewhat generic pathway. A user-community need is identified, the vendor community is canvassed, and relevant, interested companies are selected. A demonstration plan is prepared by the verification organization and circulated to participants prior to the field activities. Field trials are normally held at two geologically or environmentally different sites and typically require one week at each site. Samples (soil, soil gas, water, surface wipe etc.) provided to the vendor at the demonstration include site-specific samples and standards or "performance evaluation" samples. Sample splits are sent to a pre-selected laboratory for analysis using a reference method. Laboratory data are used for comparison with field technology results during the data analysis phase of the demonstration. Data analysis is the responsibility of the verification organization with results summarized in a Technology Verification Report and a three-page Verification Statement. Typical results from completed field demonstrations are presented in this paper to illustrate the verification process and the importance of the program in providing objective information to aid potential users in making informed choices regarding the efficacy of these technologies for their specific characterization and monitoring problems.

INTRODUCTION

Rapid, reliable, and cost-effective field screening and analysis technologies are needed to as in the complex task of characterizing and monitoring hazardous and chemical waste sites.

DISCLAIMER

Environmental regulators and site managers are often reluctant to use new technologies which have not been validated in an objective EPA-sanctioned testing program or other similar process which facilitates acceptance. Until field characterization technology performance can be verified through objective evaluations, users will remain skeptical of innovative technologies, despite their promise of better, less expensive, and faster environmental analyses.

The Environmental Technology Verification (ETV) Program was created by the U. S. Environmental Protection Agency to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, purchase, and use of environmental technologies. The ETV Program capitalizes upon and applies the lessons that were learned in the implementation of the Superfund Innovative Technology Evaluation (SITE) Program to the verification of twelve categories of environmental technology: Drinking Water Systems, Pollution Prevention/Waste Treatment, Pollution Prevention/Innovative Coatings and Coatings Equipment, Indoor Air Products, Advanced Monitoring Systems, EvTEC (an independent, private-sector approach), Wet Weather Flows Technologies, Pollution Prevention/Metal Finishing, Source Water Protection Technologies, Site Characterization and Monitoring Technologies, and Climate Change Technologies.

ETV SITE CHARACTERIZATION AND MONITORING PILOT

The mission of the Site Characterization Pilot is to identify, demonstrate, and verify the performance of innovative site characterization and monitoring technologies. The Pilot also disseminates information about technology performance to developers, environmental remediation site managers, consulting engineers, and regulators. The EPA utilizes the expertise of partner "verification organizations" to design efficient procedures for conducting performance tests of environmental technologies. The EPA selects its partners from both public and private sectors including Federal laboratories, states, and private sector entities. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from all major stakeholder/customer groups associated with the technology area. The U.S. Department of Energy's Sandia National Laboratories in Albuquerque, New Mexico, and Oak Ridge National Laboratories in Oak Ridge, Tennessee, serve as verification organizations for the Site Characterization and Monitoring Pilot.

The Technology Verification Process

The technology verification process is intended to serve as a template for conducting technology demonstrations that will generate high-quality data which the EPA can use to verify technology performance. Four key steps, discussed in more detail in the following paragraphs, are inherent in the process:

- Needs Identification and Technology Selection;
- Demonstration Planning and Implementation;
- Report Preparation; and,
- Information Distribution.

Needs Identification and Technology Selection

The first aspect of the technology verification process is to determine technology needs of the EPA and the regulated community. EPA, the U.S. Department of Energy, the U.S. Department of Defense, industry, and state agencies are asked to identify technology needs and interest in a technology area. Once a technology area is identified, a search is conducted to identify suitable technologies that will address the need. The technology search and identification process consists of reviewing responses to *Commerce Business Daily* announcements, searches of industry and trade publications, attendance at related conferences, and leads from technology developers and experts in the field. Candidate characterization and monitoring technologies are evaluated in light of the following criteria:

- Meets user needs;
- May be used in the field or in a mobile laboratory;
- Has a regulatory application;
- Applicable to a variety of environmentally impacted sites;
- High potential for resolving problems for which current methods are unsatisfactory;
- Costs are competitive with current methods;
- Performance as good or better than current methods in areas such as data quality, sample preparation, or analytical turnaround time;
- Uses techniques that are easier and safer than current methods; and,
- Is a commercially available, field-ready technology.

Demonstration Planning and Implementation

After a technology has been selected, the EPA, the verification organization, and the developer(s) agree to responsibilities for conducting the demonstration and evaluating the technology. The following issues are addressed at this time:

- Identifying at least two demonstration sites that will provide the appropriate physical or chemical attributes, in the desired environmental media;
- Identifying and defining the roles of demonstration participants, observers, and reviewers;
- Determining logistical and support requirements (for example, field equipment, power and water sources, mobile laboratory, communications network);
- Arranging field sampling and reference analytical laboratory support; and,
- Preparing and implementing a demonstration plan that addresses the experimental design, sampling design, quality assurance/quality control, health and safety considerations, scheduling of field and laboratory operations, data analysis procedures, and reporting requirements.

Report Preparation

Innovative technologies are evaluated independently and, when possible, with reference to conventional technologies. The field technologies are operated by the developers in the presence of independent technology observers affiliated with the EPA or the verification organization. Demonstration data are used to evaluate the capabilities, limitations, and field applications of each technology. Following the demonstration, all raw and reduced data used to evaluate each technology are compiled into a Technology Evaluation Report, which is a record of the demonstration. A data summary and detailed evaluation of each technology are published in an Environmental Technology Verification Report (ETVR). The ETVR includes a Verification Statement, which is a concise summary of instrument performance during the demonstration.

Information Distribution

The goal of the information distribution strategy is to ensure that ETVRs and accompanying Verification Statements are readily available to interested parties through traditional data distribution pathways, such as printed documents. Documents are also available on the World Wide Web through the ETV Web site (*http://www.epa.gov/etv*) and through a Web site supported by the EPA Office of Solid Waste and Emergency Response's Technology Innovation Office (*http://clu-in.com)*. Additional information at the ETV Web site includes a summary of the demonstration plan, test protocols (where applicable) demonstration schedule and participants, and in some cases a brief narrative and pictorial summary of the demonstrations completed.

DEMONSTRATION DESIGN ELEMENTS

The primary objective of the technology verification demonstrations is to test and verify the performance of field-portable characterization and monitoring technologies for sampling or analysis activities at contaminated sites. All Site Characterization Pilot demonstration designs incorporate the three objectives listed below:

- Verify instrument performance attributes that can be directly quantified. Such factors include blank and detection level sample performance, measurement accuracy and precision, data completeness, and sample throughput.
- Verify instrument characteristics and performance in various qualitative categories such as instrument ease of operation, required logistical support, operator training requirements, instrument transportability, and versatility.
- Compare field technology results to those from an off-site laboratory using reference analytical methods. An important underlying objective of the demonstration is an assessment of the reference laboratory data quality.

A guidance document that outlines the demonstration design process and presents critical design elements was developed in the early stages of this pilot program (1).

SITE CHARACTERIZATION PILOT DEMONSTRATIONS

Under the ETV Site Characterization Pilot, a first round of technology verifications have been completed and a second set is presently underway. A brief description of these demonstrations follows.

Cone Penetrometer Laser-Induced Fluorimetry

Two systems designed for *in situ* detection of sub-surface petroleum hydrocarbons were evaluated in 1995. Both systems utilized laser-induced fluorimetry techniques in conjunction with a truck-mounted cone penetrometer system. Laser light pulses, from a laser in the truck, are routed to a sapphire window near the cone tip through a fiber optic cable. The polycyclic aromatic hydrocarbon component of fuels in the surrounding soils yield a fluorescent response to this laser light. The fluorescence signal is collected at the probe window, routed back to the truck via a second fiber optic cable, and analyzed by a photodetector system. The technologies provide real-time indication of sub-surface fuel contamination as the cone is pushed down through the soil. Performance verification was completed for the following two systems: Rapid Optical Screening Tool--ROST (Fugro Geosciences, Houston, TX) and Site Characterization and Analysis Penetrometer System—SCAPS (US Navy Command, Control, and Ocean Surveillance Center, San Diego, CA). Tests were completed at fuel contaminated sites near Port Hueneme, CA and Albuquerque, NM. Technology performance relative to conventional methods was assessed by the collection and laboratory analysis of soil samples obtained using auger and split spoon sampler in bore holes immediately adjacent to soil probed by the cone penetrometer system.

Field-Portable GC/MS

A demonstration of field-portable gas chromatograph/mass spectrometer systems was also completed in 1995. Performance characteristics of three systems for soil, water and soil gas sample matrices were assessed at the DOE Savannah River Site, near Aiken, SC and Wurtsmith Air Force Base near Oscoda, MI. Systems evaluated were the SpectraTrak™ 672 (Viking Instruments Corp., Chantilly, VA), the EM640™ (Bruker Analytical Systems Inc., Billerica, MA), and the 3DQ Discovery™ (Teledyne Electronic Technologies, Mountain View, CA). The demonstration design included the use of performance evaluation (PE) samples as well as site samples to assess critical instrument performance parameters such as precision, accuracy and sample throughput. Sample splits were sent to a number of reference laboratories such that field instrument-laboratory comparisons could be carried out.

On-Site PCB Analysis

A technology demonstration of polychlorinated biphenyl (PCB) field analytical techniques occurred in July 1997. The demonstration was conducted at a DOE Oak Ridge site where a large repository of PCB-contaminated materials from multiple DOE sites exists. Technology developers with PCB monitoring instrumentation or chemical test kits were evaluated. These instruments and kits are suitable for the quantification of PCBs in a variety of matrices including soil and surface extracts. A fundamental objective of this demonstration was to evaluate how well the technologies can assist in regulatory decision-making processes for PCB-contaminated waste. Technologies evaluated in this demonstration included Dehalogenation/Ion Specific Electrode Analysis (Dexsil Corp., Hamden, CT), Immunoassay (Hach Corp., Loveland, CO) and (Strategic Diagnostics Inc., Newark, DE), and Gas Chromatography (Electronic Sensor Technology, Newbury Park, CA). The evaluation included PE samples of known PCB composition as well as numerous soil samples from a variety of DOE sites. As with other pilot demonstrations, an off-site commercial laboratory was selected and analyzed split samples using a reference EPA method for comparative purposes.

Wellhead Monitoring for Chlorinated VOCs in Water

Field-portable monitors for the detection of chlorinated volatile organic compounds at the wellhead were evaluated at two sites in September 1997. Instrument systems evaluated included the Voyager™ Gas Chromatograph (Perkin Elmer-Photovac, Wilton, CT), the Scentoscan Plus II™ Gas Chromatograph (Sentex Inc., Richfield, NJ), the Model 4100 Gas Chromatograph (Electron Sensor Technology, Newbury Park, CA), the HAPSITE™ GC/MS (Leybold-Inficon Inc. Syracuse, NY) and the Model 1312 Photoacoustic IR Analyzer (Innova AirTech Instruments, Naerum, Denmark). The demonstration focused on the analysis of contaminated ground water from extensive monitoring well networks at the DOE Savannah River Site and McClellan Air Force Base near Sacramento, CA. As with other technology demonstrations, the sample set included a significant number of PE samples such that an absolute measure of instrument performance in such categories as precision and accuracy could be obtained. Off-site reference laboratory measurements of sample splits were also carried out.

DATA ANALYIS TECHNIQUES

The challenge for the verification organization in the conduct of ETV Technology Verifications is essentially two-fold: 1) develop a field experiment plan that adequately tests the participating technologies and ensures an acceptable data set at moderate cost; and, 2) analyze and report the data in a concise and timely manner. Data analysis tasks fall under both quantitative or qualitative categories as outlined below.

Quantitative Factors

Quantitative instrument performance factors verified and reported include such instrument parameters as: field measurement accuracy and precision, variation of accuracy and precision over an instrument's working range or under various environmental conditions, instrument performance at sample concentrations near its level of detection, blank sample response, measurement specificity, measurement comparability with reference methods, sample throughput and others. Some illustrations of the analytical procedures used to assess quantitative instrument performance attributes in completed and ongoing demonstrations are given below.

Instrument Precision

In the field demonstrations, measurement uncertainty is assessed over the working range of the instrument by the use of replicate samples from a number of PE mixtures. Most designs incorporate four or more blind replicate analyses over a range of target analyte concentrations. For example, the Wellhead Demonstration utilized five different PE mixtures—each containing a dozen or more chlorinated VOC compounds—at each demonstration site. The volume of data from this many samples is best represented graphically, as shown in Figure 1, for the GC/MS system which participated in the demonstration. Data pooling techniques are also employed to further summarize overall instrument performance. A frequency distribution of pooled relative standard deviation values from all compounds in all PE mixture analysis at the Wellhead Demonstration is shown in Figure 2 for one of the gas chromatograph (GC) systems. The median and 95th percentile values of the distribution are computed and are presented in the summary statements concerning instrument performance.

Instrument Accuracy

Instrument accuracy is also evaluated by using results from the PE samples noted above. Comparison of field technology results to PE samples of known composition avoids the complications that can arise when technology results are only compared to laboratory results. In certain instances, the laboratory may encounter problems in sample handling or analysis which can jeopardize the integrity of the entire study. Thus PE samples with an independent certificate of analysis afford an added level of protection in the demonstration design. Mean sample recoveries, derived from replicate sample analyses, are used to evaluate instrument performance relative to PE samples. A plot of mean recoveries for one of the PE mixtures used at the Wellhead demonstration is shown for a gas chromatograph system in Figure 3. To derive summary statistics, recovery data are expressed as absolute percent difference from the true value and the data are pooled to determine median and 95th percentile values of the distribution shown in Figure 4. Although information on instrument bias can be obtained from such data, in these performance verifications a summary statement of absolute instrument accuracy (e.g. median accuracy is ±20%) is generally considered adequate.

Blank Sample Response

Blank samples are also included in the sample sets provided to demonstration participants. These are submitted blind to the participants and are used to assess false positive detection or problems with instrument contamination when moving from high to low concentration samples. The data are summarized by reporting the false positive rate of target compounds of particular interest in the study.

Method Detection Levels

The scope of these demonstrations does not include a comprehensive evaluation of instrument detection levels. However, to assess instrument performance at the lower end of its working range, ten or more replicate samples at or slightly above the expected detection level of each instrument are provided for analysis at each site. For example, in the Wellhead demonstration, ten replicates of a 14-component PE mixture were submitted blind to the participants throughout the 5-day study at each site. The mixture was prepared at a concentration level of 10 μg/L which was a factor of two or three above the detection levels reported by the instrument developers for target chlorinated volatile organic compounds. Results from these analyses are tabulated and reported as false positive detect rates on a compound-specific basis.

Sample Throughput

Sample throughput is also assessed during the demonstration, and in the context of these verifications takes into account all aspects of sample processing including sample handling and preparation, daily instrument calibration, sample analysis, occasional re-analysis, and data reduction. In some cases, instrument sample cycle times may be fast, however, extended data analysis efforts may limit sample throughput. The multi-day demonstration design permits the determination of sample throughput rates over an extended time period, and thus is representative of throughput rates likely to be observed in routine field use of the instrument.

Sample throughput rates are generally reported both in number of samples per hour and number of samples per 8-hour day.

Instrument-Laboratory Comparison

One of the most challenging aspects of data analysis is carrying out an objective analysis of technology performance in comparison with laboratory performance. As noted previously, laboratory analyses posses their own uncertainty and may not always serve as a reliable reference. Nonetheless, potential instrument users or regulatory personnel frequently desire to see how a technology performs relative to an accepted method that relies upon off-site laboratory analyses. A visual comparison of field technology and laboratory performance can be obtained through the use of an x-y scatter plot as illustrated in Figure 5. Laboratory data from a set of soil sample analysis for PCB using EPA SW846 Method 8081 (solvent extraction/gas chromatography-electron capture) are plotted relative to the results obtained from split samples by the Dexsil Model L2000 field analysis system. The diagonal line on the figure indicates a slope of unity. The correlation coefficient can also be computed from these data pairs to give a single measure of the degree of linear correlation of the data. Linear regression parameters such as slope and intercept can yield evidence of field technology-laboratory bias or non-linearity (2).

When replicate analysis of sample pairs are analyzed by both field technology and laboratory, confidence intervals about a mean value can be computed and graphically portrayed as illustrated in Figure 6. In this illustration from the PCB demonstration, laboratory 95% confidence intervals are in many cases much broader than those of the field technology suggesting that the field methodology is in fact more precise that the laboratory method. Formal statistical tests can be applied to assess the null hypothesis (e.g. field technology and laboratory results are equivalent) however, detailed statistical analyses are generally avoided in the data reduction and reporting phase in deference to an anticipated report readership with limited statistical background and training. A non-parametric test which is particularly well suited to testing the statistical significance of a suspected bias between field technology and laboratory which has occasionally been employed in data evaluation is the Wilcoxson signed rank test (3). The test examines the observed differences between the technology-laboratory sample pairs and gives a probability that observed variation between the two data sets is the result of random variation only and not caused by methodological differences. In many cases, a scatter plot such as Figure 5 can be used to communicate the same information to the reader without the use of statistical tests.

Qualitative Factors

Important qualitative instrument performance factors are instrument portability, logistical support requirements, operator training requirements, ease of operation, and others. Logistical requirements include a description of the technology's power requirements, setup time, routine maintenance requirements, and the need for ancillary equipment or supplies, such as a computers, reagent solutions, or gas mixtures. Qualitative factors such as these are assessed during the demonstration by the use of technology auditors. The auditor's role is to observe the instrument in operation during the demonstration. Operator interviews as well as extended observations give the auditor a sense of instrument performance characteristics that may not be fully detailed in technology descriptions provided by the vendor to the verification organization prior to the field demonstration. Instrument costs and associated field operational costs are also a subject of the field audit. A limited cost comparison between the use of field technology and off-site laboratory is also carried out to further assist the potential technology user in making informed choices about which technology should be applied in a given characterization or monitoring situation.

REPORTING AND INFORMATION OUTREACH

Instrument performance is summarized in a Verification Statement and in a Technology Verification Report (4 -7). The Verification Statement is a concise three-page summary of the verification effort. It gives a brief description of the demonstration design, the demonstration sites, principle of operation of the technology, and a summary of the performance attributes of the technology in areas such as accuracy, precision, sample throughput, and laboratory comparability. The Verification Statement is intended to capture the essence of the demonstration and the technology results. By virtue of its concise format, it is amenable to widespread distribution through the ETV Web Site and other electronic means. The Environmental Technology Verification Report is a relatively thorough description of the demonstration and results. The goal of the pilot is to complete the entire process, from vendor selection to completed reports, in one year. A separate report is prepared for each participating technology and no attempt is made by the verification organization to compare one technology with another. Earlier verification efforts involved a requirement for the participants to submit performance claims which were then evaluated in the demonstration. A determination was made as to whether claims were met during the demonstration. The present approach has moved away from a vendor claims evaluation approach. Alternatively, the provision is made through the inclusion of appropriate demonstration design elements, that the verification organization can objectively state the performance attributes (precision, accuracy etc.) of the technology without resorting to a pass-fail connotation. The intention of the program is to provide the prospective user with enough information about the performance characteristics of a suite of technologies such that the user can make an informed choice as to which technology to apply in light of established data quality objectives for the application of interest.

CONCLUSIONS

The Site Characterization and Monitoring Pilot of the EPA's Environmental Technology Verification Program is the longest in existence of the ten pilots currently underway. The purpose of the pilot is to accelerate the acceptance of technologies that reduce the cost and increase the speed of environmental clean-up and monitoring. The Site Characterization Pilot has completed two technology demonstrations of in-situ monitoring for sub-surface fuel contamination and field-portable GC/MS systems and is nearing completion on an additional two for field analysis of PCB in soils and monitoring for chlorinated VOCs at the wellhead. An additional two demonstrations dealing with decision support software systems and ground water sampling technologies are in the beginning stages.

Each new demonstration presents continuing challenges for the verification organization. Fundamentally, they are the development of scientifically-sound field demonstration plans that are cost effective and quickly implemented, and the use of data analysis techniques that can succinctly summarize field technology performance for a wide readership base. Achievements in these areas illustrate the importance of the EPA's Environmental Technology Verification program in providing objective information to aid potential users in making informed choices regarding the efficacy of new and innovative technologies for their specific characterization and monitoring problems.

ACKNOWLEDGEMENTS

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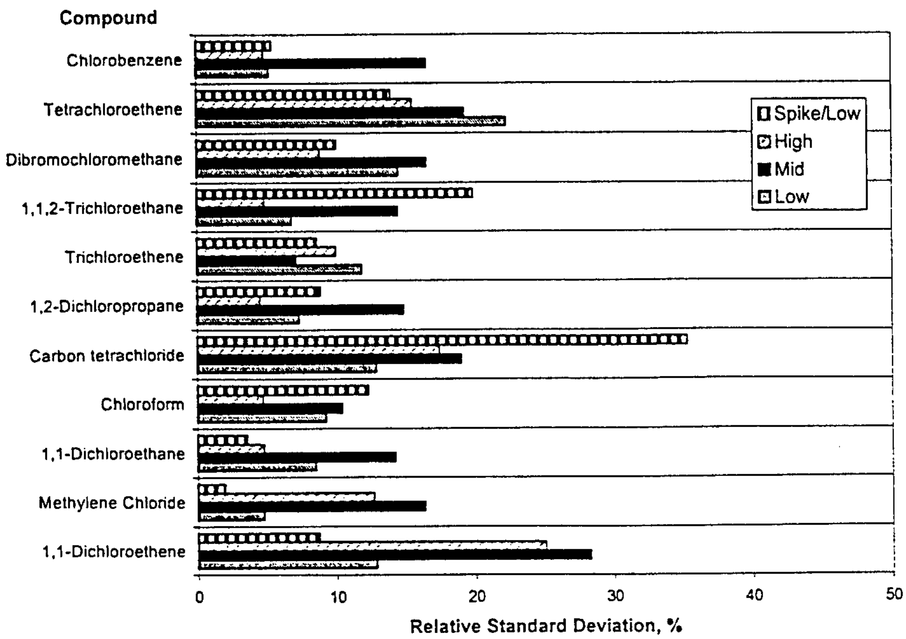

Figure 1. Precision of the HAPSITE portable GC/MS for a mixture of chlorinated VOCs in water

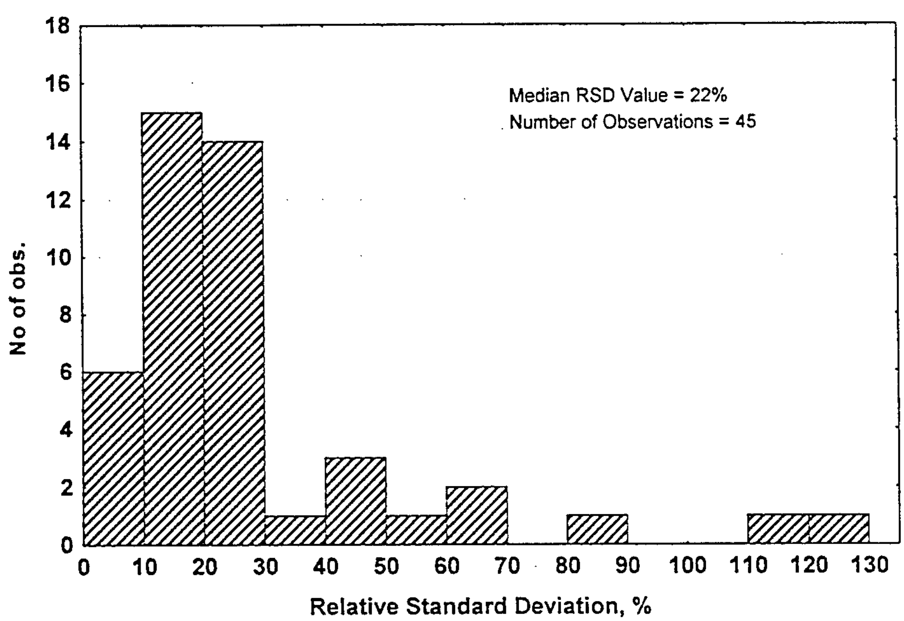

Figure 2. Frequency distribution of pooled RSD values for EST Model 4100 GC

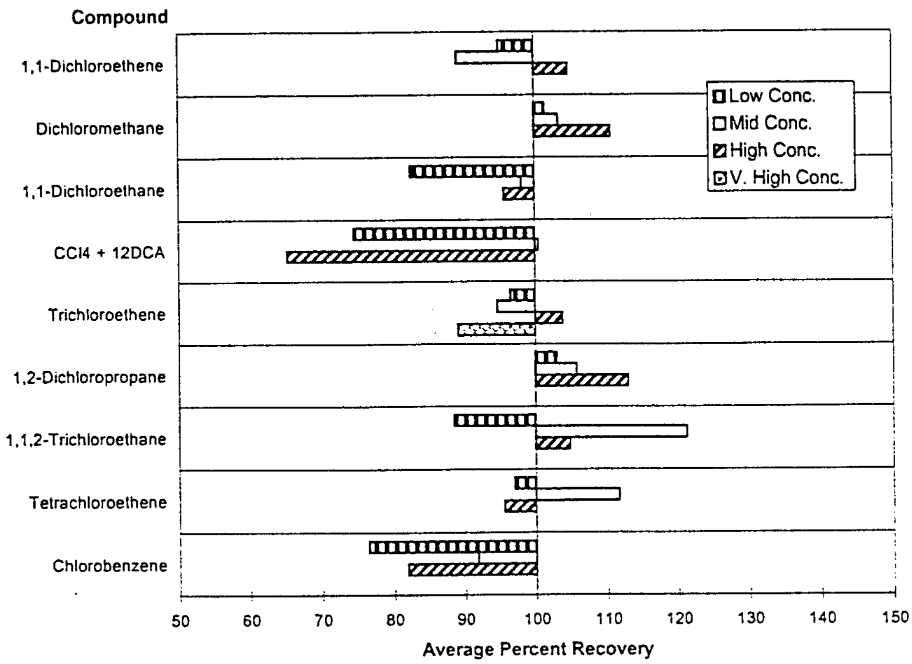

Figure 3. Average recoveries of the Scentograph Plus II gas chromatograph for selected target compounds

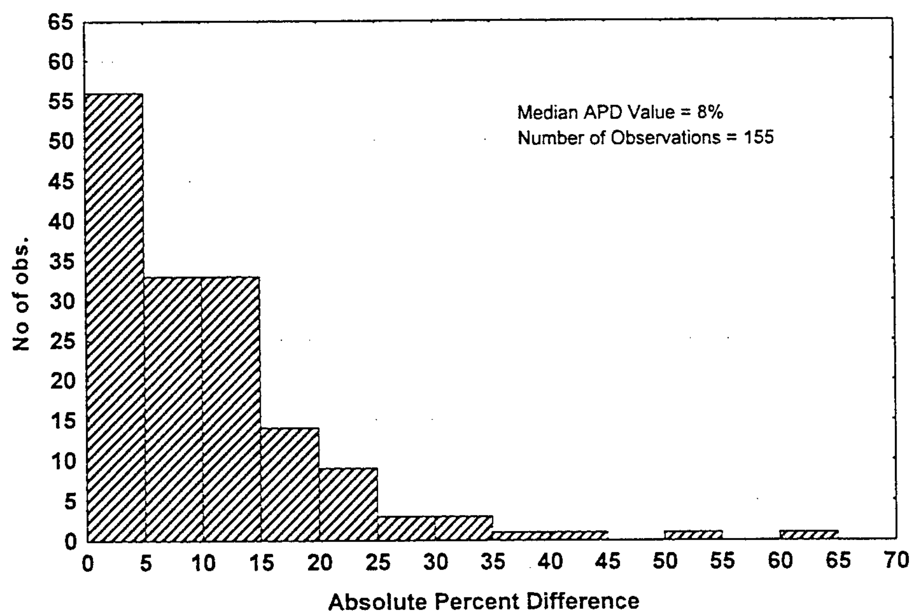

Figure 4. Frequency distribution of Absolute Percent Differences for the HAPSITE portable GC/MS

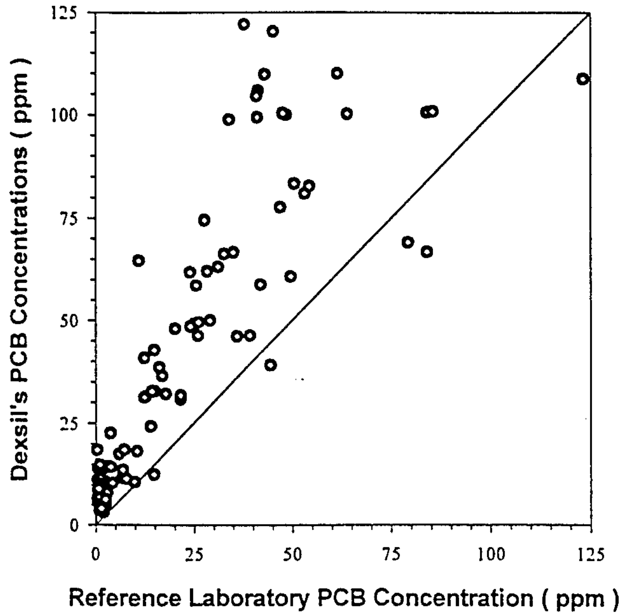

Figure 5. Scatter plot of laboratory and field technology PCB results on replicate sample splits

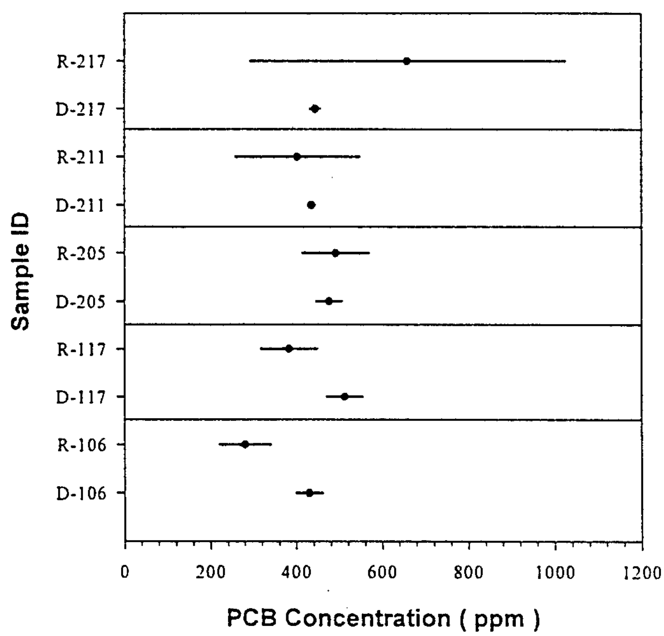

Figure 6. Comparison of laboratory (R) and field technology (D) mean and 95% confidence intervals for replicate sample splits.

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