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Superfund Innovative Technology Evaluation (SITE) Program Evaluation Report for ANTOX BTX Water Screen (BTX IMMUNOASSAY)

SITE

SUPERFUND INNOVATIVE
TECHNOLOGY EVALUATION

EPA/540/R-93/518
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SUPERFUND INNOVATIVE TECHNOLOGY EVALUATION (SITE) PROGRAM EVALUATION REPORT FOR ANTOX BTX WATER SCREEN (BTX IMMUNOASSAY)

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ABSTRACT

The results of a demonstration of a portable immunoassay for the detection of benzene, toluene, and xylene(s) (BTX) are described in this report. The BTX immunoassay was developed by Antox, Inc., (South Portland, ME) and is intended as a screening technology, with a claimed detection level of 25 ppb BTX. This demonstration was conducted under the Superfund Innovative Technology Evaluation (SITE) Program, under which measurement and monitoring technologies of interest to the U.S. Environmental Protection Agency are evaluated.

The demonstration was designed to investigate the ability of the immunoassay to perform as a portable, on-site screening method for BTX contaminated ground water samples. Samples from monitoring well fields at four sites in the Las Vegas valley provided a range of concentration levels for gasoline contaminated ground water. Sample splits were analyzed on-site by the BTX immunoassay and in the laboratory by gas chromatography (GC). The confirmatory GC analysis followed the procedures in EPA Method 8020.

The BTX immunoassay was rapid and simple to use, and it performed well in identifying high level contamination and gasoline contaminated samples having BTX concentrations greater than 100 ppb. The results from duplicate immunoassay analysis of 79 field samples were consistent with GC results for most concentrations. Only two samples were found to generate consistent false negative immunoassay results (each sample had BTX concentrations between 25 and 100 ppb). Two samples also gave consistent false positive immunoassay results. However, all false positive immunoassay results were on samples having distinct low-level gasoline signatures in their chromatographs. Both field sample and various performance evaluation sample results showed a significant tendency to generate false negative results in the 25 to 100 ppb BTX region. A more sensitive formulation of the immunoassay may need to be developed if the immunoassay is to be used for critical decisions involving samples with these levels of BTX.

This evaluation is submitted in partial fulfillment of Contract Number 68-CO-0049 by Lockheed Engineering & Sciences Company under the sponsorship of the EPA.

TABLE OF CONTENTS

Section	page
Notice	ii
Abstract	iii
List of Figures	vi
List of Tables	vi
Abbreviations and Acronyms	vii
Acknowledgments	viii
Executive Summary	ix
1. Introduction	1
Overview of the Immunoassay Program	1
Overview of the SITE Program	3
Overview of the BTX SITE Demonstration	3
Project Organization	4
2. Descriptions of Technologies	6
BTX Immunoassay	6
Gas Chromatography Analysis	6
Gas Chromatography/Mass Spectrometry Analysis	9
3. BTX Immunoassay Demonstration Design	10
Predemonstration Testing and Planning	10
Cross Reactivity	10
Colorimeter Calibration	10
Variability of Optical Density Readings	11
Study Design for the SITE Demonstration and Evaluation	13
Site Selection	13
Sample Collection Procedures	13
Quality Assurance Design	14
Data Quality Objectives	14
QA Problems and Resolutions	15
Changes to the QA Plan	15
4. BTX Demonstration Results and Discussion	17
Gas Chromatography Results	17
Field BTX Immunoassay Versus GC	17

False Negatives 20
False Positives 20
Variability of BTX Immunoassay Field Analysis Results 20
Quality Assurance and Quality Control Sample Results 22
An Assessment in Terms of Five Data Quality Elements 23
Results of the On-Site Audit 27
Additional QA/QC Observations and Conclusions 27

5. Conclusions and Recommendations 29
Conclusions 29
Advantages of the BTX Immunoassay 29
Limitations of the BTX Immunoassay 29
Minimizing False Negative and False Positive Results 30
Limitations of this Demonstration 30
Recommendations 31
Recommendations Specific to the BTX Demonstration 31
Recommendations for Future Studies 31

References 32

Appendices

Appendix A -- A Description of the BTX Immunoassay Procedure 34

Appendix B -- Procedural Details and Quality Assurance Summary for Gas Chromatography and Purge-and-Trap Methods 41

Appendix C -- Modified Procedure for Preparation of Trip Audit/Performance Audit Samples 63

Appendix D -- Field Audit Report 66

Appendix E -- Participating Personnel 70

Appendix F -- Data Tables for Field Immunoassay, Laboratory Immunoassay, and Gas Chromatography Analyses 73

LIST OF FIGURES

Figure — Page

1. Organizational structure for the SITE Program demonstration of the BTX immunoassay. . . . 5
2. BTX immunoassay test kit and field test equipment. 7
3. BTX immunoassay test procedure. 8
4. Colorimeter versus spectrophotometer comparison. 12
5. Gas chromatographs for typical field samples. 18
6. First replicate immunoassay S/R vs GC BTX concentration. 19
7. Second replicate immunoassay S/R vs GC BTX concentration. 19
8. Replicate vs original S/R ratio. 21
9. BTX immunoassay results for field performance evaluation samples. 26
10. Reference cuvette optical density vs time. 28

LIST OF TABLES

Table — page

1. BTX immunoassay cross-reactivity data 11
2. Cuvette variability results 13
3. GC results for field prepared QA samples. 23
4. Laboratory BTX immunoassay results of performance evaluation samples. 23

ABBREVIATIONS AND ACRONYMS

BTX	benzene, toluene, xylene
BTEX	benzene, toluene, ethylbenzene, xylene
CECS	Converse Environmental Consultants Southwest
ELISA	enzyme-linked immunosorbent assay
EMSL-LV	Environmental Monitoring Systems Laboratory - Las Vegas
EPA	Environmental Protection Agency
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
HP	Hewlett-Packard
HRP	horseradish peroxidase
MCL	maximum contaminant level
MCLG	maximum contaminant level goal
MDL	method detection limit
MMTP	Monitoring and Measurement Technologies Program
MTBE	methyl t-butyl ether
OD	optical density
PEM	personal exposure monitor
PID	photoionization detector
ppb	parts per billion
ppm	parts per million
QA	quality assurance
QC	quality control
SARA	Superfund Amendments and Reauthorization Act
SITE	Superfund Innovative Technology Evaluation Program
S/R	sample to reference
TMB	tetramethylbenzidine
VOA	volatile organic analysis
UV/vis	ultraviolet/visible

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EXECUTIVE SUMMARY

This evaluation report presents the results of a demonstration designed to assess the performance of a field-portable immunoassay. The immunoassay was developed by Antox, Inc. (South Portland, ME) for the analysis of benzene, toluene, and xylene(s) (BTX) in environmental water samples. The demonstration was conducted under the Monitoring and Measurement Technologies Program of the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) Program. The demonstration was performed under the guidance of the EPA Environmental Monitoring Systems Laboratory in Las Vegas, Nevada (EMSL-LV).

The demonstration took place between January and April, 1992 at four sites in the Las Vegas valley. Each site contained a well field established to monitor groundwater contaminated with gasoline. Groundwater contamination from leaking underground storage tanks, pipelines, or other sources is a significant problem of EPA's interest. The EPA and various state and local agencies are interested in preventing, detecting, and correcting problems of gasoline contamination in ground water.

The BTX immunoassay demonstration involved the assay of 79 field samples. These samples were analyzed on-site in duplicate, and sample splits were analyzed in the laboratory using gas chromatography (GC) as the confirmatory method. GC analysis following EPA Method 8020 produced quantitative estimates for benzene, toluene, and xylene. Quality control of the GC analysis was maintained by following the requirements and meeting the specifications in the method description. In addition, a variety of quality control and performance evaluation samples were included in the study design. Selected samples from each site were also analyzed by gas chromatography/mass spectrometry (GC/MS) to evaluate sample components unidentified by the GC analysis.

Immunoassays are analytical techniques based on protein molecules (antibodies). The binding of a specific antibody to its target analyte can be used to quantitatively or qualitatively determine the extent of contamination in environmental samples. Specific antibodies can be developed to detect single analytes or groups of compounds. The Antox BTX Immunoassay is a competitive enzyme-linked immunosorbent assay (ELISA), and is meant to provide a qualitative estimate of the concentration of several small aromatic compounds in environmental water samples.

The test is rapid and easy to use. It takes thirty minutes to analyze a group of up to four samples. The test is performed by placing reference and test solutions in separate antibody-coated polystyrene cuvettes in which competition for binding to the antibody occurs. After a 10 minute incubation step, the cuvettes are washed to remove unbound enzyme conjugate. Substrate and chromogen are then added to the cuvettes, and a colored reaction product is formed during a second incubation period. The enzymatic reaction is stopped by the addition of a few drops of 1 N sulfuric acid. The color intensity of the enzymatic product is inversely proportional to the BTX hydrocarbon level in the test sample. The optical density (OD) of the reference and sample cuvettes is read on a portable digital colorimeter, and the ratio of sample to reference OD values is used to estimate whether the aromatic hydrocarbon levels are above or below 25 ppb BTX. Ratios

below 0.85 are associated with high BTX concentrations and ratios above 0.85 are associated with low BTX concentrations.

Results from the GC analysis gave 36 samples with BTX concentrations above 25 ppb BTX and 43 samples with concentrations below 25 ppb BTX. Using the immunoassay, two samples in the range from 25 to 100 ppb BTX were consistently misidentified as false negatives and two samples with concentrations below 25 ppb were consistently found to be false positives. Interestingly, all false positive immunoassay results were associated with samples having low-level gasoline contamination.

An evaluation of sample to reference (S/R) OD ratios for repeated analysis of performance evaluation samples having concentrations of 2.5, 25, 40, and 100 ppb BTX showed that the BTX immunoassay performed relatively poorly in the region from 25 to 100 ppb BTX. Most of the 25 and 40 ppb performance samples were classified as below 25 ppb by the immunoassay. The variability of the S/R OD ratio in this region leads one to expect a relatively high number of false negatives in this concentration range. GC analysis of sample splits from these performance samples confirmed that they were correctly prepared and did not represent loss due to improper dilution or volatilization.

The BTX immunoassay was simple to perform, and, for the most part, provided results consistent with those from later GC analysis. If critical decisions are dependent on analysis of samples in the range from 25 to 100 ppb BTX, then a more sensitive formulation of the test should be pursued. However, for certain applications, such as mapping the distribution of contaminants at a site or monitoring changes in concentration over time, the current level of sensitivity would be adequate.

SECTION 1

INTRODUCTION

The performance of an immunoassay field kit technique was evaluated during a U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) demonstration. This semi-quantitative method was developed by Antox Inc. (South Portland, ME), to detect benzene, toluene, and xylene contaminant levels in environmental water samples. The evaluation compared results from BTX immunoassays performed in the field to quantitative results obtained by gas chromatographic analysis using EPA Method 8020 (U.S. EPA, 1986a).

There are several million underground storage tanks (USTs) in the United States that contain petroleum or hazardous chemicals. The EPA has estimated that approximately 300,000 suspected releases will be identified at UST facilities over the next five years. Leaking USTs can cause fires or explosions and can contaminate ground water, which is a primary source of drinking water. In the interest of public health and safety, the EPA and various state and local agencies have issued regulations aimed at preventing, detecting, and correcting problems associated with leaking USTs (U.S. EPA, 1990a). The EPA has set regulatory standards for toluene, xylene, and benzene under the Safe Drinking Water Act (U.S. EPA, 1990b). For toluene and xylene, the proposed National Primary Drinking Water maximum contaminant levels (MCL) are 1 and 10 parts per million (ppm) respectively, and the proposed National Secondary Drinking Water maximum contaminant level goals (MCLG) are 0.04 and 0.02 ppm, respectively. The National Primary Drinking Water MCL for benzene is 0.005 ppm and the MCLG is 0 ppm.

Many government agencies require that laboratory analyses be performed on soil and water samples collected during tank closures, from suspected leaking UST sites, or to measure progress of corrective action. The cost and delays in decision making associated with laboratory analyses has prompted a search for field monitoring methods that are less expensive and are capable of providing rapid, on-site results. In many situations, real-time results facilitate better protection of public health through quicker corrective action decisions (Midwest Research Institute, 1990). An immunoassay test for BTX has the potential for use as a quick and inexpensive screening method for the detection of gasoline components in ground and surface water.

This document includes descriptions of the BTX immunoassay method and the theory behind it (Section 2), the design of the SITE demonstration and QA plans (Section 3), and an evaluation of the results compared to a standard EPA method (Section 4). Conclusions and recommendations for the BTX immunoassay are discussed in Section 5.

OVERVIEW OF THE IMMUNOASSAY PROGRAM

The immunochemistry program at the Environmental Monitoring Systems Laboratory, Las Vegas (EMSL-LV) consists of the following major components: identification of need for an immunochemical method, identification of existing technologies, development of new technologies,

adaptations and evaluations of existing technologies, field demonstration of portable technologies, and finally, technology transfer.

Immunoassays are increasingly being recognized as cost-effective and expedient alternatives to chromatographic and spectroscopic procedures for use in large-scale environmental monitoring studies. Immunoassay techniques have been applied to the analysis of many hazardous substances and possess several attributes that make them suitable for field screening methods. In general, immunoassays have proven to be sensitive, selective, precise, rapid, cost-effective, and applicable to a wide range of contaminants. A variety of immunoassay methods and formats can be applied to environmental analysis problems.

Once an immunoassay is presented to the EPA it may undergo an evaluation. Evaluation studies are guided by the parameters of the individual immunoassay system. If an assay is not well developed or characterized by the developer, the EPA may undertake a preliminary evaluation. However, the assay must be for a compound of high priority to the EPA and there must be a well-defined need for the method. Results from the preliminary studies are made known to the developer. The developer can use this information to base further assay development to eventually provide an assay suitable for EPA's use. Assays that are mature are evaluated in more detail than those assays still in the developmental stage. If a field-portable immunoassay system has successfully undergone a laboratory evaluation, the technology is demonstrated in the field. Such demonstrations are conducted under the SITE provisions of the Superfund Amendment and Reauthorization Act of 1986 (SARA).

The majority of the immunochemistry program at the EMSL-LV is currently directed towards the development and evaluation of specific immunoassay technologies. However, as needs have been identified, the program is expanding into other areas, including: immunoaffinity personal exposure monitors (PEMs), immunoaffinity chromatography sample preparations, fiber optic immunosensors (biosensors), immunoassays for biological indicators (biomarkers) of exposure, and the more effective development of specific antibodies.

The overall goal of the immunochemistry program is to provide quality assured methods to augment the agency's monitoring capabilities. The objectives of the program are listed below:

- Provide standardized immunoassays with proper quality assurance and standard operating procedures.
- Identify existing technologies that can be applied off-the-shelf or with minimal modification.
- Identify monitoring needs that cannot be adequately addressed with current technologies; and initiate a methods development program.
- Convert existing technologies into usable monitoring and site characterization methods.
- Demonstrate those technologies that successfully emerge from the development phases of the program.
- Prepare standard operating protocols and guidelines for the implementation of methods.

- Transfer the new technology to the user community and encourage the commercialization of resulting products.

- Foster the development of a respected Center of Excellence for Immunochemistry - both within and outside of the EPA.

The EPA Environmental Monitoring Systems Laboratory at Las Vegas, Nevada (EMSL-LV), is responsible for developing and evaluating immunoassays for specific environmental applications. According to EPA guidelines for methods evaluations, this process requires the determination of performance parameters such as precision, between method bias, method detection limits, interferences, and ruggedness of the method.

To be effective as rapid screening tools, immunoassays must provide timely, cost-effective results that complement conventional analytical methods. They must be capable of measuring the target analyte with sufficient accuracy and precision to identify that samples are clearly above or below a critical concentration range.

OVERVIEW OF THE SITE PROGRAM

Under SARA, site monitoring and characterization activities are expected to increase. As a direct result, the costs associated with collecting, transporting, and analyzing environmental samples will escalate. Field analysis can significantly reduce the time and cost associated with sample collection and analysis by providing timely information about the presence or absence of environmental pollutants. The EPA established the SITE program to accelerate the development, demonstration, and use of alternative and innovative technologies at Superfund sites. The Monitoring and Measurement Technologies Program (MMTP) was created as a complementary component of the SITE program to provide rapid, low-cost field methods to support hazardous waste site monitoring and characterization activities. An overview of the MMTP is given by Koglin (1990).

The SITE program addresses two broad categories of innovative technologies: (1) monitoring and measurement technologies, rugged enough for use under field conditions, for analysis of environmental pollutants of interest in site characterization; and (2) alternative treatment technologies used in remediation at Superfund sites. The three main objectives of the MMTP element of the SITE program are to select appropriate technologies for inclusion in the program, to provide the mechanisms for demonstrating each selected technology, and to ensure that the demonstrated technology is evaluated properly.

OVERVIEW OF THE BTX SITE DEMONSTRATION

The main purpose of this demonstration was to evaluate the BTX immunoassay technology for on-site detection of benzene, toluene, and xylene (as a group) under field conditions. Attention was focused on the BTX immunoassay's performance under ambient environmental conditions, but laboratory tests were also a significant component of the study. This demonstration was designed to assess whether the technology is capable of yielding rapid, accurate, and cost-effective analysis of the above pollutants in aqueous samples.

The demonstration took place from January to April 1992, at four sites in the Las Vegas valley, NV. Each of these sites was known to have ground water contaminated with one or more hydrocarbon fuels, usually gasoline. Each site also had an existing field of sampling wells, which

provided an opportunity to generate numerous independent samples having different concentrations of contaminants. Some of the wells were expected to monitor ground water outside the contaminant plume, which provided samples with contaminant concentrations well below the detection level of the BTX immunoassay. Thus, the sample sites represented a variety of sample sources. Since the laboratory performing the comparative analysis was also within the Las Vegas valley, effects due to sample handling and shipping were minimal. And finally, logistical costs were minimized with this design.

PROJECT ORGANIZATION

The success of the immunoassay evaluation depended on the combined efforts of three primary organizations: EMSL-LV, Antox Inc., and Converse Environmental Consultants Southwest (CECS). The organizational structure for this study is shown in Figure 1.

The responsibilities of EMSL-LV, with assistance from its prime contractor, Lockheed, include:

- Designing, overseeing, and ensuring the implementation of the elements of the demonstration and quality assurance (QA) plan.
- Performing on-site BTX immunoassay analysis.
- Performing off-site analysis by GC.
- For selected samples, performing off-site analysis with the BTX immunoassay and GC/MS.
- Preparing and distributing quality assurance (QA) and quality control (QC) samples.
- Evaluating and reporting on the performance of the technologies.

Antox Inc., the developer of the immunoassay, was responsible for:

- Performing preliminary testing to assess immunoassay performance.
- Supplying a sufficient number of field kits to perform the analysis required to conduct the demonstration.
- Providing technical assistance.

CECS was responsible for:

- Site access and for providing sample splits from each well.

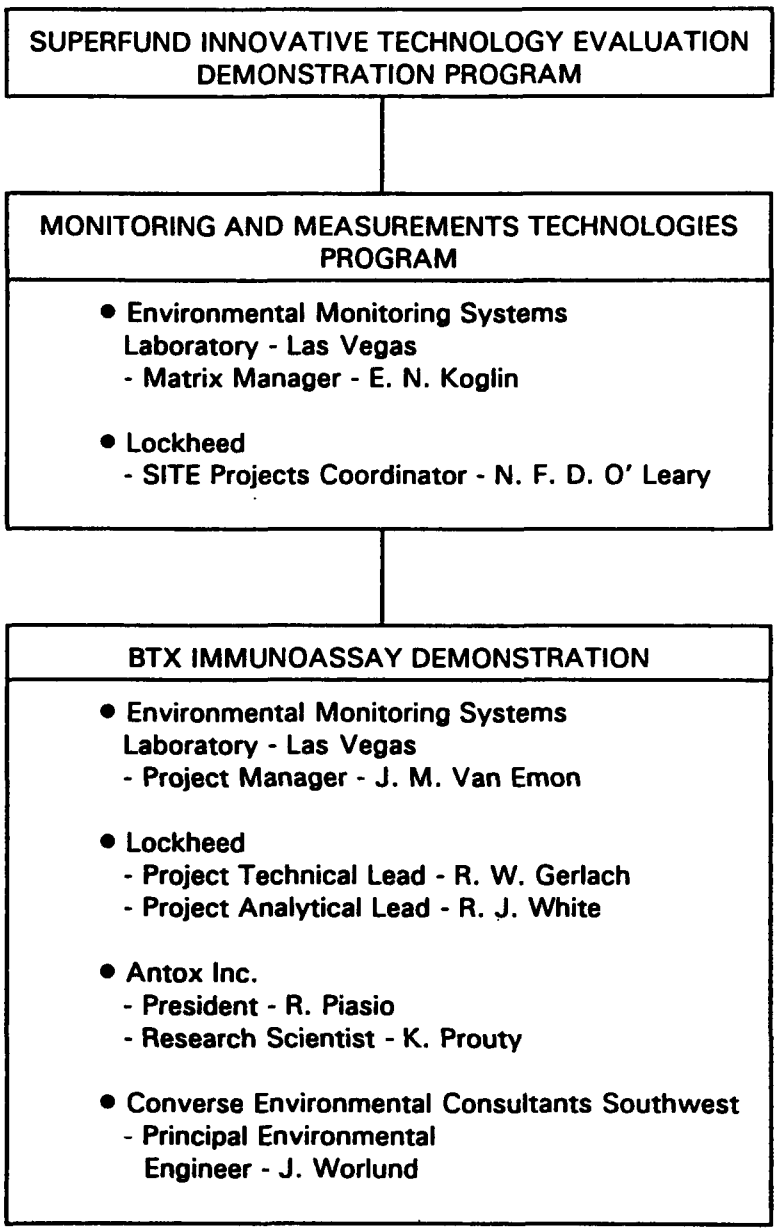

Figure 1. Organizational structure for the SITE Program demonstration of the BTX immunoassay.

SECTION 2

DESCRIPTIONS OF TECHNOLOGIES

A brief description of the immunoassay technology and of the confirmatory laboratory analysis methods is presented in this section. Figure 2 shows the complete BTX immunoassay kit supplied by Antox and various ancillary equipment needed to utilize the kit. The kit is a portable, self-contained package containing all reagents and supplies required for field use. Confirmatory GC and GC/MS laboratory analysis were traditional laboratory techniques, using approved EPA laboratory methods (EPA Methods 8020 and 8240, U.S. EPA, 1986a,b).

BTX IMMUNOASSAY

The BTX immunoassay is a competitive enzyme-linked immunosorbent assay (ELISA), which uses an antibody-coated polystyrene cuvette as the solid phase. The immunoassay uses horseradish peroxidase (HRP) as the enzyme marker, hydrogen peroxide as the substrate, and tetramethylbenzidine (TMB) as a chromogen. The rabbit polyclonal antibody binds specifically to the BTX or other closely associated aromatic hydrocarbons. A hapten-enzyme conjugate mimics the free analyte (BTX compounds) and competes with aromatic compounds in the sample for binding sites on the immobilized antibody.

The test is performed by placing reference and test solutions in separate coated cuvettes in which competition for binding to the antibody occurs. Figure 3 is a graphical presentation of the individual steps involved in the test. After a 10 minute incubation step, the cuvettes are washed to remove unbound enzyme conjugate. Substrate and chromogen are then added to the cuvettes and a colored enzymatic reaction product is formed during a second incubation period. The enzymatic reaction is stopped by the addition of a few drops of 1 N sulfuric acid. A complete description of the method is given in Appendix A.

The color intensity of the enzymatic product is inversely proportional to the BTX hydrocarbon level in the test sample. If the level of BTX hydrocarbon in the sample is high, most of the antibody binding sites will be occupied with the sample analyte, thus preventing the hapten-enzyme conjugate from binding. For less concentrated samples, more of the antibody binding sites are occupied with the hapten-enzyme conjugate, resulting in the development of more intense color in the cuvette. The optical density (OD) of the colored enzymatic product in the reference and sample cuvettes is read on a portable digital colorimeter equipped with a filter which will pass light at 450 nm (near the wavelength of peak absorption for the colored reaction product). The ratio of sample to reference OD values is used to estimate whether the aromatic hydrocarbon level in the sample is above or below 25 parts per billion (ppb).

GAS CHROMATOGRAPHY ANALYSIS

Gas chromatography analysis following the general guidelines of EPA Method 8020 (U.S. EPA, 1986a) was used as the primary confirmatory method. One modification was the use of a 30

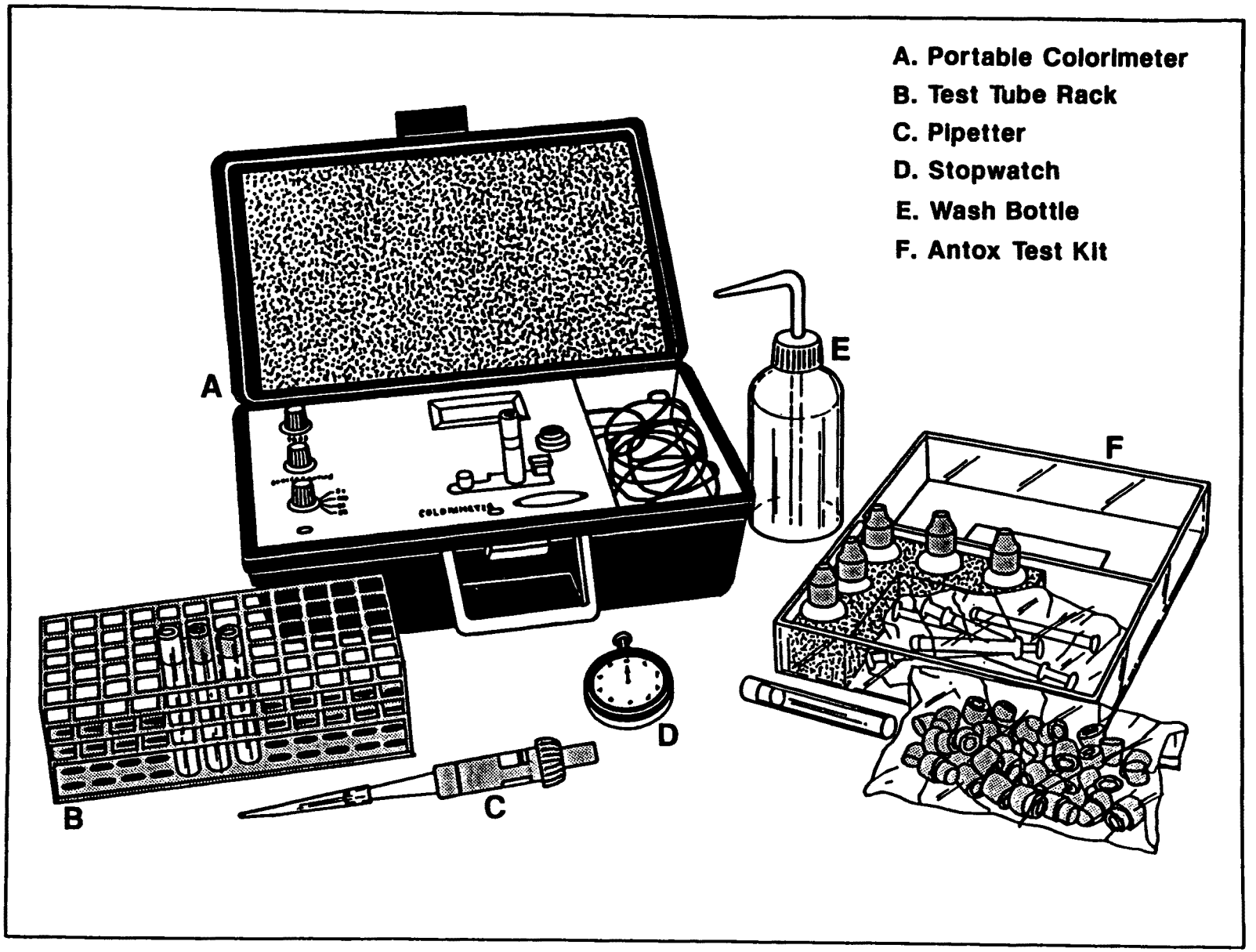

Figure 2. BTX immunoassay test kit and field test equipment.

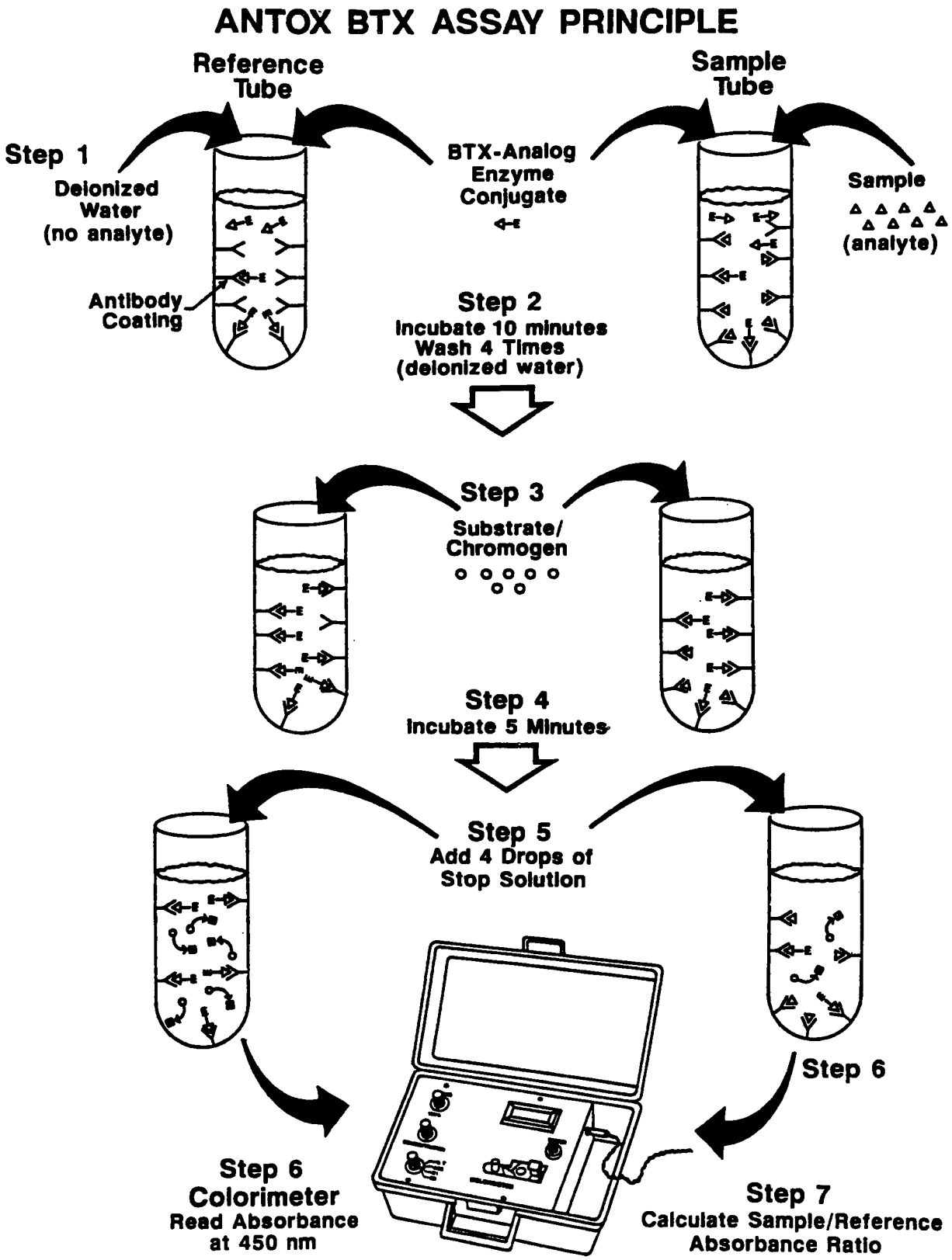

Figure 3. BTX immunoassay test procedure.

m J&W DB-624 chromatography column. Purge-and-trap sample preparation followed EPA Method 5030 (U.S. EPA, 1986c). Details of the gas chromatography and purge-and-trap procedures are given in Appendix B.

GAS CHROMATOGRAPHY/MASS SPECTROMETRY ANALYSIS

Selected samples from each site were subjected to GC/MS analysis following the general procedure given in EPA Method 8240 (U.S. EPA, 1986b). These analyses were carried out to help identify unassigned peaks from the GC analysis. Cross-reacting compounds may cause high immunoassay results compared to what is expected based on GC detection for only benzene, toluene, and xylene(s).

SECTION 3

BTX IMMUNOASSAY DEMONSTRATION DESIGN

PREDEMONSTRATION TESTING AND PLANNING

Preliminary testing of the BTX immunoassay was conducted prior to starting the field sampling. This included testing a variety of compounds and mixtures for cross-reactivity, checking the performance of the colorimeter, and determining the variability associated with reading the optical density using different cuvettes.

Cross Reactivity

Table 1 summarizes the cross reactivity data of the BTX immunoassay. Compounds are listed in descending order by strength of cross-reactivity. Percentage cross-reactivity is calculated relative to regular gasoline (leaded, from pump). These percentages are intended to give a rough estimate of the relative strength of cross-reactivity. Since these values are calculated from a single point and based on the simplifying assumptions of approximate linearity and parallelism of the standard curves, they should be considered only as rough estimates. When S/R is greater than 1.0, the above assumptions are not valid, and the cross-reactivity is assumed to be negligible. Note that the antibody interacts most strongly with BTX hydrocarbons and closely related compounds which are substituted with nitro-, chloro-, and hydroxyl groups. It is also notable that the antibody tends to cross-react moderately with some chlorinated alkanes and alkenes (e.g., 1,4-dichlorobutane, *cis*-1,3-dichloropropene). The antibody does not cross-react appreciably with most of the alkanes. The difference in cross-reactivity between regular (leaded) gasoline, white gasoline, and kerosene may reflect the relative percentages of BTX compounds in each.

Colorimeter Calibration

Another predemonstration activity involved investigation of bias associated with the portable colorimeter. A set of serially diluted samples of N-2,4-dinitrophenylglycine in water were prepared and read in both the colorimeter and a Hewlett-Packard Model 8452A diode array UV/vis spectrophotometer, at 450 nm. Both instruments were zero-adjusted with cuvettes containing deionized water. The data, plotted in Figure 4, indicate low bias with respect to the spectrophotometer at high OD values. The non-uniform bias observed in Figure 4 might introduce systematic error in estimating the Sample OD/Reference OD ratio.

There will only be a small region where this bias might effect the classification of samples. Samples will be biased toward misclassification if the reference OD is high (near 2.0) and the sample OD is slightly smaller. In all other cases the bias will not affect the classification or it will enhance the probability of correct classification. If the bias at high OD readings (a bias of 0.73) is compared to the OD bias at 0.85 of a high OD reading (a bias of 0.79), we can estimate a modified S/R OD ratio. The modified ratio represents an alternate decision level for the BTX immunoassay.

Table 1. BTX immunoassay cross-reactivity data[a].

Compound(s)	S/R[b] ratio	Percent cross-reactivity[c]
BTEX[d](1:1:1:1 by volume)	0.18	172
BTX[e](1:1:1 by volume)	0.25	124
o-Cresol (2-Methylphenol)	0.27	115
Regular gasoline[f]	0.31	100
Chlorobenzene	0.34	91
Nitrobenzene	0.43	72
1,2-Dichlorobenzene	0.43	72
Phenol	0.52	60
White gasoline	0.53	58
1,4-Dichlorobutane	0.72	43
cis-1,3-Dichloropropene	0.77	40
2,4-Dinitrotoluene	0.79	41
1,2-Dichloroethane	1.03	0
Iso-octane	1.04	0
Ethylene glycol	1.06	0
Pentane	1.06	0
trans-1,2-Dichloroethylene	1.11	0
1,1-Dichloroethane	1.12	0
1,1,2-Trichloromethane	1.12	0
Kerosene	1.12	0
cis-1,2-Dichloroethylene	1.18	0
Methylene chloride	1.26	0

[a] All cross-reactivity results were determined at 100 ppm.
[b] S/R = Sample absorbance/Reference absorbance.
[c] Percentage cross-reactivity = 100 (0.31/compound S/R ratio). The cross-reactivity is assumed to be 0 when S/R is 1.0 or greater.
[d] Benzene/toluene/ethylbenzene/xylene.
[e] Benzene/toluene/xylene.
[f] Regular (leaded gas = 100%).

When the S/R ratio is 0.85, the bias of the S/R ratio is 0.73 / 0.79 = 0.92, from which an alternative decision level of 0.85 X 0.92 = 0.78 can be calculated. The results section will show that this change in the decision level will only slightly affect the field sample analyses results.

Variability of Optical Density Readings

The background variability associated with different cuvettes was determined by measuring the OD for three sets of 10 cuvettes. One set was left empty, one set contained distilled water, and one set contained a solution of N-2,4-dinitrophenylglycine. The volume of solution was equal to that expected when using the standard immunoassay procedure. A summary of results is given in Table 2. The standard deviations are small, ranging from 0.004 to 0.014 OD units. All colorimeter readings were repeated after re-orienting the cuvettes in the reader slots. No practical difference in mean or standard deviation was observed compared to the initial readings. However, it was observed that repeated use of a plastic cuvette with the colorimeter might lead to the

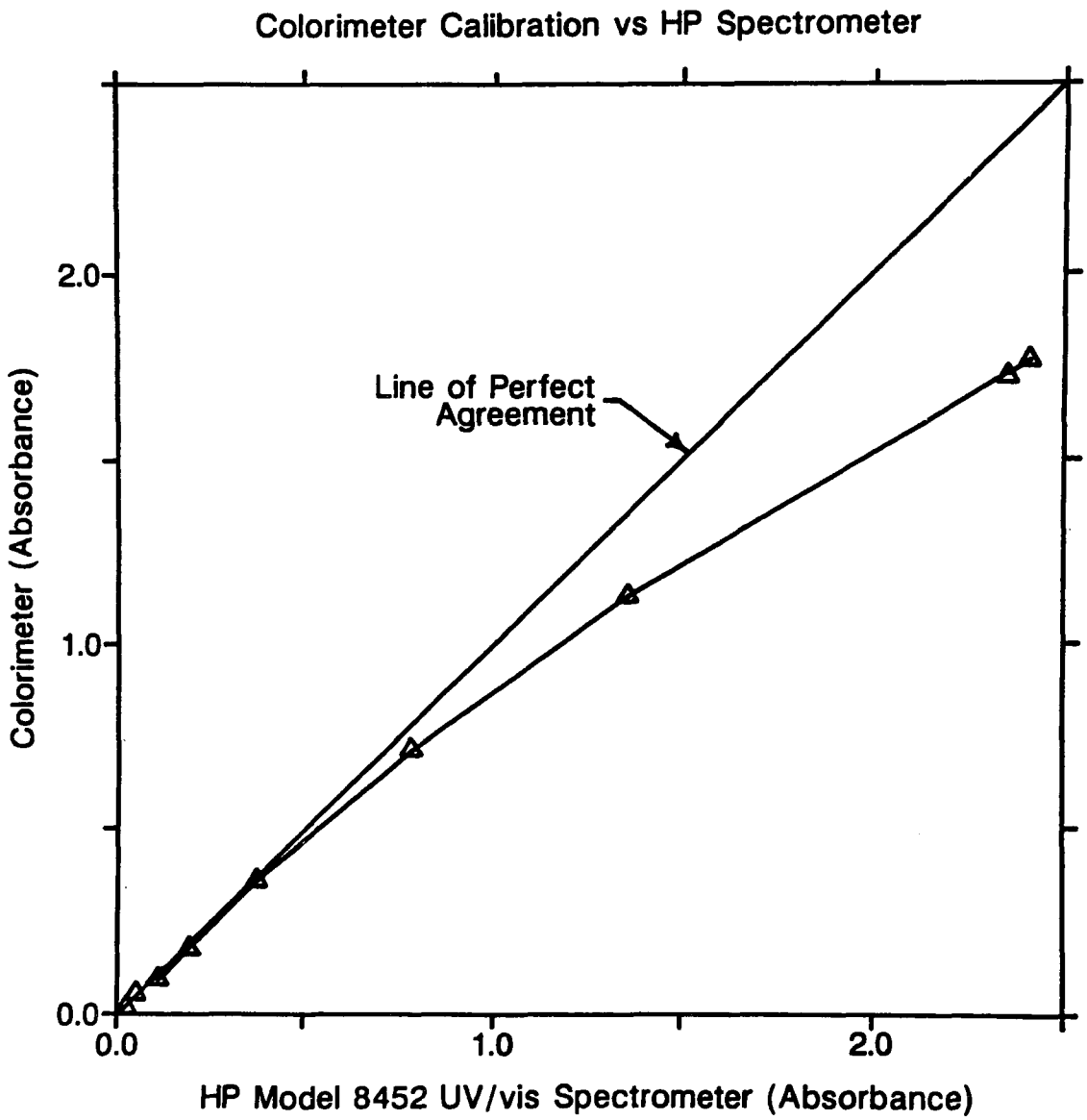

Figure 4. Colorimeter versus spectrophotometer comparison.

Table 2. Cuvette variability results.

Solution	n	Mean (OD units)	Standard Deviation (OD units)
Empty	10	0.044	0.004
Deionized water	10	-0.001	0.007
N-2,4-dinitrophenylglycine	10	0.68	0.014

development of scratches, which could affect the OD reading. This suggests that one should periodically discard cuvettes being repeatedly used to zero the colorimeter. To obtain the best performance, one should always inspect cuvettes for scratches or other blemishes before use.

STUDY DESIGN FOR THE SITE DEMONSTRATION AND EVALUATION

The primary focus of the study design was the evaluation of the accuracy, precision, and other key performance characteristics of the BTX immunoassay, under field conditions. Samples from a variety of locations around hydrocarbon plumes contaminating groundwater were included to provide representative data over a range of concentrations.

Site Selection

For this study an arrangement was negotiated with Converse Environmental Consultants Southwest (CECS) for sample acquisition from four different well fields located within the Las Vegas valley. Three of these sites represented gasoline sources and the fourth site had wells that might also be contaminated with other hydrocarbon mixtures, such as jet or diesel fuel. Each of these sites had well monitoring fields already in place, with several wells having no or very low levels of hydrocarbons present. The exact location of the sample wells is confidential due to an agreement with CECS, which controls access to the sites and represents the interests of site owners. The site owners are reluctant, for various reasons, to have their names divulged.

The four sites have been coded A, B, C, and D. Eleven wells were sampled at site A at three different times, during January 22-23, February 24-25, and April 15-16. Site B had 18 wells sampled on February 25-28. Site C, with 16 wells sampled, was visited on March 9-13. Site D had 12 wells sampled on April 17-22. A total of 79 well samples were obtained.

Sample Collection Procedures

All field samples were collected from pre-existing wells that had been previously developed to remove non-native materials from the well bore and penetrated aquifer in order to ensure that representative ground-water samples were obtained. Purging at sites A, B, and D involved the use of a purging pump. However, due to the 30-40 m depth to the water table at site C, dedicated sampling pumps were used for both purging and acquiring samples. Each well was purged of three well volumes of water prior to sampling to remove stagnant water from the well and surrounding aquifer. After purging, samples were taken by placing a clean, disposable 5.1-cm diameter teflon bailer slowly down the well. After slowly bringing the bailer to the surface, a clean Voss vial

pipette was placed on the bottom of the bailer. Clear 40-mL VOA (Volatile Organic Analysis) vials with teflon septums were filled from the bottom after triple rinsing each vial with formation water. VOA vial splits from a given well were analyzed on-site, or held at 4°C until analyzed at the laboratory. To maintain a record of sample collection, transfer, shipment, and receipt; a chain-of-custody form was filled out for each day's collection of samples. Sample tracking was accomplished by recording the sample site, well number, and date of sampling.

QUALITY ASSURANCE DESIGN

To ensure that the data resulting from the SITE demonstration are of known quality and that a sufficient number of critical measurements are taken, a quality assurance project plan (QAPjP) was prepared (Gerlach et al., 1992). The QAPjP for this demonstration describes procedures appropriate for a Category II project, as specified by EPA (U.S. EPA, 1987). Category II projects apply to measurement activities when the results are not directly used in regulatory, enforcement, legal, or policy matters, where data quality objectives may be semi-quantitative, and for which precision, accuracy, completeness, reproducibility, and representativeness may not be easily defined.

Data Quality Objectives

In order to make sound decisions about the BTX immunoassay field kit, the conclusions of the demonstration must be based on sound interpretations of the resulting data. To achieve this end, objectives were established for data quality based on the proposed end uses of the data (Stanley and Verner, 1985). The QA component of the demonstration was designed to maximize the probability that the resulting data would be suitable for its intended use. EPA has established data quality objectives (DQOs) for five indicators of data quality (Stanley and Verner, 1983): representativeness, completeness, comparability, bias, and precision. These data quality objectives are provided as guidelines. If the objectives were not met, it does not necessarily mean that the data would not meet the needs of the project.

Three analytical methods were used in this study: BTX immunoassay, GC, and GC/MS. The BTX immunoassay is the focus of the evaluation, and in this sense the entire project is a study of the performance characteristics of this method. Consideration of whether the BTX immunoassay performed well is based on a composite evaluation of all aspects of the study, including ease of use, evaluation of the protocol, performance with respect to audit (see Gerlach et al., 1992 and Section 4) and field samples, etc. On a more basic level, the study design required at least 60 environmental well samples to be analyzed on-site by the immunoassay. The study design also specified that at least 90 percent of the analysis should be free of identifiable errors associated with the immunoassay itself (for example, cuvettes which fail the manufacturer's QA/QC criteria).

Several types of quality assurance and quality control samples were used in this demonstration. Trip blanks and 40 and 100 ppb BTX trip audit samples were prepared in the field and transported with the field samples to check for contamination or loss from transportation or storage effects. Individual VOA vial splits of these samples were analyzed in the laboratory by GC and immunoassay. Performance audit samples were prepared at 2.5, 25, and 100 ppb BTX to aid in monitoring both immunoassay and GC results. These samples were prepared immediately prior to analysis. In the field, one set was run at the beginning and another set was prepared and run at the end of the day. Separate sets of performance audit samples were also prepared and run at the time of GC analysis and for laboratory immunoassay analysis. The above audit samples served to check and confirm the analysis results reported for field samples by both immunoassay and GC.

The most important DQOs are associated with the GC analysis, as this is the confirmatory method against which the BTX immunoassay results are compared. GC analysis was run in accordance with the requirements spelled out in EPA Method 8020 (U.S. EPA, 1986a). A standard curve was initially developed for each of four analytes; benzene, ethylbenzene, toluene, and *o*-xylene. Each standard curve was verified daily by injection of one or more working standards. The responses were checked to verify that they did not vary by more than ±15 percent. If so, a new calibration curve was established. Method 8020 states that it will generate estimates that represent greater than 90 percent recovery with relative interlaboratory standard deviations from 18 to 26 percent. These bias and variability estimates were considered adequate to evaluate the performance of the BTX immunoassay.

The GC/MS analysis was used primarily to identify GC peaks which might correspond to compounds that cross-react in the immunoassay. At least one positive sample (by immunoassay) from each of the sites (A, B, C, and D) was assayed by GC/MS. The samples were run following the protocol and requirements in EPA Method 8240 (U.S. EPA, 1986b).

Data quality objectives were established for the five usual data quality indicators; representativeness, completeness, comparability, bias, and precision, as well as for detection limit. These objectives are discussed in the relevant sub-sections of Section 4. Because their assessment required an analysis of both field and QA/QC sample data, it was not practical to generate an independent section of this report containing all DQO results. As has been stated previously, any study whose main function is a method evaluation is nothing more than an exercise devoted to the evaluation of quality assurance and quality control samples. In this sense field samples are merely another type of QA/QC sample.

QA Problems and Resolutions

The procedure for generating trip and performance audit samples outlined in the demonstration plan had some deficiencies. Due to the volatility of the target analytes, there was no way of determining how close the actual final concentrations were to the target values. In addition, some of the immunoassay results on splits of audit samples assayed directly after preparation and after several hours, indicated possible changes with time. For this reason, the procedure for preparing and analyzing the audit samples was modified to allow one of the splits of each audit sample to be analyzed by GC. In addition, a holding time study was conducted to check for changes to analyte concentration over time for audit samples stored in VOA vials in coolers. The section following entitled "Changes to the QA Plan" gives further details on these modifications.

Changes to the QA Plan

The procedure for generating trip audit samples and performance audit samples was modified between the February and March sampling events. Site A had been sampled once in January, and sites A and B had both been sampled in late February. There were several reasons for modifying the audit sample procedure. The BTX immunoassay results from the original protocol for the performance audit suggested there might be a time lag associated with generating the correct performance audit sample concentration. That is, the low level performance audit samples might not reflect an accurate concentration until after some period of time necessary to achieve equilibrium. In addition, there was concern about the comparability of field and laboratory results for performance evaluation samples because the initial plan involved preparing separate sets of standards for each location. With the original design, site-specific sample preparation effects might

be the source of bias between field and laboratory results. And finally, the field crew was having to spend too much of their time with the preparation of audit samples.

The plan was modified to have the trip audits made from an aliquot of the same solutions prepared for the performance audits. This meant that there were three positive trip audits; at 2.5, 25, and 100 ppb prepared from the toluene standard. Since these samples served as both trip audit and performance audit samples, they were assigned a new sample code, W1 for the 2.5 ppb, W2 for the 25 ppb, and W3 for the 100 ppb. In addition, splits of these same solutions were used as the performance audit samples analyzed at the beginning and end of each day. A detailed description of the modified audit sample preparation protocol is given in Appendix C.

A second modification was made to carry out an additional laboratory holding time study in order to determine if holding time had any effect on the BTX immunoassay results for performance evaluation samples with concentrations in the 25 to 100 ppb BTX range. This study was initiated because numerous incorrect assignments for the 25 ppb BTX samples were being made with the BTX immunoassay at the beginning of the evaluation. There was concern that the sample concentration was not stable at these levels. To address these concerns, several sample splits of 25 and 100 ppb toluene standards were prepared. These sample splits were run immediately after preparation and at several other times on the day of preparation and on the following day. There was no evidence of any change in response due to a change in sample composition. Instead, it appears from examination of the complete data set for the demonstration that the problems with the 25 ppb BTX standards resulted from the lower than expected sensitivity of the immunoassay.

SECTION 4

BTX DEMONSTRATION RESULTS AND DISCUSSION

Several sets of data were developed as part of the evaluation. The primary data set consists of duplicate field BTX immunoassay results for 79 groundwater samples and corresponding laboratory GC results for splits of these samples. Several secondary data sets were generated from different types of QA sample analyses. Sample types such as trip blanks, trip positives, and performance evaluation standards were generated to evaluate the analytical methods and to monitor procedures. A separate set of QA/QC data was generated for the GC method in order to ensure the confirmatory method was in control. The results from each of these types of data sets are presented and discussed in this section.

Gas Chromatography Results

Representative chromatographs for three different samples are displayed in Figure 5. The chromatograph on the bottom is for a typical high level sample, and was run at a five-fold dilution. Peaks for benzene, toluene, and xylene are easily distinguished in the first half of the chromatograph. Two internal standards, *cis*-1,2-dichloroethene and fluorobenzene, are also present on this chromatograph. A characteristic fingerprint associated with gasoline contaminated samples is present at the end of the run. The middle chromatograph is from an almost clean sample; barely detectable peaks can be discerned. The chromatograph on the top is thought to be an example from a weathered location. The compounds at the beginning of the chromatograph are barely detectable, perhaps they have evaporated since the gasoline came in contact with the groundwater. However, the higher boiling point constituents in the fingerprint region are still present at easily detectable levels. The BTX concentration estimates used in the following discussion are the sum of the analytical results for benzene, toluene, and xylene(s) from the GC analysis. Ethylbenzene was also quantitated by GC, and was expected to cross-react to some extent in the assay. However, it was found that adding the ethylbenzene component to the BTX value would not have changed the classification of any sample as positive or negative relative to the 25 ppb decision level. GC/MS results from selected field samples did not reveal any other compounds at levels which would affect the classification of samples by the BTX immunoassay.

Field BTX Immunoassay Versus GC

A summary of the relationship between the field BTX immunoassay results and the GC results is shown in figures 6 and 7, where the letters A, B, C, and D represent the sampling sites (see Section 3). Figure 6 uses the initial field BTX immunoassay result, and Figure 7 uses the duplicate field BTX immunoassay value. For samples where the OD reading exceeded the performance criteria of the colorimeter (recorded as >2) the S/R ratio was arbitrarily set to 1.5 so that these samples remained correctly classified but were distinguishable from the other sample results. In both figures the horizontal line is at the Sample OD / Reference OD (S/R) decision level of 0.85 and the vertical line is at 25 ppb BTX, the detection level claimed by the developer. One can see that

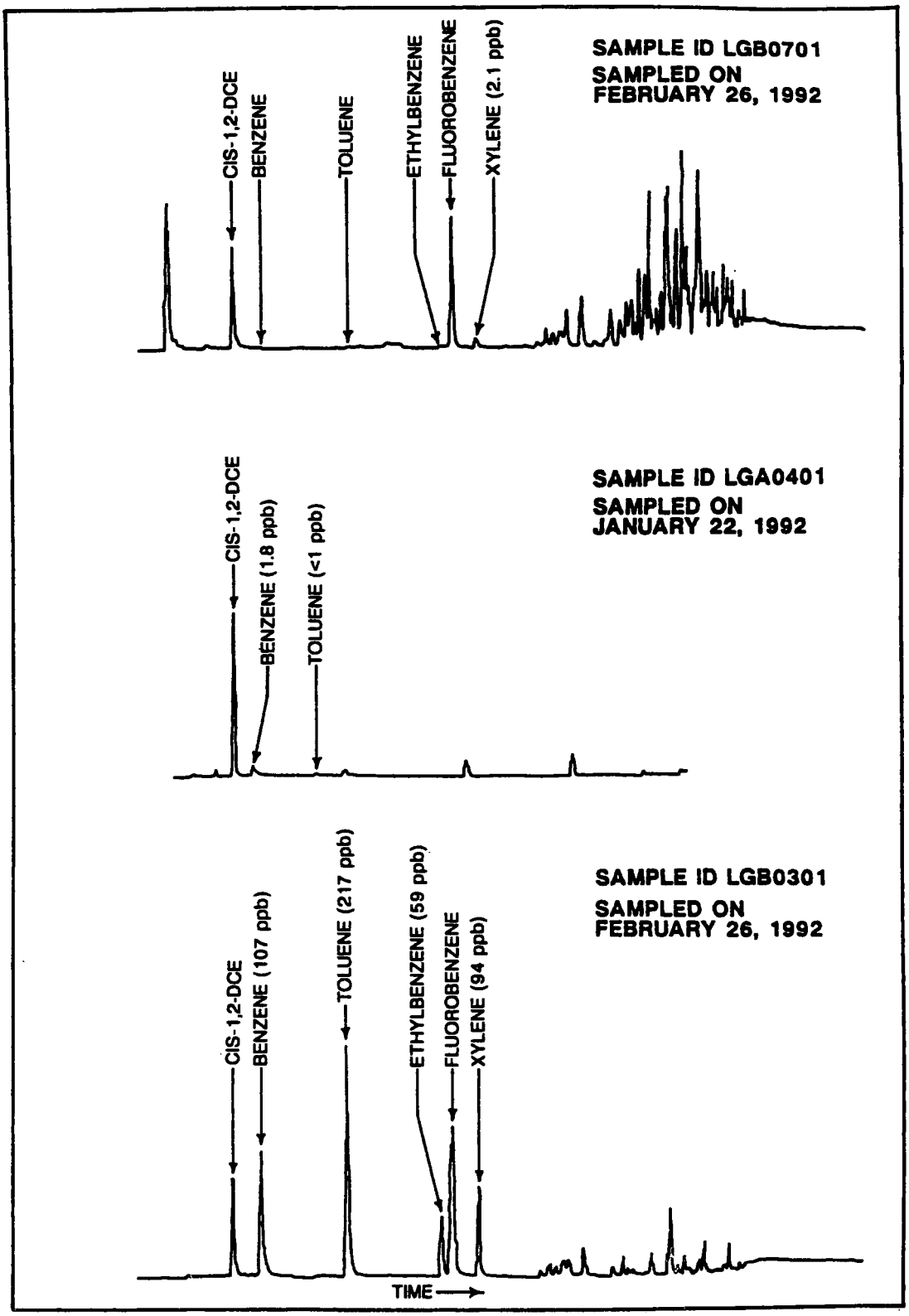

Figure 5. Gas chromatographs for typical field samples.

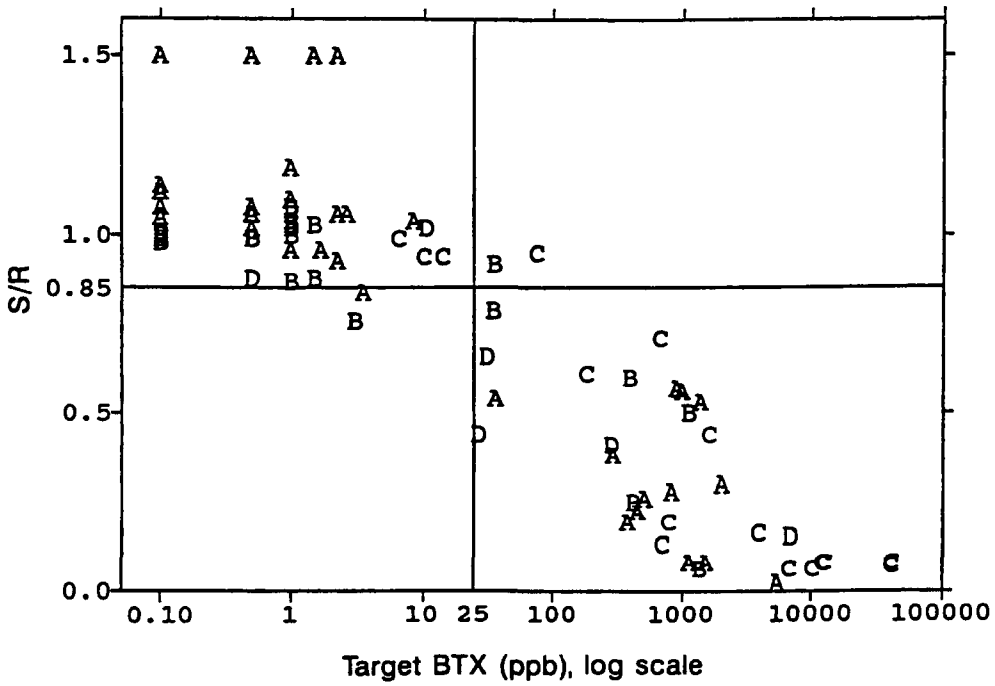

Figure 6. First replicate immunoassay S/R vs GC BTX concentration.

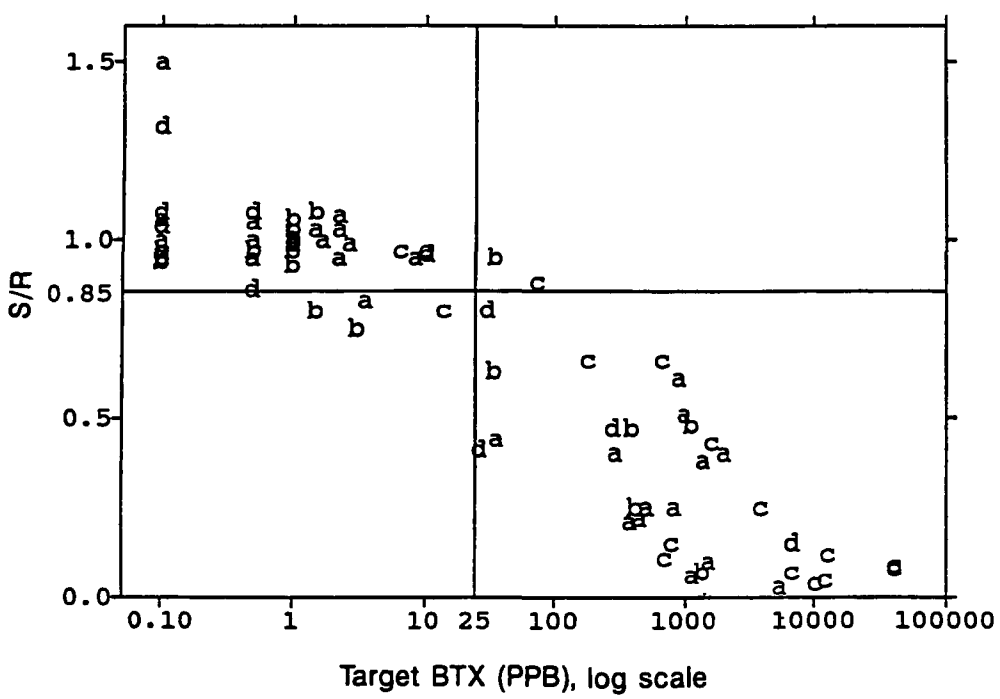

Figure 7. Second replicate immunoassay S/R vs GC BTX concentration.

both plots show similar relationships, the sigmoidal shape mirrors a typical immunoassay calibration curve. The data can be analyzed by considering the four quadrants separated by the 25 ppb BTX and 0.85 S/R lines. Samples in the upper left and lower right quadrants were correctly determined by the immunoassay with respect to the GC results. Samples in the upper right quadrant are false negatives, and samples in the lower left quadrant are false positives.

False Negatives

Of the 79 field samples, 36 samples were determined to be above 25 ppb BTX components by GC analysis. Two of these samples (6 percent) produced negative results on both replicate analysis with the BTX immunoassay. One sample had a BTX concentration of 35.5 ppb and the other a concentration of 74.8 ppb. If we include the ethylbenzene component, these estimates rise to 36 and 80.6 ppb respectively.

False Positives

Based on the BTX estimates from the GC analysis, 43 samples had concentrations below the 25 ppb criteria of the immunoassay. Overall, there was a seven percent false positive rate for these field samples (assuming replicates are independent), though only five percent of the positive samples were consistently classified incorrectly. Two of the 43 samples (five percent) were estimated as higher than 25 ppb by the initial field immunoassay determination, and four samples (including the two in the first determination), a nine percent rate, were estimated as above 25 ppb by the second immunoassay determination.

It is interesting to note that in each of the above six instances of a false positive by the BTX immunoassay, an examination of the associated gas chromatograph showed the characteristic fingerprint of gasoline contamination. An example of a chromatograph for these samples is given in Figure 5 for sample LGB0701, sampled on February 26, 1992. In this sense, the BTX immunoassay appears to be robust with respect to identifying gasoline contaminated samples. One example of a false positive occurred from a well that was sampled on three different months. The false positive was for the second sampling date, and the GC BTX results for these three dates were 0.1, 3.6, and 36.5 ppb, respectively. In this case, the use of the BTX immunoassay as a screening tool would have forwarded the second sample for further analysis and the identification of low level gasoline contamination might have provided useful information for monitoring the changing water quality of this well. However, because the contamination due to the gasoline fingerprint region is at so low a level, cross-reactivity cannot explain the false positive result in this instance. Loss during sample preparation would explain the discrepancy, but data from audit samples (see below) do not support this theory. Nevertheless, for other samples it is possible that higher molecular weight aromatic compounds (e.g. polyaromatic hydrocarbons or diethylbenzene) are causing a combined cross-reaction signaling the presence of gasoline.

Variability of BTX Immunoassay Field Analysis Results

A qualitative evaluation of the measurement variability associated with the BTX immunoassay can be obtained from the field replicate measurements. Figure 8 shows the replicate S/R measurement plotted against the original S/R value. The vertical and horizontal lines represent the 0.85 S/R decision level for the BTX immunoassay. Points in the lower left region represent samples with high BTX concentrations, and points in the upper right represent samples with low BTX concentrations. Samples which produced inconsistent results are located in the upper left or lower right

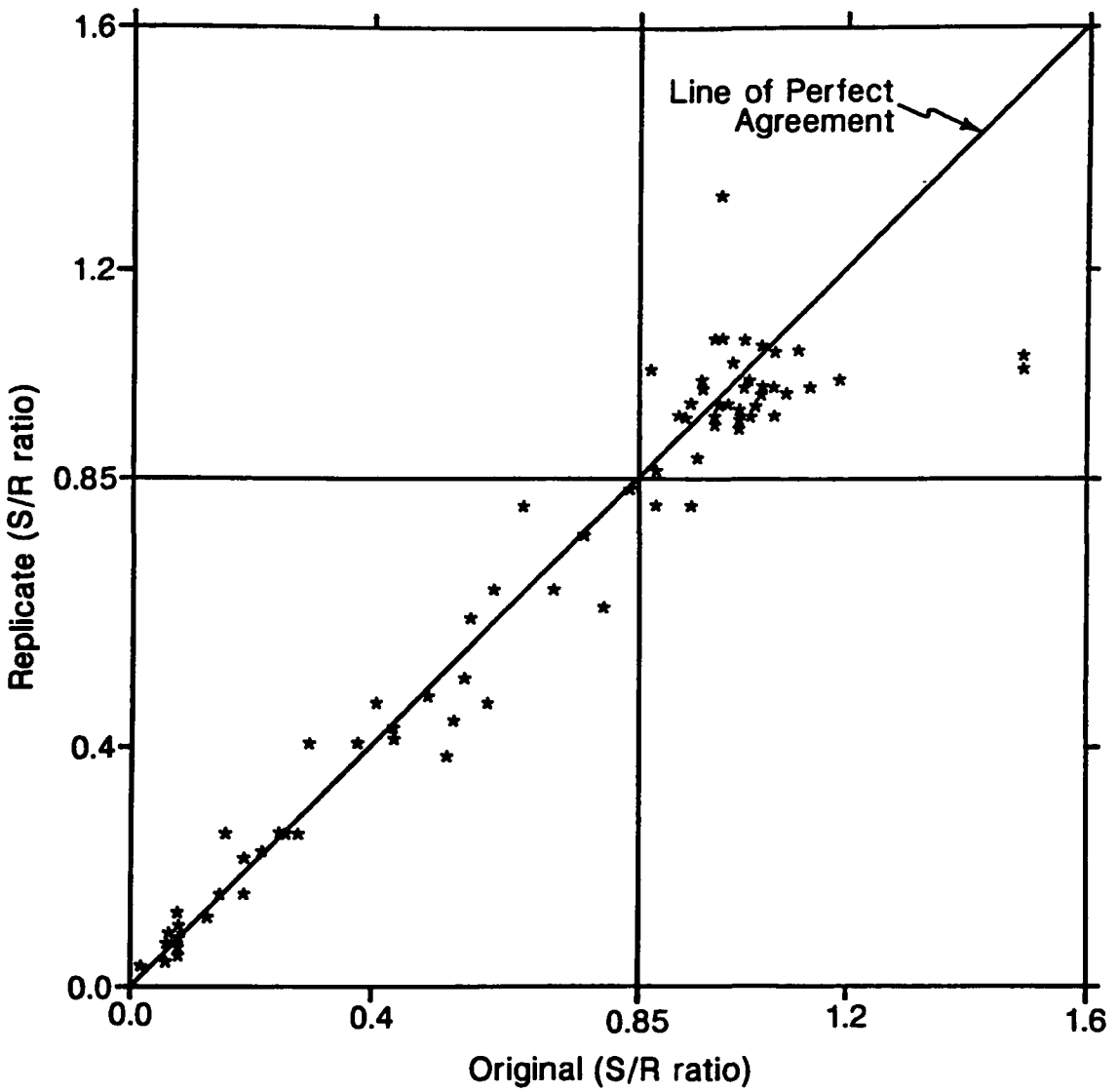

Figure 8. Replicate vs original S/R ratio.

quadrants. One can see that the variability about the line of perfect agreement is lowest for high level samples, and becomes larger as the BTX sample concentration becomes smaller. In agreement with the information in figures 6 and 7, one can also see that two samples lie in the off-diagonal quadrants. As a practical matter, we note that wherever the decision level is, a large distribution of samples near the decision level will increase the likelihood of getting an indeterminate result (for replicate analysis) or false positive and false negative results (for single analysis protocols).

QUALITY ASSURANCE AND QUALITY CONTROL SAMPLE RESULTS

Several types of QA samples were prepared in the field and transported back to the laboratory for analysis by GC. These included trip blanks, trip lows (40 ppb) and trip highs (100 ppb) during the initial portion of the evaluation, and three trip positives at 2.5, 25, and 100 ppb. All concentrations are in terms of toluene. Table 3 summarizes the results from these analyses, with the results grouped by their expected target concentration.

The mean and standard deviation obtained for the field prepared QA samples can be compared to those expected from a single analyst using EPA Method 8020 (U.S. EPA, 1986a). Except for a relatively high standard deviation for the 40 ppb samples, this study's results are relatively close to the single analyst expectations. The consistently higher variability is probably due to the fact that the single analyst expectations were determined from a round robin analysis of split samples made up at a single time while each individual sample in this study was produced independently. This suggests that a protocol for generating performance evaluation standards at the time of analysis is quite feasible. The results also show that sample integrity has not been compromised during transportation or storage. From these GC results, samples in this study had no significant or systematic loss or gain of volatile organics.

Laboratory BTX immunoassay results of sample splits from the samples with positive target values discussed in the preceding paragraph show an interesting trend. The data are summarized in Table 4 on both a per individual immunoassay basis and on a per sample basis. For the per sample results, "correct" or "incorrect" means that all individual immunoassays agreed when compared to the GC results, and "indeterminate" means that some immunoassay results agreed with and some disagreed with the GC results. Most immunoassay and GC analysis were in agreement for the 2.5 and 100 ppb standard samples. Only one of 20 (5 percent) of the 100 ppb samples was incorrectly determined. Ten of 12 of the 2.5 ppb samples were correctly assigned; with two samples having indeterminate results. However, for the 25 and 40 ppb samples, less than 50 percent of the individual immunoassay analyses, and fewer than 50 percent on a per sample basis, were in agreement with the GC results. It appears that the BTX immunoassay performs relatively poorly in the region near the 25 ppb classification level.

Laboratory immunoassay of the performance evaluation standards P1, P2, and P3 (2.5, 25, and 100 ppb toluene, respectively) carried out in the first half of the evaluation generated results in line with those above. Recall (from Section 3) that these samples were generated in the laboratory, while the samples discussed above were generated in the field. Only four samples of each type were assayed. One incorrect classification was found for each of the 2.5 and 100 ppb sample groups, while 3 of 4 samples having 25 ppb toluene were incorrectly classified. Once again, the immunoassay does not appear sensitive enough near the 25 ppb level.

Table 3. GC results for field prepared QA samples.

Target Value (ppb)	Field Prepared QA Sample Results				EPA Method 8020 Single Analyst Expectations	
	n	Mean (ppb)	Standard Deviation (ppb)	Range (ppb)	Mean (ppb)	Standard Deviation (ppb)
0	17	0.7	1.1	0 - 3.5	0.6	0.5
2.5	10	4.0	2.4	2.4 - 9.4	3.0	0.7
25	10	29	3.2	24 - 34	24	2.7
40	7	30	13	4 - 39	38	4
100	17	101	14	72 - 129	95	9.5

Table 4. Laboratory BTX immunoassay results of performance evaluation samples.

Target Value (ppb)	Per Assay Results		Per Sample Results		
	n Assays	n Correct	Correct	Indeterminate	Incorrect
2.5	26	24	10	2	0
25	28	9	1	6	6
40	13	4	1	3	3
100	40	38	19	0	1

An Assessment in Terms of Five Data Quality Elements

Accuracy

Analysis of trip blanks provided one way of evaluating accuracy and checking for contamination due to sample shipping and storage. The GC analysis was often carried out several days after the field immunoassay analysis, but well within the allowed 14-day holding time. No preservative was added to any sample. However, the samples were cooled and maintained at 4°C until analysis; which is the recommended procedure for volatile organic analysis of aqueous samples (U.S. EPA, 1986a). All thirty-three trip blanks were correctly identified by immunoassay in the laboratory. Only one determination resulted in an S/R ratio below 0.85. However, this sample was reanalyzed twice, with ratios of 1.02 and 1.01 resulting. Thus, no trip blanks were incorrectly classified by

the laboratory immunoassay. These results suggest that sample contamination was not a significant problem.

A select number of sample splits were analyzed by immunoassay in the laboratory to provide additional laboratory-to-field comparison data. These samples were analyzed at approximately the same time as the GC runs. Of 49 determinations from which comparisons could be made, only four were inconsistent with the GC results. The S/R ratios for these were 0.59, 0.60, 0.70, and 0.83. The last two results were associated with indeterminate results; that is, only one of two replicate analysis were not in agreement with the GC results. In summary, only one sample gave a false positive immunoassay result and two samples produced indeterminate immunoassay results.

Comparison of laboratory to field immunoassay results showed one pair of inconsistent laboratory results for a sample giving inconsistent field analysis results, one pair of positive laboratory results paired with a set of inconsistent field results, and three individual laboratory determinations inconsistent with the field results. However, two of these laboratory immunoassay values were associated with a sample which gave results that were consistent with the GC values. In summary, the laboratory immunoassay results from selected field samples do not give one reason to question the integrity of sample transportation and holding time. These factors do not appear to bias the results in any important way.

The false negative and false positive results were reassessed with respect to the bias measured between the colorimeter and the laboratory spectrophotometer. Classification of samples with high OD measurements for both sample and reference cuvettes was not affected by the bias. Classification of samples where the reference OD is high and the sample OD is low would also not be affected. Neither would the classification of samples with a low reference OD and a high sample OD. In all the above situations, the effect of bias is low, or it enhances the probability of correct classification. Only situations where the reference OD is high and the sample OD is slightly lower will be subject to misclassification due to bias in the OD reading. Using the alternative sample OD/reference OD ratio of 0.78 from Section 3 as the classification level, very little difference was found in the false negative and false positive rates for the field BTX immunoassay results. The alternative ratio gave six false positive determinations, which represent both replicate analyses for each of three samples. This compares with six false positives (both replicates for two samples, and one replicate for each of two other samples) for the original ratio. The alternative ratio yielded six false negatives, two samples with both replicates incorrect and two samples with only one replicate incorrect. This compares with four false negatives (both replicates for two samples) for the original ratio. Based on replicate analysis, only one more sample was consistently misidentified using the alternative S/R ratio.

Further results bearing on accuracy are presented in the following section on precision.

Precision

Precision of the BTX immunoassay analysis was assessed from the analysis results for field prepared performance evaluation standards. Three standard levels, at 2.5, 25, and 100 ppb toluene were prepared each day by the field analyst from ampulated 1000 ppm toluene standards. Usually, one set of analysis was performed before and another set was performed after the well samples were analyzed. Fresh sets of samples were prepared for each day's analysis, therefore variances reflect both dilution error and error in the assay.

There were 36 determinations (involving 19 separate days) for each concentration. The mean S/R ratio for the 2.5 ppb standards was 0.999, with a standard deviation of 0.056. All analyses had S/R ratios greater than 0.85. For the 25 ppb standard, only four analysis were less than 0.85.

The mean S/R was 0.943, with a standard deviation of 0.108. The BTX immunoassay was not sensitive enough to correctly classify most of these samples. The 100 ppb standards showed nine determinations with S/R ratios greater than 0.85. The mean ratio was 0.766, with a standard deviation of 0.104. A graphical summary is given in Figure 9. Figure 9 shows curves corresponding to the mean S/R ratio and the mean ratio plus or minus one and two standard deviations. This clearly shows why this formulation of the BTX immunoassay tends to produce a significant number of false negative and false positives in the concentration range from just below 25 ppb to just above 100 ppb.

Representativeness

The field samples were representative of typical groundwater samples. All field samples were obtained from pre-established monitoring wells. The well fields were at four different sites in the Las Vegas valley and represented sites where the aquifer was contaminated with gasoline. Naturally, these sites do not represent all the different groundwater characteristics present across the nation. For instance, variability due to differences in hardness or pH were not studied. However, it is felt that the results from this study are generally applicable to water contaminated with gasoline. Antox results with spiked seawater samples indicated that the test is not significantly affected by mineral content.

Completeness

The completeness criteria of 54 samples (90 percent of the originally specified 60 well samples) was met. Seventy-nine well samples were analyzed both by the BTX immunoassay in the field and by the laboratory GC method.

The performance of the BTX immunoassay was also assessed in terms of the percentage of cuvettes which generated acceptable results. Data quality objectives of 95 percent acceptable performance for reference and sample cuvettes had been specified in the demonstration plan. However, the developer had not provided limits on acceptable performance for the cuvettes.

For reference cuvettes the only qualification provided was that the optical density should be between 1.0 and 1.9 for optimal performance. However, this does not mean that ODs outside this range mean unacceptable performance. During the field and laboratory evaluation of the water samples 144 reference cuvettes were used. Seven had ODs less than 1.0, 99 had ODs between 1.0 and 1.9, and 38 had ODs greater than 1.9. Reference cuvettes with ODs below 1.0 were believed to be indicative of poor performance, and constituted 5 percent of the total. Reference cuvettes with results above 1.9 were accepted as valid, as it appeared the high values were a function of the levels of enzyme conjugate and antibody supplied in the kit. Antox provided instructions for modifying reagent levels and timing of assay steps to lower reference sample OD levels (see Appendix A).

No identifiable rules for determining acceptable sample cuvette behavior were provided by Antox. In this study we could not identify one problem associated with the 552 sample cuvettes used for the analysis of field or QA/QC samples. Since the results can span the OD range, it does not appear that any a priori criteria can be specified. It appears that the only types of sample cuvette problems one might find are associated with visible defects such as scratches or other marks on the cuvettes.

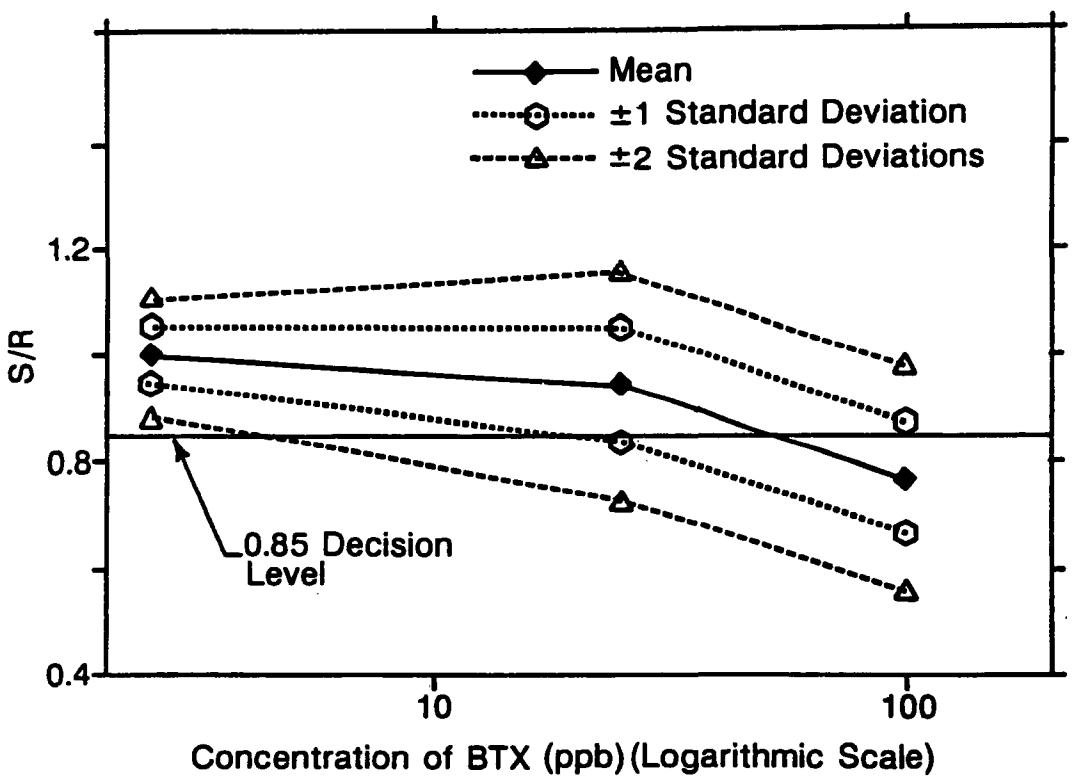

Figure 9. BTX immunoassay results for field performance evaluation samples. Mean S/R ± one and two standard deviations are shown.

Comparability

The comparability of results between GC and immunoassay was addressed by the use of a variety of QA/QC samples and through close adherence to written protocols. The QA/QC procedures described in EPA Method 8020 (U.S. EPA, 1986a) were followed for gas chromatography analysis. The results are presented in Appendix B, showing that this study was within the guidelines for the method. The results of quality control samples (presented elsewhere in this section) also demonstrated that the GC was providing high quality data. Trip audit samples, including both blank and positive BTX samples, showed that sample integrity was maintained between field and laboratory.

GC/MS analysis of representative samples indicated that a large unassigned GC peak found in several samples was methyl t-butyl ether (MTBE), an octane booster for gasoline. MTBE is one of several oxygenated compounds used to boost oxygen content of gasoline in the Las Vegas valley in the winter months. For high level samples, GC/MS confirmed the assignments of benzene, toluene, ethylbenzene, and xylene as the major aromatic compounds present in the samples. For very high level samples, much smaller amounts of other (unidentified) aromatic compounds were also reported. However, the relative amounts of these compounds were small, and would not have changed the classification of the samples with respect to the 25 ppb detection level used in this study.

Results of the On-Site Audit

A field audit was conducted on April 16, 1992. Only minor deviations from the demonstration plan were reported. The dilution protocol for preparing audit samples was different, and sample identification codes were changed for the field-analyzed performance evaluation standards. The sample identification codes were changed due to modifications to the demonstration plan after the evaluation was in progress. The dilution protocols were being followed correctly, but the auditor had misinterpreted the discussion in the demonstration plan. The auditor noted that "These small changes in procedure should not influence the results of the study". The auditor's report is included in this document as Appendix D.

ADDITIONAL QA/QC OBSERVATIONS AND CONCLUSIONS

Optical densities for the reference cuvettes are plotted as a function of time in Figure 10. Most of the reference OD values were between 1.7 and 2.0. Figure 10 shows that the range of OD values narrowed over the course of the demonstration. It is not known whether this effect is due to increased experience and reproducibility on the part of the analyst, or to changes in the reagents. Because of the non-linearity of the colorimeter in this range, it is recommended that the antibody and enzyme conjugate titers be adjusted to give reference OD values in the 1.0 to 1.5 range. It may be necessary to develop formulations of coated cuvettes and enzyme conjugates that have better stability with lower initial reference OD values.

According to the field analyst, there were indications toward the end of the study that the colorimeter drift was more pronounced. This has been associated with decline in the performance of the light source. Since the colorimeter was re-blanked before and after each run, the significance of this drift was minimal.

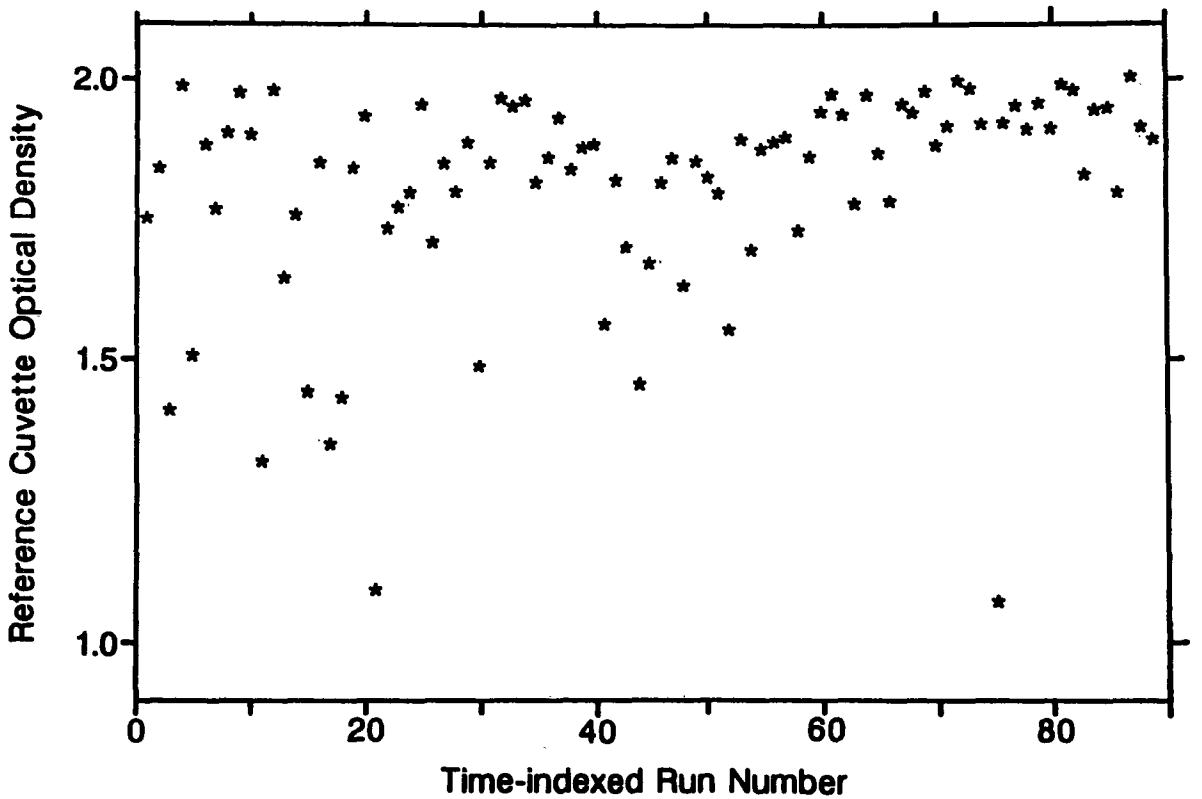

Figure 10. Reference cuvette optical density vs time.

SECTION 5

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The BTX Immunoassay performed well for most samples evaluated in this study. Only five percent of the samples were misclassified based on replicate determinations. The false negative results suggest that the current formulation of the BTX immunoassay is not robust enough to accurately deal with samples in the range of 25 to 100 ppb BTX. If one wants to be very accurate in this range, a more sensitive format should be developed. The immunoassay was easy to use and provided a quick estimate of BTX concentration that would prove useful in certain applications.

Advantages of the BTX Immunoassay

- Rapid - Up to four samples can be analyzed on-site within 30 minutes, allowing rapid field screening of environmental water samples.

- Portable - The field kit can be set up in the space of a small table. The portable colorimeter can be run from either a 120 VAC wall outlet or from batteries. However, one needs to have a cooler available to keep most of the reagents at 4°C.

- Easy to use - No extensive training is needed to use the field kit. The instructions are clear and easy to follow.

- Inexpensive - As a screening tool, use of the BTX immunoassay is much less expensive than requiring that all samples be analyzed by conventional laboratory analytical techniques.

- Effective - For certain screening functions, the BTX immunoassay appears to be very effective. Samples above approximately 100 ppb BTX were accurately identified. Low S/R ratios were related to contaminated samples, even for samples with gasoline contamination below the 25 ppb detection claim of the test. This method could prove useful in mapping the distribution of contaminants at a site or for monitoring changes in contaminant levels over time.

Limitations of the BTX Immunoassay

- Sensitivity - Results from this evaluation indicate that the BTX immunoassay does not meet the developer's claim for sensitivity. The test appears to allow a significant percentage of false negative results from samples in the range from 25 to 100 ppb BTX. If critical decisions are relying on analysis of samples with these concentration ranges, this test would not be appropriate. A sample would have to be

above 100 ppb to be in a range where there was a five percent or lower probability of getting a false negative result.

- Performance standards - Despite efforts by the developer, the kit comes with no performance standards. It is difficult to prepare stable low-level (in this case, 10 to 50 ppb) standards of volatile organic compounds. In this evaluation, low level standards were prepared on-site during each day's analysis by dilution from high-level ampulated standards. Preparation of these standards requires volumetric pipettes and glassware and careful attention to detail to prevent errors due to dilution or contamination. The use of these procedures with the kit would require the addition of several items of analytical glassware and more training, as well as a clear, written protocol. The user would also need access to an appropriate source of water free from BTX for making dilutions.

- Battery powered use - Despite the fact that the colorimeter is only used approximately every half-hour, leaving it on all the time drains the batteries. This study left the colorimeter on continuously during the day to avoid any instability that might have occurred from repeatedly turning it on and off. It was found that the batteries lasted about one and a half days (12 hours). Batteries were replaced at the beginning of each day to avoid having to monitor for unusual measurement properties during the second day, as there was no clue other than "unexpected" or "strange" readouts to show that the batteries were beginning to fail.

Minimizing False Negative and False Positive Results

In the absence of bias one can minimize the number of false positives and false negatives through two actions. One way is to reduce the variability associated with the immunoassay. As seen from Figure 8, low variability will reduce the probability that a sample will fall on the wrong side of the 0.85 S/R decision value simply from analysis error (variability). The other factor in determining false positive and false negative rates is the distribution of BTX concentrations one expects from a study. If the sample concentrations are expected to be mostly well removed from the decision level, then the rate of false negatives and false positives will be low. Alternatively, for a given study one may wish to minimize either false positive or false negative results by shifting the decision level. However, lower false negative rates will always be compensated by higher false positive rates, and vice versa.

Limitations of this Demonstration

The principal limitation of this SITE demonstration has to do with the limited matrix types evaluated. All the sites represented groundwater in the Las Vegas valley, and contamination at each site was primarily due to gasoline contamination.. Thus, the BTX immunoassay results reported here may not be fully applicable at sites with different or more complex organic contamination matrices, or at sites with large concentrations of inorganic compounds.

A second limitation of this evaluation is that it did not necessarily reflect the variability one might expect from use by a large number of field analysts. This study had only one field analyst. The evaluation did not test the robustness of the directions and procedures with respect to individuals not receiving direct training by personnel familiar with immunoassay and analytical chemistry techniques.

RECOMMENDATIONS

Recommendations Specific to the BTX Demonstration

- Quality assurance guidelines should be made very specific for an analysis which might be used by those with little experience. For the BTX immunoassay, specific guidance with respect to the minimal OD for the reference sample is needed. Though the developer told us verbally that a reference OD of less than 0.7 was unacceptable, the written guidance with respect to trouble shooting mentions only that reference ODs "should always be in the range 1.0—1.9 for optimal performance." The developer should provide more detailed and specific QC acceptance criteria based on statistically valid performance evaluations.

- It appears that the kit is not quite as sensitive as claimed. The developer needs to improve the kit's ability to perform as claimed in the 25 to 100 ppb region.

- A simple protocol for generation of performance standards needs to be developed.

- The colorimeter should be modified so as to display a warning light when battery power is too low for adequate performance. Alternately, the test procedure could include samples with known concentration. One would then be able to check the performance of the colorimeter as part of a check of the entire kit immunoassay procedure.

- Improve kit reagent stability so that reagent levels can be adjusted to give lower OD values at the beginning of the kit's shelf-life.

Recommendations for Future Studies

- Development of a more sensitive format would be advantageous. This is especially true for the detection of benzene, which has a maximum contaminant level of 5 ppb under the Safe Drinking Water Act (U.S. EPA, 1990b).

- Develop stable low-level BTX performance evaluation standards. Currently a field analyst needs extra standards and analytical glassware in order to generate performance evaluation standards.

- Evaluate the immunoassay on a variety of ground water matrices.

- Conduct a multi-laboratory or multi-site study to evaluate interlaboratory variability and assess operator dependent responses.

- Conduct additional studies with selected matrix interferences and cross-reacting substances.

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APPENDIX A

A DESCRIPTION OF THE BTX IMMUNOASSAY PROCEDURE

ANTOX BTX WATER SCREEN
25 ppb SENSITIVITY

Intended Use

The Antox BTX Water Screen is an enzyme immunoassay for the qualitative detection of BTX (Benzene, Toluene and Xylene) and similar compounds. This test is designed to reduce the number and frequency samples requiring expensive and time-consuming analytical testing by identifying those samples that have significant levels of BTX contamination.

Summary and Explanation

BTX are aromatic hydrocarbons widely used in industrial processes and products. The compounds can become environmental pollutants if misused or improperly disposed of. The detection of BTX compounds in water samples is important in determining contamination and effectiveness of remediation processes.

Principle of the Procedure

The Antox BTX Water Screen is a sensitive, fast and easy-to-use enzyme immunoassay which employs polyclonal antibodies to identify BTX and similar compounds in water samples. Using this test, a qualitative determination of BTX is possible.

The Antox BTX Water Screen is based upon immunological principles governing antigen-antibody reactions. The kit features a cuvette coated with antibody specific for BTX and an enzyme-conjugated BTX (antigen) reagent. Sample is added to the antibody coated cuvette after which the enzyme-conjugated BTX reagent is added. BTX or similar compounds present in the sample will compete with the enzyme-conjugated BTX for binding sights on the antibody-coated cuvette. The cuvette is then rinsed to remove excess reactants. Substrate and chromogen are then added which, in the presence of enzyme, turn a color that can be measured on a photometer. The amount of BTX in the sample is indirectly proportional to the color development. A comparison to a reference sample determines the relative concentration of BTX or similar compounds in the sample.

Materials Provided in the kit:

Antibody Coated Cuvettes
Buffer Solution (Gray Cap)
Enzyme Solution (Red Cap)
Color Developer 1 (Black Cap)
Color Developer 2 (Blue Cap)
Terminating Solution (Purple Cap)
Disposable Syringes/Transfer Pipettes

Materials not Provided:

VOA Transport Vials
Reference Solution (Distilled Water)
? — Felt Tip Pen — ?
Liquid Dispenser
Quality Control Samples (Available from Antox)
Timer

Caution

Do not mix reagents from different lots.
Do not use components beyond expiration date.
Do not mix reagent bottle caps.
FOR ENVIRONMENTAL TESTING ONLY.

Storage

All reagents should be stored refrigerated (2-8°C) when not in use except color developer 2 (Blue Cap) which should be stored at room temperature (20°-28°C). Prior to use, place the Buffer Solution (gray cap), Enzyme Solution (red cap), Reference Solution (distilled water) into the ice chest with the samples. Allow Color Developer 1 (black cap), Color Developer 2 (blue cap) and bagged cuvettes to come to ambient temperature.

DO NOT FREEZE

SAMPLE COLLECTION, STORAGE AND PREPARATION

Ground water samples are best, and testing should be done as the samples are collected. Testing on site is recommended due to the volatile nature of BTX and similar compounds.

The sample and reference solution (distilled water) should be maintained in a cold environment, such as an ice water bath, prior to testing. This is due to the volatility of these compounds.

Procedural Notes

Quality Control Samples Refer to Enclosed QC Procedure

Temperature Limits

REFERENCE SOLUTION, SAMPLE SOLUTION, ENZYME SOLUTION, AND BUFFER SOLUTION SHOULD BE BROUGHT TO EQUIVALENT TEMPERATURES IN AN ICE WATER BATH. IF AMBIENT TEMPERATURE IS LESS THAN 75°F BUT

MORE THAN 32°F THEN THE ASSAY CAN BE RUN AT ROOM TEMPERATURE.

IF THE TEMPERATURE IS GREATER THAN 75°F STEPS 1-8 SHOULD BE PERFORMED IN AN ICE WATER BATH.

Each reagent should be added to the reference cuvette immediately followed by the sample cuvette. This should be done as quickly as possible so there is a minimum amount of time between the addition of reagents to the reference and sample cuvette.

All components should be added to the cuvettes by holding the dropper vial in an upright inverted position so the dropper nozzle is in the center of the test cuvette. When adding the Color Developing Solutions, take care to dispense the drops into the bottom of the cuvette.

When washing the cuvettes, discard the contents of both cuvettes with a flicking motion. Using the rinse bottle provided, rinse the cuvettes four times with distilled water. Forcefully fill the cuvettes to overflowing to overflowing and forcefully empty after each wash. Flick the cuvettes well after the last addition to eliminated residual water.

The timing aspects of this test are important. DO NOT WALK AWAY!

ASSAY PROCEDURE:

(1.) LABEL ONE ANTIBODY COATED CUVETTE "R" for Reference.

(2.) Appropriately label an ANTIBODY COATED CUVETTE for each sample (up to four).

NOTE: *****COMPLETE STEPS 3 THROUGH 6 AS QUICKLY AS POSSIBLE

(3.) Use a disposable syringe or transfer pipette to dispense 4 ml of Reference Solution (or distilled water) into the "R" cuvette.

(4.) Use the same technique to measure 4 ml from sample for the VOA vial into the appropriately labeled cuvette. BE SURE TO USE A CLEAN TRANSFER PIPETTE FOR EACH SAMPLE TO AVOID CROSS CONTAMINATION.

(5.) Add 4 drops of the Buffer Solution (Gray cap) to all cuvettes.

(6.) Add 4 drops of the Enzyme Solution (Red Cap) to all cuvettes. Cap and invert cuvettes.

(7.) Incubate 10 minutes.

(8.) Using distilled water (NOT COLD) and wash bottle, wash and decant the cuvettes 4 times. Be sure to remove as much water from the cuvettes as possible (blot if necessary).

(9.) Add 4 drops of Color Developer 1 (Black cap) to each cuvette. Add 4 drops Color Developer 2 (Blue cap) to each cuvette. Rap cuvettes gently to assure all solution is in the bottom of the cuvettes.

(10.) Incubate 5 minutes.

(11.) Add 4 drops terminating solution (Purple cap) to each cuvette. Mix by swirling gently.

(12.) Read the cuvettes at 450 nm. Wipe the lower portion of each cuvette with a Kimwipe before reading.

Performance Characteristics

Sensitivity

Assay sensitivity is 25 ppb for water samples. This sensitivity will vary based on the specific compounds that are being tested, possible interfering substances or matrix effects.

Sensitivity characteristics for individual compounds may need to be determined.

Specificity

The antibody is directed at BTX (Benzene, Toluene and Xylene). Substantial cross-reactivity should be anticipated due to the fact many compounds possess similar antigenic properties.

Calculation and Interpretation of Results

$$\frac{\text{Absorbance of Sample (S)}}{\text{Absorbance of Reference (R)}} = \frac{S}{R}$$

At the 95% confidence:

If S/R is equal to or greater then .850, then the sample contains 25 ppb (parts per billion) or less of BTX.

If S/R is less than .850, then the sample contains 25 ppb or more of BTX.

Example:

Sample abs.	=	.600
Reference abs.	=	1.000

$$\frac{.600}{1.00} = .600$$

.600 is less than .850. The sample contains 25ppb or more BTX.

Manufactured By:

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(207) 772-3544 - (800)323-3199

Antox BTX Water Screen

Trouble Shooting Addendum #1 : Off Scale Absorbance Values

At the time of Quality control testing the Antox BTX Water Screen has a Reference Absorbance that averages 1.3 units at 450 nm. The Reference Absorbance values should always be in the range 1.0 - 1.9 for optimal performance.

The following suggestions are meant to assist the user when Absorbance values are consistantly greater than 1.9. It is assumed that the photometer used has been checked for effieciency.

BEFORE MANIPULATION OF THE TEST PROCEDURE, RULE OUT THE FOLLOWING:

1) PROPER WASHING It is EESENTIAL THAT ALL NONIMMUNOLOGICALLY BOUND ENZYME SOLUTION be removed before adding the color developers. Using a laboratory wash bottle and a FORCEFUL jet of water the cuvettes should be filled to overflowing and emptied at least 4 times. The cuvettes should be empties with a sharp "flicking " motion to assure removal of all rinse water.

2) CLEAN TIPS ON COLOR DEVELOPERS If the vial tips on the color developers become contaminated with enzyme solution, the enzyme conjugate could react with the color developers. This should not happen if the user practices care in replacing the caps on the proper vials. An easy way to check this is: place 2 drops of each color developer in a disposable test tube. If any enzyme conjugate is present color will develope immediately.

3) TIMING Both incubation times should be adhered to. The reactions are continuous and longer incubations will give higher Absorbance values

IF THE ABOVE ARE RULED OUT, THE FOLLOWING MANIPULATIONS MAY BE DONE

1) Reduce the Enzyme Solution from 4 to 3 drops.

2) Reduce the 2nd incubation from 5 to 4 minutes.

3) Dilute the terminated test solutions with distilled water. A 1:2 dilution should be sufficient (E.g. the terminated test solution in the cuvette has a volume of approximately 500 uL. Add 500 uL distilled water for a 1:2 dilution.

APPENDIX B

PROCEDURAL DETAILS AND QUALITY ASSURANCE SUMMARY FOR GAS CHROMATOGRAPHY AND PURGE-AND-TRAP METHODS

Table of Contents

	page
GC Method Used in the BTX SITE Demonstration	43
Instrumentation	43
Calibration Curve Generation	43
Daily Calibration Standards	44
Retention Time Windows	44
Method Detection Limits	44
Method Precision	45

List of Figures

Figure		page
B-1.	Benzene standard curve (3-12-92)	49
B-2.	Toluene standard curve (3-12-92)	49
B-3.	Ethylbenzene standard curve (3-12-92)	50
B-4.	*o*-Xylene standard curve (3-12-92)	50
B-5.	Benzene calibration control chart (32 ppb)	52
B-6.	Toluene calibration control chart (32 ppb)	52
B-7.	Ethylbenzene calibration control chart (32 ppb)	53
B-8.	*o*-Xylene calibration control chart (32 ppb)	53
B-9.	Benzene calibration control chart (60 ppb)	55
B-10.	Toluene calibration control chart (60 ppb)	55
B-11.	Ethylbenzene calibration control chart (60 ppb)	56
B-12.	*o*-Xylene calibration control chart (60 ppb)	56

List of Tables

Table page

B-1. HP 5890/PID detector calibration on 1-14-92 (Mean of three injections). 46
B-2. HP 5890/PID detector calibration on 2-24-92 (Mean of three injections). 47
B-3. HP 5890/PID detector calibration on 3-12-92 (Mean of three injections). 48
B-4. HP 5890/PID detector continuing calibrations - (32 ppb). 51
B-5. HP 5890/PID detector continuing calibrations - (60 ppb). 54
B-6. HP 5890/PID detector - retention time windows within day precision (analyzed 1-14-92). 57
B-7. HP 5890/PID detector - retention time windows precision over a two week period (analyzed 1-14-92 to 1-29-92) 58
B-8. HP 5890/PID detector - method detection limits in water spiked at 1 µg/L (ppb) (analyzed 1-15-92) 59
B-9. HP 5890/PID detector - method detection limits in water spiked at 1 µg/L (ppb) (analyzed 2-25-92). 60
B-10. HP 5890/PID detector - method detection limits in water spiked at 1 µg/L (ppb) (analyzed 3-13-92). 61
B-11. HP 5890/PID detector - precision study replicates analyzed at 20 µg/L (analyzed 1-09-92). 62

GC METHOD USED IN THE BTX SITE DEMONSTRATION

Water samples were analyzed by gas chromatography to provide comparison results for replicate samples analyzed by the Antox BTX immunoassay. The analytical procedures used were EPA Method 8020 (GC with PID detector) and EPA method 5030 (Purge and Trap).

Instrumentation

The following analytical instruments were used:

Gas Chromatograph:	Hewlett-Packard Model 5890 - series 2
GC Column:	DB 624 30 m X 0.54 mm ID (J&W Scientific)
Purge and Trap:	O.I. Analytical Model 3340 with O.I. Model MPM-16 AutoSampler
Detector:	O.I. Model 3320 P.I.D. Detector
Data Integrator:	Hewlett-Packard Model 3396A

The instrument conditions were as follows:

Gas Chromatograph:

Carrier Gas -	Helium
Flow Rate -	10 cc/min
Oven Temperature -	40°C for 2 min. Ramped at 4°C / min 100°C final
Injector Temperature -	200°C
Detector Temperature -	250°C
PID Lamp	10 eV at intensity 4

Purge and Trap:

Purge Gas -	Helium
Purge Flow -	40 cc/min
Purge Time -	11 min
Purge Temperature -	28°C (ambient)
Desorb Temperature -	180°C
Desorb Time -	2 min

Calibration Curve Generation

The GC was calibrated using the external standard calibration procedure as outlined in SW-846. Calibration standards were prepared at a minimum of five concentrations by adding volumes of stock standards to a volumetric flask and diluting with methanol. The analytical reference standards were obtained from the EPA Quality Assurance Materials Bank at Research Triangle Park, NC. The following standards were used:

Benzene	5,000 µg/mL	Lot # 04-01-06
Toluene	1,000 µg/mL	Lot # 84-01-08
Ethylbenzene	10,000 µg/mL	Lot # 36-01-04
o-Xylene	5,000 µg/mL	Lot # 201-01-05

The above solutions were diluted into a single solution containing 5 μg/mL of each compound. This working standard was used to spike deionized water to concentrations of between 1 to 200 μg/L (ppb). These water spikes were analyzed by purge-and-trap in the same manner as the actual samples.

The instrument was calibrated three times over the course of the study; on 1-14-92, 2-14-92, and 3-12-92. The results of these calibrations are presented in Tables B-1, B-2, and B-3. The response factors were calculated by dividing the concentration of the water standard by the area of the peak. The areas were obtained from the HP 3396A integrator. The method states linearity can be assumed and the average calibration factor can be used if the relative standard deviation (percent RSD) of the calibration factor is less than 20 percent over the working range of the method. As shown in the data tables, this was achieved for all analytes for all three dates. Tables B-1, B-2, and B-3 also show the squared correlation coefficient for each calibration and the slope of the prediction equation. Examples of the calibration curves for the four analytes are shown in figures B-1 to B-4, for the calibration performed on 3-12-92.

Daily Calibration Standards

EPA method 8020 requires the calibration to be verified on each working day by injection of at least one calibration standard. For this study at least two standards were injected on each day samples were analyzed, one before any samples were analyzed and one following the last sample analyzed. In addition, a standard was analyzed for each set of 12 samples. This QC standard was near the mid point of the calibration curve. The method states the analysis is in control if the response for any analyte varies from the predicted response by less than ±15 percent. If the response varies by more than ±15 percent, a new calibration curve is to be prepared.

From 1-15-92 to 3-02-92, the QC standard was a water sample spiked to contain 32 ppb of each target analyte. The analytical results are presented in Table B-4 for these daily QC standards. These results are shown as quality control charts in figures B-5 to B-8. The lines indicate the upper and lower control limits at ±15 percent of the expected concentration. There was only one analysis on March 2 which exceeded the control limits (up to 23 percent high). Prior to running any further samples, a calibration curve was prepared on 3-12-92 with the concentration range expanded to 200 ppb. Therefore, the QC standard analyzed after this date was increased to 60 ppb. The results of the daily 60 ppb QC standards are presented in Table B-5, and shown as QC charts in figures B-9 to B-12. None of the 60 ppb QC standards exceeded the control limits.

Retention Time Windows

The method requires the establishment of retention time windows for the target analytes. The procedure defines the window as three times the standard deviation of the absolute retention time for each analyte analyzed. Each standard deviation was obtained by replicate injection of a standard. Retention time precision was calculated for a single day, and over a two week period (1-14-92 to 1-29-92). These data are presented in tables B-6 and B-7. The data show very good retention time precision. Single day retention time windows were less than 0.01 minutes and retention time windows over two weeks were less than 0.05 minutes for each analyte.

Method Detection Limits

The method detection limit (MDL) was determined each time a new calibration was prepared. The MDL was determined by analyzing replicate low level standards. The low level standard was 1.0 ppb. The MDL was defined as the concentration obtained by a peak area of three times the standard deviation of the replicate low level standard. Data is presented in tables B-8, B-9, and B-

10 for the MDL calculations. All analytes show an MDL of less than 1 ppb; well below the data quality objective of 10 ppb specified for this study.

Method Precision

The overall precision of the method was determined by analyzing standards at 20 ppb. A standard was analyzed on each port of the autosampler. The results are presented in Table B-11 The percent RSD for each of the four standards was four.

Concentration (μg/L)	Area (μV X sec / 8)			
	Benzene	Toluene	Ethylbenzene	o-Xylene
4	35859	30112	27058	26786
16	125097	111768	103456	102712
32	233663	201471	188228	185906
60	440296	384755	363435	356390
80	550769	488408	461050	450415
100	694612	618535	588340	578171
120	969963	891411	842367	839200
140	1139242	1030770	972185	972051
	Response Factors			
4	1.115E-04	1.328E-04	1.478E-04	1.493E-04
16	1.279E-04	1.432E-04	1.547E-04	1.558E-04
32	1.369E-04	1.588E-04	1.700E.04	1.721E-04
60	1.363E-04	1.559E-04	1.651E-04	1.684E-04
80	1.453E-04	1.638E-04	1.735E-04	1.776E-04
100	1.440E-04	1.617E-04	1.700E-04	1.730E-04
120	1.237E-04	1.346E-04	1.425E-04	1.430E-04
140	1.229E-04	1.358E-04	1.440E-04	1.440E-04
Mean	1.31E-04	1.48E-04	1.58E-04	1.60E-04
RSD (%)	8.3	8.2	7.5	8.2
Regression Results				
R^2	0.9862	0.9821	0.9831	0.9799
Slope (μg/L)/Area	1.261E-04	1.383E-04	1.461E-04	1.465E-04

Table B-1. HP 5890/PID detector calibration on 1-14-92 (Mean of three injections).

Concentration (μg/L)	Area (μV X sec / 8)			
	Benzene	Toluene	Ethylbenzene	*o*-Xylene
4	72109	60900	52798	53579
16	242937	214254	197431	188352
32	485011	423830	388253	375529
60	888909	777131	707516	688894
100	1393942	1216043	1099833	1092868
	Response Factors			
4	5.55E-05	6.57E-05	7.576E-05	7.466E-05
16	6.59E-05	7.47E-05	8.104E-05	8.495E-05
32	6.60E-05	7.55E-05	8.242E-05	8.521E-05
60	6.75E-05	7.72E-05	8.480E-05	8.710E-05
100	7.17E-05	8.22E-05	9.092E-05	9.150E-05
Mean	6.53E-05	7.51E-05	8.30E-05	8.47E-05
RSD (%)	8.2	7.2	6.0	6.5
Regression Results				
R^2	0.9986	0.9984	0.9979	0.9991
Slope (μg/L)/Area	7.22E-05	8.27E-05	9.14E-05	9.20E-05

Table B-2. HP 5890/PID detector calibration on 2-24-92 (Mean of three injections).

Concentration (μg/L)	Area (μV X sec / 8)			
	Benzene	Toluene	Ethylbenzene	o-Xylene
1	11092	10646	10606	11348
4	38457	30907	29215	30655
16	138623	106150	107773	109891
32	272223	208115	213565	216941
60	497314	377541	392741	394126
100	837529	643061	671368	675251
140	1151019	888521	928122	932350
200	1693306	1307454	1368847	1374853
	Response Factors			
1	9.016E-05	9.393E-05	9.429E-05	8.812E-05
4	1.040E-04	1.294E-04	1.369E-04	1.305E-04
16	1.154E-04	1.507E-04	1.485E-04	1.456E-04
32	1.176E-04	1.538E-04	1.498E-04	1.475E-04
60	1.206E-04	1.589E-04	1.528E-04	1.522E-04
100	1.194E-04	1.555E-04	1.489E-04	1.481E-04
140	1.216E-04	1.576E-04	1.508E-04	1.502E-04
200	1.181E-04	1.530E-04	1.461E-04	1.455E-04
Mean	1.13E-04	1.44E-04	1.41E-04	1.38E-04
RSD(%)	9.0	14.5	12.9	14.4
Regression Results				
R^2	0.9997	0.9996	0.9995	0.9995
Slope (μg/L)/Area	1.194E-04	1.547E-04	1.475E-04	1.470E-04

Table B-3. HP 5890/PID detector calibration on 3-12-92
(Mean of three injections).

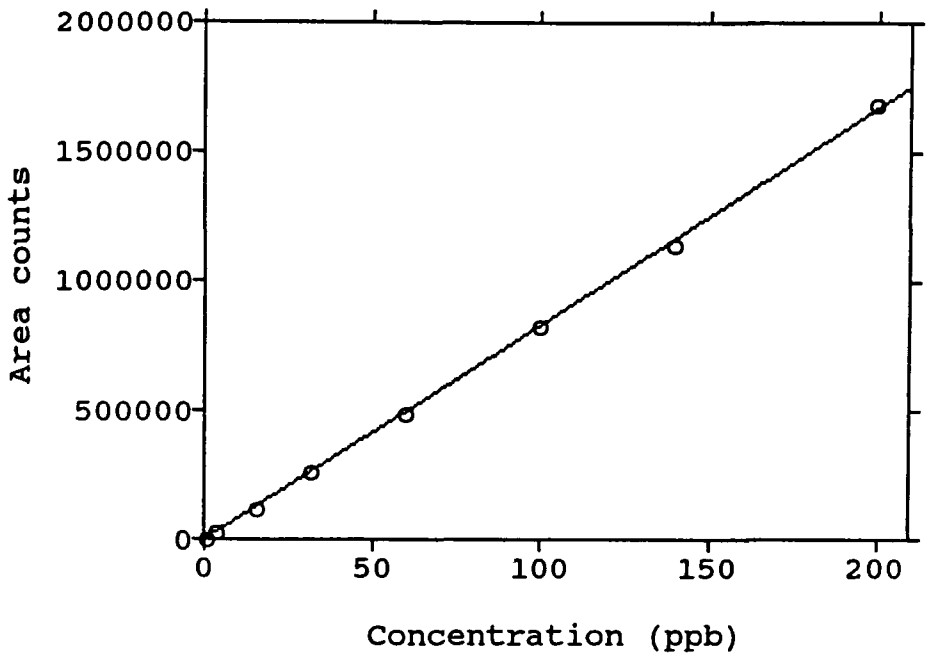

Figure B-1. Benzene standard curve (3-12-92).

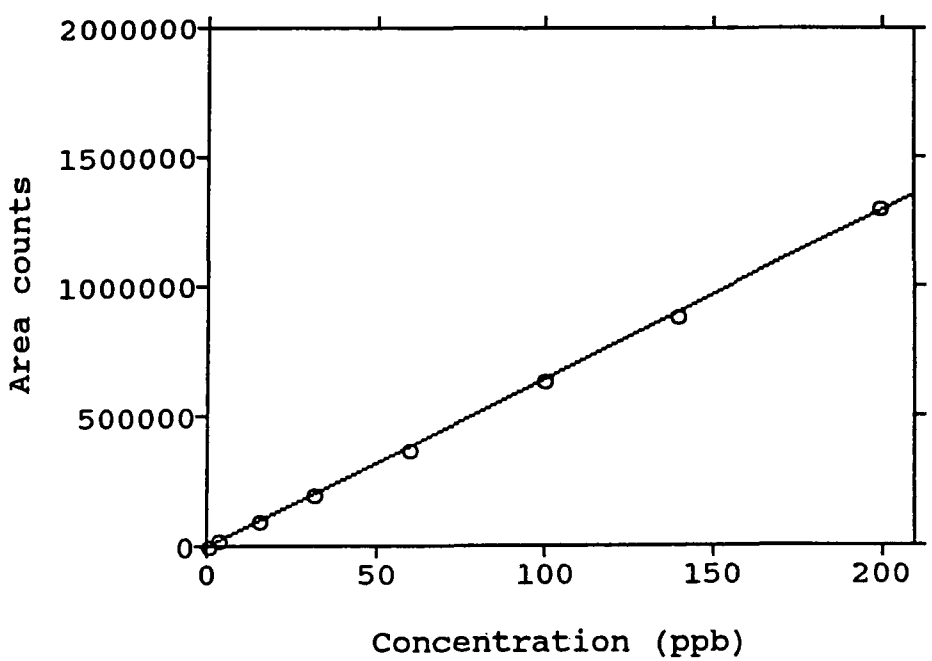

Figure B-2. Toluene standard curve (3-12-92).

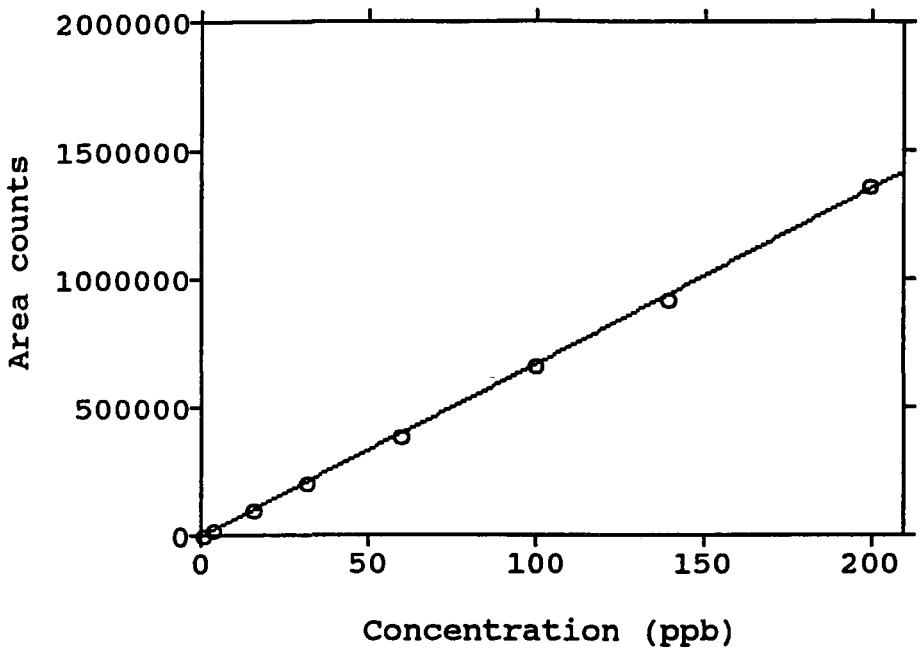

Figure B-3. Ethylbenzene standard curve (3-12-92).

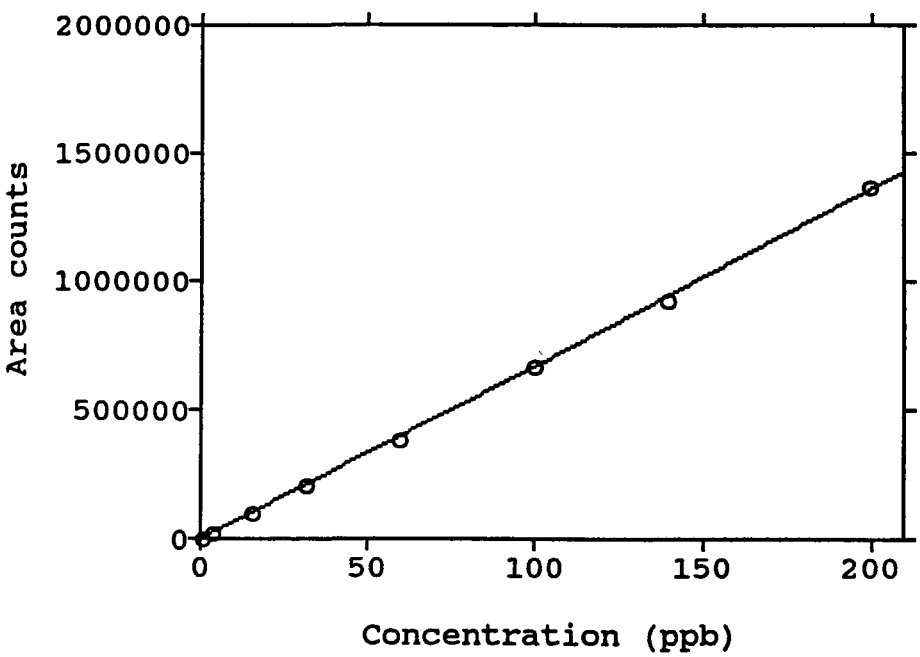

Figure B-4. *o*-Xylene standard curve (3-12-92).

Date	Run Number	Concentration (μg/L)			
		Benzene	Toluene	Ethylbenzene	o-Xylene
1-15-92	136	33.1	33.9	33.7	33.0
1-17-92	191	33.7	34.1	33.5	32.4
1-21-92	209	34.1	35.1	34.9	34.5
1-27-92	256	31.8	33.2	33.0	32.4
1-27-92	274	33.5	32.9	32.8	32.4
1-27-92	278	31.4	35.5	35.3	34.7
1-28-92	281	31.8	33.0	32.9	32.2
1-28-92	297	33.0	33.7	33.8	33.1
2-25-92	633	34.9	34.5	34.4	35.5
2-25-92	635	33.3	34.2	33.1	34.3
2-26-92	675	36.3	36.9	36.4	36.5
2-26-92	653	32.7	33.3	32.4	33.2
2-28-92	724	35.5	36.1	34.4	35.5
2-28-92	739	31.9	32.8	29.8	27.9
2-28-92	740	34.5	34.6	32.3	33.1
2-29-92	750	32.3	33.6	31.8	32.7
2-29-92	764	32.2	33.4	31.0	31.8
3-01-92	767	34.0	35.2	33.5	34.3
3-01-92	781	32.3	32.7	30.8	31.4
3-01-92	782	34.5	35.2	32.6	33.7
3-02-92	795	35.1	36.1	33.8	34.6
3-02-92	796	33.4	34.2	32.2	32.7
3-02-92	798	34.2	35.3	34.0	34.6
3-02-92	812	35.9	36.8	35.2	36.0
3-02-92	813	38.2	39.3	37.6	38.4
3-02-92	814	30.9	32.3	30.8	31.8

Table B-4. HP 5890/PID detector continuing calibrations - (32 ppb).

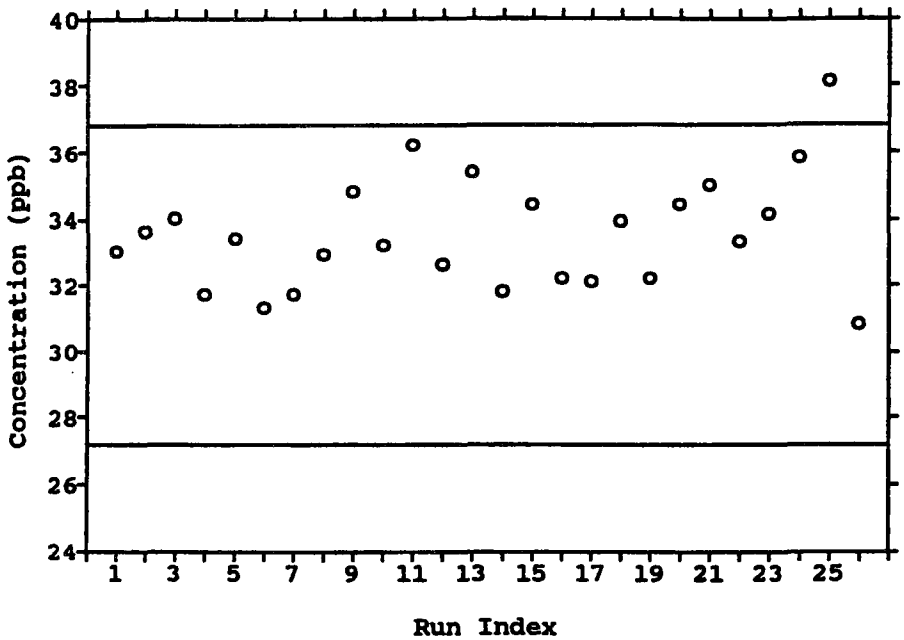

Figure B-5. Benzene calibration control chart (32 ppb).

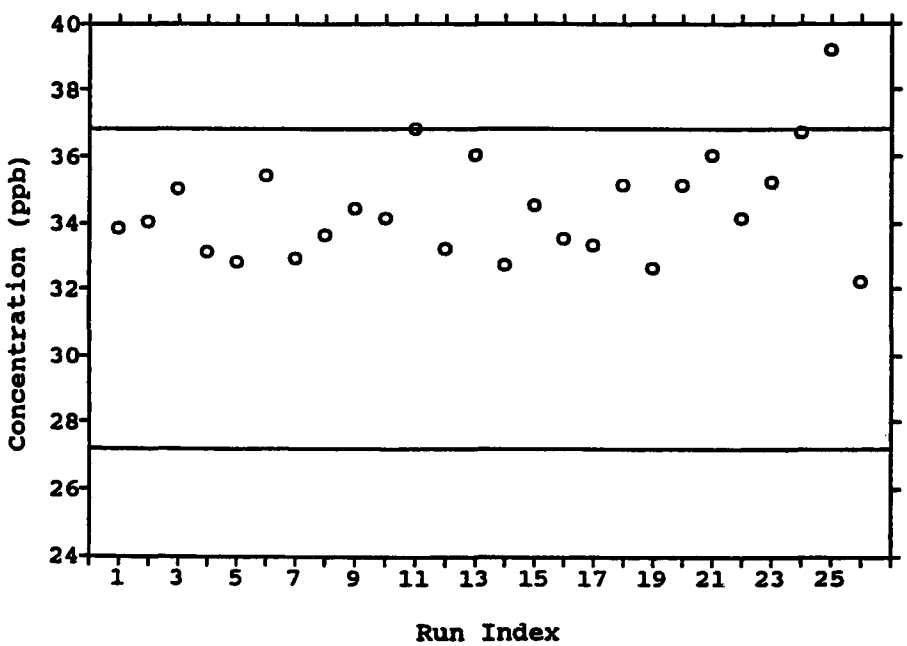

Figure B-6. Toluene calibration control chart (32 ppb).

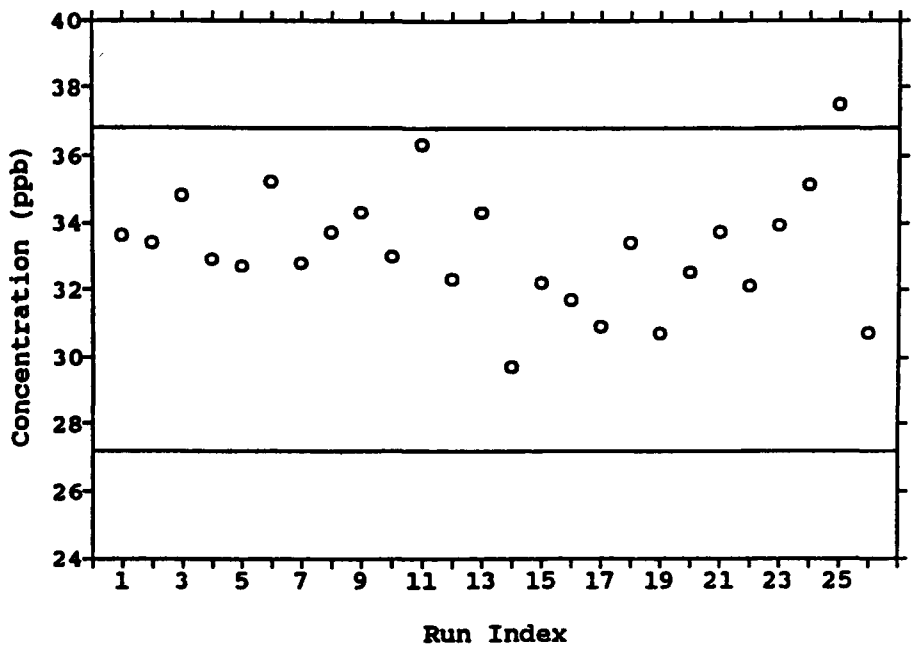

Figure B-7. Ethylbenzene calibration control chart (32 ppb).

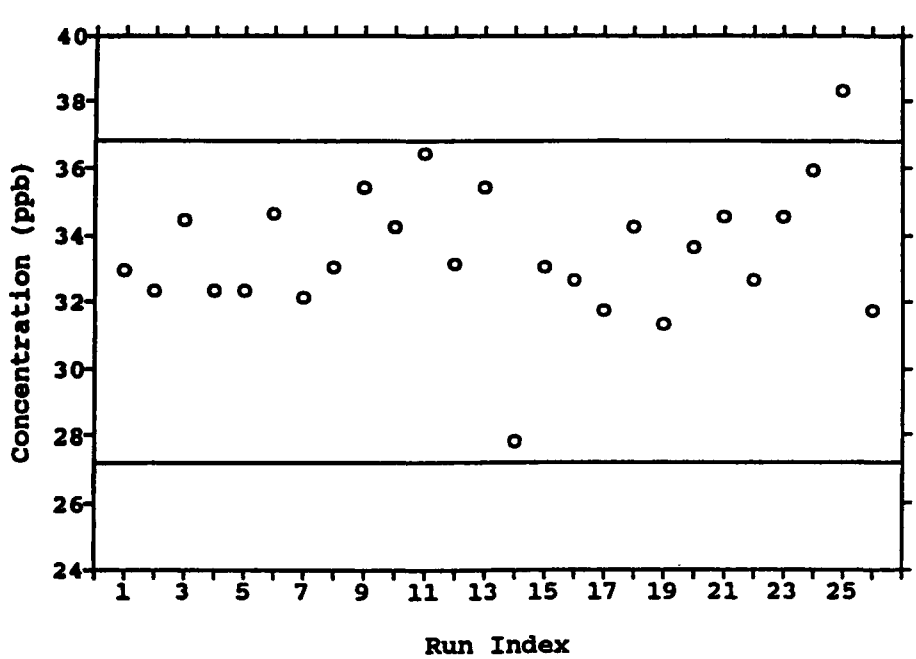

Figure B-8. *o*-Xylene calibration control chart (32 ppb).

Date	Run Number	Concentration (μg/L)			
		Benzene	Toluene	Ethylbenzene	*o*-Xylene
3-13-92	1053	57.0	55.1	56.6	56.9
3-13-92	1068	61.7	58.5	60.9	59.8
3-14-92	1071	54.3	52.4	54.9	54.4
3-14-92	1086	60.1	59.0	57.6	57.5
3-15-92	1094	64.2	62.1	64.3	63.9
3-16-92	1109	60.7	60.3	59.0	58.6
3-16-92	1113	61.4	62.1	60.7	60.0
3-16-92	1120	62.3	62.4	61.5	61.4
3-17-92	1124	67.6	64.7	68.5	67.2
3-17-92	1131	61.9	59.9	63.6	62.6
3-18-92	1134	58.4	56.7	59.5	58.6
3-23-92	1201	58.7	56.4	59.1	57.8
3-23-92	1210	60.4	57.6	60.2	59.1
4-20-92	1413	53.5	53.2	56.3	54.9
4-20-92	1426	57.2	53.5	56.5	55.7
4-20-92	1427	58.2	56.2	59.5	58.1
4-20-92	1441	57.8	54.5	56.5	55.1
4-21-92	1445	55.1	52.6	55.7	54.6
4-23-92	1461	53.0	51.3	54.3	53.4
4-23-92	1475	55.6	53.0	55.8	54.9
4-23-92	1490	54.3	51.5	54.3	53.1
4-24-92	1495	57.1	55.5	57.3	57.7
4-24-92	1499	59.0	56.3	59.5	58.3

Table B-5. HP 5890/PID detector continuing calibrations - (60 ppb).

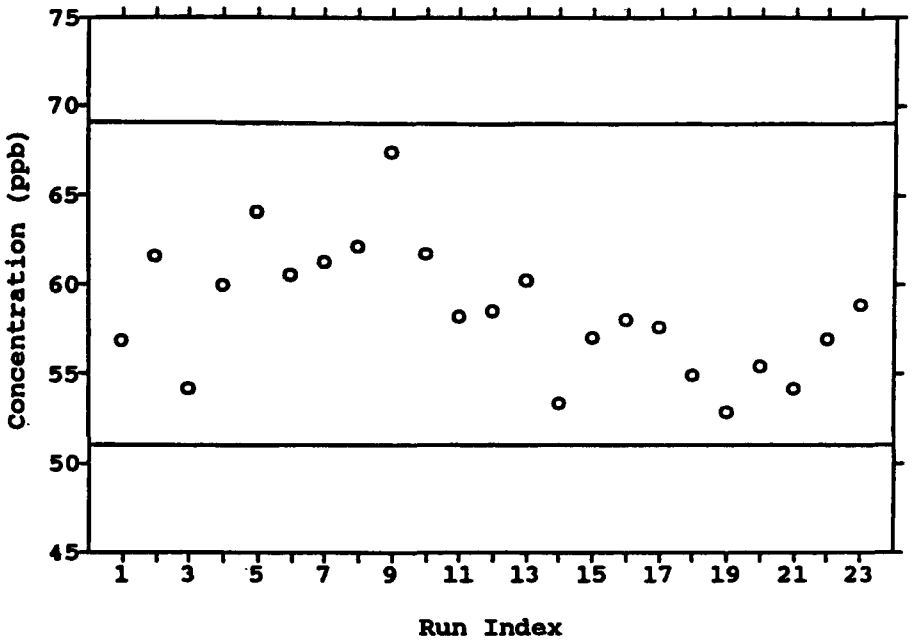

Figure B-9. Benzene calibration control chart (60 ppb).

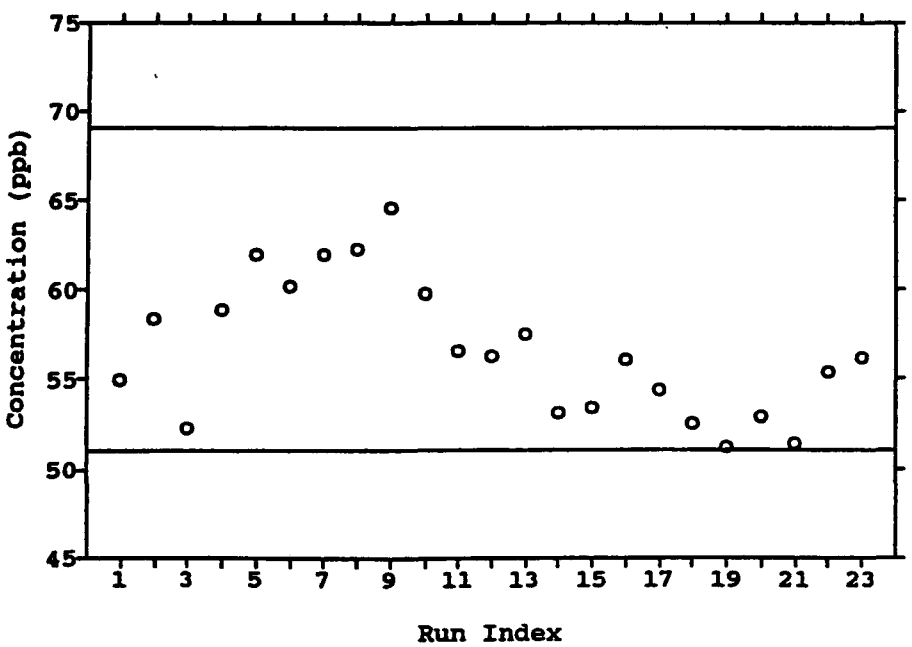

Figure B-10. Toluene calibration control chart (60 ppb).

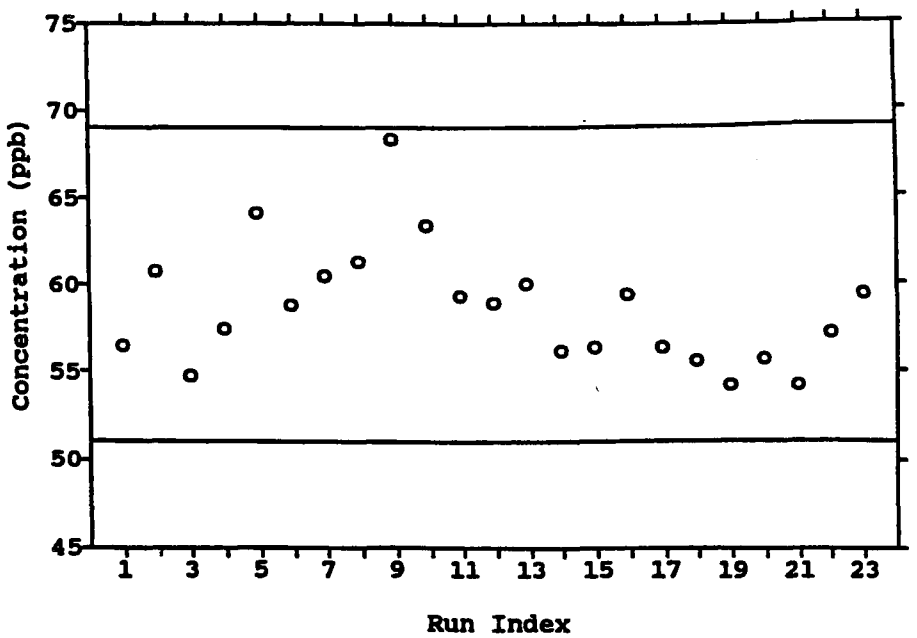

Figure B-11. Ethylbenzene calibration control chart (60 ppb).

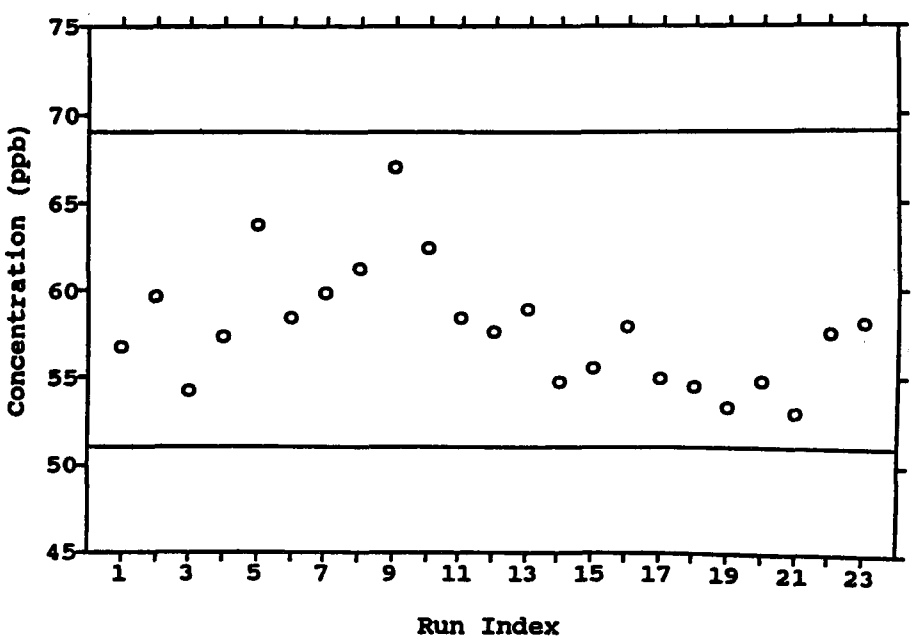

Figure B-12. *o*-Xylene calibration control chart (60 ppb).

Run Number	Retention Time (min)			
	Benzene	Toluene	Ethylbenzene	*o*-Xylene
109	3.022	5.345	7.850	8.786
111	3.015	5.340	7.843	8.777
112	3.016	5.340	7.845	8.780
113	3.015	5.340	7.843	8.779
115	3.016	5.342	7.843	8.779
118	3.020	5.345	7.849	8.785
119	3.016	5.343	7.848	8.783
120	3.018	5.346	7.850	8.785
121	3.019	5.345	7.849	8.786
122	3.019	5.345	7.848	8.785
124	3.019	5.346	7.848	8.786
125	3.012	5.340	7.846	8.785
126	3.016	5.344	7.849	8.786
127	3.015	5.344	7.850	8.785
128	3.019	5.345	7.850	8.786
129	3.015	5.345	7.850	8.785
130	3.017	5.345	7.851	8.785
133	3.019	5.346	7.853	8.789
Mean	3.017	5.344	7.848	8.784
SD	0.002	0.002	0.003	0.003
RSD (%)	0.08	0.04	0.04	0.03
Retention Time Windows				
3*SD	0.007	0.007	0.009	0.009
Percent	0.2	0.1	0.1	0.1

Table B-6. HP 5890/PID detector - retention time windows within day precision (analyzed 1-14-92).

Run Number	Retention Time (min)			
	Benzene	Toluene	Ethylbenzene	*o*-Xylene
109	3.022	5.345	7.850	8.786
126	3.016	5.344	7.849	8.786
130	3.017	5.345	7.851	8.785
256	3.030	5.367	7.877	8.816
274	3.045	5.376	7.882	8.817
278	3.045	5.376	7.884	8.818
281	3.035	5.370	7.879	8.815
297	3.041	5.370	7.878	8.813
315	3.040	5.376	7.887	8.825
Mean	3.032	5.363	7.871	8.807
SD	0.012	0.014	0.016	0.016
RSD (%)	0.38	0.27	0.20	0.18
Retention Time Windows				
3*SD	0.035	0.043	0.048	0.049
Percent	1.1	0.8	0.6	0.6

Table B-7. HP 5890/PID detector - retention time windows precision over a two week period (analyzed 1-14-92 to 1-29-92).

Run Number	Area (μV X sec / 8)			
	Benzene	Toluene	Ethylbenzene	*o*-Xylene
142	12006	9317	7102	7185
144	10335	10020	7933	8103
145	10107	10297	7972	8449
146	9808	10025	7892	8266
147	12704	13085	11132	12037
148	9189	9515	7603	7588
149	10468	9939	7629	7879
150	9332	10045	7149	7702
151	9853	9785	7470	7716
Mean	10422	10225	7987	8325
SD	1117	1049	1151	1360
RSD (%)	10.7	10.3	14.4	16.3
Method Detection Limits				
3*SD	3352	3147	3454	4080
MDL (ppb)	0.322	0.308	0.432	0.490

Table B-8. HP 5890/PID detector - method detection limits in water spiked at 1 μg/L (ppb) (analyzed 1-15-92).

Run Number	Area (μV X sec / 8)			
	Benzene	Toluene	Ethylbenzene	*o*-Xylene
642	22579	16394	22516	16769
643	30941	16240	--	12248
644	21331	17621	19826	18652
645	22365	16696	31269	27580
646	21414	17217	20022	18467
648	21964	17234	20745	18392
649	27709	17359	11256	13867
650	24352	17815	20863	20351
651	26192	18367	28222	29876
Mean	24316	17216	21840	19579
SD	3138	647	5608	5460
RSD (%)	12.9	3.8	25.7	27.9
Method Detection Limits				
3*SD	9413	1941	16825	16380
MDL (ppb)	0.39	0.11	0.77	0.84

Table B-9. HP 5890/PID detector - method detection limits in water spiked at 1 μg/L (ppb) (analyzed 2-25-92).

Run Number	Area (μV X sec / 8)			
	Benzene	Toluene	Ethylbenzene	*o*-Xylene
1035	10850	9826	9717	10126
1036	10650	10173	10195	10752
1037	11775	11940	11906	13165
1044	12935	11559	11596	11513
1045	13946	12768	12416	12588
1046	12547	11828	11747	12001
1047	11903	10987	10790	12197
1048	11721	12345	12120	13023
1049	10825	8980	8094	8365
1050	11003	9194	8186	8552
1051	11588	10337	9271	9249
Mean	11796	10903	10549	11048
SD	972	1227	1491	1673
RSD (%)	8.2	11.3	14.1	15.1
Method Detection Limits				
3*SD	2917	3861	4472	5020
MDL (ppb)	0.25	0.34	0.42	0.45

Table B-10. HP 5890/PID detector - method detection limits in water spiked at 1 μg/L (ppb) (analyzed 3-13-92).

Port Number	Area (μV X sec / 8)			
	Benzene	Toluene	Ethylbenzene	*o*-Xylene
1	164449	159916	139537	135173
2	139872	135061	119468	117475
3	149744	145185	127832	127576
4	147412	141334	127001	124080
5	150194	145009	127682	128535
6	145834	139233	120861	122467
7	152719	143833	125823	125822
8	157745	144081	126124	125652
9	148831	140996	121540	122972
10	157991	141619	124077	121615
11	147731	143255	124178	123363
12	148059	143060	123665	121287
13	142816	135900	120035	117943
14	147917	140173	119087	119240
15	151058	141917	120680	121075
Mean	150158	142711	124507	123618
SD	5965	5415	4968	4392
RSD (%)	4.0	3.8	4.0	3.6

Table B-11. HP 5890/PID detector - precision study replicates analyzed at 20 μg/L (analyzed 1-09-92).

APPENDIX C

MODIFIED PROCEDURE FOR PREPARATION OF TRIP AUDIT/PERFORMANCE AUDIT SAMPLES

MODIFIED PROCEDURE FOR PREPARATION OF TRIP AUDIT/PERFORMANCE AUDIT SAMPLES

1. Place 1.1 L of deionized water in a glass bottle and chill in ice to 4°C.

2. Prepare 10 ppm BTX standard as per SOP in the Demonstration Plan.

3. Label one clean 250-mL volumetric flask as the 2.5 ppb standard. Fill flask 9/10 full with ice-cold deionized water.

4. With a 100-μL (gas-tight) syringe, transfer 62.5-μL of the 10 ppm standard into the flask and bring the volume to the mark with ice-cold deionized water. While pipetting, keep the syringe tip under water.

5. Quickly stopper the flask. Wrap Parafilm around the stopper seal and thoroughly mix the contents by repeated inversion. Make sure the solution is well mixed.

6. Transfer the contents to four clean, appropriately labelled* VOA vials and quickly cap each vial and place on ice in a cooler.

7. For the 25 ppb standard, proceed as in steps 3 through 6 above with the exception that in step 3 the 250-mL volumetric flask is labelled as the 25 ppb standard and in step 4, 625 μL of the 10 ppm standard is pipetted into the flask using a 1.0-mL (gas-tight) syringe. While pipetting, keep the syringe under water.

8. For the 100 ppb standard, proceed as in steps 3 through 6 above except that in step 3 the 250-mL volumetric flask is labelled as the 100 ppb standard and in step 4, 2.5 mL of the 10 ppm standard is pipetted into the flask using a 2-mL graduated glass pipet.

9. Assay two of the sets of three standards (2.5, 25, and 100 ppb) as the first and last runs of the day in the BTX immunoassay field analysis.

10. Transport the other two sets of standards back to the laboratory in the cooler, along with the other field samples. Store the sets of standards at 4°C.

11. Within 7 days of the collection date, analyze one set of standards by the BTX immunoassay in the laboratory and analyze the other set by GC using EPA Method 8020.

* Sample codes for these samples are: W1 for 2.5 ppb sample
W2 for 25 ppb sample
W3 for 100 ppb sample
Run code for the field analysis at the beginning of the day is 01, and 02 for the field analysis at the end of the day.

Additional supplies needed:

5 -- 250-mL volumetric flasks with stoppers.
2 -- 2-mL graduated glass pipets.
1 -- 100-μL (gas-tight) syringe.

APPENDIX D

FIELD AUDIT REPORT

FIELD AUDIT REPORT
ANTOX BTX WATER SCREEN

by

Neal Amick

1. SUMMARY

An on-site evaluation of field operations in support of the Superfund Innovative Technology Evaluation (SITE) for Antox BTX water screen was conducted on April 16, 1992. Sample collection procedures, field analytical procedures, and data documentation were carefully observed. Overall, the field work was of very good quality, and nothing was observed which would adversely effect the study results. Only minor deviations from the Demonstration Plan were observed, which are recorded in this report.

2. PERSONNEL AND EQUIPMENT

The personnel involved and the supporting field equipment were more than adequate to fulfill the requirements of the study. The operation was organized and neat. All supplies required for the study were present and set up or stored in designated spaces.

3. SAMPLING PROCEDURES

Sampling procedures were performed well and according to the plan. Samples were stored on ice in coolers. Temperature of the coolers were checked regularly.

4. FIELD PROCEDURES

The field analyst took ultimate care that the procedure was performed precisely the same way every time. The field kit standard operating procedure was followed to the letter for the analysis of samples. Minor differences were noted in the procedures for preparing audit samples between those in the Demonstration Plan and as performed in the field. For example, the plan calls for preparing audit samples by diluting a stock solution of BTX into 4 mL of distilled water. Instead, the stock solution was diluted into volumetric flasks. From the plan, one might assume the audit sample contained benzene, toluene, and xylene (BTX). Only toluene was used for a spike for the audit samples. The ampulated stock solution used for preparing the audit samples was 1000 ppm, instead of 10 ppm as stated in the plan. This 1000 ppm solution was diluted in two steps (one to ten dilutions) to obtain the 10 ppm stock solution.

These small changes in procedure should not influence the result of the study.

5. DOCUMENTATION

All facets of the field study were well documented. Chain-of-Custody forms were completed in a timely manner after sample collection. The field data form as provided in the Demonstration Plan was not used; however, all information provided by that form was recorded either in the sample data log sheet, or in a field logbook.

Samples were labeled with identification codes as specified in the Demonstration Plan, except the standards were labeled as W1, W2, and W3 instead of P1, P2, and P3.

6. CONCLUSION

All observations made during this field audit indicate good quality data was obtained.

Figure D-1 is a copy of the audit sheet completed in the field.

ON-SITE AUDIT CHECKLIST FORM (F-2)

LOCATION:	YES	YES, WITH NUMEROUS EXCEPTIONS	YES, WITH FEW EXCEPTIONS	NO	PROBLEM
Field Forms Filled Out Completely And Accurately			X		Form not the same as [illegible]
Field Kit SOP Followed	X				
QC Acceptance Criteria Observed	X				
Sampling And Analysis Plan Followed			X		Some deviations from [illegible]
Sample Handling, Labeling, Tracking And Archiving Done According To Instructions	X				
Sample Packaging And Shipment Handled Properly	X				
Safety Observed	X				
Cleanliness, Adherence To GLP Observed	X				

Auditor's Signature [illegible] Date 4-16-92

BTEX Sample Collection Tube Form

Figure D-1. Field audit data sheet completed by auditor.

APPENDIX E

PARTICIPATING PERSONNEL

PARTICIPATING PERSONNEL

Many individuals from various organizations contributed to the success of the BTX immunoassay demonstration and participated in the preparation of this report. Their names, organizations, and contributions are provided below.

Responsibility	Name	Affiliation*
Authors	R. W. Gerlach	Lockheed
	R. J. White	Lockheed
	N. F. D. O' Leary	Lockheed
	J. M. Van Emon	U.S. EPA
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	R. Piasio	ANTOX
	N. F. D. O' Leary	Lockheed
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Quality Assurance	V. A. Ecker	Lockheed
	L. R. Williams	U.S. EPA
Experimental Design	R. W. Gerlach	Lockheed
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Logistical Support	D. Alford	CECS
	E. Noack	CECS
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	J. H. Zimmerman	Lockheed
	M. Brand	CECS
Analytical Support	R. J. White	Lockheed
	P. A. Amick	Lockheed
	E. N. Amick	Lockheed
Data Base Management	G. I. Martucci	Lockheed
Statistical Analysis	R. W. Gerlach	Lockheed

Graphics	S. O. Garcia	Lockheed

* ANTOX	Antox Inc., South Portland, ME.
CECS	Converse Environmental Consultants Southwest, Las Vegas, NV.
U.S. EPA	U.S. Environmental Protection Agency, Las Vegas, NV.

APPENDIX F

DATA TABLES FOR FIELD IMMUNOASSAY, LABORATORY IMMUNOASSAY, AND GAS CHROMATOGRAPY ANALYSES

List of Tables

Table — page

F-1. Data for field immunoassay analysis 75
F-2. Data for laboratory immunoassay analysis 82
F-3. Data for gas chromatography analysis 87

Abbreviations

*	exceeds calibration
B	benzene
C	co-elution
DIL	dilution
DCE	*cis*-1,2-dichloroethene
FB	fluorobenzene
E	ethylbenzene
nd	not detected
T	toluene
X	xylene

Sample ID Number coding scheme for the BTX SITE demonstration:

Location of Analysis	F	= Field
	L	= Laboratory
Analysis Type	G	= GC
	I	= Immunoassay
Site Identification	A	= Site A
	B	= Site B, etc.
Water Type	01	= Well 1
	02	= Well 2, etc.
	P1	= 2.5 ppb Standard
	P2	= 25 ppb Standard
	P3	= 100 ppb Standard
	TB	= Trip Blank
	TL	= Trip Audit Low
	TH	= Trip Audit High
	W1	= 2.5 ppb Audit Sample
	W2	= 25 ppb Audit Sample
	W3	= 100 ppb Audit Sample
Replicate Number	01	= 1st Analysis
	02	= 2nd Analysis, etc.

Example = FIA0101

F = Field Location
I = Immunoassay Analysis
A = Site A
01 = Well Number 1
01 = Replicate Number 1

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
1/22/92	FIA0401	1.746	1.854	1.06		
1/22/92	FIA0402	1.836	1.961	1.07		
1/22/92	FIA0403	1.809	>2		*	
1/22/92	FIA0601	1.746	0.517	0.30		
1/22/92	FIA0602	1.836	0.742	0.40		
1/22/92	FIA0603	1.809	0.794	0.44		
1/22/92	FIA0701	1.408	1.600	1.14		
1/22/92	FIA0702	1.984	1.993	1.00		
1/22/92	FIA0703	1.865	1.972	1.06		
1/22/92	FIA0901	1.408	0.539	0.38		
1/22/92	FIA0902	1.984	0.802	0.40		
1/22/92	FIA0903	1.865	1.041	0.56		
1/22/92	FIA1001	1.746	1.947	1.12		
1/22/92	FIA1002	1.836	1.947	1.06		
1/22/92	FIA1003	1.809	1.963	1.09		
1/22/92	FIA1101	1.408	1.467	1.04		
1/22/92	FIA1102	1.984	1.890	0.95		
1/22/92	FIA1103	1.865	1.280	0.69		
1/22/92	FIAP101	1.506	1.387	0.92		
1/22/92	FIAP102	1.880	1.907	1.01		
1/22/92	FIAP201	1.506	1.184	0.79		
1/22/92	FIAP202	1.880	1.849	0.98		
1/22/92	FIAP301	1.506	0.753	0.50		
1/22/92	FIAP302	1.880	1.356	0.72		
1/23/92	FIA0101	1.766	0.383	0.22		
1/23/92	FIA0102	1.900	0.416	0.22		
1/23/92	FIA0201	1.766	1.869	1.06		
1/23/92	FIA0202	1.900	1.905	1.00		
1/23/92	FIA0301	1.972	1.039	0.53		
1/23/92	FIA0302	1.898	0.712	0.38		
1/23/92	FIA0501	1.766	1.912	1.08		
1/23/92	FIA0502	1.900	1.807	0.95		
1/23/92	FIA0801	1.972	>2		*	
1/23/92	FIA0802	1.898	1.992	1.05		
1/23/92	FIAP103	1.319	1.451	1.10		
1/23/92	FIAP104	1.977	1.981	1.00		
1/23/92	FIAP203	1.319	1.255	0.95		
1/23/92	FIAP204	1.977	1.809	0.92		
1/23/92	FIAP303	1.319	0.876	0.66		
1/23/92	FIAP304	1.977	1.592	0.81		
2/24/92	FIA0101	1.640	0.434	0.26		
2/24/92	FIA0102	1.754	0.434	0.25		
2/24/92	FIA0103	1.817	0.484	0.27		
2/24/92	FIA0201	1.440	1.580	1.10		
2/24/92	FIA0202	1.847	1.833	0.99		

Table F-1. Data for field immunoassay analysis. (Page 1 of 7)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
2/24/92	FIA0203	1.838	1.869	1.02		
2/24/92	FIA0301	1.640	0.940	0.57		
2/24/92	FIA0302	1.754	1.078	0.61		
2/24/92	FIA0303	1.817	0.976	0.54		
2/24/92	FIA0401	1.440	1.378	0.96		
2/24/92	FIA0402	1.847	1.869	1.01		
2/24/92	FIA0403	1.838	1.889	1.03		
2/24/92	FIA0501	1.440	1.715	1.19		
2/24/92	FIA0502	1.847	1.874	1.01		
2/24/92	FIA0503	1.838	1.775	0.97		
2/24/92	FIA0601	1.640	0.925	0.56		
2/24/92	FIA0602	1.754	0.901	0.51		
2/24/92	FIA0603	1.817	0.855	0.47		
2/24/92	FIA0701	1.640	1.570	0.96		
2/24/92	FIA0702	1.754	1.754	1.00		
2/24/92	FIA0703	1.817	1.500	0.83		
2/24/92	FIAP101	1.348	1.531	1.14		
2/24/92	FIAP102	1.430	1.345	0.94		
2/24/92	FIAP201	1.348	1.373	1.02		
2/24/92	FIAP202	1.430	0.991	0.69		
2/24/92	FIAP301	1.348	1.125	0.83		
2/24/92	FIAP302	1.430	0.732	0.51		
2/25/92	FIA0801	1.837	1.940	1.06		
2/25/92	FIA0802	1.932	1.912	0.99		
2/25/92	FIA0901	1.837	0.353	0.19		
2/25/92	FIA0902	1.932	0.410	0.21		
2/25/92	FIA1001	1.837	1.539	0.84		
2/25/92	FIA1002	1.932	1.601	0.83		
2/25/92	FIA1101	1.837	1.707	0.93		
2/25/92	FIA1102	1.932	1.835	0.95		
2/25/92	FIAP101	1.799	1.75	0.97		
2/25/92	FIAP10X	1.092	1.14	1.04	BAD RUN	
2/25/92	FIAP201	1.799	1.828	1.02		
2/25/92	FIAP20X	1.092	1.529	1.40	BAD RUN	
2/25/92	FIAP301	1.799	1.510	0.84		
2/25/92	FIAP30X	1.092	1.032	0.95	BAD RUN	
2/25/92	FIB0101	1.730	1.507	0.87		
2/25/92	FIB0102	1.769	1.814	1.03		
2/25/92	FIB0201	1.730	1.868	1.08		
2/25/92	FIB0202	1.769	1.882	1.06		
2/25/92	FIBP102	1.794	1.777	0.99		
2/25/92	FIBP202	1.794	1.665	0.93		
2/25/92	FIBP302	1.794	1.440	0.80		
2/26/92	FIB0301	1.952	0.496	0.25		
2/26/92	FIB0302	1.705	0.431	0.25		

Table F-1. Continued. (Page 2 of 7)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
2/26/92	FIB0401	1.844	1.904	1.03		
2/26/92	FIB0402	1.796	1.933	1.08		
2/26/92	FIB0501	1.844	1.455	0.79		
2/26/92	FIB0502	1.796	1.138	0.63		
2/26/92	FIB0701	1.952	1.478	0.76		
2/26/92	FIB0702	1.705	1.277	0.75		
2/26/92	FIB0901	1.952	1.790	0.92		
2/26/92	FIB0902	1.705	1.617	0.95		
2/26/92	FIB1001	1.952	1.179	0.60		
2/26/92	FIB1002	1.705	0.809	0.47		
2/26/92	FIBP101	1.882	1.863	0.99		
2/26/92	FIBP102	1.486	1.504	1.01		
2/26/92	FIBP10X	0.064	0.100	1.56	BAD RUN	
2/26/92	FIBP201	1.882	1.741	0.93		
2/26/92	FIBP202	1.486	1.493	1.00		
2/26/92	FIBP20X	0.064	0.270	4.22	BAD RUN	
2/26/92	FIBP301	1.882	1.357	0.72		
2/26/92	FIBP302	1.486	1.007	0.68		
2/26/92	FIBP30X	0.064	0.344	5.38	BAD RUN	
2/27/92	FIB1101	1.847	1.847	1.00		
2/27/92	FIB1102	1.964	1.909	0.97		
2/27/92	FIB1401	1.951	1.935	0.99		
2/27/92	FIB1402	1.960	1.897	0.97		
2/27/92	FIB1501	1.951	1.919	0.98		
2/27/92	FIB1502	1.960	1.840	0.94		
2/27/92	FIB1601	1.847	1.816	0.98		
2/27/92	FIB1602	1.964	1.862	0.95		
2/27/92	FIB1701	1.813	1.928	1.06		
2/27/92	FIB1702	1.855	1.842	0.99		
2/27/92	FIB1801	1.813	1.882	1.04		
2/27/92	FIB1802	1.855	1.867	1.01		
2/27/92	FIB1901	1.951	1.955	1.00		
2/27/92	FIB1902	1.960	1.894	0.97		
2/27/92	FIB2001	1.847	0.116	0.06		
2/27/92	FIB2002	1.964	0.129	0.07		
2/27/92	FIB2101	1.813	1.873	1.03		
2/27/92	FIB2102	1.855	1.856	1.00		
2/27/92	FIBP101	1.927	1.883	0.98		
2/27/92	FIBP102	1.835	1.668	0.91		
2/27/92	FIBP10X	0.477	0.554	1.16	BAD RUN	
2/27/92	FIBP201	1.927	1.677	0.87		
2/27/92	FIBP202	1.835	1.662	0.91		
2/27/92	FIBP20X	0.477	0.784	1.64	BAD RUN	
2/27/92	FIBP301	1.927	1.252	0.65		
2/27/92	FIBP302	1.835	1.320	0.72		

Table F-1. Continued.(Page 3 of 7)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
2/27/92	FIBP30X	0.477	0.627	1.31	BAD RUN	
2/28/92	FIB0801	1.875	0.938	0.50		
2/28/92	FIB0802	1.882	0.895	0.48		
2/28/92	FIB1201	1.875	1.655	0.88		
2/28/92	FIB1202	1.882	1.505	0.80		
2/28/92	FIB1203	1.820	1.391	0.76		
2/28/92	FIB1301	1.875	1.907	1.02		
2/28/92	FIB1302	1.882	1.746	0.93		
2/28/92	FIBP101	1.559	1.809	1.16		
2/28/92	FIBP102	1.820	1.720	0.95		
2/28/92	FIBP201	1.559	1.656	1.06		
2/28/92	FIBP202	1.820	1.578	0.87		
2/28/92	FIBP301	1.559	1.331	0.85		
2/28/92	FIBP302	1.820	1.238	0.68		
3/9/92	FIC1601	1.696	0.099	0.06		
3/9/92	FIC1602	1.452	0.097	0.07		
3/9/92	FIC1701	1.696	0.129	0.08		
3/9/92	FIC1702	1.452	0.068	0.05		
3/9/92	FIC1801	1.696	0.315	0.19		
3/9/92	FIC1802	1.452	0.223	0.15		
3/9/92	FICW101	1.666	1.708	1.03		
3/9/92	FICW102	1.812	1.668	0.92		
3/9/92	FICW201	1.666	1.562	0.94		
3/9/92	FICW202	1.812	1.522	0.84		
3/9/92	FICW301	1.666	1.434	0.86		
3/9/92	FICW302	1.812	1.352	0.75		
3/10/92	FIC0801	1.856	0.247	0.13		
3/10/92	FIC0802	1.629	0.177	0.11		
3/10/92	FIC1001	1.856	0.129	0.07		
3/10/92	FIC1002	1.629	0.142	0.09		
3/10/92	FICW101	1.85	1.888	1.02		
3/10/92	FICW102	1.823	1.729	0.95		
3/10/92	FICW102	1.896	1.855	0.98		
3/10/92	FICW201	1.85	1.833	0.99		
3/10/92	FICW202	1.823	1.566	0.86		
3/10/92	FICW202	1.896	1.75	0.92		
3/10/92	FICW301	1.85	1.683	0.91		
3/10/92	FICW302	1.823	1.482	0.81		
3/10/92	FICW302	1.896	1.394	0.74		
3/11/92	FIC0401	1.794	1.703	0.95		
3/11/92	FIC0402	1.548	1.357	0.88		
3/11/92	FIC0901	1.794	1.68	0.94		
3/11/92	FIC0902	1.548	1.246	0.80		
3/11/92	FIC0903	1.692	1.485	0.88		
3/11/92	FIC2301	1.794	1.273	0.71		

Table F-1. Continued.(Page 4 of 7)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
3/11/92	FIC2302	1.548	1.014	0.66		
3/11/92	FIC2401	1.794	1.693	0.94		
3/11/92	FIC2402	1.548	1.498	0.97		
3/11/92	FIC2601	1.794	0.1	0.06		
3/11/92	FIC2602	1.548	0.06	0.04		
3/11/92	FICW101	1.89	1.887	1.00		
3/11/92	FICW102	1.692	1.756	1.04		
3/11/92	FICW201	1.89	1.867	0.99		
3/11/92	FICW202	1.692	1.392	0.82		
3/11/92	FICW301	1.89	1.615	0.85		
3/11/92	FICW302	1.692	1.241	0.73		
3/12/92	FIC0601	1.87	0.819	0.44		
3/12/92	FIC0602	1.884	0.817	0.43		
3/12/92	FIC0701	1.87	1.846	0.99		
3/12/92	FIC0702	1.884	1.831	0.97		
3/12/92	FIC1101	1.87	0.154	0.08		
3/12/92	FIC1102	1.884	0.219	0.12		
3/12/92	FICW101	1.891	1.938	1.02		
3/12/92	FICW102	1.725	1.823	1.06		
3/12/92	FICW102	1.88	1.899	1.01		
3/12/92	FICW201	1.891	1.949	1.03		
3/12/92	FICW202	1.725	1.772	1.03		
3/12/92	FICW202	1.88	1.795	0.95		
3/12/92	FICW301	1.891	1.665	0.88		
3/12/92	FICW302	1.725	1.363	0.79		
3/12/92	FICW302	1.88	1.632	0.87		
3/13/92	FIC0201	1.857	0.306	0.16		
3/13/92	FIC0202	1.937	0.48	0.25		
3/13/92	FIC0301	1.857	0.141	0.08		
3/13/92	FIC0302	1.937	0.161	0.08		
3/13/92	FIC0501	1.857	1.129	0.61		
3/13/92	FIC0502	1.937	1.271	0.66		
3/13/92	FICW101	1.971	1.998	1.01		
3/13/92	FICW102	1.934	1.948	1.01		
3/13/92	FICW102	1.943	1.922	0.99		
3/13/92	FICW201	1.971	1.936	0.98		
3/13/92	FICW202	1.934	1.835	0.95		
3/13/92	FICW202	1.943	1.837	0.95		
3/13/92	FICW301	1.971	1.726	0.88		
3/13/92	FICW302	1.934	1.573	0.81		
3/13/92	FICW302	1.943	1.778	0.92		
4/15/92	FIA0101	1.773	0.03	0.02		
4/15/92	FIA0102	1.968	0.065	0.03		
4/15/92	FIA0201	1.773	1.914	1.08		
4/15/92	FIA0202	1.968	1.975	1.00		

Table F-1. Continued. (Page 5 of 7)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
4/15/92	FIA0301	1.773	0.142	0.08		
4/15/92	FIA0302	1.968	0.19	0.10		
4/15/92	FIA0401	1.773	1.868	1.05		
4/15/92	FIA0402	1.968	1.914	0.97		
4/15/92	FIA0501	1.773	1.81	1.02		
4/15/92	FIA0502	1.968	1.86	0.95		
4/15/92	FIAW101	1.862	1.883	1.01		
4/15/92	FIAW102	1.777	1.863	1.05		
4/15/92	FIAW201	1.862	1.848	0.99		
4/15/92	FIAW202	1.777	1.767	0.99		
4/15/92	FIAW301	1.862	1.481	0.80		
4/15/92	FIAW302	1.777	1.518	0.85		
4/16/92	FIA0601	1.95	0.542	0.28		
4/16/92	FIA0602	1.935	0.485	0.25		
4/16/92	FIA0701	1.95	>2		*	
4/16/92	FIA0702	1.935	>2		*	
4/16/92	FIA0703	1.944	1.785	0.92		
4/16/92	FIA0801	1.974	>2		*	
4/16/92	FIA0802	0.658	1.425	2.17	BAD RUN	
4/16/92	FIA0803	1.877	1.936	1.03		
4/16/92	FIA0804	1.375	1.312	0.95	DIL 1:2	
4/16/92	FIA0901	1.974	0.164	0.08		
4/16/92	FIA0902	0.658	0.131	0.20	BAD RUN	
4/16/92	FIA0903	1.877	0.107	0.06		
4/16/92	FIA0904	1.375	0.057	0.04	DIL 1:2	
4/16/92	FIA1001	1.974	1.063	0.54		
4/16/92	FIA1002	0.658	0.536	0.81	BAD RUN	
4/16/92	FIA1003	1.877	0.818	0.44		
4/16/92	FIA1004	1.375	0.539	0.39	DIL 1:2	
4/16/92	FIA1101	1.95	>2		*	
4/16/92	FIA1102	1.935	1.992	1.03		
4/16/92	FIA1103	1.944	1.836	0.94		
4/16/92	FIAW101	1.91	1.928	1.01		
4/16/92	FIAW102	1.993	1.934	0.97		
4/16/92	FIAW201	1.91	1.831	0.96		
4/16/92	FIAW202	1.993	1.738	0.87		
4/16/92	FIAW301	1.91	1.558	0.82		
4/16/92	FIAW302	1.993	1.557	0.78		
4/17/92	FID1001	1.978	1.992	1.01		
4/17/92	FID1002	1.913	1.994	1.04		
4/17/92	FID1101	1.978	1.312	0.66		
4/17/92	FID1102	1.913	1.531	0.80		
4/17/92	FID1201	1.978	1.955	0.99		
4/17/92	FID1202	1.067	1.409	1.32	DIL	
4/17/92	FIDW101	1.917	1.771	0.92		

Table F-1. Continued. (Page 6 of 7)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
4/17/92	FIDW102	1.948	1.901	0.98		
4/17/92	FIDW201	1.917	1.713	0.89		
4/17/92	FIDW202	1.948	1.755	0.90		
4/17/92	FIDW301	1.917	1.203	0.63		
4/17/92	FIDW302	1.948	1.553	0.80		
4/20/92	FID0101	1.905	1.945	1.02		
4/20/92	FID0101X	2	1.933		*	
4/20/92	FID0102	1.952	1.865	0.96		
4/20/92	FID0201	1.905	0.788	0.41		
4/20/92	FID0201X	2	0.696		*	
4/20/92	FID0202	1.952	0.911	0.47		
4/20/92	FID0301	1.905	0.278	0.15		
4/20/92	FID0301X	2	0.269		*	
4/20/92	FID0302	1.952	0.29	0.15		
4/20/92	FID9901	1.905	1.685	0.88		
4/20/92	FID9901X	2	1.081		*	
4/20/92	FID9902	1.952	1.673	0.86		
4/20/92	FIDW101	1.907	1.871	0.98		
4/20/92	FIDW102	1.985	1.987	1.00		
4/20/92	FIDW201	1.907	1.73	0.91		
4/20/92	FIDW202	1.985	1.866	0.94		
4/20/92	FIDW301	1.907	1.166	0.61		
4/20/92	FIDW302	1.985	1.722	0.87		
4/21/92	FID0501	1.975	0.861	0.44		
4/21/92	FID0502	1.825	0.756	0.41		
4/21/92	FID0801	1.975	1.927	0.98		
4/21/92	FID0802	1.825	1.973	1.08		
4/21/92	FID0901	1.975	1.949	0.99		
4/21/92	FID0902	1.825	1.979	1.08		
4/21/92	FIDW101	1.938	1.948	1.01		
4/21/92	FIDW102	1.944	1.92	0.99		
4/21/92	FIDW201	1.938	1.839	0.95		
4/21/92	FIDW202	1.944	1.9	0.98		
4/21/92	FIDW301	1.938	1.368	0.71		
4/21/92	FIDW302	1.944	1.563	0.80		
4/22/92	FID0401	1.792	0.981	0.55		
4/22/92	FID0402	1.998	1.263	0.63		
4/22/92	FID0701	1.792	0.013	0.01		
4/22/92	FID0702	1.998	0.045	0.02		
4/22/92	FIDW101	1.91	1.817	0.95		
4/22/92	FIDW102	1.887	1.835	0.97		
4/22/92	FIDW201	1.91	1.778	0.93		
4/22/92	FIDW202	1.887	1.803	0.96		
4/22/92	FIDW301	1.91	1.36	0.71		
4/22/92	FIDW302	1.887	1.558	0.83		

Table F-1. Continued. (Page 7 of 7)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
1/22/92	LIA0401	1.875	1.900	1.01		1/27/92
1/22/92	LIA0402	1.795	1.862	1.04		1/27/92
1/22/92	LIA0601	1.875	0.648	0.35		1/27/92
1/22/92	LIA0602	1.795	0.810	0.45		1/27/92
1/22/92	LIA0701	1.924	>2		*	1/27/92
1/22/92	LIA0901	1.924	0.94	0.49		1/27/92
1/23/92	LIA0101	1.875	1.102	0.59		1/28/92
1/23/92	LIA0101	1.941	0.492	0.25		1/29/92
1/23/92	LIA0102	0.505	0.526	1.04	BAD RUN	1/28/92
1/23/92	LIA0102	1.672	0.605	0.36		1/29/92
1/23/92	LIA0201	1.875	1.946	1.04		1/28/92
1/23/92	LIA0201	1.941	1.888	0.97		1/29/92
1/23/92	LIA0202	0.505	1.793	3.55	BAD RUN	1/28/92
1/23/92	LIA0202	1.672	1.897	1.13		1/29/92
1/23/92	LIA0301	1.875	1.163	0.62		1/28/92
1/23/92	LIA0301	1.941	0.736	0.38		1/29/92
1/23/92	LIA0302	0.505	0.795	1.57	BAD RUN	1/28/92
1/23/92	LIA0302	1.672	0.735	0.44		1/29/92
1/23/92	LIATB01	1.803	1.621	0.90		1/28/92
1/23/92	LIATB02	1.406	1.258	0.89		1/28/92
1/23/92	LIATH01	1.803	0.949	0.53		1/28/92
1/23/92	LIATH02	1.406	1.037	0.74		1/28/92
1/23/92	LIATL01	1.803	1.479	0.82		1/28/92
1/23/92	LIATL02	1.406	1.236	0.88		1/28/92
1/27/92	LIAP101	1.962	1.875	0.96		1/27/92
1/27/92	LIAP102	1.924	1.890	0.98		1/27/92
1/27/92	LIAP201	1.962	1.854	0.94		1/27/92
1/27/92	LIAP202	1.924	1.689	0.88		1/27/92
1/27/92	LIAP301	1.962	1.579	0.80		1/27/92
1/27/92	LIAP302	1.924	1.500	0.78		1/27/92
1/27/92	LIATH01	1.875	1.451	0.77		1/27/92
1/27/92	LIATH02	1.795	1.515	0.84		1/27/92
1/27/92	LIATL01	1.875	1.636	0.87		1/27/92
1/27/92	LIATL02	1.795	1.606	0.89		1/27/92
1/28/92	LIAP101	1.785	1.787	1.00		1/28/92
1/28/92	LIAP201	1.785	1.617	0.91		1/28/92
1/28/92	LIAP301	1.785	1.582	0.89		1/28/92
1/29/92	LIAP101	1.788	1.452	0.81		1/29/92
1/29/92	LIAP201	1.788	1.305	0.73		1/29/92
1/29/92	LIAP301	1.788	0.981	0.55		1/29/92
2/24/92	LIA0101	0.818	0.238	0.29		3/3/92
2/24/92	LIA0102	1.738	0.590	0.34		3/3/92
2/24/92	LIA0201	1.812	1.840	1.02		3/3/92
2/24/92	LIA0202	1.686	1.824	1.08		3/3/92
2/24/92	LIA0701	1.812	1.729	0.95		3/3/92
2/24/92	LIA0702	1.686	1.179	0.70		3/3/92
2/24/92	LIATB01	0.818	1.110	1.36		3/3/92
2/24/92	LIATB02	1.738	1.805	1.04		3/3/92
2/24/92	LIATH01	0.818	0.443	0.54		3/3/92
2/24/92	LIATH02	1.738	1.168	0.67		3/3/92

Table F-2. Data for laboratory immunoassay analysis. (Page 1 of 5)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
2/24/92	LIATL01	0.818	0.686	0.84		3/3/92
2/24/92	LIATL02	1.738	1.733	1.00		3/3/92
2/25/92	LIA1001	1.812	1.696	0.94		3/3/92
2/25/92	LIA1002	1.686	1.475	0.87		3/3/92
2/25/92	LIATB01	1.918	1.870	0.97		3/3/92
2/25/92	LIATB02	1.835	1.909	1.04		3/3/92
2/25/92	LIATH01	1.918	1.598	0.83		3/3/92
2/25/92	LIATH02	1.835	1.551	0.85		3/3/92
2/25/92	LIATL01	1.918	1.703	0.89		3/3/92
2/25/92	LIATL02	1.835	1.621	0.88		3/3/92
2/26/92	LIB0301	1.270	0.406	0.32		3/3/92
2/26/92	LIB0302	1.580	0.612	0.39		3/3/92
2/26/92	LIB0401	1.593	1.580	0.99		3/3/92
2/26/92	LIB0402	1.566	1.778	1.14		3/3/92
2/26/92	LIBTB01	1.270	1.535	1.21		3/3/92
2/26/92	LIBTB02	1.580	1.579	1.00		3/3/92
2/26/92	LIBTH01	1.270	1.071	0.84		3/3/92
2/26/92	LIBTH02	1.580	1.280	0.81		3/3/92
2/26/92	LIBTL01	1.270	1.309	1.03		3/3/92
2/26/92	LIBTL02	1.580	1.467	0.93		3/3/92
2/27/92	LIB2001	1.593	0.103	0.06		3/3/92
2/27/92	LIB2002	1.566	0.101	0.06		3/3/92
2/27/92	LIBTB01	1.694	1.656	0.98		3/3/92
2/27/92	LIBTB02	1.688	1.675	0.99		3/3/92
2/27/92	LIBTH01	1.694	1.188	0.70		3/3/92
2/27/92	LIBTH02	1.688	1.358	0.80		3/3/92
2/27/92	LIBTL01	1.694	1.303	0.77		3/3/92
2/27/92	LIBTL02	1.688	1.454	0.86		3/3/92
2/28/92	LIB1201	1.593	0.932	0.59		3/3/92
2/28/92	LIB1202	1.566	0.932	0.60		3/3/92
2/28/92	LIBTB01	-0.025	0.593	-23.72	BAD TUBE	3/3/92
2/28/92	LIBTB02	0.852	1.051	1.23		3/3/92
2/28/92	LIBTH01	-0.025	0.855	-34.20	BAD TUBE	3/3/92
2/28/92	LIBTH02	0.852	0.581	0.68		3/3/92
2/28/92	LIBTL01	-0.025	0.392	-15.68	BAD TUBE	3/3/92
2/28/92	LIBTL02	0.852	0.488	0.57		3/3/92
3/3/92	LIAP101	1.220	1.349	1.11		3/3/92
3/3/92	LIAP201	1.220	1.246	1.02		3/3/92
3/3/92	LIAP301	1.220	0.888	0.73		3/3/92
3/9/92	LIC1701	1.593	0.051	0.03		3/19/92
3/9/92	LIC1702	1.666	0.054	0.03		3/19/92
3/9/92	LICW101	1.593	1.404	0.88		3/19/92
3/9/92	LICW102	1.666	1.481	0.89		3/19/92
3/9/92	LICW201	1.593	1.333	0.84		3/19/92
3/9/92	LICW202	1.666	1.359	0.82		3/19/92
3/9/92	LICW301	1.593	1.016	0.64		3/19/92
3/9/92	LICW302	1.666	1.018	0.61		3/19/92
3/9/92	LITB101	1.593	1.473	0.92		3/19/92
3/9/92	LITB102	1.666	1.638	0.98		3/19/92
3/10/92	LIC1001	1.657	0.038	0.02		3/18/92

Table F-2. Continued. (Page 2 of 5)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
3/10/92	LIC1002	1.689	0.094	0.06		3/18/92
3/10/92	LICW101	1.657	1.514	0.91		3/18/92
3/10/92	LICW102	1.689	1.648	0.98		3/18/92
3/10/92	LICW201	1.657	1.52	0.92		3/18/92
3/10/92	LICW202	1.689	1.463	0.87		3/18/92
3/10/92	LICW301	1.657	1.13	0.68		3/18/92
3/10/92	LICW302	1.689	1.144	0.68		3/18/92
3/10/92	LITB101	1.657	1.478	0.89		3/18/92
3/10/92	LITB102	1.689	1.636	0.97		3/18/92
3/11/92	LIC0901	1.439	1.272	0.88		3/19/92
3/11/92	LIC0902	1.486	1.232	0.83		3/19/92
3/11/92	LIC2301	1.439	0.88	0.61		3/19/92
3/11/92	LIC2302	1.486	0.992	0.67		3/19/92
3/11/92	LICW201	1.439	1.114	0.77	ICY	3/19/92
3/11/92	LICW202	1.486	1.355	0.91		3/19/92
3/11/92	LICW301	1.439	0.979	0.68	ICY	3/19/92
3/11/92	LICW302	1.486	0.893	0.60		3/19/92
3/12/92	LICW101	1.374	1.456	1.06		3/18/92
3/12/92	LICW102	1.701	1.562	0.92		3/18/92
3/12/92	LICW201	1.374	1.164	0.85		3/18/92
3/12/92	LICW202	1.701	1.368	0.80		3/18/92
3/12/92	LICW301	1.374	0.906	0.66		3/18/92
3/12/92	LICW302	1.701	0.946	0.56		3/18/92
3/12/92	LITB101	1.374	1.339	0.97		3/18/92
3/12/92	LITB102	1.701	1.603	0.94		3/18/92
3/13/92	LIC0301	1.621	0.063	0.04		3/19/92
3/13/92	LIC0302	1.493	0.068	0.05		3/19/92
3/13/92	LICW101	1.621	1.527	0.94		3/19/92
3/13/92	LICW102	1.493	1.478	0.99	ICY	3/19/92
3/13/92	LICW201	1.621	1.039	0.64		3/19/92
3/13/92	LICW202	1.493	1.411	0.95	ICY	3/19/92
3/13/92	LICW301	1.621	0.958	0.59		3/19/92
3/13/92	LICW302	1.493	1.1	0.74	ICY	3/19/92
3/13/92	LITB101	1.621	1.714	1.06		3/19/92
3/13/92	LITB102	1.493	1.615	1.08	ICY	3/19/92
3/18/92	LICW101	1.4	1.718	1.23		3/18/92
3/18/92	LICW102	1.529	1.506	0.98		3/18/92
3/18/92	LICW201	1.4	1.271	0.91		3/18/92
3/18/92	LICW202	1.529	1.375	0.90		3/18/92
3/18/92	LICW301	1.4	1.099	0.79		3/18/92
3/18/92	LICW302	1.529	1.226	0.80		3/18/92
3/19/92	LICW101	1.594	1.547	0.97		3/19/92
3/19/92	LICW102	1.635	1.679	1.03		3/19/92
3/19/92	LICW201	1.594	1.377	0.86		3/19/92
3/19/92	LICW202	1.635	1.503	0.92		3/19/92
3/19/92	LICW301	1.594	1.382	0.87		3/19/92
3/19/92	LICW302	1.635	1.538	0.94		3/19/92
4/15/92	LIA0301	1.82	0.205	0.11		4/27/92
4/15/92	LIA0302	1.827	0.167	0.09		4/27/92
4/15/92	LIA0501	1.82	1.715	0.94		4/27/92

Table F-2. Continued. (Page 3 of 5)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
4/15/92	LIA0502	1.827	1.890	1.03		4/27/92
4/15/92	LIDTB01	1.678	1.234	0.74		4/23/92
4/15/92	LIDTB02	1.47	1.502	1.02		4/23/92
4/15/92	LIDTB03	1.866	1.883	1.01		4/23/92
4/15/92	LIDW101	1.678	1.159	0.69		4/23/92
4/15/92	LIDW102	1.47	1.458	0.99		4/23/92
4/15/92	LIDW103	1.866	1.774	0.95		4/23/92
4/15/92	LIDW201	1.678	1.283	0.76		4/23/92
4/15/92	LIDW202	1.47	1.251	0.85		4/23/92
4/15/92	LIDW203	1.866	1.868	1.00		4/23/92
4/15/92	LIDW301	1.678	1.007	0.60		4/23/92
4/15/92	LIDW302	1.47	0.905	0.62		4/23/92
4/15/92	LIDW303	1.866	1.226	0.66		4/23/92
4/16/92	LIA0601	1.836	0.324	0.18		4/27/92
4/16/92	LIA0602	1.924	0.362	0.19		4/27/92
4/16/92	LIATB01	1.866	1.872	1.00		4/23/92
4/16/92	LIAW101	1.866	1.836	0.98		4/23/92
4/16/92	LIAW201	1.866	1.743	0.93		4/23/92
4/16/92	LIAW301	1.866	1.320	0.71		4/23/92
4/16/92	LIDTB02	1.849	1.829	0.99		4/23/92
4/16/92	LIDW101	1.849	1.714	0.93		4/23/92
4/16/92	LIDW202	1.849	1.688	0.91		4/23/92
4/16/92	LIDW302	1.849	1.196	0.65		4/23/92
4/17/92	LID2301	1.918	1.237	0.64		4/27/92
4/17/92	LIDTB01	1.918	1.766	0.92		4/27/92
4/17/92	LIDTB02	1.907	1.766	0.93		4/27/92
4/17/92	LIDW101	1.918	1.906	0.99		4/27/92
4/17/92	LIDW102	1.907	1.764	0.93		4/27/92
4/17/92	LIDW201	1.918	1.844	0.96		4/27/92
4/17/92	LIDW202	1.907	1.643	0.86		4/27/92
4/17/92	LIDW302	1.907	1.145	0.60		4/27/92
4/20/92	LID0301	1.82	0.195	0.11		4/27/92
4/20/92	LID0302	1.827	0.255	0.14		4/27/92
4/20/92	LID9901	1.836	1.598	0.87		4/27/92
4/20/92	LID9902	1.924	1.414	0.73		4/27/92
4/20/92	LID9903	1.82	1.458	0.80		4/27/92
4/20/92	LIDTB01	1.607	1.142	0.71	BAD TUBE	4/23/92
4/20/92	LIDTB02	1.948	1.996	1.02		4/23/92
4/20/92	LIDTB03	1.917	1.847	0.96		4/23/92
4/20/92	LIDW101	1.607	0.825	0.51	BAD TUBE	4/23/92
4/20/92	LIDW102	1.948	1.958	1.01		4/23/92
4/20/92	LIDW103	1.917	1.883	0.98		4/23/92
4/20/92	LIDW201	1.607	0.834	0.52	BAD TUBE	4/23/92
4/20/92	LIDW202	1.948	1.848	0.95		4/23/92
4/20/92	LIDW203	1.917	1.791	0.93		4/23/92
4/20/92	LIDW301	1.607	0.016	0.01	BAD TUBE	4/23/92
4/20/92	LIDW302	1.948	1.341	0.69		4/23/92
4/20/92	LIDW303	1.917	1.342	0.70		4/23/92
4/21/92	LIDTB01	1.863	1.737	0.93		4/23/92
4/21/92	LIDTB02	1.799	1.828	1.02		4/23/92

Table F-2. Continued. (Page 4 of 5)

SAMPLING DATE	SAMPLE ID NO.	REFERENCE OD	SAMPLE OD	S/R RATIO	FLAGS	RUN DATE
4/21/92	LIDTB03	1.712	1.886	1.10		4/23/92
4/21/92	LIDW101	1.863	1.639	0.88		4/23/92
4/21/92	LIDW102	1.799	1.471	0.82		4/23/92
4/21/92	LIDW103	1.712	1.782	1.04		4/23/92
4/21/92	LIDW201	1.863	1.366	0.73		4/23/92
4/21/92	LIDW202	1.799	1.361	0.76		4/23/92
4/21/92	LIDW203	1.712	1.697	0.99		4/23/92
4/21/92	LIDW301	1.863	0.908	0.49		4/23/92
4/21/92	LIDW302	1.799	0.966	0.54		4/23/92
4/21/92	LIDW303	1.712	1.310	0.77		4/23/92
4/22/92	LID0701	1.836	0.064	0.03		4/27/92
4/22/92	LID0702	1.924	0.012	0.01		4/27/92
4/22/92	LIDTB01	1.754	1.817	1.04		4/23/92
4/22/92	LIDTB02	1.75	1.857	1.06		4/23/92
4/22/92	LIDW101	1.754	1.614	0.92		4/23/92
4/22/92	LIDW102	1.75	1.650	0.94		4/23/92
4/22/92	LIDW201	1.754	1.417	0.81		4/23/92
4/22/92	LIDW202	1.75	1.494	0.85		4/23/92
4/22/92	LIDW301	1.754	0.965	0.55		4/23/92
4/22/92	LIDW302	1.75	1.052	0.60		4/23/92
4/23/92	LIZTB01	1.961	1.858	0.95	LAB STD	4/23/92
4/23/92	LIZTB02	1.634	1.896	1.16	LAB STD	4/23/92
4/23/92	LIZW101	1.961	1.872	0.95	LAB STD	4/23/92
4/23/92	LIZW102	1.634	1.652	1.01	LAB STD	4/23/92
4/23/92	LIZW201	1.961	1.696	0.86	LAB STD	4/23/92
4/23/92	LIZW202	1.634	1.556	0.95	LAB STD	4/23/92
4/23/92	LIZW301	1.961	1.360	0.69	LAB STD	4/23/92
4/23/92	LIZW302	1.634	1.269	0.78	LAB STD	4/23/92
4/27/92	LIZTB01	1.835	1.914	1.04	LAB STD	4/27/92
4/27/92	LIZTB02	1.916	1.851	0.97	LAB STD	4/27/92
4/27/92	LIZW101	1.835	1.930	1.05	LAB STD	4/27/92
4/27/92	LIZW102	1.916	1.846	0.96	LAB STD	4/27/92
4/27/92	LIZW201	1.835	1.587	0.86	LAB STD	4/27/92
4/27/92	LIZW202	1.916	1.657	0.86	LAB STD	4/27/92
4/27/92	LIZW301	1.835	1.568	0.85	LAB STD	4/27/92
4/27/92	LIZW302	1.916	1.233	0.64	LAB STD	4/27/92

Table F-2. Continued. (Page 5 of 5)

SAMPLING DATE	SAMPLE ID	RUN	RUN DATE	DIL	CONCENTRATION (ug/L) B	T	E	X	SURROGATE % RECOVERY CIS-1,2 DCE	FB	FLAGS
1/22/92	LAGTH01	266	1/27/92	1	nd	86.30	nd	nd	82		
1/22/92	LAGTL01	265	1/27/92	1	2.3	25.60	nd	nd	85		
1/22/92	LGA0401	275	1/27/92	1	1.8	<1	nd	nd	110		
1/22/92	LGA0601	277	1/27/92	20	1820.3	220.80	nd	nd	85		
1/22/92	LGA0701	273	1/27/92	1	nd	nd	nd	nd	95		
1/22/92	LGA0901	271	1/27/92	5	231	14.00	94.4	49.3	97		
1/22/92	LGA1001	268	1/27/92	1	nd	nd	nd	nd	96		
1/22/92	LGA1101	276	1/27/92	1	6.5	2.10	1.9	nd	91		
1/22/92	LGATB01	264	1/27/92	1	2	nd	nd	nd	102		
1/23/92	LGA0101	289	1/28/92	5	320.3	112.10	59.4	26	81		
1/23/92	LGA0201	286	1/28/92	1	nd	<1	nd	nd	97		
1/23/92	LGA0301	295	1/28/92	20	1393	<20	90.1	nd	97		
1/23/92	LGA0501	287	1/28/92	1	nd	<1	nd	nd	97		
1/23/92	LGA0801	288	1/28/92	1	nd	<1	nd	nd	101		
1/23/92	LGATB01	282	1/28/92	1	nd	<1	nd	nd	105		
1/23/92	LGATH01	284	1/28/92	1	nd	89.70	nd	nd	79		
1/23/92	LGATL01	283	1/28/92	1	nd	28.70	nd	nd	84		
2/24/92	LGA0101	772	3/1/92	10	455.1	41.10	93.4	17.9	69	95	
2/24/92	LGA0201	727	2/28/92	1	<1	<1	<1	nd	95	98	
2/24/92	LGA0301	773	3/1/92	50	889	<50	<50	nd	91	97	
2/24/92	LGA0401	729	2/28/92	1	<1	<1	<1	nd	94	131	
2/24/92	LGA0501	726	2/28/92	1	<1	<1	<1	nd	99	104	
2/24/92	LGA0601	774	3/1/92	50	983.9	<50	<50	nd	91	96	
2/24/92	LGA0701	730	2/28/92	1	1.2	<1	nd	nd	90	120	
2/24/92	LGATB01	728	2/28/92	1	<1	<1	nd	nd	92	118	
2/24/92	LGATH01	759	2/29/92	1	nd	100.80	nd	nd	155	93	
2/24/92	LGATL01	736	2/28/92	1	2.4	39.40	<1	nd	90	101	
2/25/92	LGA0801	731	2/28/92	1	1.7	<1	nd	<1	92	124	
2/25/92	LGA0901	771	3/1/92	10	305.4	22.40	127.1	54.7	94	133	
2/25/92	LGA1001	737	2/28/92	1	1.1	2.50	<1	nd	75	101	
2/25/92	LGA1101	732	2/28/92	1	1.3	<1	<1	<1	81	109	
2/25/92	LGATB01	733	2/28/92	1	<1	<1	<1	nd	92	117	

Table F-3. Data for gas chromatography analysis. (Page 1 of 5)

SAMPLING DATE	SAMPLE ID	RUN	RUN DATE	DIL	CONCENTRATION (ug/L) B	T	E	X	SURROGATE % RECOVERY CIS-1,2 DCE	FB	FLAGS
2/25/92	LGATH01	760	2/29/92	1	nd	95.10	nd	nd	82	92	
2/25/92	LGATL01	756	2/29/92	1	<1	4.00	nd	nd	92	97	
2/25/92	LGB0101	734	2/28/92	1	<1	<1	<1	nd	91	110	
2/25/92	LGB0201	735	2/28/92	1	<1	<1	<1	nd	92	100	
2/26/92	LGB0301	787	3/1/92	5	106.9	217.20	58.9	93.8	98	125	
2/26/92	LGB0401	751	2/29/92	1	<1	<1	<1	<1	99	103	
2/26/92	LGB0501	754	2/29/92	1	21.7	5.00	28.8	8.1	101	124	
2/26/92	LGB0701	783	3/1/92	1	<1	<1	<1	2.1	100	110	
2/26/92	LGB0901	770	3/1/92	1	28.7	3.40	<1	3.4	90	104	
2/26/92	LGB1001	778	3/1/92	20	382.4	nd	<20	<20	78	81	
2/26/92	LGBTB01	753	2/29/92	1	<1	<1	nd	nd	93	97	
2/26/92	LGBTH01	786	3/1/92	2	<1	99.20	nd	nd	91	98	
2/26/92	LGBTL01	769	3/1/92	1	<1	32.60	nd	nd	94	103	
2/27/92	LGB1101	805	3/2/92	1	nd	nd	nd	nd	98	99	
2/27/92	LGB1401	803	3/2/92	1	<1	nd	nd	nd	101	103	
2/27/92	LGB1501	806	3/2/92	1	nd	nd	nd	nd	98	99	
2/27/92	LGB1601	804	3/2/92	1	nd	nd	nd	nd	101	103	
2/27/92	LGB1701	794	3/2/92	1	<1	<1	<1	nd	91	99	
2/27/92	LGB1801	789	3/1/92	1	<1	<1	nd	nd	92	97	
2/27/92	LGB1901	790	3/1/92	1	<1	<1	nd	nd	93	99	
2/27/92	LGB2001	779	3/1/92	20	1067.5	90.10	1047.6	225.5	96	297	
2/27/92	LGB2101	791	3/2/92	1	<1	<1	nd	nd	92	97	
2/27/92	LGBTB01	792	3/2/92	1	<1	<1	nd	nd	93	98	
2/27/92	LGBTH01	788	3/1/92	2	<2	110.20	nd	nd	91	97	
2/27/92	LGBTL01	793	3/2/92	1	1.2	42.80	nd	nd	89	93	
2/28/92	LGB0801	811	3/2/92	50	1113.6	20.00	nd	nd	91	91	
2/28/92	LGB1201	784	3/1/92	1	<1	<1	<1	<1	92	101	
2/28/92	LGB1301	809	3/2/92	1	<1	<1	nd	nd	98	99	
2/28/92	LGBTB01	807	3/2/92	1	<1	nd	nd	nd	97	97	
2/28/92	LGBTH01	810	3/2/92	2	2.1	106.80	nd	nd	91	92	
2/28/92	LGBTL01	808	3/2/92	1	1	38.70	nd	nd	97	98	
3/9/92	LGC1601	1073	3/14/92	40	1641.8	4,312.10	689	864.8	107	295	F*

Table F-3. Data for gas chromatography analysis. (Page 2 of 5)

SAMPLING DATE	SAMPLE ID	RUN	RUN DATE	DIL	CONCENTRATION (ug/L) B	T	E	X	SURROGATE % RECOVERY CIS-1,2 DCE	FB	FLAGS
3/9/92	LGC1701	1074	3/14/92	40	5762.2	4,661.60	1825.8	1796.6	106	484	F*
3/9/92	LGC1801	1064	3/13/92	20	443.3	228.30	136.1	124	106	132	
3/9/92	LGCW101	1056	3/13/92	1	nd	3.30	nd	nd	102	99	
3/9/92	LGCW201	1057	3/13/92	1	nd	30.00	nd	nd	106	104	
3/9/92	LGCW301	1058	3/13/92	1	nd	113.00	nd	nd	87	87	
3/9/92	GCTB101	1054	3/13/92	1	nd	nd	nd	nd	115	108	
3/10/92	LGC0801	1076	3/14/92	20	395.6	200.40	68.4	109.4	98	147	F*
3/10/92	LGC1001	1075	3/14/92	200	7252	28,603.00	3031	5254	105	379	F*
3/11/92	LGC2301	1097	3/15/92	40	437.7	202.40	55.6	37.5	102	101	
3/10/92	LGCW101	1059	3/13/92	1	nd	3.10	nd	nd	106	102	
3/10/92	LGCW201	1060	3/13/92	1	nd	29.90	nd	nd	103	102	
3/10/92	LGCW301	1061	3/13/92	1	<1	113.90	nd	<1	97	102	
3/10/92	GCTB101	1055	3/13/92	1	nd	nd	nd	nd	114	106	
3/11/92	LGC0401	1106	3/15/92	1	14.4	49.40	5.8	11	101	108	
3/11/92	LGC0901	1101	3/15/92	1	8.4	4.50	<1	1.3	119	100	
3/11/92	LGC2401	1117	3/16/92	1	3.1	6.00	nd	1.2	107	116	
3/11/92	LGC2601	1095	3/15/92	40	2304.4	6,515.80	1097.6	1404.5	113	511	F*
3/11/92	LGCW101	1105	3/15/92	1	2.3	9.40	nd	<1	101	100	
3/11/92	LGCW201	1108	3/15/92	1	1	32.00	<1	1.4	92	96	
3/11/92	LGCW301	1107	3/15/92	1	2.3	119.80	<1	1.4	98	106	
3/11/92	GCTB101	1116	3/16/92	1	nd	3.50	nd	<1	97	94	
3/12/92	LGC0601	1098	3/15/92	40	1271.8	313.50	51.4	39.5	105	103	
3/12/92	LGC0701	1114	3/16/92	1	1.5	4.60	<1	<1	111	104	
3/12/92	LGC1101	1096	3/15/92	40	5484.4	6,168.20	1038.2	1059.4	109	329	F*
3/12/92	LGCW101	1102	3/15/92	1	1.4	7.50	nd	<1	102	99	
3/12/92	LGCW201	1103	3/15/92	1	3.2	34.00	nd	<1	101	100	
3/12/92	LGCW301	1104	3/15/92	1	2.9	112.70	nd	1.4	94	99	
3/12/92	GCTB101	1115	3/16/92	1	1.2	3.20	nd	<1	105	101	
3/13/92	LGC0201	1100	3/15/92	200	930.8	2,520.20	277	481.8	110	118	
3/13/92	LGC0301	1099	3/15/92	400	12359.6	25,060.40	1966.8	3675.6	104	115	
3/13/92	LGC0501	1119	3/16/92	1	144.6	28.90	9.2	8.7	102	133	
3/13/92	LGCW101	1081	3/14/92	1	<1	3.80	nd	nd	98	97	

Table F-3. Data for gas chromatography analysis. (Page 3 of 5)

SAMPLING DATE	SAMPLE ID	RUN	RUN DATE	DIL	CONCENTRATION (ug/L) B	T	E	X	SURROGATE % RECOVERY CIS-1,2 DCE	FB	FLAGS
3/13/92	LGCW201	1082	3/14/92	1	1.1	30.80	nd	nd	95	94	
3/13/92	LGCW301	1084	3/14/92	1	5.3	128.80	1.4	4.2	87	136	
3/13/92	GCTB101	1118	3/16/92	1	1.3	2.40	nd	<1	104	97	
4/15/92	LGA0101	1439	4/20/92	20	1529.1	3,420.60	391.9	551.5	83	356	FC, T*
4/15/92	LGA0201	1418	4/20/92	1	nd	nd	nd	nd	94	92	
4/15/92	LGA0301	1440	4/20/92	20	1286.7	162.30	652.2	103.6	99	128	FC
4/15/92	LGA0401	1420	4/20/92	1	nd	nd	nd	nd	96	92	
4/15/92	LGA0501	1419	4/20/92	1	nd	<1	nd	nd	79	79	
4/15/92	LGATB01	1421	4/20/92	1	nd	<1	nd	nd	98	94	
4/15/92	LGAW101	1425	4/20/92	1	nd	2.70	nd	nd	96	93	
4/15/92	LGAW201	1430	4/20/92	1	nd	27.00	nd	nd	93	96	
4/15/92	LGAW301	1431	4/20/92	1	2.3	93.50	nd	<1	96	94	
4/16/92	LGA0601	1438	4/20/92	20	800.8	<20	339.3	23.3	92	96	
4/16/92	LGA0701	1422	4/20/92	1	nd	nd	nd	nd	97	93	
4/16/92	LGA0801	1424	4/20/92	1	1.8	<1	<1	nd	96	92	
4/16/92	LGA0901	1437	4/20/92	20	899.5	61.10	470.2	191.3	98	138	FC
4/16/92	LGA1002	1470	4/23/92	1	34.6	1.40	72.6	<1	56	108	DC
4/16/92	LGA1101	1423	4/20/92	1	<1	<1	<1	<1	65	94	DC
4/16/92	LGATB01	1432	4/20/92	1	nd	nd	nd	nd	97	96	
4/16/92	LGAW101	1433	4/20/92	1	nd	2.60	nd	nd	93	92	
4/16/92	LGAW201	1434	4/20/92	1	nd	26.60	nd	nd	92	94	
4/16/92	LGAW301	1435	4/20/92	1	2.2	97.10	nd	<1	92	92	
4/17/92	LGD1002	1472	4/23/92	1	nd	nd	nd	nd	94	94	
4/17/92	LGD1101	1485	4/23/92	1	29.3	<1	3.7	1.3	114	129	
4/17/92	LGD1202	1471	4/23/92	1	nd	nd	nd	nd	94	95	
4/17/92	LGDTB01	1465	4/23/92	1	nd	nd	nd	nd	96	97	
4/17/92	LGDW101	1463	4/23/92	1	nd	2.40	nd	nd	80	83	
4/17/92	LGDW201	1464	4/23/92	1	nd	25.60	nd	nd	92	98	
4/17/92	LGDW301	1469	4/23/92	1	2.4	71.80	nd	nd	97	94	
4/20/92	LGD0102	1473	4/23/92	1	10.7	nd	<1	nd	93	92	
4/20/92	LGD0201	1488	4/23/92	10	231.9	<10	40.2	46.6	91	106	
4/20/92	LGD0301	1498	4/24/92	100	6821.4	44.00	261.8	30.5	95	98	

Table F-3. Data for gas chromatography analysis. (Page 4 of 5)

SAMPLING DATE	SAMPLE ID	RUN	RUN DATE	DIL	CONCENTRATION (ug/L) B	T	E	X	SURROGATE % RECOVERY CIS-1,2 DCE	FB	FLAGS
4/20/92	LGD9902	1474	4/23/92	1	nd	<1	nd	nd	91	95	
4/20/92	LGDTB01	1466	4/23/92	1	nd	nd	nd	nd	96	98	
4/20/92	LGDW101	1467	4/23/92	1	nd	2.80	nd	nd	95	95	
4/20/92	LGDW201	1468	4/23/92	1	nd	23.60	nd	nd	90	95	
4/20/92	LGDW301	1484	4/23/92	1	3.6	92.40	<1	<1	106	93	
4/21/92	LGD0501	1496	4/24/92	1	11.6	2.50	94.4	12.6	121	157	DC, FC
4/21/92	LGD0801	1481	4/23/92	1	nd	nd	nd	nd	95	96	
4/21/92	LGD0901	1483	4/23/92	1	nd	<1	nd	nd	91	92	
4/21/92	LGDTB01	1482	4/23/92	1	nd	nd	nd	nd	92	93	
4/21/92	LGDW101	1479	4/23/92	1	nd	2.50	nd	nd	96	98	
4/21/92	LGDW201	1480	4/23/92	1	nd	26.50	nd	nd	88	95	
4/21/92	LGDW301	1486	4/23/92	1	2.2	91.30	nd	nd	96	94	
4/22/92	LGD0401										MISSING
4/22/92	LGD0701										MISSING
4/22/92	LGW101										MISSING
4/22/92	LGW201										MISSING
4/22/92	LGW301										MISSING

Table F-3. Data for gas chromatography analysis. (Page 5 of 5)

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