PUBLIC WORKS TECHNICAL BULLETIN 200-1-61
1 OCTOBER 2008

AN EVALUATION OF FIELD TEST KITS FOR ENVIRONMENTAL SAMPLING

Public Works Technical Bulletins are published by the U.S. Army Corps of Engineers, 441 G Street NW, Washington, DC 20314-1000. They are intended to provide information on specific topics in areas of Facilities Engineering and Public Works. They are not intended to establish new DA policy.

DEPARTMENT OF THE ARMY
U.S. Army Corps of Engineers
441 G Street, NW
Washington, DC 20314-1000

CECW-CE

Public Works Technical Bulletin No. 200-1-61 — 1 October 2008

Facilities Engineering
Environmental

AN EVALUATION OF FIELD TEST KITS FOR ENVIRONMENTAL SAMPLING

1. Purpose.

a. This Public Works Technical Bulletin (PWTB) identifies some field test kits available on the market and their effectiveness in the identification of contaminants. This document could be used as guidance to illustrate appropriate uses of these test kits and the limitations of such field measurements.

b. All PWTBs are available electronically (in Adobe® Acrobat® portable document format [PDF]) through the World Wide Web (WWW) at the National Institute of Building Sciences' Whole Building Design Guide web page, which is accessible through URL:

http://www.wbdg.org/ccb/browse_cat.php?o=31&c=215

2. Applicability. This PWTB applies to all Corps of Engineers Districts and Department of the Army installations responsible for characterization and monitoring of hazardous waste sites.

3. References.

a. Army Regulation (AR) 200-1, "Environmental Protection and Enhancement," 13 December 2007.

b. Other references are listed at the end of Appendix A.

4. Discussion.

a. AR 200-1 contains policy for preparing spill response plans as well as for regular monitoring and measuring of key characteristics in operations or activities that can have an impact on the environment. Finding efficient technologies to accomplish such requirements can sometimes be difficult and time consuming.

b. Polychlorinated biphenyls (PCBs); heavy metals; petroleum, oils, and lubricants (POL) spills; and explosives are common contamination problems Army wide. Quick decisions are often necessary in scoping an environmental emergency and determining an appropriate response. Additionally, screening a large area (or large number of items) requires efficient tools. Field test technologies are often the best alternative for providing the user with a quick response. Potential uses include spill response, screening of recycling scrap or waste building materials, compliance, long-term monitoring, and pre-screening of contaminated areas to determine future actions.

c. Modern technical approaches, such as the Triad for characterization and remediation of hazardous waste sites, encourage the use of real-time measurement technologies, including field analytical instrumentation. Some of the benefits of this modern approach are acceleration of the project schedule, cost reduction, and outcome improvement.

d. Appendix A provides the most commonly used field testing technologies. Instruments based on these technologies are presented (Figures A-1, A-2, and A-4 through A-14). For each technology and instrument, a summary of performance is provided. Advantages and disadvantages are also discussed based on EPA evaluations as well as information provided by each technology vendor. However, citation and inclusion of vendors does not constitute an official endorsement.

e. Appendix B lists some Army examples where implementation of field testing technologies has saved operational costs and project time.

f. Before selecting any field testing technology to be used at a hazardous waste site, the specific objectives of the study and the quality of data needed should be evaluated, since different technologies will give different quality of results. Additionally, it is highly recommended to support at least 10% of the results obtained by any field testing technology with laboratory confirmatory testing.

5. Points of Contact (POCs). Headquarters, U.S. Army Corps of Engineers (HQUSACE) is the proponent for this document. The POC at HQUSACE is Malcolm E. McLeod, CEMP-II, 202-761-0632, or e-mail: Malcolm.E.Mcleod@ usace.army.mil.

Questions and/or comments regarding this subject should be directed to the technical POC:

U.S. Army Engineer Research and Development Center
Construction Engineering Research Laboratory
ATTN: CEERD-CN-E (Giselle Rodriguez)
2902 Newmark Drive
Champaign, IL 61822-1072
Tel: (217) 373-3434
Fax: (217) 373-3430
e-mail: Giselle.Rodriguez@usace.army.mil

FOR THE COMMANDER:

JAMES C. DALTON, P.E.
Chief, Engineering and Construction
Directorate of Civil Works

APPENDIX A:
FIELD TEST KITS TECHNOLOGIES

IMMUNOASSAY TECHNOLOGIES

Immunoassay technologies are characterized by the use of biologically engineered antibodies to bind with a target compound. The purpose of the assay is to identify and quantify these organic and inorganic compounds. Immunoassay technologies have been described as effective and inexpensive methods making their use preferable for on-site environmental analysis. Immunoassay techniques more commonly used for field testing are:

1. Enzyme-linked immunosorbent assay (ELISA)
2. Enzyme immunoassay (EI)
3. Fluorescent immunoassay (FIA)
4. Radioimmunoassay (RIA)

ELISA is the immunoassay technique most often used for environmental field testing. ELISA offers high sample throughput, low detection limits, and it can be optimized for selectivity. This technique is preferable because it does not contain radioactive materials and is the simplest of all immunoassay methods. ELISA-based test kits are lightweight and more portable than the other methods.

In ELISA immunoassay, antibodies are developed specifically to bind with a selected environmental contaminant or contaminants, and that selective response is used to confirm the presence of the contaminant in samples. Presence and concentrations of contaminants are determined by the use of a sensitive colorimetric reaction. First, to determine if a contaminant is present in the sample, the color developed is compared with a standard color form by a sample of known concentration. The contaminant concentration is determined by the intensity of color in the sample, which can be estimated by visually comparing the color intensity to a standardized color card or by reading a photometer or spectrophotometer.

Sample analysis by this method requires multiple steps. EPA summarized the steps as follows (http://www.clu-in.org/char/technologies/immunoassay.cfm):

1. Enzyme Conjugate and Sample Extract Addition

   If the antibodies are coated on the inside surface of the test tube, the sample and enzyme conjugate are combined directly in the test tube. If the antibodies are coated on magnetic particles or latex particles, a carefully measured amount of the solution that contains the coated particles is added to the test tube. Measured amounts of both the enzyme conjugate and the actual sample containing the target analyte are added to the test tube. The action is a timed incubation step. During the incubation, the analyte in the sample competes with the known amount of labeled antigen in the enzyme conjugate for the limited number of antibody binding sites.

2. Washing

   After incubation, the excess unbound enzyme conjugate is washed (removed) from the test tube.

3. Color Development

   The amount of the enzyme conjugate that remains in the test tube is measured through the use of a colorimetric reaction. An enzyme substrate and a chromogen are added to the test tube to cause the formation of the color. That action also is a timed step, after which a solution is added to stop the formation of color. Because the amount of bound enzyme conjugate determines the amount of color, the amount of color is inversely proportional to the amount of analyte present in the sample.

There are some factors that can affect the time required for analysis and the sample throughput. It will depend mostly on the sample matrix, whether the samples are soil or water, the detection limits being looked for, and the accuracy required. Soil samples will need an extraction of the target contaminant into a solution. Generally extraction kits are sold or rented separately. Operator experience and size of the sample batches analyzed are also key factors for sample throughput. Additionally, the operator must be very careful to follow the manufacturer’s instructions regarding to effective temperature range since it have a significant impact in the outcome of the analysis, some times defining whether the test works or not.

Field Test Kits Based on Immunoassay

EnSys – Strategic Diagnosis

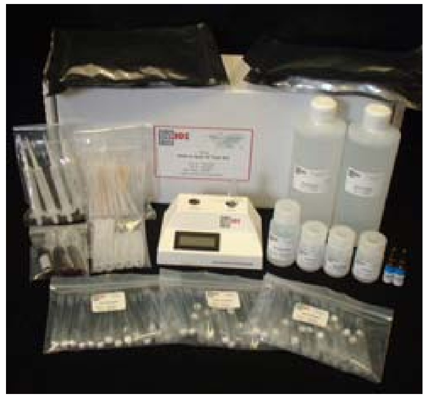

Figure A-1. Strategic Diagnosis Ensys - Field Test Kit and Reagents (www.cysense.com).

Ensys Test Kits, produced by Strategic Diagnosis Incorporated (SDI) (Newark, DE), are kits based on immunoassay technology. These kits are available for the detection of different contaminants including Total Petroleum Hydrocarbons (TPHs) and polychlorinated biphenyls (PCBs). These devices are also available for the detection of explosives such as cyclotrimethylene-trinitramine (RDX) and trinitrotoluene (TNT), although they are not immunoassay-based technologies. In addition to the systems mentioned above, some other EnSys Test Kits are commercially available for the identification of other common contaminants such as Dioxin and Pentachlorophenol (PCP).

EnSys Petro Test System

Based on information captured from the EPA Superfund Innovative Technology Evaluation (SITE) document, EnSys Petro Test System is a technology intended to measure TPH in samples including gasoline, diesel, Jet A fuel, JP-4, kerosene, No. 2 fuel oil, No.6 fuel oil, and mineral spirits. The system complies with the standards established by EPA SW-846 Method 4030 for screening TPHs using immunoassay detection. One important characteristic of this system is that the antibodies necessary for the assay are coated on the test tube walls. Therefore, no additional pipette use is needed.

The system includes the SDI Sample Extraction Kit, the EnSys Petro 12T Soil Test Kit, and the EnSys/EnviroGard® Common Accessory Kit. Accessory equipment may be rented or purchased from SDI. The EnSys Petro Test System includes enough antibody-coated test tubes to perform 12 sample evaluations. The operating procedure for this system is generally as described above for ELISA and includes five steps: extraction of contaminant, preparation of the standards, washing the sample with detergent solution, colorimetric reaction, and finally the color measurement for estimation of TPH concentration. More detailed information about these steps can be found in the EPA SITE program report or on the vendor’s (SDI’s) website.

The EnSys Petro Soil Test System offers different assay ranges for each TPH component evaluated. Detection levels may vary for each contaminant. For instance, assay range stands between 10 to 10,000 ppm for gasoline and 15 to 10,000 ppm for diesel fuel.

Advantages: EnSys Petro Test System can be easily operated by one person with basic wet chemistry skills allowing the user to evaluate 1 to 12 samples within 45 minutes. In addition the device can be easily moved, and its components are battery-operated making it suitable for field testing.

Limitations: EnSys Petro Test System does not generate quantitative results. The results provided can indicate only whether a sample is above or below the concentration of a reference standard containing 3 mg/l m-xylene. Even though the system is able to measure petroleum hydrocarbons, but not all TPH fuel types, it is unable to differentiate between different petroleum compounds.

EnSys PCB Soil Test Kit

Unlike the EnSys Petro Test System, the EnSys PCB Soil Test Kit had no EPA evaluation report that could be found. Thus, this evaluation will be based mostly on vendor and literature information. However, test principles and procedures are very similar to EnSys Petro Test System and comply with EPA SW-846 Method 4020 standards. This technology is also based on the use of antibodies that are immobilized to the test tube wall and colorimetric reaction for the estimation of specific PCB Aroclor.

According to the EnSys PCB Soil Test Kit vendor (SDI), this technology is able to correctly identify 95% of samples that are PCB-free and those containing 1 ppm or more of PCBs. Different PCB Aroclors are detected with varying sensitivity (Table A-1).

Table A-1. Minimum and maximum detection levels that can be accurately achieved with Ensys PCB Test Kit (SDI).

Aroclor	Minimum Detection Level	Maximum Detection Level
1260	0.5 ppm	500 ppm
1254	0.5 ppm	500 ppm
1248	1.0 ppm	500 ppm
1242	2.0 ppm	500 ppm
1232	4.0 ppm	500 ppm
1016	4.0 ppm	500 ppm

- Kit standard is Aroclor 1248

Advantages: Two detection levels are provided: assay ranges standing between (1) 0.5 to 500 ppm for soil samples and (2) 5 to 5,000 ug/100cm^2 for wipe samples. As with the EnSys Petro Test, the EnSys PCB Soil Test Kit hardware comes in a plastic, portable briefcase with boxes of test and extraction kits available, which makes it suitable for field testing.

Limitations: According to vendor information, soil containing high levels of petroleum fuels or transformer oil may affect results; this can be seen if the sample is added to the buffer and a cloudy suspension forms. Moreover, the color substrate can be affected by direct sunlight.

RaPID - Strategic Diagnosis

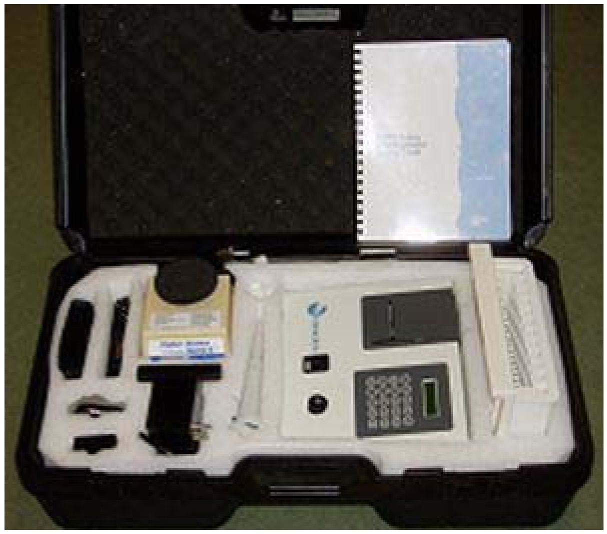

Figure A-2. RaPID Assay by SDI (www.cysense.com).

RaPID Assay PCB Test Kit

The RaPID Assay PCB Test Kit follows the same immunoassay principles (ELISA) as the other technologies already mentioned for the detection of PCBs on samples. However, this technology differs with EnSys PCB Soil Test Kit because the antibodies have to be added to the sample, along with an enzyme conjugate, since they are coated on paramagnetic particles and are not attached to the test tube walls. Besides this additional step, the test can be performed following the general guidelines described on page A-2.

According to SDI, the RaPID Assay PCB Test Kit user’s guide lists the minimum detection level for this technology as 0.2 ppb as Aroclor 1254. However, based on EPA Environmental Technology Verification program performance evaluation results, RaPID Assay PCB Test Kit method detection limit (MDL) was calculated to be 1.5 ppm.

Advantages: A single operator with minimum chemical skills and brief training can make use of this technology and evaluate approximately 10 to 11 samples per hour. The overall performance of the RaPID Assay PCB Test Kit was characterized by the EPA Environmental Technology Verification (ETV) Program as “slightly biased and precise, under a given set of environmental conditions.” The ETV evaluation demonstrated that the bias is evenly distributed (positive and negative); therefore, it is not reflected in the overall accuracy. Additionally, this evaluation found this test kit capable of providing accurate data either as a complement to data generated in a fixed laboratory or as generating stand-alone data in decision-making processes.

Limitations: This immunoassay test does not differentiate between PCB and related compounds. Therefore, their presence can affect measurement results.

RaPID BTEX/TPH Assay

Following the same immunoassay principles as the other technologies already mentioned, and using the same hardware and operational logistics as for the RaPID Assay PCB Test Kit, RaPID BTEX/TPH Assay is a tool for measuring Benzene, Toluene, Ethylbenzene, and Xylenes (BTEX) and TPH in soil and water. This technology has not been evaluated by the EPA. However, this system complies with the EPA SW-846 Method 4030 (EPA 1996) standards for the screening of soil samples for petroleum hydrocarbons.

Figure A-3 lists all the BTEX and TPH related compounds that RaPID BTEX/TPH Assay is able to detect. According to SDI, detection limits range between 0.9 to 30 ppm as the Total BTEX standard for soils and 0.02 to 3.0 ppm as the Total BTEX standard for water samples. Similar to RaPID Assay PCB Test Kit, this technology does not differentiate between BTEX and related compounds.

Total BTEX**
m-Xylene
p-Xylene
o-Xylene
Ethylbenzene
Toluene
Benzene
TPH
Naphthlene
1,2,4-Trimethylbenzene
Anathrazene
Styrene
Hexachlorobenzene
Phenanthrene
Creosote
1,3,5-trimethylbenzene
Acenapthene
n-propylbenzene
n-Hexane
n-Octane
n-Nonane
n-Heptane
Cyclohexane
n-Decane
Methylene chloride
Trichloroethylene
Mineral Spirits
Household Lubricant

Figure A-3. BTEX and TPH related compounds (www.cysence.com).

COLORIMETRIC TESTS

Organochlorine Detection Based on Colorimetric Tests

The following technologies are based on colorimetric methods. According to vendor (Dexsil Corporation) information, this method is based on the addition of a fixed amount of mercuric nitrate. After adding mercuric nitrate, an indicator very sensitive to free mercuric ions is added to the solution. Chlorine absence in the sample will be determined by the development of a purple color, which means free mercuric ions are present. However, no color development means all the mercury has been complexed with chloride. In other words, chlorine is present in the sample.

<u>Clor-N-Soil Test Kit</u>

Clor-N-Soil Test Kit is designed to detect extracted PCB compounds from soil samples based on the principle of total organic chlorine detection. As shown in Figure A-4, this test kit is sold in a portable cardboard box. As described by the EPA ETV program report, all the products necessary to perform the analysis are contained inside the box: a stainless steel scoop, a hand-held scale, a plastic pipette bulb, an extraction test tube, organic extraction solvent, a filter syringe assembly, a reaction test tube containing two ampoules, an indicator test tube containing two ampoules and 7 mL of buffer solution, step-by-step instructions, and a color chart.

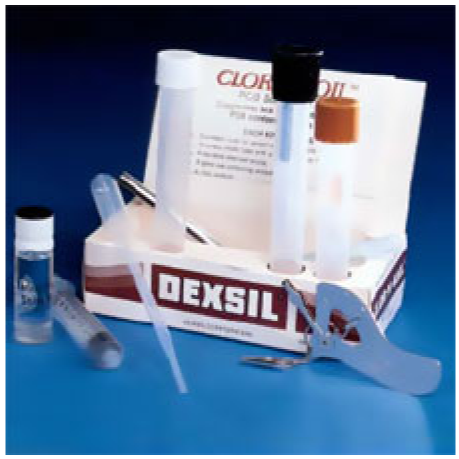

Figure A-4. Clor-N-Soil products (www.dexsil.com).

The following list describes all the steps necessary to perform this test:

1. PCBs are extracted from the sample using butyl diglyme. This step will liberate the sample from any inorganic chlorine, water, soil particles, and compounds containing polar chlorine. The sample will be treated with metallic sodium to strip chlorine from the biphenyl compound.

2. The extract is transferred to an indicator tube to measure and detect total organic chlorine.

3. The addition of an indicating solution of mercuric nitrate and diphenyl carbazone will generate a colorimetric reaction to measure chlorine content.

4. Color development is inversely proportional to the chlorine content. Purple indicates the absence of chloride and therefore PCBs; yellow indicates the presence of chloride.

Advantages: Clor-N-Soil test kit is a low-cost per sample alternative for PCB screening. Additionally, little or no technical expertise is required to use it. Another important advantage of this method is its high sample throughput, which is estimated by the EPA ETV program report as 48 samples per day.

Limitations: As with some other field detection technologies, one of the major limitations related to this technology is the tendency for false positives. These are more likely when interferants such as halogenated organic compounds are present in the sample. Mercury can cause false negatives when it is present with PCBs, which is why the EPA SITE program report recommends the use of this technology where the Aroclor of concern is known and where halogenated organics or mercury are not present.

Clor-N-Oil Test Kit

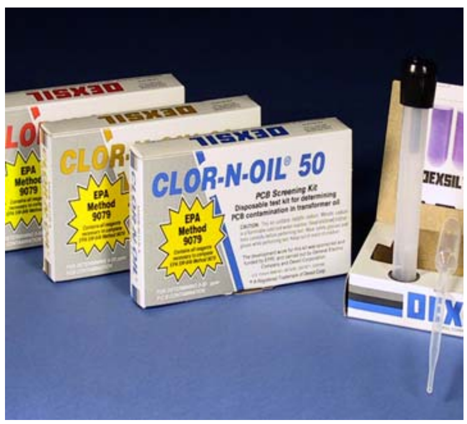

Figure A-5. Clor-N-Oil products (www.dexsil.com).

This technology works on the same principles and logistics as Clor-N-Soil Test Kit. However, it is intended for the detection of PCBs in oil samples. The Clor-N-Oil Test Kit follows EPA SW-846 Method 9079 (EPA 1996).

Both Clor-N-Oil and Clor-N-Soil Test Kits are designed to detect if the sample is above or below a specific concentration: 20, 50, and 100 ppm for Clor-N-Oil and 50 ppm for Clor-N-Soil. (Note: The use of this technique as a quantitative test is not recommended by either EPA or the product vendor.)

Explosives and Energetic Materials Detection Based on Colorimetric Tests

Expray and DropEx Test Kits

Expray and DropEx Test Kits (Figures A-6 and A-7), products from Plexus Scientific, are one of the easier to operate technologies evaluated in this PWTB. Based on the principle of colorimetric analysis and alkaline reactions, this technology is able to qualitatively identify explosive and energetic materials.

Each kit includes different regents that are capable of detecting different explosive groups. For instance, Expray and DropEx 1 detect Group A explosives, which include TNT, Tetryl, trinitrobenzene (TNB), dinitrotoluene (DNT), picric acid, and its salts. Expray and DropEx 2 detect Group B explosives, which include dynamite, nitroglycerine (NG), RDX, pentaerythritol tetranitrate (PETN), Semtex, nitrocellulose (NC), smokeless powder, and Tetryl. Expray and DropEx 3 detect Group I explosives, which include any nitrate-based explosive, black powder, flash powder, potassium nitrate, and ammonium nitrate. The DropEx kit also includes two extra reagents, DropEx A and B, which detects chlorates and peroxide-based explosives.

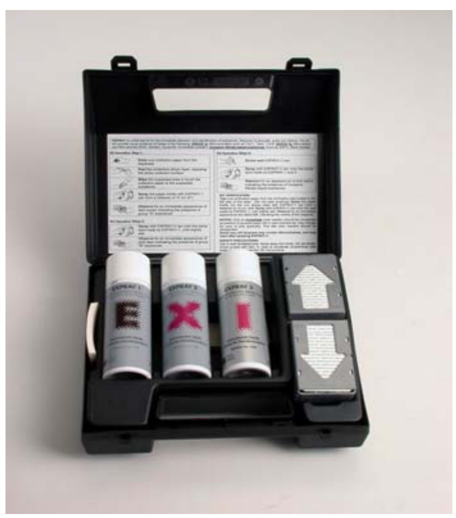

Figure A-6. Expray Test Kit (Mistral Security, Inc).

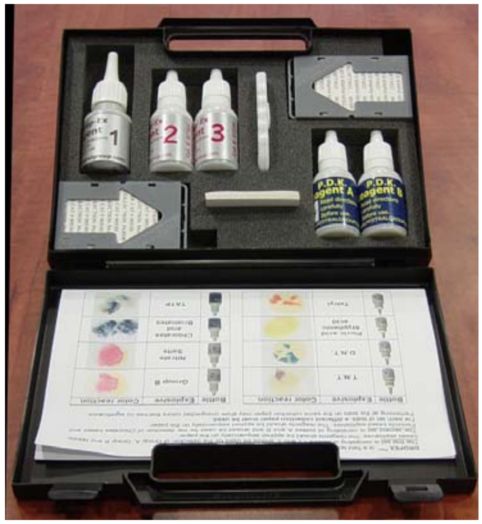

Figure A-7. DropEx Test Kit (Mistral Security, Inc).

Both tests are based on the same principles and logistics. Although, according to Plexus Scientific, there are four major differences between them. First, the Expray kit is more sensitive than the DropEx kit. The second difference relates to shelf life. Expray can be used throughout an entire year after first opened; however, the DropEx kit has a shelf life of only 1 month after first opened. A drop solvent pretreatment may be necessary when using Expray on surfaces with a considerable amount of plasticizer (e.g., large caliber gun propellant grains). Additionally, due to Expray flammability, DropEx is easier to ship and does not need special handling.

Even though this technology has been widely used for numerous applications where explosive screening is needed and extensive documentation was found describing all the advantages and potential uses for this product, an EPA method or evaluation associated with it could not be found.

<u>EnSys TNT Soil Test System/EnSys RDX Soil Test System</u>

EnSys TNT and RDX methods where initially developed by Dr. Thomas Jenkins in 1990 at the U.S. Army Engineer Research and Development Center laboratory in Hanover, NH (Cold Regions Research and Engineering Laboratory). The method is now commercialized by SDI. These devices can detect RDX and TNT in concentrations between 1 and 30 ppm, with 0.8 and 0.7 ppm being the minimum detectable concentrations, respectively. This test is based on the extraction of the explosives and the addition of a developer that induces a chemical reaction producing color. This color development is proportional to the concentration of RDX or TNT. EnSys TNT will also detect 2,4-DNT, Tetryl and TNB while EnSys RDX will also react to NG, PETN, NC, and Tetryl. Both devices are widely used for the detection of explosives and follow EPA methods 8510 for RDX and 8515 for TNT screening in soil.

Advantages: If using both test methods together, the same sample extract generated with the TNT test can be reused with the RDX test and nearly all the explosive types that could be present in the site would be screened.

Limitations: In the RDX test, nitrates and nitrites can cause false positives. If used for water samples, a solid phase extraction is required.

ELECTROCHEMICAL ANALYSIS FOR ORGANOCHLORINE DETECTION

The following technology determines the presence of chlorine based on electrochemical analysis. Removing the chloride from the PCB sample requires an initial extraction with a sodium-based compound. To quantitatively detect chloride ion concentration, an ion-specific electrode is used, and then with the appropriate instrumentation (described below) the concentration of chloride ions can be translated to ppm of PCB (Dexsil).

L2000 PCB/Chloride Analyzer

The L2000 PCB/Chloride Analyzer (Figure A-8), a technology developed by Dexsil Corporation, provides the user quantitative results for the detection of PCBs. As with Clor-N-Soil and Clor-N-Oil Test Kits, this technology is designed to detect extracted PCB compounds from soil samples based on the principle of total organic chlorine detection. The same organic solvent as for the previous technologies, butyl diglyme, is used to extract PCBs from samples.

Figure A-8. L2000 PCB/Chloride Analyzer (www.dexsil.com).

After calibration, total organic chlorine is then measured by an ion-specific electrode that quantifies chlorinated organic compounds.

Advantages: Four matrices can be evaluated with this single instrument: transformer oil, soil, water, and wipe samples. To facilitate the use of this instrument in the field, the L2000 PCB/Chloride Analyzer is powered by a rechargeable 8 volt battery.

Limitations: According to the EPA ETV program report, the presence of sources of organic chlorine other than PCBs can result in higher concentration readings or even false positives. False negatives are also possible when analyzing Aroclors other than 1242. Another limitation of this technology is that the device loses its calibration easily and must be recalibrated frequently.

The EPA recommends the use of this technology where the Aroclor of concern is known and where halogenated organics are not present. Additionally it is recommended that the quantitative results reported by the L2000 PCB/Chloride Analyzer be corrected by determining a correction factor that can be obtained by sending 10 to 20 percent of the samples for laboratory analysis.

FRIEDEL-CRAFTS REACTION

Reaction Process

The technology shown in Figure A-9 is based on the Friedel-Crafts Alkylation Reaction. This reaction involves an aromatic substitution. As described in the EPA SITE program report evaluation, the reaction process consists of the reaction of an alkyl halide, for example, dichloromethane (CH_2CL_2) with an aromatic hydrocarbon such as benzene (C_6H_6). To successfully occur, the reaction has to take place in the presence of a solid-phase metal halide catalyst, such as anhydrous aluminum chloride ($AlCl_3$).

Remedi Aid Starter Kit

Figure A-9. Remedi Aid Starter Kit (http://www.chemetrics.com/TPH.html).

Based on a modified version of the Friedel-Crafts Alkylation Reaction described above, the Remedi Aid Starter Kit, a technology developed by CHEMetrics, provides the user with quantitative results for the detection of TPHs. According to vendor information, by performing different calibrations this technology is able to quantify specific petroleum products such as: BTEX, PAH, diesel fuel, leaded and unleaded gasoline, weathered gasoline, Brent crude, and lubricating oil. Table A-2 lists vendor-identified detection ranges.

Table A-2. Remedi Aid Starter Kit Detection Ranges.

Petroleum Product	Detection Range (mg/kg)
Unleaded Gasoline	40-370
Weathered Gasoline	40-360
Diesel	60-880
Brent Crude	60-770
Lube Oil	160-2160
BTEX	20-150
Leaded Gasoline	40-470
PAH (18 component mixture)	8-70

Advantages: The Remedi Aid Starter Kit can be operated easily by one person with basic wet chemistry skills. To prevent chemical degradation and to facilitate its use, the system consists of pre-measured vacuum-sealed ampoules. Therefore, no pipette skills are required and the user’s contact with reagents is minimized. Also, to facilitate the use of this instrument in the field, the Remedi Aid Starter Kit is powered by a 9 volt battery; therefore, no alternating current power source is needed for operation.

Limitations: A limitation of this technology is that different calibrations are required to evaluate specific types of contaminants. Based on the EPA SITE program report evaluation, when calibration is performed, the kit can measure aliphatic hydrocarbons in the presence of aromatic compounds. However, it does not measure aliphatic hydrocarbons when aromatic compounds are not present in the sample.

In general, Remedi Aid Starter Kit was highly rated by the EPA SITE program report evaluation, which described this technology as a “reliable field measure device for TPH in soil.”

INFRARED ANALYSIS

Infrared (IR) analysis is another technology used to evaluate concentrations of aromatic and aliphatic hydrocarbons. Since carbon-hydrogen bonds absorb IR light, the process consists of using light in the IR wavelength range to measure petroleum hydrocarbons.

Infracal TOG/TPH Analyzer

The Infracal TOG/TPH Analyzer, a technology developed by Wilks Enterprise Inc., uses IR analysis to determine TPH concentrations in samples of diverse matrices including soil and liquid samples. Two different models can be operated using the same device: Model CVH and Model HATR-T2.

The two models are differentiated by the sample stage with which they work. As shown in Figure A-10, Model CVH uses a quartz cuvette cell with Teflon stopper, while model HART-T2 uses a built-in cubic zirconia horizontal attenuated total reflection (HATR) stainless steel sample stage. Samples are extracted with Freon 113 or Vertrel® MCA for Model CVH and Model HATR-T2 respectively. Before extraction, samples have to be dried with a silica gel. Freon 113 is a chlorofluorocarbon (CFC) and its production has been banned. However, substitutions such as a hydrocarbon-free grade of perchloroethylene, AK-225 S-316, or other IR transparent solvent can be used as the extracting solvent.

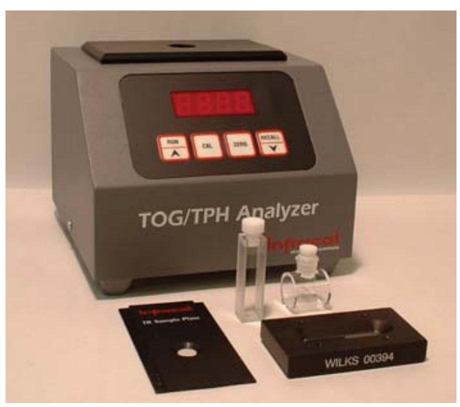

Figure A-10. Infracal TOG/TPH Analyzer (http://www.wilksir.com/analyzers.htm).

Advantages: The user can connect the device to a personal computer, and download and save measurement results. Also, the device can be connected to an external battery pack or to an automobile cigarette lighter. Contrary to conventional spectrophotometers, this device uses a pulse, infrared device instead of a chopper to prevent measurement fluctuations resulting from mechanical wear (EPA SITE program report).

Limitations: Accuracy of the device could be affected by naturally occurring oil and grease present in soil that is not removed by silica gel. Additionally, soil moisture can affect accuracy of TPH results for diesel soil samples. Fingerprints also absorb in the infrared region. Therefore, care must be taken to remove fingerprints from the cuvette cell.

FIELD PORTABLE XRF

Field portable x-ray fluorescence (FPXRF) spectroscopy analysis is a technique based on the exposure of solid samples to an x-ray source. Every element present in a given sample will emit a unique set of characteristic x-rays. Therefore, a qualitative analysis can be performed from characteristic energy, or wavelength of the fluorescent x-rays emitted. If a quantitative elemental analysis is desired, it can be determined from the intensity of the x-rays at a given wavelength (EPA 2006).

According to the EPA, an XRF analyzer consists of three major components:

1. The source – where the x-rays are generated (it could be a radioisotope or x-ray tube)

2. The detector – where the x-rays emitted from the sample are converted into measurable electronic signals

3. The data processing unit – where the emission or fluorescence energy signals are saved and where the elemental concentrations in the sample are calculated.

The XRF technique is intended mostly for the detection of heavy metals from various matrices (soil, alloy metal, filters, other solids, and thin samples). The EPA, under the SITE program, has recently evaluated some of the XRF technologies available on the market. As part of this document, however, only the fully portable devices will be compared: The X-MET 3000TX XRF Analyzer by Oxford Instruments Analytical, Inc, the XLt 700 Series XRF Analyzer and XLi 700 Series XRF Analyzer by Niton, and the XT400 XRF Analyzer by Innov-X Systems, Inc.

These devices present some common characteristics. First, no analytical chemistry skills are needed to operate the devices. Basic training, usually for 1 to 3 days, is offered by the equipment vendor. Additionally, the cost of these devices varies form $30,000 to $42,000, and rental is also available for approximately $1,500 to $2,000 per week. Regarding operator safety, the vendors claim that radiation exposure due to handling and operation of these devices is minimal and would never exceed exposure limits. To minimize x-ray tube emissions, however, shielding and other safety measures are strongly recommended.

The following performance descriptions are based on the EPA SITE program report. For simplicity, when describing accuracy descriptions, just the high and very low limits are mentioned. More detailed information can be found in the source documents.

X-MET 3000TX XRF Analyzer

The X-MET 3000TX XRF Analyzer (Figure A-11), manufactured by Oxford Instruments Analytical, Inc is an XRF technology that works with a miniature x-ray tube and a Peltier-cooled silicon-PiN (Si-PiN)diode x-ray detector. Soils, sediments, and other thick homogenous samples can be evaluated for a variety of elements, from Titanium to Uranium, simultaneously. Elements from Potassium to Scandium can be also evaluated but with higher detection limits. To facilitate data collection in the field, data can be managed and stored in a removable personal data assistant (PDA) attached to the instrument.

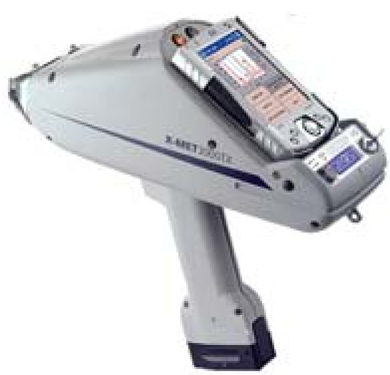

Figure A-11. X-MET 3000TX XRF Analyzer (www.oxford-instruments.com).

Instrument accuracy was also evaluated on the EPA SITE program report. Accuracy was ranked as high, moderate, low and very low. The demonstration concluded that the X-MET 3000TX XRF Analyzer has high accuracy for cadmium and selenium but very low accuracy for antimony and mercury. According to the demonstration, an average of 80 to 120 samples can be evaluated per 8 hour workday.

Niton XLt 700 Series XRF Analyzer

The XLt 700 Series XRF Analyzer (Figure A-12), a Niton Analyzers product, also works with a miniaturized x-ray tube source and a Peltier-cooled diode. However, x-rays are detected with a Si-PiN diode x-ray detector. Soil and sediment samples can be evaluated for a total of 25 elements. With the installation of optional software, the XLt 700 Series XRF Analyzer is capable of analyzing light elements, which include vanadium and chromium. To facilitate data storage, the instrument has an integrated touch-screen display and data can be transferred via Bluetooth wireless communication to a laptop computer or PDA.

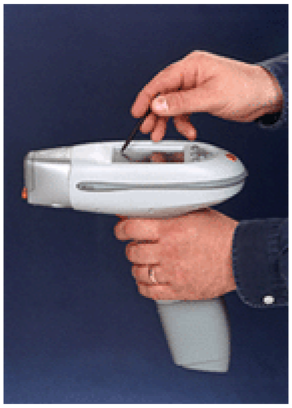

Figure A-12. XLt 700 Series XRF Analyzer (www.environmental-expert.com).

Instrument accuracy was also evaluated on the EPA SITE program report. Accuracy was ranked as high, moderate, low, or very low. The evaluation concluded that the XLt 700 Series XRF Analyzer has high accuracy for copper and selenium but is very low for antimony and mercury. EPA estimated that an average of 91 samples can be evaluated per 8 hour workday.

XLi 700 Series XRF Analyzer

Unlike the previous instrument, the XLi 700 Series XRF Analyzer, also manufactured by Niton Analyzers, determines element concentrations with an isotope-based XRF. The instrument (Figure A-13) can be adjusted with different isotope options depending on the environmental application needed. Up to 25 elements can be evaluated with this technology, depending on the isotope selected. As with the XLt 700 Series XRF Analyzer, data storage is facilitated with an integrated touch-screen display and data can be transferred via Bluetooth wireless communication to a laptop computer or PDA.

Figure A-13. XLi 700 Series XRF Analyzer (www.environmental-expert.com)

Instrument accuracy was also evaluated on the EPA SITE program report. Accuracy is ranked as high, moderate, low, or very low. The evaluation concluded that the XLi 700 Series XRF Analyzer was highly accurate for selenium. However, the instrument showed very low accuracy for antimony, mercury, silver, and vanadium. EPA estimated that an average of 53 samples can be evaluated per 8 hour workday.

XT400 XRF Analyzer

The XT400 XRF Analyzer, manufactured by Innov-X Systems, Inc, combines a rugged x-ray tube excitation source and a Light Element Analysis Program (LEAP) for the detection of up to 25 elements simultaneously, from Potassium to Uranium. To facilitate data collection in the field, data can be managed and stored in a removable PDA attached to the instrument (Figure A-14).

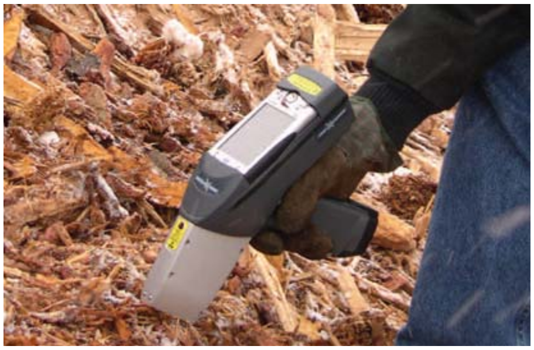

Figure A-14. XT400 XRF Analyzer
(www.innovxsys.com/products).

Instrument accuracy was also evaluated on the EPA SITE program report. Accuracy is ranked as high, moderate, low, or very low. The demonstration concluded that the XT400 XRF Analyzer was highly accurate for Cadmium. However, the instrument showed very low accuracy for Vanadium. EPA estimated that an average of 86 samples can be evaluated per 8 hour workday.

SUMMARY

Tables A-3 and A-4 summarize the technologies evaluated in this PWTB and the advantages and limitations for each.

Table A-3. Summary of Technologies Evaluated.

Technology	Field Testing Device	Vendor
Immunoassay (ELISA)	EnSys Petro Test System	Strategic Diagnosis Incorporated
	EnSys PCB Soil Test Kit	Strategic Diagnosis Incorporated
	RaPID Assay PCB RaPID BTEX/TPH Test Kits	Strategic Diagnosis Incorporated
Colorimetric Tests	Clor-N-Soil/Clor-N-Oil Test Kits	Dexsil Corporation
	Expray and DropEx Test Kits	Plexus Scientific
	EnSys TNT/RDX Soil Test System	Strategic Diagnosis Incorporated
Electrochemical Analysis	L2000 PCB/Chloride Analyzer	Dexsil Corporation
Friedel-Crafts Reaction	Remedi Aid Starter Kit	CHEMetrics
Infrared Analysis	Infracal TOG/TPH Analyzer	Wilks Enterprise Inc.
Field Portable XRF	X-MET 3000TX XRF Niton XLt 700 Series XRF XLi 700 Series XRF XT400 XRF	Oxford Instruments Analytical,Inc. Niton Analyzers Niton Analyzers Innov-X Systems

Table A-4. Summary of Advantages and Limitations for Field Test Kits Evaluated.

Technology	Field Testing Device	Vendor	Analytes	Advantages	Limitations
Immunoassay (ELISA)	EnSys Petro Test System	Strategic Diagnosis Inc.	Gasoline Diesel Jet A fuel Kerosene No. 2 fuel oil No. 6 fuel oil Mineral spirits	Basic wet chemistry skills needed. 1 to 12 samples can be evaluated within 45 min. The device can be easily moved. The components are battery-operated.	Does not generate quantitative results. Unable to differentiate between different petroleum compounds
	EnSys PCB Soil Test Kit	Strategic Diagnosis Inc.	PCB compounds	Two detection levels provided: (1) 0.5-500 ppm for soil samples (2) 5-5,000 ug/100cm^2 for wipe samples Hardware comes in a plastic, portable briefcase with boxes of test and extraction kits.	Soil containing high levels of petroleum fuels or transformer oil may affect results. The color substrate can be affected by direct sunlight.
	RaPID Assay PCB RaPID BTEX/TPH Test Kits	Strategic Diagnosis Inc.	PCB Aroclors	Minimum chemical skills and brief training needed. Approximately 10 to 11 samples can be evaluated per hour. Test kit provides accurate results either as a complement to lab generated data or as stand-alone data.	The test does not differentiate between PCB and related compounds. Their presence can affect measurement results.
Colorimetric Tests	Clor-N-Soil/ Clor-N-Oil Test Kits	Dexsil Corporation	PCB compounds	Low-cost per sample. Little or no technical expertise required to operate. High sample throughput (48 samples/day)	Tendency for false positives. These are more likely when interferants such as halogenated organic compounds are present in the sample. Mercury can cause false negatives when it is present with PCBs.

Technology	Field Testing Device	Vendor	Analytes	Advantages	Limitations
	Expray and DropEx Test Kits	Plexus Scientific	Expray and DropEx 1 TNT Tetryl Trinitrobenzene (TNB) Dinitrotoluene (DNT) Picric acid and its salts Expray and DropEx 2 Dynamite Nitroglycerine (NG)RDX Pentaerythritol tetranitrate (PETN) Semtex Nitrocellulose(NC) Smokeless powder Tetryl Expray and DropEx 3 Nitrate-based explosives Black powder Flash powder Potassium nitrate Ammonium nitrate DropEx A and B Chlorates Peroxide-based explosives.	No analytical chemistry skills are needed to operate.No sample preparation required.High sample throughput.	The system does not generate quantitative results.Tendency to false positive when fertilizers are present. The kit does not distinguish between fertilizer and nitrate based explosives.Some field samples may not react fully with the Expray solution, yielding false negatives.
	EnSys TNT/RDX Soil Test System	Strategic Diagnosis Inc.	TNT and RDX	If using both tests together, the same sample extract generated with the TNT test can be reused with the RDX test.	In the RDX test, nitrates and nitrites can cause false positives. If used for water samples, a solid phase extraction is required.
Electrochemical Analysis	L2000 PCB/Chloride Analyzer	Dexsil Corporation	PCB compounds	Four matrices can be evaluated: transformer oil, soil, water, and wipe samples. Powered by a rechargeable 8 volt battery.	The presence of sources of organic chlorine other than PCBs can result in higher concentration readings or even false positives. False negatives are also possible when analyzing Aroclors other than 1242. The device loses its calibration easily and must be recalibrated frequently.

Technology	Field Testing Device	Vendor	Analytes	Advantages	Limitations
Friedel-Crafts Reaction	Remedi Aid Starter Kit	CHEMetrics	BTEX PAHsDiesel fuelLeaded and unleaded gasolineWeathered gasoline Brent crudeLubricating oil	Basic wet chemistry skills. No pipette skills are required and the user’s contact with reagents is minimized by premeasured ampoules. The kit is powered by a 9 volt battery.	Different calibrations are required to evaluate specific types of contaminants. The device does not measure aliphatic hydrocarbons when aromatic compounds are not present in the sample.
Infrared Analysis	Infracal TOG/TPH Analyzer	Wilks Enterprise Inc.	BTEX PAHs Diesel fuel Leaded and unleaded gasoline Weathered gasoline Brent crude Lubricating oil	The device can be connected to a personal computer, external battery pack or to an automobile cigarette lighter. This device uses a pulse, infrared device instead of a chopper to prevent measurement fluctuations resulting from mechanical wear.	Accuracy of the device could be affected by naturally occurring oil and grease present in soil that is not removed by silica gel. Soil moisture can affect accuracy of TPH results for diesel soil samples. Fingerprints also absorb in the infrared region. Therefore, care must be taken to remove fingerprints from the cuvette cell.
Field Portable XRF	X-MET 3000TX XRF Niton XLt 700 Series XRF XLi 700 Series XRF XT400 XRF	Oxford Instruments Analytical Niton Analyzers Niton Analyzers Innov-X Systems	Heavy metals	Can detect heavy metals from various matrices (soil, alloy metal, filters, other solids, and thin samples). No analytical chemistry skills are needed to operate the devices. High sample throughput - More than 50 samples/day	Radiation exposure is possible. Shielding and other safety measures are strongly recommended.

APPENDIX B:

ARMY EXAMPLES

The Innovative Technologies section of the U.S. Army Corps of Engineers Environmental Community of Practice (eCoP) website presents some successful Army examples of cleanup and remediation efforts at hazardous waste sites. In all these cases, the use of field testing technologies has saved time and money in characterization and remediation projects. These examples are described below. Contact information and more detailed descriptions of these cases are available on the eCoP website.

Fort Campbell, KY
Immunoassay technologies were used at Fort Campbell to determine the extent of PCB contamination at various sites. This project, executed by the U.S. Army Corps of Engineers' Nashville District under the Installation Restoration Program with a dynamic sample screening approach, accelerated the remedial action by several months.

Presidio of San Francisco, CA
In the case of the Presidio of San Francisco, immunoassay technologies were also implemented successfully. The Sacramento District used this field testing technology to verify compliance with the Presidio's Site Cleanup Requirements. Cost difference between using a traditional laboratory approach and the use of immunoassay was approximately $600,000-$700,000.

Santa Rosa Army Airfield, CA
In this effort, the Sacramento District recommended the use of real-time soil sample analysis for the characterization of a firing range and skeet range at the Santa Rosa Army Airfield. Soil samples were analyzed with Field Portable Energy Dispersive X-ray Fluorescence. The ranges were characterized in less than 2 weeks saving the Sacramento District approximately $30,000.

West Virginia Ordnance Works
Immunoassay field testing technologies were used to determine the extent of dinitrotoluene and TNT contamination at the West Virginia Ordnance Works in Pt. Pleasant, WV. This approach saved time and lowered analytical costs to the Nashville District.

APPENDIX C

REFERENCES

Bjella, K. L. 2005. Pre-Screening for explosives residues prior to HPLC analysis utilizing expray. ERDC/CRREL TN 05-2. Hanover, NH.

CHEMetrics, Inc website: http://www.chemetrics.com/TPH.html

Dexsil Corp. website: https://secure.dexsil.com/

Environmental Protection Agency (EPA). 2007. SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, United States EPA Office of Solid Waste, February 2007.

EPA Technology Innovation Program (CLU-IN website) http://www.clu-in.org/char/technologies/color.cfm

EPA. 1995. Innovative Technology Evaluation Report. EPA/540/R-95/518. Clor-N-Soil PCB Test Kit, Dexsil Corp.

EPA. 1998. EPA Environmental Technology Verification Report. EPA/600/R-98/111. Immunoassay Kit. Strategic Diagnosis Inc. RaPID Assay System for PCB Analysis.

EPA. 1998. EPA Environmental Technology Verification Report. EPA/600/R-98/109. Electrochemical Technique/Ion Specific Electrode. Dexsil Corporation L2000 PCB/Chloride Analyzer.

EPA. 2000. United States Environmental Protection Agency (EPA). SITE Demonstration Plan. Field Measurement Technologies for Total Petroleum Hydrocarbons in Soil. EPA/600/R-01.

EPA. 2006. EPA Innovative Technology Verification Report. EPA/540/R-06/008. XRF Technologies for Measuring Trace Elements in Soil and Sediment. Oxford X-Met 3000TX XRF Analyzer.

EPA. 2006. EPA Innovative Technology Verification Report. EPA/540/R-06/004. XRF Technologies for Measuring Trace Elements in Soil and Sediment. Niton XLt 700 Series XRF Analyzer.

EPA. 2006. EPA Innovative Technology Verification Report. EPA/540/R-06/002. XRF Technologies for Measuring Trace Elements in Soil and Sediment. Innov-X XT400 Series XRF Analyzer.

Jenkins, T. F., and M. E. Walsh. Field-Based Analytical Methods for Explosive Compounds. Hanover, NH:U.S. Army Engineer Research and Development Center.

Strategic Diagnostics Inc. website: http://www.sdix.com/

U.S. Army Corps of Engineers Environmental Community of Practice (eCoP) website: https://ekopowered.usace.army.mil/ecop/tools_info/it/

Wilks Enterprise Inc. website: http://www.wilksir.com/analyzers.htm