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FUELS IN SOIL TEST KIT: FIELD USE OF DIESEL DOG® SOIL TEST KITS

Topical and Final Report

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DISCLAIMER

ABSTRACT

Western Research Institute (WRI) has developed a new commercial product ready for technology transfer, the *Diesel Dog*® Portable Soil Test Kit, for performing analysis of fuel-contaminated soils in the field. The technology consists of a method developed by WRI (U.S. Patents 5,561,065 and 5,976,883) and hardware developed by WRI that allows the method to be performed in the field (patent pending). The method is very simple and does not require the use of highly toxic reagents. The aromatic components in a soil extract are measured by absorption at 254 nm with a field-portable photometer. WRI added significant value to the technology by taking the method through the American Society for Testing and Materials (ASTM) approval and validation processes. The method is designated as ASTM Method D 5831-96, Standard Test Method for Screening Fuels in Soils. This ASTM designation allows the method to be used for federal compliance activities. In June 2001, the *Diesel Dog* technology won an American Chemical Society Regional Industrial Innovations Award.

To gain field experience with the new technology, *Diesel Dog* kits have been used for a variety of site evaluation and cleanup activities. Information gained from these activities has led to improvements in hardware configurations and additional insight into correlating *Diesel Dog* results with results from laboratory methods. The Wyoming Department of Environmental Quality (DEQ) used *Diesel Dog* Soil Test Kits to guide cleanups at a variety of sites throughout the state. ENSR, of Acton, Massachusetts, used a *Diesel Dog* Portable Soil Test Kit to evaluate sites in the Virgin Islands and Georgia. ChemTrack and the U.S. Army Corps of Engineers successfully used a test kit to guide excavation at an abandoned FAA fuel-contaminated site near Fairbanks, Alaska. Barenco, Inc. is using a *Diesel Dog* Portable Soil Test Kit for site evaluations in Canada. A small spill of diesel fuel was cleaned up in Laramie, Wyoming using a *Diesel Dog* Soil Test Kit.

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EXECUTIVE SUMMARY

Western Research Institute (WRI) is commercializing *Diesel Dog*® Portable Soil Test Kits for performing analysis of fuel-contaminated soils in the field. The method that the kit is used to perform involves mixing 5 g of soil with 5 g of calcium oxide, which dries the soil and binds humic materials to prevent interference. A 50-mL portion of isopropyl alcohol is added, and the mixture is mechanically stirred for three minutes. The kit includes a unique stirrer/extractor. The extract is then filtered using a disposable syringe and filter, and the aromatic components are measured by reading the absorbance of the extract at 254 nm. A portable UV photometer is included in the soil test kit to measure absorbance. The method is very simple and does not require the use of highly toxic reagents. In previous years, WRI added significant value to the technology by taking the new soil screening method through the American Society for Testing and Materials (ASTM) approval and validation processes. The method is designated as ASTM Method D 5831, Standard Test Method for Screening Fuels in Soils. This ASTM designation allows the method to be used for federal compliance activities. The technology consists of a method developed by WRI (U.S. Patents 5,561,065 and 5,976,883) and hardware developed by WRI that allows the method to be performed in the field (patent pending). In June 2001, the *Diesel Dog* technology won an American Chemical Society Regional Industrial Innovations Award.

Early in the current task, twenty-five preproduction *Diesel Dog* Soil Test Kits were produced. This included designing and constructing portable extractors and photometers. Technical computer-assisted design (CAD) drawings were prepared. Component fabrication was outsourced. All individual parts and components were assigned part numbers. The preproduction kits were constructed in cooperation with CF Electronics of Laramie, Wyoming.

To gain field experience with the new technology, *Diesel Dog* kits have been used for a variety of site evaluation and cleanup activities. This has provided insight on correlating *Diesel Dog* results with results from laboratory methods. The Wyoming Department of Environmental Quality (DEQ) used *Diesel Dog* Soil Test Kits to guide cleanups at a variety of sites throughout the state. ENSR, of Acton, Massachusetts, used a *Diesel Dog* Portable Soil Test Kit to evaluate sites in the Virgin Islands and Georgia. ChemTrack and the U.S. Army Corps of Engineers successfully used a test kit to guide excavation at an abandoned FAA fuel-contaminated site near Fairbanks, Alaska. Barenco, Inc. is using a *Diesel Dog* Portable Soil Test Kit for site evaluations in Canada. A diesel fuel spill was cleaned up in Laramie, Wyoming using a *Diesel Dog* Soil Test Kit. Based on field analysis results, the kits were upgraded with circuit modifications, new polyethylene foam inserts, alternate extractor impellers, and updated instruction manuals. The option of using reagent-grade n-heptane was added to the method to allow for the analysis of peaty plant material.

OBJECTIVES

The objectives of the three-year effort included finalizing the soil test kit design and assembling twenty-five preproduction units for evaluating fuel contamination in soil using the patented technology. This involved working closely with a manufacturing firm to commercialize the technology. Another objective was to provide units to the cosponsors who performed field evaluations using the test kits as opportunities arose. Western Research Institute (WRI) also provided technical support, assisted in interpreting and communicating results, and modified the test kits based on feedback from field experience.

INTRODUCTION

Background

WRI has developed *Diesel Dog*® Portable Soil Test Kits for performing analysis of fuel-contaminated soils in the field. The technology consists of a method developed by WRI (U.S. Patents 5,561,065 and 5,976,883) and hardware developed by WRI that allows the method to be performed in the field (patent pending). The method is very simple and does not require the use of highly toxic reagents. The aromatic components in a soil extract are measured by absorption at 254 nm using a field-portable photometer. WRI added significant value to the technology by taking the method through the American Society for Testing and Materials (ASTM) approval and validation processes. The method is designated as ASTM Method D 5831-96, Standard Test Method for Screening Fuels in Soils (ASTM 2001). This ASTM designation allows the method to be used for federal compliance activities. In June 2001, the *Diesel Dog* technology won an American Chemical Society Regional Industrial Innovations Award.

The presence of diesel fuel and heavier fuels in soils is an important environmental issue. State cleanup standards are typically site specific, and are either health-based or arbitrary. A typical recommended cleanup standard is 100 mg/Kg, although these vary from state to state (Nascarella et al. 2002). Health-based guidelines range from 1,166 to 11,287 mg/Kg (Millner et al. 1992). As the diesel in soil is weathered and subjected to bacterial degradation, the remaining fuel is more aromatic than the starting material and less volatile (Douglas et al. 1992). Unlike more volatile gasoline, diesel fuel cannot be readily measured using a portable organic vapor analyzer (OVA) based on either photoionization detection (PID) or flame ionization detection (FID).

U.S. Environmental Protection Agency (EPA) laboratory methods are based on Soxhlet or ultrasonic extraction of soils prior to measurement of organics using gas chromatographic (GC) separation (U.S. EPA 1996a). The Massachusetts Department of Environmental Protection extractable petroleum hydrocarbon (MADEP EPH) method (MADEP 1995) involves an additional step of removing solvent from a soil extract and redissolving the extract in heptane prior to separation into saturate-rich and aromatic-rich fractions using a silica-based Sep Pak™ syringemounted cartridge (Schabron et al. 1997). These methods provide information on the type and distribution of fuel components; however, they are not suitable for field use. A variety of field screening methods have been introduced to define the boundaries of contamination at a site and to allow informed decisions to be made as to where samples should be taken for more expensive and extensive laboratory analyses. Such methods can also result in cost savings for site excavations by minimizing the incidence and costs of unnecessary removal of uncontaminated soils. These methods are described in the following section.

All field and laboratory methods for analyzing soils for hydrocarbon contaminants are only as good as the information available about the specific contaminant and the availability of a portion of the contaminant for standardization (Rhodes et al. 1996). Since the contaminant fuel is rarely available for calibration, the results are dependent on the method and calibration material used. Thus, the various methods rarely provide comparable or truly accurate results. The common laboratory methods, which involve gas chromatographic separations, usually disregard the presence of materials heavier than diesel-range contaminants. As a result, used motor oil and fuel oil contaminants are rarely reported. No two fuel analysis methods (laboratory or field) should be expected to give the same answer, because of the tremendous variation in fuels and the different principles of measurement. Typically, a laboratory reference method is agreed upon by a regulatory agency, site owner, and contractor. A field screening method is often used to select the locations from which samples are collected for laboratory analysis. The manner in which a field screening method relates to the laboratory method can become an issue in site cleanup. It is important that the field method provides no false negative results when guiding rapid cleanup and excavation activities. In project planning, selection of the field and laboratory methods to be used is very important to site remediation.

Current Field Methods

Several approaches are available for field testing for various types of fuel contaminants in soils. Typically the acronym TPH, Total Petroleum Hydrocarbons, is used interchangeably and loosely in the methods. Each approach has strengths and weaknesses that must be understood so that it is used appropriately. As mentioned, none of the methods available are necessarily “correct,” unless the exact fuel that is being determined is used for calibration. Fuels purchased at different filling stations and at different times from locations around the country show variations in composition. This is especially true of diesel fuels, which have a higher boiling range than gasoline and can contain a greater variety of components. Any field or laboratory method for fuels, including the ones described in this report, will yield results dependent on the fuel source and type.

A kit for measuring TPH in soil using a chlorofluorocarbon solvent extraction followed by slow gravity filtration and infrared (IR) spectroscopy measurement has been described (Grant and Taliadourous 1992). This methodology requires a solvent that does not contain the C-H functionality since that is what is measured by IR. For this reason, chlorofluorocarbon solvents such as Freon^{TM} have been used in the past. The use of such solvents has been phased out due to the deleterious effect of the solvent vapors on the earth's ozone layer and their persistence in the environment. Recently, IR methods have been modified to use tetrachloroethylene (a carcinogen) as a solvent. A kit is available for performing a soil extraction with methanol using manual agitation. The extract is mixed with salt water, which causes the hydrocarbons to be insoluble and results in a cloudy, emulsion-like appearance of the extract (Dexsil 1995). Fuel content is estimated by measuring the extract turbidity. Since solubility is the key to this measurement, any factor that affects solubility can affect the test results. For example, water acts as a negative interference because it is difficult to extract hydrocarbons from a wet soil. Solubility is also affected by the aliphatic components of the fuel and the temperature during analysis.

An immunoassay field test kit for hydrocarbon contamination has been developed (Allen et al. 1992). Immunoassay involves manual extraction of the sample with methanol followed by several steps for visual color development. The color development components must be kept refrigerated until they are used. Reaction rates for the multiple steps are temperature dependent, and skilled operators are required. For these reasons, the usefulness of immunoassay methods for field analysis has been questioned (Friedman 1996).

A kit based on the Friedel-Krafts alkylation reaction for detecting aromatic rings is also available (Hanby 1995). The aluminum chloride catalyst used in this kit reacts violently with water, and one of the solvents used in the kit, carbon tetrachloride, is a carcinogen. In addition, wet clay soils are difficult to extract due to poor mixing with the mixture of heptane and carbon tetrachloride used for extraction.

ASTM Method D 5831, Standard Test Method for Screening Fuels in Soils

As mentioned, a new field screening method for determining the presence of fuels containing aromatic components, particularly diesel and heavier fuels, in soils was developed by WRI (Schabron et al. 1995, Sorini and Schabron 1997), along with the *Diesel Dog* Portable Soil Test Kit for performing the method. National acceptance of the technology has been facilitated by the existence of ASTM Method D 5831-96, Standard Test Method for Screening Fuels in Soils (ASTM 2001), because Public Law 104-113 (March 7, 1996) states, "...all federal agencies and departments shall use technical standards that are developed by voluntary consensus standards bodies."

The ASTM method measures the aromatic components in a soil extract by absorption at 254 nm using a laboratory spectrophotometer or a field-portable photometer. The method is very simple. It does not involve complicated color development steps or require the use of highly toxic reagents. The method involves mixing 5 g of soil with 5 g of calcium oxide, which dries the soil and binds humic materials to minimize interference. The soil is then extracted for three minutes with a 10:1 ratio (v:w) of reagent-grade isopropyl alcohol using mechanical mixing. Extraction is not performed by manual agitation, as is the common practice for most field analysis methods, because manual agitation has been shown to be inefficient and non-repeatable (Schabron et al. 1995). The extract is filtered using a disposable Teflon® syringe filter, and its absorbance is read at 254 nm using a UV photometer. This provides a direct measurement of the aromatic components, which are the most persistent in the environment over time and represent the most toxic of fuel components. New or old sites contaminated with diesel or heavier fuels (fuel oil, petroleum, coal tar) are best evaluated using this method. Lighter fuels, such as gasoline, are best screened with OVAs. Since OVAs are not suitable for measuring diesel or heavier fuels, both an OVA and a *Diesel Dog* Soil Test Kit should be used in the field.

ASTM Method D 5831 and its performance with various soils and fuel types has been discussed in detail elsewhere (Schabron et al. 1995, Schabron et al. 1998). Prototype test kits were used in a national study to validate the new ASTM method (Sorini and Schabron 1997). The method has been tested successfully with a variety of soils, fuel types, and conditions (Butler et al. 1997; Schabron et al. 1997; Sorini et al. 1997; Sorini and Schabron 1996).

The ASTM method can be used for three tiers of analysis. If a sample of the contaminant fuel is available for calibration of the photometer, a quantitative analysis can be performed. If the contaminant fuel type is known but a sample of the contaminant fuel is not available for calibration, an estimate of the contaminant concentration can be determined using average response factors provided in the method. If the nature of the contaminant is unknown, the screening method can be used to identify the possible presence of contamination.

RESULTS AND DISCUSSION

Interpreting Data and Comparing Results of Various Methods

The results from ASTM Method D 5831 are not expected to be quantitatively identical to results from other measurement methods, which in turn may not be identical to one another. This is due to the variation in composition of diesel and other fuels from various sources, the different measurement principles and calibration standards used by various laboratory and screening methods, and differences between the portions of soil samples analyzed. When compared with laboratory GC methods, the results from screening methods should not be expected to be numerically identical. The EPA SW846 GC method, 8015B (U.S. EPA 1996b), states that calibration should be performed using the specific fuel that is contaminating the site, and that when such a standard is not available, a recently purchased commercially available fuel should be used. Therefore, in cases where the actual contaminant fuel is not available for calibration, the results are estimates at best. However, for field screening methods to be useful, their results must correlate with laboratory results.

In many cases, EPA Method 8015B is the benchmark method for analysis of fuel-contaminated soils in the laboratory, and as a result, data from analyses using this method are often the basis for decisions concerning site activities. Therefore, in many cases, data generated using ASTM Method D 5831 are compared to 8015B laboratory data, and it is important that the data from the two methods correlate, even when they provide only estimates of fuel contamination.

Determination of a Response Factor for Weathered Diesel Fuel for Inclusion in ASTM D 5831

For a fresh diesel spill, the diesel response factor of 209 (mg/L)/AU that is given in ASTM Method D 5831 is appropriate to use in calculations to estimate diesel contamination in cases where the actual contaminant fuel is not available for calibration. Use of this response factor for fresh diesel contamination generally provides an estimate of the diesel concentration in the soil that correlates with laboratory GC results. However, as previously mentioned, as diesel fuel in soil is subjected to bacterial degradation and weathering processes, the remaining fuel is more aromatic than the starting material and less volatile (Douglas et al. 1992). These weathered fuels can contain heavier hydrocarbon materials ($>C_{22}$). Components in fuels above C_{22} are not detected well by the 8015B GC method. Therefore, a different response factor is required for use in calculations to estimate weathered diesel concentrations in soil that will correlate with laboratory GC results. For this reason, laboratory data from analysis of weathered diesel-contaminated soils using EPA Method 8015B were used to calculate a response factor for weathered diesel fuel in soil for use in ASTM Method D 5831. This value, which is 58 (mg/L)/AU, was calculated using data from analysis of nine samples collected from various locations at four different field sites. The response factors that were used to calculate the value of 58 (mg/L)/AU are shown in Table 1. A proposal to include this response factor for weathered diesel fuel in the table listing response factors in the ASTM method has been balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization and ASTM Main Committee D 34 on Waste Management.

Determination of a Site-Specific Response Factor for Using ASTM D 5831

Field screening results are site specific and dependent on the contaminant. As discussed, it is important that when performing ASTM Method D 5831, the appropriate response factor that relates to the aromaticity of the contaminant fuel is used. If site data from laboratory analyses are available, and if there is time, field analyses using D 5831 can be performed prior to the start of field work to determine an appropriate response factor for the particular site. In this way, a response factor that is specific to the contaminant fuel, soil type, laboratory method, etc. can be selected for use in screening soil samples from the site.

Diesel Dog Soil Test Kit

The *Diesel Dog* Soil Test Kit is portable and easy to use in the field. Figure 1 shows the components of the soil test kit, and Figure 2 shows the 12-volt portable soil extractor and 12-volt portable UV photometer that are major components of the kit. The portable soil extractor is a unique unit in that soils can be extracted efficiently in the field without manual shaking. As mentioned, manual agitation has been shown to be inefficient and non-repeatable (Schabron et al. 1995). The portable soil extractor has only glass, Teflon®, and stainless steel wetted parts. The portable UV photometer is used to measure soil extract absorbance at 254 nm in the field. In addition, for convenience, individual foil-lined packets containing approximately 5 grams of calcium oxide are available. As mentioned, calcium oxide dries the soil and binds humic materials to minimize interference. ASTM Method D 5831 specifies the addition of 5-gram portions of calcium oxide to the soil sample prior to extraction. A typical setup of the soil test kit on the tailgate of a pickup truck at a sampling site is shown in Figure 3.

Performing ASTM Method D 5831

Consideration of the Method Quantitation Limit

The quantitation limit for a fuel type depends on four main factors. These factors are: (1) the solvent-to-soil ratio used for extraction, (2) the absorptivity of the fuel, (3) the instrumental detection limit, and (4) the background level in the blank soil extracts. Background level is a function of the soil type and amount of humic material and can be controlled by adding calcium oxide to the soil or selecting a different solvent (see the sections below on running blank analyses and solvent extraction). The sensitivity of ASTM Method D 5831 is related to the aromatic content of the fuel, because the method is based on measurement of absorbance at 254 nm due to the presence of aromatic rings in the fuel. The actual response will vary somewhat, even for similar fuel types from different sources, because fuels are complex mixtures of varying compositions within a boiling range. The method quantitation limits for highly aliphatic materials, such as aviation gasoline and synthetic motor oil, are much higher than those for more aromatic materials, such as coal oil and diesel fuel.

The quantitation limit of 75 mg/Kg for fresh diesel cited in ASTM D 5831 is fairly conservative and is based on three times the standard deviation of blank extract absorbance from a variety of soils (sand, silt, clay, potting soil) obtained under a variety of conditions using a response factor of 209 (mg/L)/AU. The quantitation limit reflects an extract absorbance of 0.036 AU (absorbance units) for a 10:1 solvent (mL):soil (g) extract, which corresponds to about 7.5 mg/L of diesel fuel in solution. The instrumental detection limit for a 10-mm pathlength quartz cell is about ten times lower than this. Some soils have essentially no blank extract absorbance, resulting in a lower quantitation limit. Using the response factor of 58, the method quantitation limit for weathered diesel fuel is 21 mg/Kg.

Selecting an Appropriate Response Factor

As mentioned, if the contaminant fuel type is known, but the contaminant fuel is not available, the concentration of the fuel in soil can be estimated using an average response factor. As discussed, it is important that when performing ASTM Method D 5831, the appropriate response factor that relates to the aromaticity of the contaminant fuel is used. If site data from laboratory analyses are available, and if there is time, field analyses can be performed prior to the start of field work to determine an appropriate response factor for the particular site. In this way, a response factor that is specific to the contaminant fuel, soil type, laboratory method, etc. can be determined for use in screening soil samples from the site. However, in many cases, there is not time for such preparatory work prior to starting field work. As a result, care should be taken in selecting the appropriate response factor from the list given in ASTM Method D 5831, with particular consideration given to whether the fuel contamination is fresh or has undergone weathering and/or biodegradation processes. Another factor to consider is whether the contamination is a mixture of one or more fuel types. If this is the case, and a site-specific response factor cannot be determined, the response factors for the individual fuel types in the mixture should be used to estimate the contaminant concentrations, and a decision on how to interpret the test results should be made.

Running Blank Analyses

Prior to the start of field work, a blank analysis of an uncontaminated soil sample should be performed. A soil sample collected from an area at the site known to be uncontaminated should be analyzed. Results from the blank analysis provide information on the blank soil absorbance value, the amount of calcium oxide required to dry the soil, and the time it takes for the soil and calcium oxide to settle after agitation. The blank absorbance value can also be used to set a criterion to define areas of contamination at a site.

For the first blank sample, approximately 5 grams of calcium oxide should be used. If the absorbance value of the first soil blank extract is less than 0.05, extraction of the soil samples at the site should be performed using 5 grams of calcium oxide. If the absorbance value of the first blank extract is greater than 0.05, a second blank sample should be extracted using additional calcium oxide. If a second blank analysis is required, approximately 10 grams of calcium oxide should be added to the soil sample. If the absorbance value of the second blank extract is lower than the first blank extract, but is still greater than 0.05, a third blank sample should be tested using approximately 15 grams of calcium oxide. These steps can be repeated until the blank absorbance value is less than 0.05. In this way, the amount of calcium oxide required to inhibit interferences from humic material and moisture in the soil can be determined. Excess calcium oxide will not affect the analysis results. If the absorbance value of the second blank extract is not decreased by the addition of 10 grams of calcium oxide to the blank sample or if the addition of calcium oxide does not lower the absorbance of the blank extract to less than 0.05, even with the addition of a large quantity of the conditioning agent, and the absorbance of the blank extract is less than 0.1, the blank absorbance value can be subtracted from the sample absorbance values. If this is done, blank samples from around the site should be tested to ensure that blank absorbance is constant by ± 0.02 absorbance units. If the blank absorbance for the second blank is not decreased by the addition of 10 grams of calcium oxide and the absorbance of the blank extract is greater than 0.1 or if blank correction is not desired, use of a different extraction solvent, such as n-heptane, should be considered. This is discussed in the section below on extraction solvent.

In preparation for testing to be performed in Alaska, the guidance given above was used for testing Alaskan tundra peat, a large part of which is plant material. When a 5-gram packet of calcium oxide was added to a 5-gram sample of uncontaminated Alaskan tundra peat, the blank absorbance was 0.13; however, after the addition of three packets of calcium oxide to a blank Alaskan tundra peat sample, the blank absorbance value was reduced to 0.07. At this point in the testing, it was decided that no additional calcium oxide would be used because the extraction slurries were becoming very hot. This was most likely due to reaction of the calcium oxide with moisture in the sample and possibly some hydrolysis reactions with the plant material. It was also decided that blank correction would not be an option for the testing in Alaska, because of the variation in soil samples that would be tested. As a result, a decision was made that n-heptane should be used as the extraction solvent for samples of Alaskan tundra peat. This is discussed in the section below on extraction solvent.

Another example of soil that may also have high blank absorbance values is soil located under coniferous trees, because pine tar and turpentine respond as fuel when tested by this method. In testing these types of soil, it is recommended that if the blank absorbance value cannot be lowered to less than 0.05 by the addition of calcium oxide, the blank absorbance value should be subtracted from the sample absorbance values. However, as stated, this should only be done if the blank absorbance is less than 0.1. If the blank absorbance is greater than 0.1, the method should not be used to test the soil.

As mentioned, the soil blank is also used to determine the time required for the soil and calcium oxide mixture to settle after extraction, so the extract can be drawn into the syringe and filtered into the sample cuvette for analysis. A typical settling time is about three minutes. If the mixture is not allowed to settle, the filter could clog, and use of multiple filters would be required.

Extraction Solvent

The solvent specified by ASTM Method D 5831 for extraction of soil samples is reagent-grade isopropyl alcohol (IPA). IPA is a good solvent for many fuels, and is also a strong chromatographic solvent that can readily displace weathered fuel materials adsorbed onto soil surfaces. IPA works especially well with sandy soils, wet clays, and humic soils, such as potting and other organic-rich soils. However, for some soils, IPA does not perform well as a solvent for use in the ASTM method. For example, as mentioned, when D 5831 was used to test samples of Alaskan tundra peat, the absorbance value of the blank extract was very high. It was determined that this high absorbance value was due, in part, to the IPA extracting UV-absorbing plant material from the tundra. As a result, a decision was made to use n-heptane as the extraction solvent for testing samples of the Alaskan tundra peat. Heptane is an acceptable solvent for use in the method as long as the soil being tested is not so wet as to prevent good mixing.

Whether the solvent being used is IPA or n-heptane, it must be uncontaminated; that is, it must not have an absorbance value vs. air that is greater than 0.1. A solvent with a higher absorbance value should not be used. Typically, reagent-grade IPA and ACS n-heptane have absorbance values vs. air of less than 0.05. To maintain solvent purity, solvents should be stored in clean glass, metal, or inert Teflon® containers with lids that do not leach UV-absorbing contaminants. Solvents should not be stored for more than one week in plastic containers or containers with plastic lids, because UV-absorbing contaminants can be leached from plastics.

Sample Extraction

D 5831 specifies that the soil sample, calcium oxide, and IPA slurry is to be stirred for three minutes using a portable stirring device or magnetic stir bar and stirrer so that the solids are in motion during stirring. The portable soil extractor that is part of the *Diesel Dog* Soil Test Kit has a turbine blade impeller that works very well for stirring most soil slurries so that the solids are in motion. However, in testing the Alaskan tundra peat that consisted of stringy plant material, the blade impeller of the soil extractor did not stir the sample well. As a result, a short cylindrical impeller was designed for stirring this type of material using the portable extractor. The alternate impeller can be easily fitted to the soil extractor shaft by unscrewing the stainless steel screw and lock washer used to attach the turbine blade to the shaft. When performing ASTM Method D 5831, close attention should be paid to the extraction step to make sure that the solids are in motion. Testing soils that have an unusual composition may require use of the shorter, cylindrical impeller for extraction.

Syringes and Filters

The syringe used to collect the extract for injection into the sample cuvette for analysis must be made of polyethylene/polypropylene (disposable) or glass to prevent contamination of the extract. Hospital-style plastic syringes with lubricated elastomer plungers should not be used because these will contaminate the extract. The filters used to filter the extract as it is injected into the cuvette should be made of PTFE with nylon or polyolefin body construction. They should have a 25-mm diameter and a 0.45-micron pore size. A blank portion of solvent should be drawn into a syringe and passed through a filter into the cuvette for measurement of absorbance prior to testing to ensure that no UV-absorbing materials are being leached from the syringe and filter. The photometer absorbance reading should be less than 0.03 for the syringe/filter blank.

Proposed Revision of ASTM Method D 5831

Since development and approval of ASTM Method D 5831 in 1996, a great deal has been learned about application of the method from its use at many field sites throughout the country. Much of this information is discussed in the previous sections of this report. This information must also be incorporated into the ASTM method to improve its usefulness and update it. As a result, a proposed revision to ASTM Method D 5831 was prepared and balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization and ASTM Main Committee D 34 on Waste Management, so that the method will be updated with the important information that has been learned about its application. The proposed revision includes the response factor for weathered diesel fuel and information on the options of adding extra calcium oxide to inhibit interference, subtracting out blank absorbance values, and using n-heptane as the extraction solvent, as well as information on determining a site-specific response factor that is specific to the contaminant fuel, soil type, laboratory method, etc.

Case Studies with the *Diesel Dog* Soil Test Kits

Evaluation of Samples from a Weathered Site in Alaska

A *Diesel Dog* Soil Test Kit was evaluated at the U.S. Army Corps of Engineers facility in Anchorage, Alaska at Elmendorf Air Force Base. The conditions were austere; nevertheless, the equipment performed well. The extractions were performed on a two-foot square table top, and the photometer was operated on top of a cooler (Figure 4). The short cylindrical impeller designed for stirring samples containing fibrous material was used with the portable soil extractor.

Several different sample types were tested. These had been collected in previous sampling episodes, and many of the samples had been analyzed in the past for diesel range organics (DRO, C_{11}-C_{22}) by a single GC analysis using a non-site specific fresh diesel fuel standard for calibration. Several of the samples contained pea-size or larger rocks. To avoid error due to weighing rocks with small surface areas, the samples containing rocks were screened through a number 10 (2 mm) stainless steel screen. At an actual field site, a disposable bulk screen material could be used to screen out rocks and debris from samples.

Data generated by testing the various samples using ASTM Method D 5831 and the soil test kit, as well as laboratory data that were available for some of the samples, are presented in Table

2. The weathered diesel fuel response factor of 58 (mg/L)/AU was used for ASTM Method D 5831. As shown by Table 2, there is a reasonably close correlation between the soil test kit UV data and the GC data. This supports use of the UV method and soil test kit in the field to detect highly contaminated areas quickly.

In one case (Sample 6), the sample had a definite fuel odor; however, the GC method failed to detect the presence of significant quantities of fuel (>100 mg/Kg). This is possibly due to a rock being included in the weighed portion of sample analyzed by GC. No replicate GC analyses data were available for any of the samples.

As shown in Table 2, Sample 8, the uncontaminated tundra peat, was tested using n-heptane as the extraction solvent based on the preliminary tests previously discussed in this report. Heptane worked very well as the extraction solvent for this sample. No fuel contamination was detected in the sample, and there were no interferences from organic plant material.

Aged-Diesel Pond Site Evaluation

ENSR Corporation of Acton, Massachusetts used a *Diesel Dog* Soil Test Kit at a field site in the U.S. Virgin Islands at a Domino Oil/Enighed one-year-old pond site impacted by diesel fuel. The soil tested in the area was very wet and sandy. Data from *Diesel Dog* (ASTM Method D 5831) field analyses using the weathered diesel fuel response factor were compared with data generated by more expensive and lengthy MADEP EPH laboratory analyses. These data are shown in Table 3. Both the field method and the laboratory method detected hydrocarbon contamination in all of the samples. No false negatives were reported by the field method. As discussed, the results generated by the ASTM and MADEP EPH methods can be expected to vary because of the differences in measurement techniques.

Evaluation of a Diesel-Contaminated Site

ENSR Corporation also used a *Diesel Dog* Soil Test Kit at a site in Georgia that was contaminated with diesel fuel. The kit was used to screen samples collected at drilling points. Data from testing the samples in the field using the *Diesel Dog* (ASTM D 5831) Soil Test Kit and data from laboratory analysis of the samples using EPA Method 8015B are shown in Table 4. Using the response factor for weathered diesel fuel, the quantitation limit for the ASTM method is 21mg/Kg. Of the 17 samples analyzed by both methods, 12 were determined to contain <36 mg/Kg of contamination by both methods. These samples would be considered clean. Two samples with contamination levels of <12 mg/Kg by the EPA 8015B laboratory method were shown to contain contamination levels of 255 mg/Kg and 476 mg/Kg by ASTM Method D 5831. These samples probably contained relatively high molecular weight aromatic materials that were not detected by the GC method. Testing with the soil test kit gave no false negative results. The results from this study show that the *Diesel Dog* Soil Test Kit could be used to guide an excavation at this site, and that the user could be confident that laboratory data generated by EPA Method 8015B would show that the cleanup had been performed completely and successfully.

Excavation of a Decades-Old Diesel and Road Tar Site

The Wyoming Department of Environmental Quality (DEQ) used a *Diesel Dog* Soil Test Kit to guide excavation for cleanup at a decades-old, fuel-contaminated site near Jackson, Wyoming. The site was contaminated with diesel fuel and road oil from prior department of transportation activities. The soil at the site was a heavy, wet clay. Under supervision of the Wyoming DEQ, the engineering firm, Dames and Moore, excavated approximately 6,000 cubic yards of soil, including about 2,000 cubic yards of overburden. OVAs based on photoionization were not able to detect the contamination because the fuel was severely weathered. A *Diesel Dog* Portable Soil Test Kit was used by a chemical and civil engineer on the tailgate of a pickup truck to provide rapid field analysis. The *Diesel Dog* Soil Test Kit provided data within minutes.

Testing results using the ASTM method and GC laboratory method are shown in Table 5. The data for the ASTM method were calculated using the response factor for weathered diesel fuel (58 (mg/L)/AU). As mentioned, the other contaminant at the site was road tar. The location of the site in Wyoming suggests that the road tar came from highly aromatic oil from Recluse, Wyoming. To calculate the concentration of contaminant in the samples as road tar oil, the response factor for coal oil, which is 59 (mg/L)/AU, could also be used. As a result, contaminant concentrations that are very similar to those calculated using the weathered diesel fuel response factor would be calculated using the coal oil response factor. The laboratory data were obtained by a purge-and-trap sampling method for total volatile petroleum hydrocarbons (TVPH, C_6 - C_{10}) and by solvent extraction for total extractable petroleum hydrocarbons (TEPH, C_{11} - C_{28}). The data presented in Table 5 show no significant contamination in Samples 1–6. Data generated for Samples 1 and 3 using the ASTM method are significantly higher than the laboratory results. This is most likely due to the presence of relatively high molecular weight aromatic materials in the samples that would not be detected by the GC method. Significant contamination requiring action is shown in Samples 7 and 8 by both methods. This contamination was detected by the *Diesel Dog* Soil Test Kit as a "hot spot" area within two days before termination of site activities. The highly contaminated area was successfully excavated using *Diesel Dog* Soil Test Kit results, and confirmation of site cleanup was obtained from laboratory analysis more than a week after the effort was terminated. The *Diesel Dog* equipment performed reliably, and the field personnel stated that the ASTM method and soil test kit successfully guided their site cleanup efforts. The Wyoming DEQ provided a letter describing their experiences with the test kits (Figure 5).

Evaluation of a Fuel-Contaminated Site in Alaska

Three series of samples, 100, 300, and 400, from a fuel-contaminated airfield site in Alaska were tested using a PID sniffer, Method 8015B for DRO, and ASTM Method D 5831 using a

Diesel Dog Soil Test Kit. The series 100 and 300 samples were believed to be contaminated with weathered diesel fuel. As a result, the response factor of 58 (mg/L)/AU was used to determine the method D 5831 estimated concentrations of contamination in the samples. Data from analysis of the series 100 and 300 samples using the three analytical procedures are presented in Table 6. For discussion purposes, samples are grouped in the table according to the results from their analysis by the three procedures. As shown in Table 6, contamination was detected by all three analytical procedures in the first four series-100 samples listed in the table. For the next seven series-100 samples listed in Table 6, the three procedures gave contaminant concentrations well below 100 mg/Kg. For the last set of four series-100 samples listed in Table 6, the ASTM method and PID detected contamination in three of the samples, 100-4, 100-10, and 102-10. No GC data are available for samples 100-4 and 100-10, and the GC result for sample 102-10 is <11 mg/Kg. The ASTM method showed no contamination in sample 102-03, while the GC and PID methods showed considerable contamination in the sample. The reason for the GC method not detecting contamination in sample 102-10 and the ASTM method not detecting contamination in sample 102-03 may be that nonrepresentative sample splits were analyzed in these cases.

Also, as shown in Table 6, contamination was detected by all three analytical procedures in the first three series-300 samples listed in the table. For the last two series-300 samples, no GC data are available; however, results from testing using D 5831 and PID show very low contamination in sample 301-08 and the presence of higher contamination in sample 301-11. Although smelling samples to determine if they are contaminated is not recommended, for most of the samples listed in Table 6, the odor observations that were reported match the analytical results.

The series-400 samples from the Alaskan airfield site gave low responses with both the PID and D 5831 using the weathered diesel response factor when compared to the GC results. This is strongly suggestive of the presence of an aliphatic jet fuel contaminant. Method D 5831 has a very high quantitation limit for jet fuel of 380 mg/Kg if the jet fuel response factor of 1,050 (mg/L)/AU is used. As a result, this method is not particularly suited for screening samples contaminated with jet fuel unless they are very contaminated. Data from analysis of the series-400 samples using PID, GC Method 8015B, and D 5831 using the *Diesel Dog* Soil Test Kit are presented in Table 7. Once again, for discussion purposes, the samples are grouped in the table based on the analysis results. The D 5831 data are reported using the jet fuel response factor for these samples. As shown in Table 7, contamination was detected by all three analytical procedures in the first four series-400 samples listed. For the next set of two samples listed in the table, 400-1 and 400NW-K-7, the ASTM and GC methods both detected significant contamination in the samples; however, the PID did not.

For the third set of samples listed in the table (400E-H-4, 400NW-E-7, 400-5A, 400-12A, 400-16A, 400-2A, 402X1-1, and 402X1-10), the ASTM method indicated significant concentrations of contamination using the jet fuel response factor, while the concentrations detected by the GC and PID methods are very low by comparison. These samples may have contained relatively small amounts of weathered aromatic material such as diesel fuel that could not be detected by the GC and PID techniques. This would result in artificially high values with D 5831 if the jet fuel response factor is used. Data for the next set of samples listed in Table 7, 400NW-M12, 400-7, 400-5, and 402X1-E show that the GC method detected contamination that was not detected by the ASTM method and that was detected at significantly lower concentrations by the PID method. As previously stated, the ASTM method is not particularly suited for detecting jet fuel because of its low aromaticity. In the case of these four samples, it appears that the contaminant material was aliphatic in nature and could not be detected by either the ASTM method or the PID sniffer. Samples 402X1-D, 402X1-F, 402X2-A, and 402X2-G listed in Table 7 are shown to contain contaminant concentrations well below 100 mg/Kg by the GC and PID methods. Results from analyzing these samples using the soil test kit and ASTM Method D 5831 are all less than the very high quantitation limit of 380 mg/Kg. It is difficult to determine the concentrations of contamination in the next 18 samples listed in Table 7, 400-2 through 400-15A, because no GC data are available, the ASTM method has such a high quantitation limit, and there is uncertainty about the PID values based on the performance of the PID to detect contamination in the other samples listed in Table 7. For samples 400E-K-10 and 400-6A, method D 5831 detected contamination that was not detected by PID. There are no GC data for these samples. There are also no GC data for sample 400E-I-10. As a result, it is difficult to determine if the contaminant concentration in the sample was greater than 100 mg/Kg, because of the high quantitation limit of the ASTM method with jet fuel and the uncertain performance of the PID for this series of samples.

The data shown in Tables 6 and 7 for analysis of the fuel-contaminated soil in Alaska show that the ASTM method performed well for analysis of the series 100 and 300 samples, which were contaminated with weathered diesel fuel. For most of the samples listed in Table 6, the estimated values of contamination determined by the ASTM method correlate well with the results determined by the laboratory GC method. For most of the samples listed in Table 6 showing contamination, the PID data are significantly lower than the D 5831 and GC results.

The data presented in Table 7 show that the contamination in the series-400 samples was detected fairly well by ASTM Method D 5831 if the jet fuel response factor is used, considering the method's high quantitation limit for jet fuel. Once again, for most of the samples showing contamination, the PID values are significantly lower than the D 5831 and GC results. Data from analysis of the series-400 samples from this site emphasize that when considering use of ASTM Method D 5831, the method's quantitation limit for the fuel type and the aromaticity of the contaminant fuel must be considered.

Filling Station Site Excavation

The Wyoming DEQ conducted an excavation project involving the removal and disposal of 9,200 cubic yards of contaminated soils at a filling station site. Contamination was from two recent gasoline spills and several smaller, older spills. The area of the plume was known from earlier site investigations. Depth to groundwater varied from seven to nine feet below the surface. Four soil samples were taken from within the excavation site, two from the bottom and two from the sidewalls. It was assumed that the sidewall samples were not contaminated. The results from the *Diesel Dog* Soil Test Kit analyses of the sidewall samples showed no contamination. The samples from the bottom of the excavation contained 50 mg/Kg and 61 mg/Kg of contamination according to *Diesel Dog* Soil Test Kit results using the response factor of 58 (mg/L)/AU for weathered diesel fuel. The corresponding laboratory GC results showed 33 mg/Kg and 67 mg/Kg of gasoline to diesel-range fuels in the samples, respectively. As a result, the *Diesel Dog* Soil Test Kit results correlated well with the laboratory GC results.

Bus Barn Site Excavation

The Wyoming DEQ conducted an excavation of 2,670 cubic yards of contaminated soil from a transportation depot facility. The ground was contaminated by leakage from an underground storage tank that had been removed in 1989. Following the excavation, two soil samples were collected from a sidewall and analyzed using ASTM Method D 5831 and a *Diesel Dog* Soil Test Kit. No contamination was detected, and it was deemed not necessary to follow up with laboratory analyses.

Emergency Response for a Fresh Diesel Spill

A contractor was hired at a private residence in Laramie, Wyoming to steam clean carpets and upholstery. The steam cleaning equipment was in a trailer that contained a diesel engine to power the equipment and heat a tank of water. After the contractor had finished and left the premises, a strong smell of diesel fuel was noted in the area where the trailer had been parked. Closer inspection revealed a diesel spill that had flowed to the edge of the driveway into the soil and gravel. The exact perimeter of the spill was difficult to ascertain because a lawn sprinkler had soaked the location after the contractor left. The spill was only about 15 feet away from the home's private water well. The contractor was called, and he estimated the spill to be about 5 to 10 gallons. Although there was little chance that this amount of diesel could penetrate the soil to the aquifer 150 feet below, there was the potential that the well and aquifer could become contaminated via the well casing. If the outside of the casing and the surrounding bedrock had not been sealed with concrete at the time the well was drilled, surface water could flow down the outside of the well casing into the aquifer below.

A *Diesel Dog* Portable Soil Test Kit was used at the residence on the tailgate of a Jeep® to determine the level of diesel contamination and provide a reliable means to determine the extent of excavation needed to remediate the spill to proper cleanup standards. The Wyoming DEQ regulatory level of 100 mg/Kg was used as the remediation standard. The most concentrated area of the spill, where the soil and gravel were visibly stained dark, was excavated with a shovel. The soil and gravel in this area smelled quite strongly of diesel. A semicircle at the edge of the driveway that was about 5 feet wide and 2 feet deep was excavated. Three soil samples equally spaced around the inside perimeter of the 5 ft x 2 ft semicircular hole were analyzed. Since the spill was fresh, the fresh diesel response factor of 209 (mg/L)/AU was used. The results from the analyses are shown in Table 8. Data for Samples 1 and 2 showed that the first excavation was sufficient to collect all of the spill that had absorbed depth and lengthwise into the soil. Data from the third sample showed extensive diesel contamination under the concrete driveway where the diesel had drained. As a result, additional excavation was necessary. Two more analyses were performed after the second excavation (Samples 4 and 5). These showed the site to be in compliance with the Wyoming DEQ cleanup level of 100 mg/Kg. Once it was established that the level of diesel in the soil was <100 mg/Kg, the cavity was backfilled with clean soil.

Both the homeowner and contractor were confident that the spill had been cleaned to proper levels. The possibility of contamination and litigation had been avoided. Each analysis using the ASTM method and soil test kit took only a few minutes, and was conducted just a few steps from the excavated spill location. The contractor, who had no laboratory or field analysis experience, volunteered to perform the analyses for Samples 4 and 5. He found the procedure easy to learn.

Laboratory Method Comparison

En Chem, Inc., of Green Bay, Wisconsin analyzed a series of soil samples by ASTM Method D 5831 using a *Diesel Dog* Soil Test Kit and by gas chromatography for DRO. The results are provided in Table 9. The diesel response factor of 209 (mg/L)/AU was used for the UV method. According to D 5831, the quantitation limit (LOQ) for fresh diesel is about 75 mg/kg. However, as shown in Table 9, much lower diesel concentrations were determined for these soil samples. As explained in D 5831, the LOQ for the method can vary due to the variability in absorptivity between fuels of the same type and variations in soil background levels. Results show that the ASTM method would be useful for finding contaminated “hot spots” in the field, and that if a *Diesel Dog*

Soil Test Kit was used in the field to guide a rapid cleanup operation, highly contaminated areas would not be missed.

Communication of Results

A poster describing field use of the *Diesel Dog S*oil Test Kit was presented at the EPA/Waste Policy Institute Waste Testing and Quality Assurance Symposium in Washington, D.C. in August 2000 (Sorini et al. 2000). An invited presentation on method development activities leading to the *Diesel Dog* Soil Test Kit development was made at the Dine’ Navajo Nation College at Shiprock, New Mexico in August 2000. A *Diesel Dog* Soil Test Kit was shown at contaminated soils and groundwater conferences in San Diego, California in March 2000 and Amherst, Massachusetts in October 2000. An article describing case studies with the test kits appeared in the June/July 2001 issue of *Contaminated Soil Sediment and Water* (Sorini et al. 2001). A poster on *Diesel Dog* Soil Test Kit use was prepared with Eric Butler of Gradient Corp., Cambridge, Massachusetts, who presented it at the First International Congress on Petroleum Contaminated Soils, Sediments & Water, which was held in London in August 2001. In June 2001, the *Diesel Dog* technology won an American Chemical Society Regional Industrial Innovations Award.

CONCLUSIONS

In prior work, a simple and rapid field screening method, ASTM Method D 5831, was developed for screening soils for diesel and heavier fuel contamination. The *Diesel Dog* Portable Soil Test Kit was developed for performing the screening method in the field. The current effort involved design and construction of twenty-five preproduction soil test kit units. Field evaluation work was conducted using *Diesel Dog* Portable Soil Test Kits to perform ASTM Method D 5831 in the field. The field evaluation showed that the ASTM method performed using the *Diesel Dog* Soil Test Kits provided contaminant concentration values that are comparable to values determined using the more costly and time-consuming laboratory MADEP EPH and EPA 8015B GC methods. The *Diesel Dog* Soil Test Kit field analysis provides a rapid and inexpensive screening tool for site evaluation and cleanup activities. The kit is ideally suited for use at petroleum refinery, pipeline, and distribution sites. Other uses include emergency response and cleanup activities, and home-heating oil tank and petroleum field site assessments.

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Table 1. Determination of a Response Factor for Weathered Diesel Fuel for Inclusion in ASTM Method D 5831

Weathered-Diesel Contaminated Soil	Response Factor, (mg/L)/AU[a]
Rocky Alaskan soil A	39
Rocky Alaskan soil B	75
Wet, sandy soil A	49
Wet, sandy soil B	26
Wet, sandy soil C	88
Southern soil A	78
Southern soil B	51
Wet clay A	30
Wet clay B	90
Average:	58

[a] Determined using laboratory data from analysis of soil samples using EPA Method 8015B.

Table 2. *Diesel Dog* Test Kit (ASTM D 5831) Field Data and EPA Method 8015B Laboratory Data for Corps of Engineers Samples in Alaska, mg/Kg

Soil Sample	ASTM D 5831 *Diesel Dog*[a]	Extraction Solvent	EPA SW846 8015B GC DRO (C_{11}-C_{22})
1. Umiat Sieved[b] Soil	51	IPA	-
Replicate	71	IPA	-
2. Kodiak ($KMnO_4$ treated, 2,000 mg/Kg prior to treatment)	730	IPA	-
Replicate	710	IPA	-
3. Sieved[b] Rocky Soil	270	IPA	179
Replicate	260	IPA	-
4. Sieved[b] Rocky Soil	120	IPA	154
Replicate	110	IPA	-
Replicate	140	IPA	-
5. Sieved[b] Rocky Soil	120	IPA	59
6. Soil (Fuel Odor Evident)	660	IPA	99
Replicate	690	IPA	-
Replicate	600	n-Heptane	-
7. Sieved[b] Soil	24	IPA	89
Replicate	42	n-Heptane	-
8. Wet Umiat Tundra Peat (Uncontaminated Plant Material)	<2	n-Heptane	-

[a] Weathered diesel fuel response factor of 58 (mg/L)/AU was used for ASTM Method D 5831.

[b] Sieved through a #10 screen (2mm).

Table 3. *Diesel Dog* Test Kit (ASTM D 5831) Field Data and MADEP EPH Laboratory Data for Domino Oil/Enighed Pond Site, mg/Kg

	ASTM D 5831	MADEP EPH		
Sample	*Diesel Dog*[a]	Aliphatics (C9-C36)	Aromatics (C11-C22)	Total
1AA	620	110	10	120
2AA	970	12,600 est.	<9	12,600 est.
3AA	-	390	98	488
4AA	1,600	6,300	1,600	7,900
4AB	1,000	2,150	460	2,610
6AA	2,100	14,000	3,200	17,200
7AA	580	388 est.	92 est.	480 est.
7AA Replicate	-	284	41 est.	325 est.
8AA	380	-	-	-
9AA	1,400	299	71	370
10AA	340	43	13	56

[a] Weathered diesel fuel response factor of 58 (mg/L)/AU was used for ASTM Method D 5831.

Table 4. *Diesel Dog* Test Kit (ASTM D 5831) Field Data and EPA Method 8015B Laboratory Data for Proprietary Diesel Spill Site in Georgia, mg/Kg

Sample	ASTM D 5831 *Diesel Dog*[a]	EPA SW846 8015B GC
101	36	<12
102	25	<12
103	23	<12
104	300	410
105	250	<12
106	<21	<12
107	110	570
108	<21	<12
109	23	13
110	22	<12
111	<21	5.3 est., 34 dup.
112	<21	<12
113	480	<12
114	<21	3.7 est.
115	21	4.1 est.
117	560	-
118	1,500	1,300
119	34	<12

[a] Weathered diesel fuel response factor of 58 (mg/L)/AU was used for ASTM Method D 5831.

Table 5. *Diesel Dog* Test Kit (ASTM D 5831) Field Data and EPA Method 8015B Laboratory Data for Decades-Old Diesel and Road Tar Site in Jackson, WY, mg/Kg

Sample	ASTM D 5831 *Diesel Dog*[a]	EPA SW846 8015B GC TVPH		
		TVPH	TEPH	Total
1	140	1.1	<9	1.1
2	42	1.3	<9	1.3
3	160	12	<9	12
4	84	5.2	9	14
5	42	<0.5	<9	<10
6	84	<0.5	<9	<10
7	1,600	800	870	1,670
8	2,000	880	3,100	3,980

[a] Weathered diesel fuel response factor of 58 (mg/L)/AU was used for ASTM Method D 5831.

Table 6. *Diesel Dog* Test Kit (ASTM D 5831) Field Data, EPA Method 8015B Laboratory Data, and PID Data for Fuel-Contaminated Samples in Alaska, mg/Kg

Soil Sample	ASTM D 5831 *Diesel Dog*[a]	EPA SW846 8015B GC DRO (C_{11}-C_{22})	PID	Observation
100-A	340	408	117	High Odor
100-L	540	4,190	213	High Odor
100-D9	1,600	519	103	High Odor
102-08	840	145	93	Med. Odor
101-01	<21	<11	25	Low Odor
101-02	<21	<11	20.1	Low Odor
101-06	<21	<11	19.3	Low Odor
102-A8	<21	<11	4.2	No Odor
102-D9	23	<11	21	No Odor
100-5-7	52	20.9	2.0	No Odor
100-9-10	<21	42.9	2.3	No Odor
100-4	630	NA[b]	125	High Odor
100-10	92	NA	330	High Odor
102-03	<21	962	330	Low Odor
102-10	170	<11	210	Low Odor
301-01	1,200	3,510	176	High Odor
301-02	900	846	160	High Odor
301-03	1,500	1,350	163	High Odor
301-08	<21	NA	51	Odor
301-11	330	NA	96	Odor

[a] Weathered diesel fuel response factor of 58 (mg/L)/AU was used for ASTM Method D 5831.

[b] Data not available.

Table 7. *Diesel Dog* Test Kit (ASTM D 5831) Field Data, EPA Method 8015B Laboratory

Data, and PID Data for Alaskan Soil Samples Contaminated with Jet Fuel, mg/Kg

Soil Sample	ASTM D 5831 *Diesel Dog*[a]	EPA SW846 8015B GC DRO (C_{11}-C_{22})	PID	Observation
402X1-A	21,000	12,800	338	High Odor
400E-J-10	2,400	1,770	137	High Odor
400NW-M11	1,600	2,700	210	No Observation
400-14A	970	589, 316	177	High Odor
400-1	1,600	994	6.1	High Odor
400NW-K-7	2,800	3,800	12	Low Odor
400E-H-4	670	<11	1.7	Odor
400NW-E-7	1,400	97.2	1.3	High Odor
400-5A	2,700	74.4	0.6	Odor
400-12A	840	<11	0.9	Slight Odor
400-16A	1,100	<11	2	Slight Odor
400-2A	880	<11	0.4	Slight Odor
402X1-1	4,100	<11	8.8	Medium Odor
402X1-10	3,400	20.6	6.8	Low Odor
400NW-M12	<380	2,530	121	No Observation
400-7	<380	9,210	1.4	High Odor
400-5	<380	1,240	1.7	High Odor
402X1-E	<380	531	14.2	No Odor
402X1-D	<380	<11	1.8	No Odor
402X1-F	<380	58.9	66.2	No Odor
402X2-A	<380	66.4, 156	19.2	No Odor
402X2-G	<380	<12	8.3	No Odor

[a] Jet Fuel response factor of 1,050 (mg/L)/AU was used for ASTM Method D 5831.

Table 7. *Diesel Dog* Test Kit (ASTM D 5831) Field Data, EPA Method 8015B Laboratory

Data, and PID Data for Alaskan Soil Samples Contaminated with Jet Fuel, mg/Kg (continued)

	ASTM D 5831	EPA SW846 8015B	PID	Observation
Soil Sample	*Diesel Dog*[a]	GC DRO (C_{11}-C_{22})		
400-2	<380	NA[b]	1.5	No Odor
400-3	<380	NA	1.3	No Odor
400-4	<380	NA	1.1	No Odor
400-6	<380	NA	1.0	No Odor
400-8	<380	NA	2.9	No Odor
400-9	<380	NA	1.3	No Odor
400-10	<380	NA	3.1	No Odor
400NW-I-10	<380	NA	36	No Odor
400-1A	<380	NA	0.3	No Odor
400-3A	<380	NA	0.3	No Odor
400-4A	<380	NA	0.6	No Odor
400-7A	<380	NA	0.7	No Odor
400-8A	<380	NA	0.9	No Odor
400-9A	<380	NA	0.7	No Odor
400-10A	<380	NA	0.5	No Odor
400-11A	<380	NA	0.7	No Odor
400-13A	<380	NA	0.7	No Odor
400-15A	<380	NA	8.7	No Odor
400E-K-10	440	NA	1.8	No Odor
400-6A	690	NA	14	Low Odor
400E-I-10	<380	NA	61	No Odor

[a] Jet fuel response factor of 1,050 (mg/L)/AU was used for ASTM Method D 5831.
[b] Data not available.

Table 8. *Diesel Dog* Test Kit (ASTM D 5831) Analysis Results at a Diesel Spill Emergency Response Site

Sample ID	mg/Kg as Diesel[a]
First Excavation	
1. 8” deep	51
2. 4” deep	98
3. 1” behind concrete edge	20,000
Second Excavation	
4. 8” behind concrete edge	73
5. 8” behind concrete edge	69

[a] Fresh diesel fuel response factor of 209 (mg/L)/AU was used for ASTM Method D 5831.

Table 9. *Diesel Dog* Test Kit (ASTM D 5831) and EPA Method 8015B DRO Data for Soil Samples, mg/Kg

Sample	ASTM D 5831 *Diesel Dog*[a]	EPA SW846 8015B DRO
4471-1	72	<4.9
4503-2	29	3.9[b]
4635-1	190,000	160,000[b]
4637-2	60	<8.0[b]
4690-3	910	3,800
4750-15	12,000	7,600
replicate	10,000	-
4750-18	390	410
4752-4	6	6.4
4752-6	330	960
4752-7	1,600	2,800
4756-4	1,800	2,000
4756-5	97	190
4792-3	3,200	330[b]
replicate	2,700	-
4792-4	1,800	19
4792-5	1,700	1,100
4792-8	26,000	13,000

[a] Diesel fuel response factor of 209 (mg/L)/AU was used for ASTM Method D 5831.

[b] Late hump observed in chromatogram, possible heavy material present.

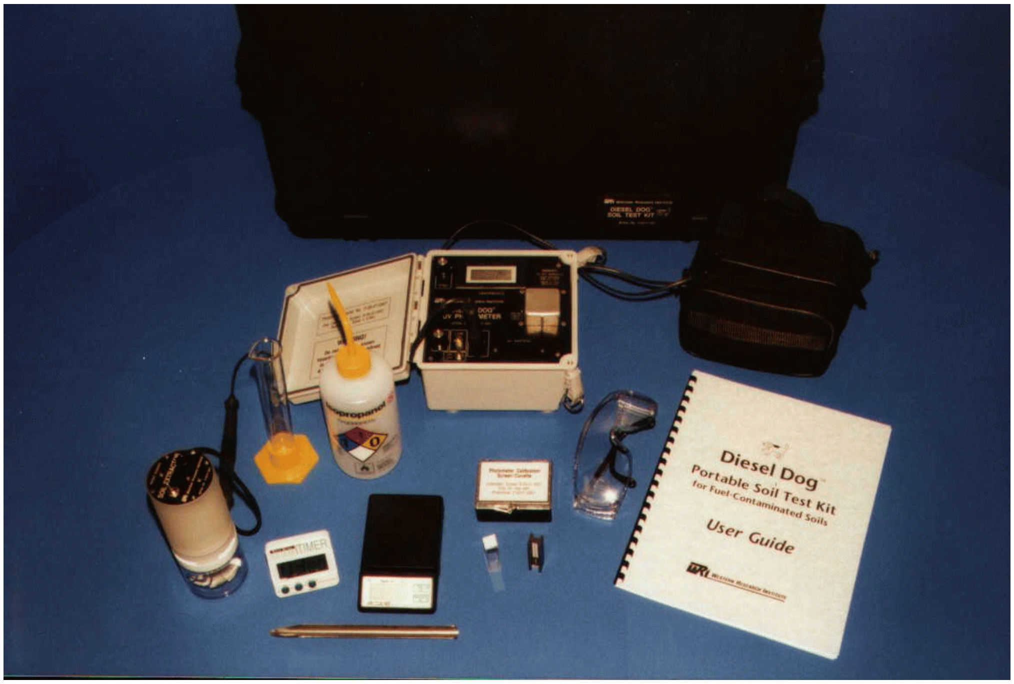
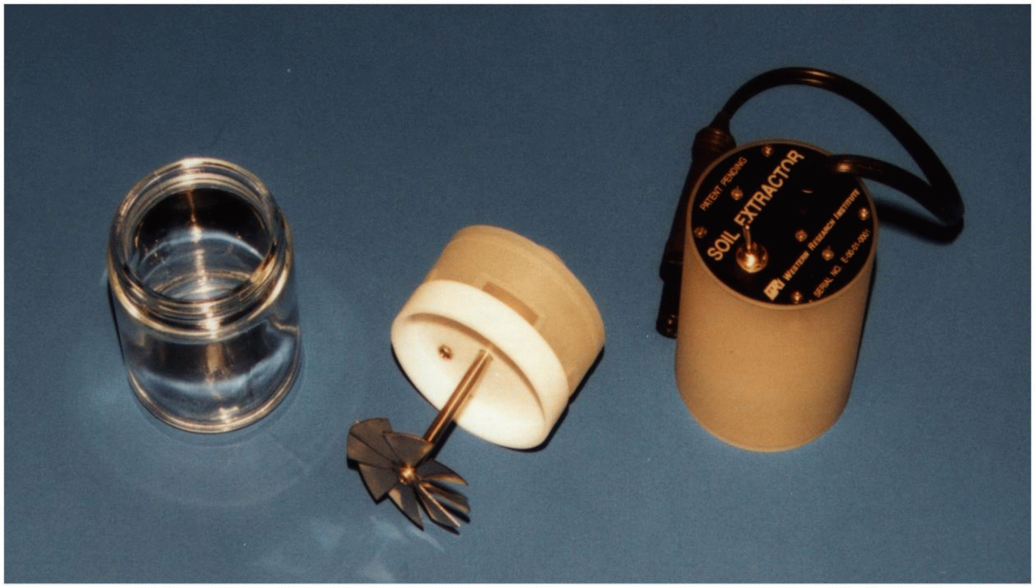


Figure 1. *Diesel Dog* Soil Test Kit Components



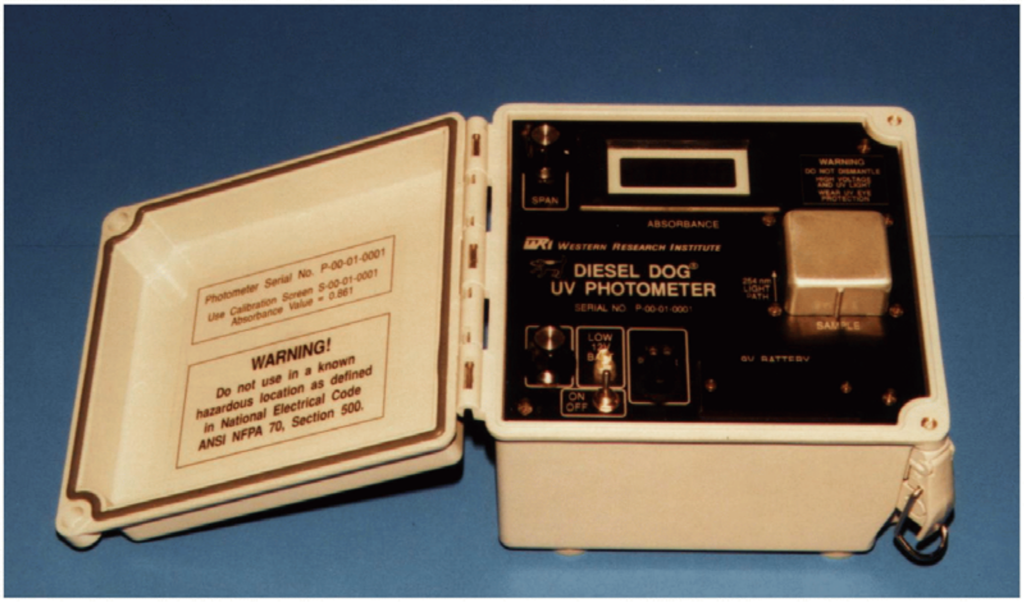
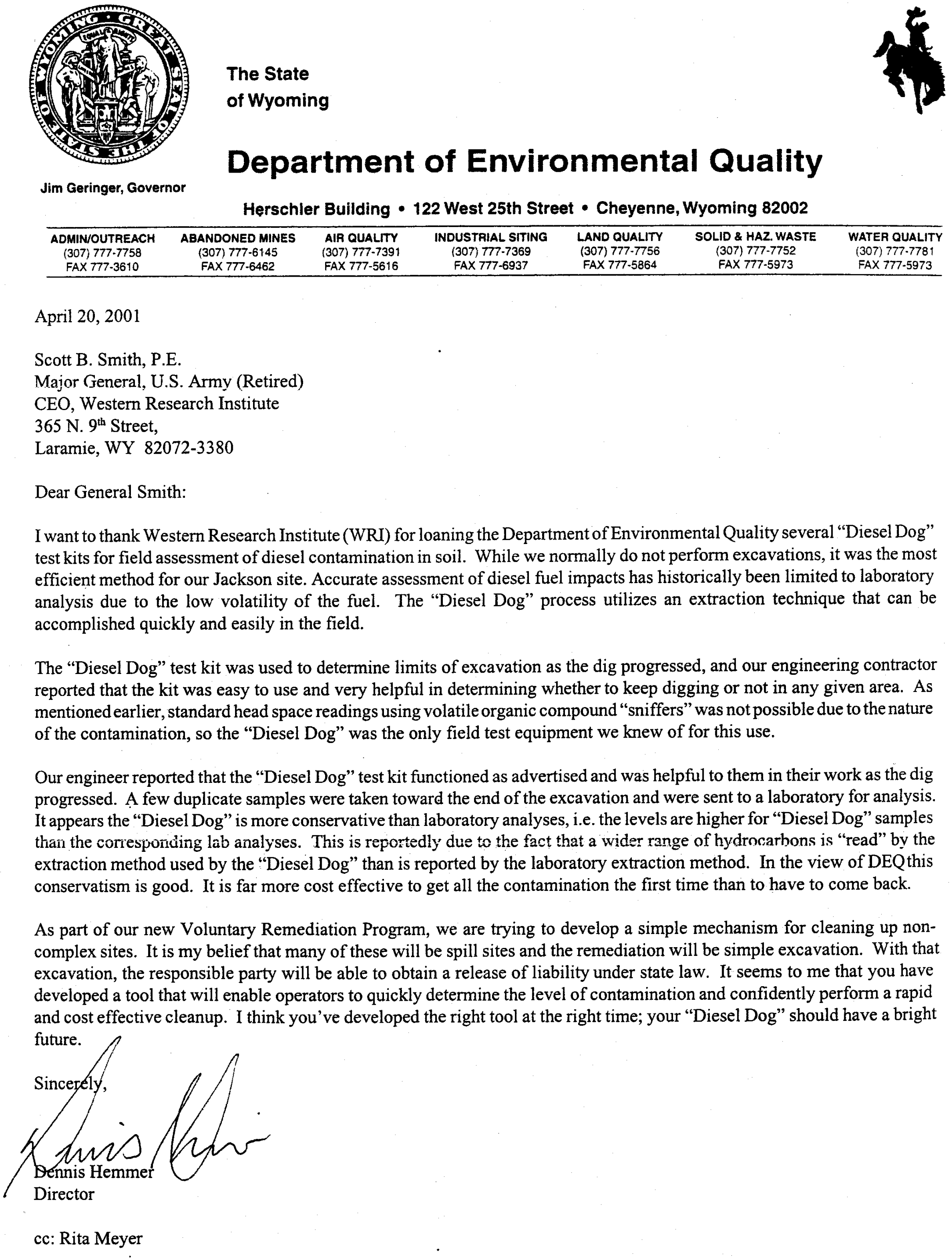


Figure 2. **Portable Soil Extractor (top) and Portable UV Photometer (bottom)**

Figure 3. ***Diesel Dog*** **Soil Test Kit Field Use**

Figure 4. *Diesel Dog* Soil Test Kit In Use In Alaska



The State
of Wyoming

Department of Environmental Quality

Jim Geringer, Governor

Herschler Building • 122 West 25th Street • Cheyenne, Wyoming 82002

ADMIN/OUTREACH	ABANDONED MINES	AIR QUALITY	INDUSTRIAL SITING	LAND QUALITY	SOLID & HAZ. WASTE	WATER QUALITY
(307) 777-7758	(307) 777-6145	(307) 777-7391	(307) 777-7369	(307) 777-7756	(307) 777-7752	(307) 777-7781
FAX 777-3610	FAX 777-6462	FAX 777-5616	FAX 777-6937	FAX 777-5864	FAX 777-5973	FAX 777-5973

April 20, 2001

Scott B. Smith, P.E.
Major General, U.S. Army (Retired)
CEO, Western Research Institute
365 N. 9th Street,
Laramie, WY 82072-3380

Dear General Smith:

I want to thank Western Research Institute (WRI) for loaning the Department of Environmental Quality several "Diesel Dog" test kits for field assessment of diesel contamination in soil. While we normally do not perform excavations, it was the most efficient method for our Jackson site. Accurate assessment of diesel fuel impacts has historically been limited to laboratory analysis due to the low volatility of the fuel. The "Diesel Dog" process utilizes an extraction technique that can be accomplished quickly and easily in the field.

The "Diesel Dog" test kit was used to determine limits of excavation as the dig progressed, and our engineering contractor reported that the kit was easy to use and very helpful in determining whether to keep digging or not in any given area. As mentioned earlier, standard head space readings using volatile organic compound "sniffers" was not possible due to the nature of the contamination, so the "Diesel Dog" was the only field test equipment we knew of for this use.

Our engineer reported that the "Diesel Dog" test kit functioned as advertised and was helpful to them in their work as the dig progressed. A few duplicate samples were taken toward the end of the excavation and were sent to a laboratory for analysis. It appears the "Diesel Dog" is more conservative than laboratory analyses, i.e. the levels are higher for "Diesel Dog" samples than the corresponding lab analyses. This is reportedly due to the fact that a wider range of hydrocarbons is "read" by the extraction method used by the "Diesel Dog" than is reported by the laboratory extraction method. In the view of DEQ this conservatism is good. It is far more cost effective to get all the contamination the first time than to have to come back.

As part of our new Voluntary Remediation Program, we are trying to develop a simple mechanism for cleaning up non-complex sites. It is my belief that many of these will be spill sites and the remediation will be simple excavation. With that excavation, the responsible party will be able to obtain a release of liability under state law. It seems to me that you have developed a tool that will enable operators to quickly determine the level of contamination and confidently perform a rapid and cost effective cleanup. I think you've developed the right tool at the right time; your "Diesel Dog" should have a bright future.

Sincerely,

Dennis Hemmer
Director

cc: Rita Meyer

Figure 5. Wyoming DEQ Letter