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Agency

Office of Research and
Development
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Innovative Technology Verification Report

Field Measurement Technologies for Total Petroleum Hydrocarbons in Soil

Strategic Diagnostics Inc. EnSys Petro Test System

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, DC 20460

Environmental Technology Verification Program
Verification Statement

TECHNOLOGY TYPE: **FIELD MEASUREMENT DEVICE**

APPLICATION: **MEASUREMENT OF TOTAL PETROLEUM HYDROCARBONS**

TECHNOLOGY NAME: **EnSys PETRO TEST SYSTEM**

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VERIFICATION PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Superfund Innovative Technology Evaluation (SITE) and Environmental Technology Verification (ETV) Programs to facilitate deployment of innovative technologies through performance verification and information dissemination. The goal of these programs is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. These programs assist and inform those involved in design, distribution, permitting, and purchase of environmental technologies. This document summarizes results of a demonstration of the EnSys Petro Test System developed by Strategic Diagnostics Inc. (SDI).

PROGRAM OPERATION

Under the SITE and ETV Programs, with the full participation of the technology developers, the EPA evaluates and documents the performance of innovative technologies by developing demonstration plans, conducting field tests, collecting and analyzing demonstration data, and preparing reports. The technologies are evaluated under rigorous quality assurance (QA) protocols to produce well-documented data of known quality. The EPA National Exposure Research Laboratory, which demonstrates field sampling, monitoring, and measurement technologies, selected Tetra Tech EM Inc. as the verification organization to assist in field testing seven field measurement devices for total petroleum hydrocarbons (TPH) in soil. This demonstration was funded by the SITE Program.

DEMONSTRATION DESCRIPTION

In June 2000, the EPA conducted a field demonstration of the EnSys Petro Test System and six other field measurement devices for TPH in soil. This verification statement focuses on the EnSys Petro Test System; a similar statement has been prepared for each of the other six devices. The performance and cost of the EnSys Petro Test System were compared to those of an off-site laboratory reference method, "Test Methods for Evaluating Solid Waste" (SW-846) Method 8015B (modified). To verify a wide range of performance attributes, the demonstration had both primary and secondary objectives. The primary objectives included (1) determining the method detection limit, (2) evaluating the accuracy and precision of TPH measurement, (3) evaluating the effect of interferents, and (4) evaluating the effect of moisture content on TPH measurement for each device. Additional primary objectives were to measure sample throughput and estimate TPH measurement costs. Secondary objectives included (1) documenting the skills and training required to properly operate the device, (2) documenting the portability of the device, (3) evaluating the device's durability, and (4) documenting the availability of the device and associated spare parts.

The EnSys Petro Test System was demonstrated by using it to analyze 66 soil environmental samples, 89 soil performance evaluation (PE) samples, and 36 liquid PE samples. In addition to these 191 samples, 12 extract duplicates prepared using the environmental samples were analyzed. The environmental samples were collected in four areas contaminated with gasoline, diesel, or other petroleum products, and the PE samples were obtained from a commercial provider. SDI chose not to analyze soil samples collected in a fifth area because according to SDI, the EnSys Petro Test System was not designed to measure the heavy lubricating oil present in the area.

Collectively, the environmental and PE samples provided the different matrix types and the different levels and types of petroleum hydrocarbon contamination needed to perform a comprehensive evaluation of the EnSys Petro Test System. A complete description of the demonstration and a summary of its results are available in the "Innovative Technology Verification Report: Field Measurement Devices for Total Petroleum Hydrocarbons in Soil—Strategic Diagnostics Inc., EnSys Petro Test System" (EPA/600/R-01/084).

TECHNOLOGY DESCRIPTION

The EnSys Petro Test System manufactured by SDI is based on a combination of immunoassay (specifically, enzyme-linked immunosorbent assay) and colorimetry. The EnSys Petro Test System includes the SDI Sample Extraction Kit, the EnSys Petro 12T Soil Test Kit, and the EnSys/EnviroGard® Common Accessory Kit. With this device, methanol is used for extraction of petroleum hydrocarbons from soil samples. Each sample extract is mixed with an enzyme conjugate solution. The reaction mixture is then transferred to an antibody-coated test tube. The hydrocarbons in the sample extract and those in the enzyme conjugate competitively bind to specific antibody sites on the test tube. The test tube is rinsed with a dilute detergent solution to remove any enzyme conjugate and hydrocarbons not bound to the antibodies. A color developer solution and hydrogen peroxide are added to the test tube in order to give yellow color to the enzymes that remain attached to the test tube. The color intensity is inversely proportional to the concentration of hydrocarbons in the extract. To accomplish color measurement, the absorbance of the antibody-coated tube containing the sample extract and an antibody-coated tube containing a reference standard (m-xylene) is compared using a differential photometer. A positive reading on the photometer indicates that the total concentration of petroleum hydrocarbons in the sample extract is less than that in the reference standard. Similarly, a negative reading on the photometer indicates that the total concentration of petroleum hydrocarbons in the sample extract is greater than that in the reference standard.

During the demonstration, extraction of petroleum hydrocarbons in a given soil sample was completed by adding 20 milliliters of methanol to 10 grams of the sample. SDI performed each analysis at three detection levels by diluting the sample extract twice during sample and standard preparation. The reference standard concentrations for gasoline (10 milligrams per kilogram [mg/kg]) and diesel (15 mg/kg) were multiplied by the dilution factors used. Thus, the concentration ranges used to estimate sample TPH concentrations were (1) less than (<)10; greater than (>)10 to <100; >100 to <1,000; and >1,000 mg/kg for samples containing gasoline range organics and (2) <15; >15 to <100; >100 to <1,000; and >1,000 mg/kg for samples containing extended diesel range organics.

VERIFICATION OF PERFORMANCE

To ensure data usability, data quality indicators for accuracy, precision, representativeness, completeness, and comparability were assessed for the reference method based on project-specific QA objectives. Although the reference method results generally exhibited a negative bias, based on the results for the data quality indicators, the reference method results were considered to be of adequate quality. The bias was considered to be significant primarily for low- and mediumconcentration-range soil samples containing diesel, which made up only 13 percent of the total number of samples analyzed during the demonstration. The reference method recoveries observed during the demonstration were typical of the recoveries obtained by most organic analytical methods for environmental samples. In general, the user should exercise caution when evaluating the accuracy of a field measurement device by comparing it to reference methods because the reference methods themselves may have limitations. Key demonstration findings are summarized below for the primary objectives.

Method Detection Limit: Based on the TPH results for seven low-concentration-range diesel soil PE samples, the method detection limit for the reference method was determined to be 6.32 mg/kg. Because the EnSys Petro Test System is a semiquantitative device, a method detection limit could not be determined for the device; however, the device's TPH concentration ranges for six of seven samples overlapped the reference method results.

Accuracy and Precision: The EnSys Petro Test System results for 16 of 66 soil environmental samples were inconclusive. Of the remaining 50 results, the device's TPH concentration ranges overlapped the reference method results for only 8 samples (16 percent); 36 EnSys Petro Test System results were biased high, and 6 results were biased low. The EnSys Petro Test System results for 12 of 28 soil PE samples were inconclusive. Of the remaining 16 results, the device's TPH

concentration ranges overlapped the reference method results for only 5 samples (31 percent); 9 EnSys Petro Test System results were biased high, and 2 results were biased low. The EnSys Petro Test System results for all 6 liquid PE samples were inconclusive.

The EnSys Petro Test System results for 3 of 66 soil environmental samples used to draw conclusions regarding whether the TPH concentrations in a given sampling area or sample type exceeded a specified action level were inconclusive. Of the remaining 63 results, the device's conclusions agreed with those of the reference method for 41 samples (65 percent); 21 EnSys Petro Test System conclusions were false positives, and 1 was a false negative. The EnSys Petro Test System results for 14 of 34 soil PE samples were inconclusive. Of the remaining 20 results, the device's conclusions agreed with those of the reference method for 15 samples (75 percent); 3 EnSys Petro Test System conclusions were false positives, and 2 were false negatives.

Both the EnSys Petro Test System and reference method exhibited good precision. Specifically, for 17 of 19 soil sample replicate sets and 2 of 2 liquid sample replicate sets, the EnSys Petro Test System TPH concentration ranges were the same for each replicate set. For 12 of 12 extract duplicate sets, the device's TPH concentration ranges were the same for each extract duplicate set. For the reference method, the median relative standard deviation ranged from 5.5 to 16 percent for 18 soil and 2 liquid sample replicate sets, and the median relative percent difference was 3 for 12 extract duplicate sets.

Effect of Interferents: The EnSys Petro Test System showed a mean response of at least 24 percent for neat tetrachloroethene (PCE); Stoddard solvent; turpentine; and 1,2,4-trichlorobenzene. The device showed no response for neat methyl-tert-butyl ether (MTBE) or soil spiked with humic acid. The reference method showed varying mean responses for MTBE (39 percent); PCE (17.5 percent); Stoddard solvent (85 percent); turpentine (52 percent); 1,2,4-trichlorobenzene (50 percent); and humic acid (0 percent). For the demonstration, MTBE and Stoddard solvent were included in the definition of TPH.

Effect of Moisture Content: The EnSys Petro Test System TPH results were inconclusive with regard to the effect of soil moisture content. The reference method TPH results were unaffected when the soil moisture content was increased from (1) 9 to 16 percent for weathered gasoline soil PE samples and (2) less than 1 to 9 percent for diesel soil PE samples.

Measurement Time: From the time of sample receipt, SDI required 39 hours, 35 minutes, to prepare a draft data package containing TPH results for 191 samples and 12 extract duplicates compared to 30 days for the reference method, which was used to analyze 199 samples and 13 extract duplicates.

Measurement Costs: For the EnSys Petro Test System, the TPH measurement cost for 191 samples and 12 extract duplicates was estimated to be $10,210 (including the daily rental cost of the EnSys/EnviroGard® Common Accessory Kit, whose purchase price is $1,999) compared to $41,290 for the reference method.
Key demonstration findings are summarized below for the secondary objectives.

Skill and Training Requirements: The EnSys Petro Test System can be operated by one person with basic wet chemistry skills. The sample analysis procedure for the device can be learned in the field with a few practice attempts.

Portability: The EnSys Petro Test System is battery-operated and requires no alternating current power source. The device can be easily moved between sampling areas in the field, if necessary.

Durability and Availability of the Device: All items in the EnSys Petro Test System are available from SDI. During a 1-year warranty period, SDI will supply replacement parts for the device by overnight courier service at no cost. During the demonstration, none of the device's reusable items malfunctioned or was damaged.

In summary, during the demonstration, the EnSys Petro Test System exhibited the following desirable characteristics of a field TPH measurement device: (1) good precision and (2) high sample throughput. In addition, the EnSys Petro Test System exhibited moderate measurement costs. However, a significant number of the EnSys Petro Test System TPH results were determined to be inconclusive because the detection levels used by SDI were not appropriate to address the demonstration objectives. Overall, the device's results did not compare well with those of the reference method; in general, the device exhibited a high positive bias. Collectively, the demonstration findings indicated that the user should exercise caution when considering the device for a site-specific field TPH measurement application.

Original
signed by

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