Evaluation of a Rapid Field Analytical Test Kit for Assessing Hydrocarbon Soil Contamination

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Presented at the 'Third International Conference On-Site Analysis'. January 22-25, 1995 Houston, TX.

ABSTRACT

When compared to conventional lab methods, the use of field analytical tests for site assessments and for tracking the remediation of contaminated soil can benefit the environmental professional by saving time and expenses. This study evaluated the analytical performance of Dexsil[1] Corporation's PetroFLAG™ field test kit at an oil and grease contaminated soil bioremediation site. The results of the field tests were compared to EPA method 418.1 for Total Recoverable Petroleum Hydrocarbons (TRPH). The sampling plan was approved and the field activities were witnessed by the lead regulatory official. To save time and costs, environmental field studies and remedial efforts are dependent upon direct reading (real time) instrumentation and the observation skills of the environmental professional. Some commonly used field detectors such as PIDs and FIDs are expensive and can only provide a semi-quantitative evaluation of the volatile fraction of petroleum-affected soil. The PetroFLAG test kit for petroleum fuels provides fast and accurate quantitative onsite field analysis comparable to laboratory methods. Field-split analysis showed that the PetroFLAG kit produced no false negatives and only 10% false positives when compared to EPA method 418. 1. Duplicate analysis of homogenized laboratory split samples resulted in a 90% correlation between the PetroFLAG results and the 418.1 results.

INTRODUCTION

Environmental site assessments, either at the Phase 11 level, or the remediation phase (Phase III), benefit from the use of quantitative field analytical tests and portable instrumentation that provide accurate on-site data of high quality. The benefits include on-site data for same day project evaluation, the saving of time and remobilization costs, and potentially substantial overall project cost savings allowing more money and resources to be directed to the actual clean-up of polluted sites. Field test kits are available for a variety of compounds, however, many kits are semi-quantitative, cumbersome to use, expensive, require extensive training, and/or serve more as a present/not present indication of the chemical compound of interest. The PetroFLAG kit was developed to provide a simple-to-use, fast, accurate, and inexpensive tool for on-site

[1]Dexsil is a registered trademark and PetroFLAG is a trademark of Dexsil Corporation, One Hamden Park Drive, Hamden, CT 06517, (203) 288-3509

evaluation of the concentration of petroleum hydrocarbons in contaminated soil. The PetroFLAG kit provides the environmental professional with a quick determination of the petroleum present so that costly and unnecessary work is avoided. The limits of contamination can be ascertained in the field and additional borings and/or monitoring wells may be accurately positioned. Phase III activities also benefit from accurate field tests that generate high quality data during site remediation. The PetroFLAG kit can quickly provide the environmental professional with a quantitative assessment of the limits of the soil contamination and the concentration of the contamination in ppm, at a fraction of the cost of providing a mobile laboratory or sending samples off to a conventional laboratory.

BACKGROUND

The environmental consultant for this study was *Youngdahl & Associates, Inc.* and Mr. David Jermstad was the project engineering geologist. The lead regulatory official for this project was Mr. Jeff Rusert, Senior Environmental Health Specialist for the Solid Waste and Hazardous Materials Division of the County of El Dorado.

On June 8, 1994, sampling was performed at a site located in El Dorado County, California. The site, a fenced vacant lot, contained approximately 1000 cubic yards of oil and grease contaminated soil. The contaminated soil was prepared for bioremediation in November 1993 and had been cultivated several times. The beginning hydrocarbon concentration (oil and grease) in the soil was approximately 1600 ppm. A clean-up level of 100 ppm was approved by the lead regulatory agency, El Dorado County Environmental Management Department (EDCEMD). The bioremediation site was selected to evaluate the PetroFLAG kit's ability to detect low levels of heavy fraction hydrocarbon contamination in soil at a real world remediation site. The site was also selected to demonstrate the ability of the kit to provide high quality reproducible data comparable to laboratory method EPA 418. 1. The PetroFLAG kit provided on-site identification of hot spots and clean areas within the bioremediation cell, while providing an easy to use and accurate overall assessment of the hydrocarbon concentrations throughout the soil remediation pile.

SAMPLING PROTOCOL

The regulatory sampling requirements for confirmation sampling at the site required that one sample be collected for every 50 cubic yards of treated soil. Twenty samples were collected and analyzed. A random grid pattern was used to determine the ten (10) sampling locations. Soil samples were collected from each location at two depths; 0.5 to 1.0 feet deep and 1.5 to
2.0 feet deep.

The largest source of variability in soil samples is the inherent non-homogeneity of the soil itself. Much of the variability can be overcome by thorough homogenization of the sample before splitting and analysis. Subsampling techniques have been developed for soils that are effective, however, most would not be appropriate for soils contaminated

with volatile hydrocarbon fuels such as gasoline (See: Francis F. Pitard, Pierre Guy's Sampling Theory and Sampling Practice, Volumes I and 2, CRC Press, Inc., Boca Raton, FL, 1992). As part of this field study a unique sampling technique was employed in the field. Although this site contained only heavier hydrocarbons, if the technique proved effective, it would have application at other sites.

The samples were collected, by driving a 2.5 inch outside diameter hammer type core sampler containing two 3 inch long brass liners six inches into the soil producing a six inch long undisturbed core sample. The two 3 inch long brass tubes were then removed from the sampler and gently twisted to split the core in the middle. The previously joined ends of the core sample were covered and marked to indicate the end of the tube where the sample was to be collected for laboratory and field analysis. This sampling method was designed and performed in an effort to reduce sample variability while minimizing the risk of volatile organic losses. Decontamination procedures between sampling locations included an alkanox soap and water wash followed by two clean water rinses. The samples, upon labeling and capping, were immediately placed into a cooler containing crushed ice. All 20 samples were delivered to an accredited environmental laboratory within 24 hours of collection and under strict chain-of-custody documentation. The laboratory was instructed to sample the marked end of the tube and analyze the samples using EPA method 418.1 for TRPH. Soil sampling was approved and witnessed by Mr. Jeff Rusert, Senior Enviroranental Health Specialist, Solid and Hazardous Waste Division, EDCEMD. Duplicates of the twenty soil samples delivered to the laboratory were analyzed in the field using the PetroFLAG test kit.

In addition to the experimental field-split-sample analysis, a duplicate laboratory analysis was performed on the remaining portion of the sample that was used for the PetroFLAG analysis. After the field analysis was performed, the remainder of the sample was recapped, packed on ice and shipped to a second lab. Upon receiving the samples, each brass tube was emptied into a glass jar. The jars were sealed and the sample homogenized by tumbling the jar under controlled conditions. Each sample was then split and analyzed by EPA Method 418.1 and the PetroFLAG kit in the lab. These results provided a control set of data for comparison with the data obtained using the field splitting technique.

SAMPLE DESCRIPTION AND FIELD OBSERVATIONS

The average soil texture within the bioremediation cell consisted of a coarse sandy loam, containing approximately 10% rocks by volume (silty sand with gravel, SM). The average soil moisture content was 3 to 5 % within the samples at the time of sampling. The native soil was derived from a parent material of quartz diorite (granite). The soil within the bioremediation cell contained very little organic matter. A clean native soil sample was collected adjacent to the contaminated soil as a field blank. The site specific zero sample is used for the preparation of on-site spiked soil standards as part of the PetroFLAG field test quality control.

Three soil samples 2-2, 3-2 and 6-1 were observed to have a slight hydrocarbon odor, but no unusual soil discoloration was observed in these sample locations. Discolored soil encountered at sample location 8 gave off a strong hydrocarbon odor. This discoloration abruptly ended at the 20-21 inch depth. Soil sample 8-2 appeared to be discolored in

both tubes at the ends where they had been previously joined.

The use of visual and olfactory detection and observation methods used routinely during site assessments proved to be ineffective and unreliable as tools to identify oil and grease contamination at the site with the exception of sample location 8. Although the site had been cultivated several times prior to sampling, the corresponding low and high concentrations in the samples collected at location 8 illustrate how variable contamination concentrations can be over very short distances.

THE PETROFLAG FIELD TEST KIT

The PetroFLAG field test kit for petroleum hydrocarbons is a portable test kit designed to provide on-site analysis of the hydrocarbon concentrations in soil samples. The test requires approximately 15 minutes to perform on a single sample after it is collected. The test takes approximately 6 minutes per sample when conducting multiple tests concurrently. The PetroFLAG field test procedure is easy to follow and quickly memorized.

The test comes prepackaged with plastic screw top test tubes, premeasured extraction solutions, filtering syringes and premeasured development solutions in screw top cuvettes. The test also requires the use of a specially configured calorimeter and prepackaged ampulized spikes for making site-specific hydrocarbon standards. The ampulized spikes utilized for this study were oil and grease. The spikes were added to 10 grams of clean native soil of the same type as the contaminated soil. This produced accurate site specific standards of 100 and 500 ppm while factoring out any potential naturally occurring background interferences. The soil standards once spiked, are tested exactly the same as an unknown soil sample. The standards are used to produce a standard curve from which the hydrocarbon concentrations were calculated. The procedure for the PetroFLAG test is as follows:

1. Weigh 10 grams of soil into a plastic screw top test tube using a clean spatula.

2. Add premeasured extraction solution to the soil in the test tube and mix for 5 minutes.

3. Add the mixture to the filter syringe and filter the solution into the screw top cuvette containing the premeasured development solution.

4. At the end of a 10 minute development period place the cuvette into the specially designed Dexsil calorimeter and record the reading.

PETROFLAG FIELD TESTING

The PetroFLAG analysis for each of the 20 samples collected at the site was performed by Mr. Jermstad. Mr. Jermstad followed the procedure of the test as described above and analyzed all of the twenty duplicate soil samples. Mr. Jermstad also prepared and tested four (4) standards using the site specific clean soil making the total of 24 samples analyzed. Mr. Rusert witnessed Mr. Jennstad's testing of each of the duplicate soil samples.

ANALYTICAL RESULTS

The results of the PetroFlag field test and the laboratory analysis of the field-split samples are summarized in Table 1. As shown in Table 1, the PetroFLAG data indicated oil and grease concentrations in the soil to be between 10 and 500 ppm. Laboratory data presented in Table I indicate oil and grease concentrations in the soil to be between ND< 20 and 740 ppm. A paired t test of the two data sets indicates that they are statistically equivalent. As shown in Figure 1 the PetroFLAG field test kit produced no false negatives and only 10% false positives, indicating that the field PetroFLAG data compared very well to laboratory results.

The laboratory split analysis resulted in a much lower intra-sample variability than the field split data. This is due entirely to the homogenization prior to splitting the sample for analysis. The results from the laboratory PetroFLAG and EPA 418.1 analysis are presented in Table 2 and plotted in Figure 2. A regression analysis indicates that the PetroFLAG kit correlates very well with EPA 418.1. (R^2=0.897)

REGULATORY COMMENT

Mr. Jeff Rusert, Senior Environmental Health Specialist, Solid Waste & Hazardous Materials Division, EDCEMD was presented the data in Table I to evaluate. After examining and comparing the data, Mr. Rusert found the PetroFLAG test data to be comparable to EPA 418.1 laboratory method. Mr. Rusert was further encouraged by the fact that the data was generated on-site and at a considerable cost and time savings to the client. Mr. Rusert stated that as a field test, the PetroFLAG test was very accurate and he said that he looks forward to evaluating the PetroFLAG test kit performance on other contaminants, such as diesel fuel. In summary, Mr. Rusert was pleased with the design of the sampling program and with the performance of the PetroFLAG kit.

CONCLUSIONS

The testing of Dexsil Corporation's PetroFLAG field test kit prototype for petroleum hydrocarbons proved it to be an accurate, cost effective, rapid and simple to use field test for determining oil and grease hydrocarbon concentrations at a bioremediation site. The test provided the environmental professional with on-site data comparable to EPA 418.1 laboratory method, without the extra cost and waiting period. The ability of the PetroFLAG kit to provide high quality accurate data allows the environmental professional the luxury of on-site decision making while saving time, remobilization costs and overall project costs. These savings may then be directed toward remedial work or used to complete an ongoing remediation.

When sampling a dynamic media such as soil, variability from one sample area to another within the same soil type can be great. As a result, sample variability is a very real and unpredictable factor to consider when a sampling plan is being designed. This also points out that although the laboratory is well equipped to produce very accurate results by using highly sophisticated instrumentation, the resulting data is not the absolute final word. The PetroFLAG test, being inexpensive and capable of yielding

rapid high quality data for on-site decision making, will allow the environmental professional the luxury of collecting and testing more soil samples and the resulting data can be used in a statistical evaluation of the site. The sampling protocol utilized in this study attempted to reduce the effect of sample variability on the two test methods. The difference in the intra-sample variability between the two splitting techniques indicates that only by thorough homogenization can the variability be effectively eliminated.

Mr. Jeff Rusert of the EDCEMD, the lead regulatory agency, was impressed with the PetroFLAG data and is very interested in the cost saving aspects of using the kit at Phase II and Phase III sites. Mr. Rusert is also interested in evaluating the performance of the kit on other hydrocarbon contaminants such as diesel.

The ability of a field test kit to accurately detect low levels of heavy fraction hydrocarbon contamination in soil samples is unique to the environmental testing and site assessment field. The use of conventional site assessment methods for locating and defining the lateral and vertical extent of heavy fraction hydrocarbon soil contamination is at best a very difficult task. The PetroFLAG field test kit for petroleum hydrocarbons is a new tool to make the task easier.

Table 1: Field Split Data

Sample ID	PetroFLAG Field Data (ppm)	Laboratory (1) 418.1 Data (ppm)
1-1	180	230
1-2	758	410
2-1	290	420
2-2	800	170
3-1	425	230
3-2	911	740
4-1	60	60
4-2	375	93
5-1	55	31
5-2	10	ND<20
6-1	420	390
6-2	330	280
7-1	110	400
7-2	568	380
8-1	345	660
8-2	580	120
9-1	45	62
9-2	195	82
10-1	280	190
10-2	330	220

Table 2: Lab Split Data

Sample ID	PetroFLAG Data (ppm)	Laboratory (2) 418.1 Data (ppm)
1-1	391	185
1-2	573	542
2-1	329	176
2-2	1069	986
3-1	460	321
3-2	733	422
4-1	130	31
4-2	290	197
5-1	253	119
5-2	173	97
6-1	491	328
6-2	460	376
7-1	353	218
7-2	491	241
8-1	316	160
8-2	494	453
9-1	70	39
9-2	284	215
10-1	81	49
10-2	330	330