# Validation of RaPID Assay<sup>®</sup> Immunoassay TPH Test Kits against Standard DFG Laboratory Techniques- Final Report for FY 2005-2006 Work (extended to FY 2006-2007)

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#### Introduction and Background

This report summarizes the immunoassay test kit work done between July 1, 2005, and February, 2007. The immunoassay test kits used in this work were RaPID Assay<sup>®</sup> Immunoassay TPH test kits purchased from Strategic Diagnostics Incorporated (SDI), headquartered at 111 Pencader Drive, Newark, DE 19702. They can be reached at (800) 544-8881. The proposal for this work is attached to this report (Attachment 1).

Immunoassay test kits have been used in many applications to provide rapid and inexpensive data related to contaminants in soil and water. These types of test kits allow for a throughput of up to 50 samples to be analyzed in 1 hour, with a final cost of approximately \$13 dollars per soil sample and \$5 per water sample (CMECC 1996).

Immunoassay technology allows for rapid analysis of a relatively large amount of samples on-site for a relatively low cost per sample. This capability could be of significant utility to the Department of Fish and Game, Office of Spill Prevention and Response (DFG-OSPR). It could allow on-site decision makers to view data quickly, and decide whether a site is "clean" or which samples should be sent in for laboratory analysis based on the screening results that the test kit provides. The use of immunoassay kits for the screening of soil samples for petroleum hydrocarbons is listed as method 4030 in EPA publication SW-846, entitled *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* which is the Office of Solid Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with the RCRA regulations (USEPA 1996).

The hypothesis tested was; the results of the RaPID Assay<sup>®</sup> Immunoassay TPH test kit compare favorably with the standard laboratory results generated by the Department of Fish and Game laboratories on fortified (spiked) sediment samples. The immunoassay test kit will be referred to hereafter as "the kit". Two deviations from the stated proposal objectives are noteworthy. First, water sample results were not reported. The reason for this has to do with the relative insolubility of hydrocarbons and the differences in how samples are prepared for the laboratory method and the immunoassay test. This will be addressed in greater detail in the following sections. It is also notable that fortified or spiked samples were used, instead of real spill samples. This allowed for a comparison to known concentrations, which is helpful at this stage of the evaluation.

#### Methods

RaPID Assay<sup>®</sup> Immunoassay TPH test kits, soil extraction kits to extract the diesel from fortified sediments, and a RPA-1 Analyzer were purchased from Strategic Diagnostics Incorporated (SDI). Sediment and water were collected at two locations. The ocean

location was a beach, North Salmon Creek Beach in Sonoma County, CA. The freshwater location was a beach at Lake Natoma near the Nimbus Dam in Rancho Cordova, CA. The goal was to test water and sediments at both high and low concentrations of petroleum product using three replicates per treatment. Originally, freshwater and saltwater matrices were to be studied as well as sand and freshwater sediments. However, due to the insolubility of petroleum in water, only sediment results were comparable to laboratory data and are being reported.

Testing began with water spikes of number two diesel (#2 diesel fuel) from the Union 76 station in Pollock Pines, CA. Dilutions of the diesel fuel were made in acetone to attempt to make the diesel soluble in water. This proved unsuccessful. A visible sheen was apparent in the spiked water samples. The immunoassay kit requires only a 200  $\mu$ L sample size and since the sample is not homogeneous, this volume will not necessarily be representative of the entire sample. Since the standard DFG laboratory method 8015 uses a 1 liter sample size and extracts the sheen on the water surface, it became apparent that the two methods would produce results that would not be comparable. The decision was made to move forward with sediment sample analysis only.

All samples were spiked with either 10 µL or 40 µL of diesel fuel. Spiking was done using gas-tight syringes (Hamilton Company) to maximize precision and accuracy. Three replicates of beach sand and freshwater sediment were spiked in each trial. Some trials with acetone carrier-based spiking solutions were performed early in the work, but only results of sediment spikes made using pure diesel fuel have been reported, eliminating the possibility of effects from the carrier solutions. All spikes were made into the extraction jars containing the 10 grams of sediment, which is the mass of sediment that is called for in the test instructions. It was determined that 10 microliters was a low concentration spike within the kit's test range and that 40 microliters was a high range spike that would require a 1:10 dilution to be within the concentration range of the immunoassay test kit. Two different types of kits were tested. They were the BTEX/TPH (benzene, toluene, ethyl benzene, xylene / total petroleum hydrocarbon) kit and the polycyclic aromatic hydrocarbon (PAH) kit. The PAH kit was provided to OSPR at no charge by SDI after the BTEX/TPH kit results were reviewed by the company. Diesel fuel is within the carbon range for both of these immunoassay kits. Results that did not meet the quality control parameters specified in the kit instructions were discarded or noted as appropriate.

All laboratory analyses used the standard Department of Fish and Game Petroleum Chemistry Laboratory protocol U.S. EPA Method 8015B "Nonhalogenated Organics Using GC/FID" employing a gas chromatograph with a flame ionization detector. Sediments were extracted with methylene chloride and resulting extracts were dried with sodium sulfate, concentrated and analyzed.

#### Dewatering Study

This study included a dewatering procedure evaluation that could be relevant and meaningful for sediment tests following a petroleum release incident where sediments

will likely contain more than 30% moisture (an upper limit for the kits). A study was conducted to determine whether the company's procedure for dewatering sediment samples before analysis with the kit would produce reliable results. The freshwater sediment was used in this study. Three treatments were established, and each was conducted in triplicate. The treatments included; 1) less than 30% moisture (site sediment), 2) greater than 30% moisture (wetted) samples where water was added to site sediments, and 3) greater than 30% moisture which were dewatered (dewatered) treatments using the company's procedure. The dewatering procedure involved placement of the sediment sample into a coffee filter and applying pressure to the sample to force the water out, essentially squeezing the sample to remove moisture. Each sediment treatment was spiked with 10  $\mu$ L of diesel fuel and analyzed using the BTEX/TPH immunoassay kit following extraction.

#### Results

#### Preliminary sediment moisture tests

The kit instructions indicate that sediment samples must contain no more than 30% moisture to achieve accurate results. On 1/12/06 the fresh water sediment sample was determined to contain 23.1% moisture, and the beach sand was found to contain 5.2% moisture. On 6/23/06 a second fresh water sediment sample was determined to contain 24.7% moisture.

#### Gravimetric determination of diesel mass and spike concentrations

A measured volume of diesel fuel, 40  $\mu$ L, was weighed on an analytical balance at the laboratory (California Department of Fish and Game Petroleum Chemistry Laboratory, Rancho Cordova, California) to determine the actual mass of diesel fuel being spiked into the sediment samples. This 40  $\mu$ L is one of the volumes used for spiking sediment samples in this investigation. The three measured masses were:

#1 - 0.03290 grams #2 - 0.03210 grams #3 - 0.03260 grams

The average of these 3 values was 0.03253 grams for a 40  $\mu$ L aliquot of diesel fuel or 0.81333 mg/ $\mu$ L (equivalent to grams/mL) of diesel. An MSDS from Phillips Petroleum Company lists the specific gravity of no. 2 diesel fuel as ranging from 0.81–0.88 @ 60°F (Phillips Petroleum Co., 2002).

Since 10µL was spiked into the "10 µL" samples, there was a total of 0.0081333 grams or 8.1333 mg of diesel spiked onto the 10 grams of soil. Converting this to mg/kg units (ppm), this is 813.33 mg of diesel per kg of soil. The laboratory used fewer significant figures, arriving at 810 mg/kg of diesel for the actual spike concentration. For the "40 µL" spiked samples, 40 µLs of diesel fuel was applied to the 10 grams of soil in each extraction jar. So the concentration was 3253 mg/kg (ppm) in the "40 µL" spikes. Using fewer significant figures the lab used 3250 mg/kg (ppm) as the actual spike concentration.

#### Chemistry Results

The DFG laboratory results are presented in Table 1 below, which also lists the actual spiked concentration for each sample.

Sample	Spiked Conc. Meas. Conc. Rec (ppm) (ppm)		Recovery (%)	Treatment Avg. (mg/kg, ppm)
Freshwater Sediment-1 (10µL)	810	420	52	
Freshwater Sediment-2 (10µL)	810	580	72	
Freshwater Sediment-3 (10µL)	810	570	70	523
Freshwater Sediment-1 (40µL)	3250	2800	86	
Freshwater Sediment-2 (40µL)	3250	2800	86	
Freshwater Sediment-3 (40µL)	3250	2800	86	2800
Beach Sand-1 (10µL)	810	640	79	
Beach Sand-2 (10µL)	810	602	74	
Beach Sand-3 (10µL)	810	590	73	611
Beach Sand-1 (40µL)	3250	3020	92	
Beach Sand-2 (40µL)	3250	3010	93	
Beach Sand-3 (40µL)	3250	2800	85	2943

Table 1. Measured Concentration and Percent Recovery of Spiked Diesel Fuel from Sediment and Sand Using EPA Method 8015.

The BTEX/TPH Kit results are presented in Table 2 below. All samples were run in triplicate on the date indicated. The kit reports BTEX concentration directly (1<sup>st</sup> and 3<sup>rd</sup> columns), and the diesel concentration was calculated using the calibration factors provided by SDI (2<sup>nd</sup> and 4<sup>th</sup> columns). The average result of the triplicate analysis, as well as the standard deviation and coefficient of variation are reported as well. The coefficient of variation is the standard deviation divided by the mean. This statistic allows comparison of the variation of populations that have significantly different mean values, as is the case here with two different spike concentrations used.

Samples	10 µL diesel s	pikes (3/23/06)	40 µL diesel spikes (6/16/	
	BTEX (ppm)	Diesel (ppm)	BTEX (ppm)	Diesel (ppm)
Freshwater Sediment-1	0.61	65	0.25	278
Freshwater Sediment-2	0.92	98	0.29	323
Freshwater Sediment-3	1.05	111	0.26	288
Avg. of Replicates	0.86	91	0.27	297
Std. Deviation	0.23	24	0.02	24
Coefficient of Variation (CV)	26.7%	25.9%	8.2%	8.0%
Beach Sand-1	1.89	197	0.40	435
Beach Sand-2	1.37	143	0.37	406
Beach Sand-3	1.38	145	0.38	413
Avg. of Replicates	1.55	161	0.38	418
Std. Deviation	0.30	30	0.01	15
Coefficient of Variation (CV)	19.4%	18.8%	3.7%	3.5%

Table 2. Total Petroleum Hydrocarbon (TPH) results using the SDI Total BTEX/TPH RaPID ASSAY<sup>®</sup> kit.

The PAH Kit results are presented in Table 3 below. All samples were run in triplicate on the date indicated, however at the 40  $\mu$ L spike level one of the beach sand results had to be excluded since the result was greater than the value of the highest calibration standard (indicated as "hi" next to the result from the spectrophotometer). The kit reports PAH concentration directly, and the total fuel (diesel) concentration was computed later using the calibration factors provided by SDI. Note that PAH concentrations are in parts per billion (ppb) or  $\mu$ g/kg.

Table 3.	Total Petroleum Hydrocarbons	(TPH) results using the SDI PAH RaPID
ASSAY®	kit.	· · · ·

Samples	10 µL dies	sel spikes (12/19/06)	12/19/06) 40 µL diesel spikes (12/21/06	
	PAH (ppb)	Total Fuel (ppm)	PAH (ppb)	Total Fuel (ppm)
Freshwater Sediment-1	27.7	87.6	211	656
Freshwater Sediment-2	28.7	91.0	907 <sup>1</sup>	<del>3082</del> 1
Freshwater Sediment-3	29.5	93.7	154	470
Avg. of Replicates	28.6	90.8	182	563
Std. Deviation	0.91	3.1	40.4	132
Coefficient of Variation (CV)	3.2%	3.4%	22.2%	23.4%
Beach Sand-1	27.3	86.4	406	1313
Beach Sand-2	27.3	86.4	187	578
Beach Sand-3	28.2	89.3	751	2522
Avg. of Replicates	27.6	87.3	448.0	1471
Std. Deviation	0.5	1.7	284.2	982
Coefficient of Variation (CV)	1.8%	1.9%	63.4%	66.8%

<sup>1</sup> Results with strikethrough text are invalid because the concentration exceeded the high calibration standard

A comparison of all results for the  $10\mu$ L spikes of diesel fuel, including the laboratory (EPA 8015) method, the BTEX kit, and the PAH kit, are shown in Table 4 below. The percentage that the test kit results represent compared to the laboratory results are also shown in this table. The t-test results showed that all of the BTEX and PAH kit results from both freshwater sediment and beach sand were significantly different from the laboratory results (p< 0.05). Figure 1 displays these results graphically.

Table 4. Comparison of EPA Method 8015 (Lab) Results with BTEX Kit and PAH Kit TPH Results for Sediment and Sand Fortified with 10  $\mu$ L of Diesel Fuel

Average	Average Values, all chemistry results are in units of ppm TPH					
Samples	Spike Amt. (ppm)	Lab Result (8015) (ppm)	BTEX Kit Result (ppm)	PAH Kit Result (ppm)	BTEX Kit % of Lab Result	PAH Kit % of Lab Result
Freshwater Sediment	810	523	91.5*	90.8*	17.5%	17.3%
Beach Sand	810	611	161*	87.3*	26.4%	14.3%

\*= significantly different from the laboratory result by a t-test (p< 0.05).

Figure 1. Comparison of EPA Method 8015 (Lab) Results with BTEX Kit and PAH Kit TPH Results for Sediment and Sand Fortified with 10  $\mu$ L Diesel Fuel



A comparison of all results for the 40µL spikes of diesel fuel, including the laboratory (EPA 8015) method, the BTEX kit, and the PAH kit, are shown in Table 5 below. The percentage that the test kit results represent compared to the laboratory results are also shown in this table. The t-test results showed that all of the BTEX and the freshwater sediment PAH kit results were significantly different from the laboratory results (p< 0.05). Figure 2 displays these results graphically.

Table 5.	Comparison of EF	A Method 8015	(Lab) Res	sults with	BTEX Kit a	and PAH Kit
TPH Res	sults for Sediment a	and Sand Fortifie	ed with 40	µL Diese	el Fuel	

Average	Values, a	II chemisti	ry results ar	e in units of	ppm TPH	
Samples	Spike Amt. (ppm)	Lab Result (8015) (ppm)	BTEX Kit Result (ppm)	PAH Kit Result (ppm)	BTEX Kit % of Lab Result	PAH Kit % of Lab Result
Freshwater Sediment	3250	2800	297*	563*	10.6%	20.1%
Beach Sand	3250	2943	418*	1471	14.2%	50.0%

\*= significantly different from the laboratory result by a t-test (p< 0.05).

Figure 2. Comparison of EPA Method 8015 (Lab) Results with BTEX Kit and PAH Kit TPH Results for Sediment and Sand Fortified with 40  $\mu$ L Diesel Fuel



#### Dewatering Procedure

The dewatering procedure data indicates that the dewatering process (dewatered sediment treatment) produces recovery results that are close to (86%) the untreated (site sediment treatment) results. All three samples were fortified with the same amount (10  $\mu$ L) of diesel fuel. These results are presented in Table 6 below.

Table 6.	Results of the dewatering test,	showing u	untreated	site se	diment,	wetted
sediment	t, and dewatered sediment tests	S				

Treatment	n	Average Conc. (ppm diesel)	Std. Dev.
Site Sediment	3	57.4	47.4
Wetted Sediment	3	30.2 (53%) <sup>1</sup>	10.4
Dewatered Sediment	3	49.3 (86%) <sup>1</sup>	16.1

value in parentheses is the percentage of the untreated "site sediment" value for comparison purposes

#### Discussion

The immunoassay test kit was relatively easy to use, but the user should either receive the SDI-sponsored training or be proficient with laboratory work including repeat pipettor technique, the use of an analytical balance, and be experienced in using the test kits before attempting to use this on an actual case.

The kits will not give comparable results to the laboratory data for water samples since the immunoassay procedure only tests the dissolved fraction of the petroleum product, where the laboratory procedure captures the dissolved fraction as well as the surface film or sheen. In instances where the determination of dissolved hydrocarbon concentrations are the goal, the kit may produce useful data.

There was a notable amount of variability in test kit results for all test conditions. The amount of variability observed in sample results from a given test condition (*ie.* freshwater 40uL spikes with the BTEX kit), with coefficients of variation ranging from 1.8 to 66.8%, may be acceptable when this kit is evaluated as a screening tool. Considering the amount of variability observed, laboratory validation of some of the sample concentrations is important, and should be considered a recommended practice for any spill site where these kits are used.

The data suggest that the matrix (sediment or soil type) may influence the results, suggesting that a site soil should be used in generating the correction factor. Ocean beach results, using  $40\mu$ L spikes with the PAH kit, were 50% of the laboratory result while all other recoveries ranged from 10-26% of the laboratory result. This particular treatment result was the only one that was not significantly different than the laboratory result.

Our data suggests that the drying procedure is effective, producing an average results that was 86% of the unaltered site sediment (all analyses done with immunoassay kit only). If sediment or soil samples contain 30% moisture or greater, then the drying procedure is recommended for use on the samples prior to analysis with the kits.

The accuracy of the kit results produced using the manufacturer's published procedure was not found to be sufficient for OSPR's data needs. Results were shared with SDI, and a company technical representative agreed that a site-specific correction factor was a recommended practice to be applied to test kit results. The application of a correction factor should be performed with site sediments or soils using a sample of the spilled petroleum product so that there can be a gravimetrically determined (weighed) concentration to compare kit results to. Unitless correction factors based on the results from Tables 4 and 5 are reported in Table 7 below. They were computed by dividing the actual concentration by the kit-reported result. They vary between 2.2 and 11.0, due to the notable variability in the results. These are the factors that would be appropriate to apply to the test kit results to arrive at a more accurate total petroleum hydrocarbon result for any sediment samples tested with the kits. The variation in these correction factors, especially between the two concentrations tested in each treatment, is of concern to OSPR. Correction factors that vary with concentration are not very useful when environmental samples where the concentrations are unknown. Further investigation may help clarify the reasons for the variability in computed correction factors, and whether the correction factors can be used to improve the accuracy enough to meet the data quality needs of OSPR. The result of applying the correction factors to the results from both the BTEX/TPH and PAH kits are shown in Table 8.

Samples	BTEX Kit Correction Factor (10µL)	PAH Kit Correction Factor (10µL)	BTEX Kit Correction Factor (40µL)	PAH Kit Correction Factor (40µL)
Freshwater Sediment	8.9	8.9	11.0	5.8
Beach Sand	5.0	9.3	7.8	2.2

Table 7. Correction factors to apply to the kit results. These are calculated from the average of the kit results in Tables 4 and 5.

	10 µL diesel s	pikes (810 ppm)	40 µL diesel sp	ikes (3250 ppm)
Samples	BTEX kit (diesel ppm)	PAH kit Total Fuel (ppm)	BTEX kit (diesel ppm)	PAH kit Total Fuel (ppm)
Freshwater Sediment-1	578	782	3051	3789
Freshwater Sediment-2	867	812	3544	<del>17794</del>
Freshwater Sediment-3	985	836	3156	2711
Avg. of Replicates	810	810	3250	3250
Std. Deviation	210	27.3	260	762
Coefficient of Variation (CV)	25.9%	3.4%	8.0%	23.4%
Beach Sand-1	986	801	3380	2900
Beach Sand-2	718	801	3160	1278
Beach Sand-3	726	828	3210	5572
Avg. of Replicates	810	810	3250	3250
Std. Deviation	152	15.7	115	2169
Coefficient of Variation (CV)	18.8%	1.9%	3.5%	66.7%

Table 8. Total Petroleum Hydrocarbon (TPH) results following application of correction factors using the SDI Total BTEX/TPH and PAH RaPID ASSAY<sup>®</sup> kits.

#### Costs per Sample

The cost of all the work done in this investigation, not including salaries or the costs of an analytical balance or Vortex Genie (existing PCL equipment), was \$6036.80. A total of 95 samples were analyzed, not including the calibration standards and controls. That equates to a per sample cost of \$63.55 per sample. That cost per sample would decrease with an increase in the number of samples, since much of the cost was for one-time equipment costs such as the RPA-1 RaPID Analyzer (a spectrophotometer) that cost \$1951.34. Costs per sample, at \$63.55 per sample, were higher than those reported by the California Military Environmental Coordination Committee (\$13 per soil sample, CMECC 1996).

#### Conclusions

- Because of sampling differences in water matrices and the fact that the carrier solvents tested did not make diesel fuel completely soluble in water, results from this kit and the DFG laboratories are not comparable. No laboratory data was collected on water phase tests for such a comparison. The immunoassay provides results only for the dissolved phase and excludes the product on the surface (sheen), where the laboratory method would have captured both the dissolved portion and the product on the surface (sheen).
- Our data revealed that the reproducibility (precision) of the kits did not meet OSPR's expectations using the number of replicates used in this investigation (n=3). More investigation into the precision is recommended before any recommendation to OSPR is made regarding the use of these kits in spill response or NRDA data collection.
- 3. Prior to the application of a correction factor, the accuracy of the kit results as tested, was not acceptable for OSPR's data needs. As recommended by the company, a site specific and product specific calibration factor should be

developed for each application of the test kit. The correction factors developed in this study, however, were not consistent (*e.g.* there were different factors for the two different spike concentrations). So, unless this is resolved in further studies, the application of correction factors to results is not currently a solution that we can recommend to OSPR that will make the results from these kits acceptable for data collection in spill-related work.

- 4. Beach sand results appear different than the freshwater sediment results, especially for the 40µL spikes using the PAH kit. Most recoveries were in the 10-26% range of the laboratory result for the BTEX and PAH test kits, except for the beach sand test of the 40µL spike using the PAH test kit which yielded 50% of the laboratory result.
- 5. Our data suggests that the drying procedure is effective, producing a measured average that was 86% of the unaltered site sediment which contained less than 30% moisture. If sediment or soil samples contain 30% moisture or greater, then the drying procedure is recommended for use on the samples prior to analysis using the immunoassay kit.

**Recomendations:** 

- 1. Laboratory staff or other staff trained to use the kit should be the ones to perform the analyses during spill situations.
- 2. Tests in other environments with other fuel products would be very useful in determining the broader applicability and durability of this kit in the work that OSPR performs.
- 3. An investigation to better quantify the precision of the kits, as noted in item #2 in the conclusions above, is recommended. This should be done both with and without sediment extractions. We anticipate that these experiments would be done with three treatments; 1) 7-10 samples fortified with the kit's control solution (contains a known concentration of the analyte) run without any soil or sediment extractions (added directly to the tubes used in the immunoassay procedure), 2) 7-10 samples run on the kits following sediment extractions, and 3) 7-10 sediment extractions run with the U.S. EPA Method 8015B (GC-FID) method commonly used by the laboratory. The results will help quantify the precision of the immunoassay kits as well as the extraction kits, and allow for a comparison to the standard laboratory analytical method.

#### Acknowledgements

Susan Sugarman, Shane Stahl, and Jim McCall all assisted with this work and we appreciated and acknowledge their valued assistance. We also thank Marida Martin for her review of this report.

#### References

California Military Environmental Coordination Committee (CMECC), Chemical Data Quality/Cost Reduction Process Action Team, 1996. *Field Analytical Measurement Technologies, Applications, and Selection.* April. Available at <u>http://www.epa.gov/Region9/qu/pdfs/measure-technol.pdf</u> Appendix A available at <u>http://www.epa.gov/Region9/qu/pdfs/mtappendixa.pdf</u>

Phillips Petroleum Co., 2002, Material Safety Data Sheet, No. 2 Diesel Fuel

United States Environmental Protection Agency (USEPA), 1996. *Method 4030 Soil Screening for Petroleum Hydrocarbons by Immunoassay.* December. Available at <a href="http://www.epa.gov/epaoswer/hazwaste/test/4\_series.htm">http://www.epa.gov/epaoswer/hazwaste/test/4\_series.htm</a>

### **Attachment 1- Original Proposal**

## Validation of RaPID Assay<sup>®</sup> Immunoassay TPH Test Kits against Standard DFG Laboratory Techniques

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**Hypothesis and Objectives**: Immunoassay test kits have been used in many applications to provide rapid and inexpensive data related to contaminants in soil and water. For example, the Central Coast Regional Water Quality Control Board has accepted data generated using immunoassays for total petroleum hydrocarbon (TPH) screening because of demonstrated good correlation with laboratory data and overall effectiveness at Vandenberg Air Force Base (CMECC 1996). These test kits allow for a throughput of up to 50 samples to be analyzed in 1 hour, with a final cost of approximately \$13 dollars per soil sample and \$5 per water sample (CMECC 1996). Immunoassay kits for the screening of soil samples for petroleum hydrocarbons have a USEPA test method number of SW-846, method 4030 (USEPA 1996).

The hypothesis that we propose to examine is; The results of the RaPID Assay<sup>®</sup> Immunoassay TPH test kit compare favorably with the standard laboratory results generated by the Department of Fish and Game laboratories on real oil spill sediment and water samples. This is simply a validation to see if these kits work as well on actual oil spill samples as they do on more weathered types of oil found at other contaminated sites. Additionally, we would like to evaluate some spiked samples with known concentrations of oil to assess test kit accuracy. Common interferences such as biogenic sheen, high turbidity, variable salinity, and various forms of organic matter content are all very common in oil spill samples, and we are interested in evaluating the ability of these test kits to perform given these real-world conditions. If they do it well, the objective would be to inform OSPR response and NRDA personnel that they may utilize these test kits to increase the efficiency of sample collection and analysis onscene. The findings may also have bearing on whether we eventually seek to utilize the PAH test kits that are also available on the market.

#### **Experimental Plan:**

After purchasing the test kits and the necessary analytical instrument (the RPA-1 Analyzer and accessories), along with the material items needed such as solvent and disposable gloves, work will be completed in collaboration with Dave Crane and the Staff at the DFG labs.

1. We will run a series of spiked water and sediment samples using petroleum products (*e.g.* diesel). The results of the test will be compared to the known (nominal) amounts spiked into the sediment or water, and an evaluation of accuracy and reproducibility will be summarized.

- 2. Next, a number of water and sediment samples will be analyzed that have previously been analyzed by the DFG laboratory. A comparison table showing the results from both tests will be compiled, along with a calculated difference between the two.
- 3. A report will be written that summarizes the findings of this work, and will include a recommendation relating to the utility of this test method in oil spill response and NRDA sampling work.

**Significance to OSPR**: This technology allows for rapid analysis of a relatively large amount of samples on-site for a relatively low cost per sample. This capability would allow on-site decision makers to view data quickly, and decide whether a site is "clean" or which samples should be sent in for laboratory analysis based on the screening results that the test kit provides. This information could be used to reduce the amount of non-detect or below reporting limit results that are received from site sampling, and can provide rapid results for clean up crews that have a real interest in knowing when a site is "clean." This last point is especially relevant when rental equipment used in the cleanup effort is on scene adding to response cost by the day or hour. Results from this type of analysis kit can also help NRDA sampling teams decide which samples warrant more definitive analysis for injury assessment purposes, and which are likely to contain undetectable concentrations of TPH.

**Project Duration**: This project is proposed to take 1 year.

- two months to purchase equipment and supplies
- two months to train up and familiarize ourselves with this equipment
- six months to run the validation samples
- two months to summarize the results.

BUDGET				
Personnel	\$0			
Equipment	\$6,250 <sup>1</sup>			
Supplies	\$750 <sup>2</sup>			
Travel	\$0			
Other Expenses	\$0			
Overhead	\$0 (no contract required)			
Total	\$7,000			

#### Estimated budget:

<sup>1</sup> Includes the RPA-1 RaPID Analyzer(photometer), 60 position magnetic separator, Eppendorff repeating pipettor, digital balance and timer, Eppendorff Adjustable Volume Pipettor, Vortex Genie, Test Tubes (polystyrene, 500/box, 12x75mm)

<sup>2</sup> Includes RaPID Assay Total BTEX/TPH 100 tube test kit, pipette tips, nitrile gloves, and other consumables such as absorbent paper)

#### Supplemental Funds: None

#### Literature Citations:

California Military Environmental Coordination Committee (CMECC), Chemical Data Quality/Cost Reduction Process Action Team, 1996. *Field Analytical Measurement* 

*Technologies, Applications, and Selection.* April. Available at <u>http://www.epa.gov/Region9/qu/pdfs/measure-technol.pdf</u> Appendix A available at <u>http://www.epa.gov/Region9/qu/pdfs/mtappendixa.pdf</u>

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