EVALUATION OF A NEW NON-IMMUNOASSAY FIELD TEST KIT FOR TOTAL PETROLEUM HYDROCARBONS IN SOIL

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Abstract

A new field test kit for TPH in soils has been evaluated and compared to SW-846 methods. Soils spiked with known amounts of both diesel fuel and number 6 fuel were analyzed using the new method as well as 418.1 and 8015. The experimental results were then compared to the known concentrations to determine the relative response factors for the two different analytes for all methods as well as the method precision and bias for the new method. The results indicate that the response factor for the new method is very good compared to method 8015 and is much better than 418.1 for both analytes. The replicate data for the new method indicates that the repeatability is better than 10% and there was very little bias found.

Introduction

A novel new analytical procedure has been developed to determine the hydrocarbon content of soil samples using environmentally safe reagents which will be simpler and less expensive than alternative methods. The data presented here compares the new method to standard methods and illustrates the effectiveness of the new PetroFLAG" technology on two analytes; diesel fuel and number 6 fuel oil.

The PetroFLAG technology has been tested in the field in a non-commercial form for over two years. A commercial version will be available soon for use in the field by environmental professionals. The calorimetric test is easy to use and contains no hazardous chemicals. A specially designed hand-held calorimeter will be available to provide a digital readout in ppm of the analyte. Using the prepackaged reagents 10 to 20 samples can be run in one batch in under 30 minutes. The anticipated cost per test is $10 to $15 and the calorimeter will cost under $300. The patent pending kit chemistry relies on a unique system of extraction solvents and color-forming reagents. Because of its broad linear response range the PetroFLAG test kit can be used on a wide variety of hydrocarbon analytes including fuels, lubricants, hydraulic fluids and greases. The PetroFLAG kit does not only test for specific compounds such as aromatics, rather all hydrocarbons. This makes the kit useful as a fast, low cost general screening tool for hydrocarbons, as well as for the quantitative determination of hydrocarbon contamination in soil samples, while providing the user with real time data for on-site decision making.

The test is easily performed at contaminated sites where a variety of sampling strategies are to be used. The test is useful at sites requiring lateral and vertical definition of the soil contamination plume throughout the vadose zone, including sites that are being drilled and sampled or excavated and sampled prior to the collection of expensive laboratory confirmation samples. The test is especially useful during underground storage tank (UST) removals to screen the excavated area during the excavation and prior to collecting expensive confirmation samples.

The test is well suited for use at large sites where a grid sampling plan is the strategy of choice. In many cases a grid sampling plan would be ruled out due to the high cost of laboratory samples. Use of the PetroFLAG kit will allow the grid strategy to be used while providing the user with the meaningful data for cost and time saving on-site decision making prior to collecting and submitting samples for expensive laboratory analysis. The use of the PetroFLAG test kit for grid sampling will help to economically locate and define the extent of the soil contamination, locate and define "hot spots" and help to delineate the zero line. This could help save unnecessary drilling and sampling costs and equipment re-location costs caused by waiting for laboratory analytical data, and allow for more area to be tested thus reducing the possibility of future liability caused by failing to identify contaminated areas. It is also possible that by using this low cost kit as a screening tool, less money will have to be spent on unnecessary expensive laboratory analysis resulting in more money being available for actual clean-up and remediation of the contaminated site.

The PetroFLAG test is also useful for tracking and evaluating the success of soil remediation projects, such as bioremediation. The test uses field calibration standards to achieve a high degree of accuracy in a large variety of soil types. The PetroFLAG technology is currently in the beta-testing stage. Evaluations on different soil types with different analytes indicate that the extraction efficiencies are very high for most petroleum hydrocarbon contaminants.

Preparation of Spiked Soil Samples

To simulate typical soils, an 8 kg mixture of clay soils and sand, approximately 75:25 w/w was prepared. The soils and sand were obtained from residential areas and were analyzed for total petroleum hydrocarbons (TPH) using EPA Method 418.1 and found to contain less than 10 ppm TPH. The clay soil was broken up by hand and allowed to air dry for 24 hours. The clay and sand were sieved to pass a 0.850 um sieve, mixed together, and tumbled for 24 hours in a rotating pail. Most of the clay particles were observed to be considerably smaller than 0.850 um. The water content of the freely flowing mixture was I %. The soil was transferred to aluminum cake pans prior to addition of contaminants. The soils were then spiked with either diesel fuel or number 6 fuel oil. The fuels were dissolved in an excess of hexane to facilitate uniform mixing throughout the soil. The soil mixtures were air dried to a constant weight, bottled in previously unused clean 8 oz. glass bottle with PTFE lined caps and tumbled for 4 hours on a rotating tumbler.

Analysis

Each of the spiked soils were analyzed in triplicate using the PetroFLAG test kit and the Sw-846 methods. Both of the standard methods were run according to standard procedures. The solvent used for method 8015 was the acetone hexane mixture. The PetroFLAG analyses used the following procedure: 5 grams of soil were weighed into the extraction tube, 10 grams of extraction solvent were added and the sample was extracted for 5 minutes with intermittent shaking. The extract was then filtered using a 0.2 um filter fitted with a glass wool pre-filter. The filtered extract was added directly to the analysis vial containing the premeasured color reagent. The vial was then capped and shaken vigorously to ensure mixing. The color was allowed to develop for a minimum of 10 minutes, shaking intermittently. After the color development step, the vial was placed in the calorimeter. The absorbance reading was then used to quantify the TPH content of the sample using the standard calibration curve.

The calibration solutions for the PetroFLAG tests were made up using diesel fuel in the extraction solvent at 50 ppm and 250 ppm. For the 5 gram sample size used for this study this is equivalent to 100 ppm and 500 ppm in the soil. In the field the PetroFLAG kit will use a soil spike at two levels to determine the response factors and background corrections for site specific soil samples. For this study, using solvent standards allowed for an estimation of the extraction efficiencies for the new method.

Results and Discussion

The PetroFLAG diesel fuel results are presented in table 1 and plotted in figure 1. As indicated by the relative standard deviation between replicates the method is very reproducible. The comparison with the gravimetric value indicates that the extraction efficiency is greater than 95%. There is also very little bias.

The number 6 fuel oil results shown in table 2 and plotted in figure 2, indicate that the response factor is approximately 90% at 500 ppm. The repeatability as indicated by the standard deviation is again at least +/- 10%.

The diesel data for all three methods are plotted in figure 3. It can be seen that the PetroFLAG results are in good agreement with the 8015 values at the lower concentrations, but at the higher concentrations the two methods diverge slightly. The 418.1 results are very poor, with an average extraction efficiency of only 14%. A GC analysis of the Freon extract confirmed this extraction efficiency. The results were much the same for number 6 fuel. These data are plotted in figure 4.

Summary

The data for the two analytes investigated show that the PetroFLAG method is very reproducible and compares well with method 8015. The 95% confidence intervals for the replicates indicate the new method should be expected to have a repeatability of better than 10%. Although the two analytes differ in composition they give a response, using the same calibrator, that is within 10% at the 500 ppm level. The poor performance of method 418.1 indicates that it should be used with caution on these analytes.

Table 1: PetroFLAG **Diesel Results**

Conc. (ppm)	**Trial A (ppm)**	**Trial B (ppm)**	**Trial C (ppm)**	**Mean (ppm)**	**Std. Dev. (ppm)**
54	69	69	71	70	1.09
106	107	114	114	112	3.82
255	239	254	245	246	7.58
1516	504	507	516	509	6.43

Table 2 Petro- Flag **Number 6 Fuel Results**

Conc. (ppm)	**Trial A** (ppm)	**Trial B** (ppm)	**Trial C** (ppm)	**Mean** (ppm)	**Std. Dev**. (ppm)
50	75	71	75	73	2.18
100	*105*	117	117	112	6.55
251	222	234	234	230	6.55
1500	438	449	449	446	6.55

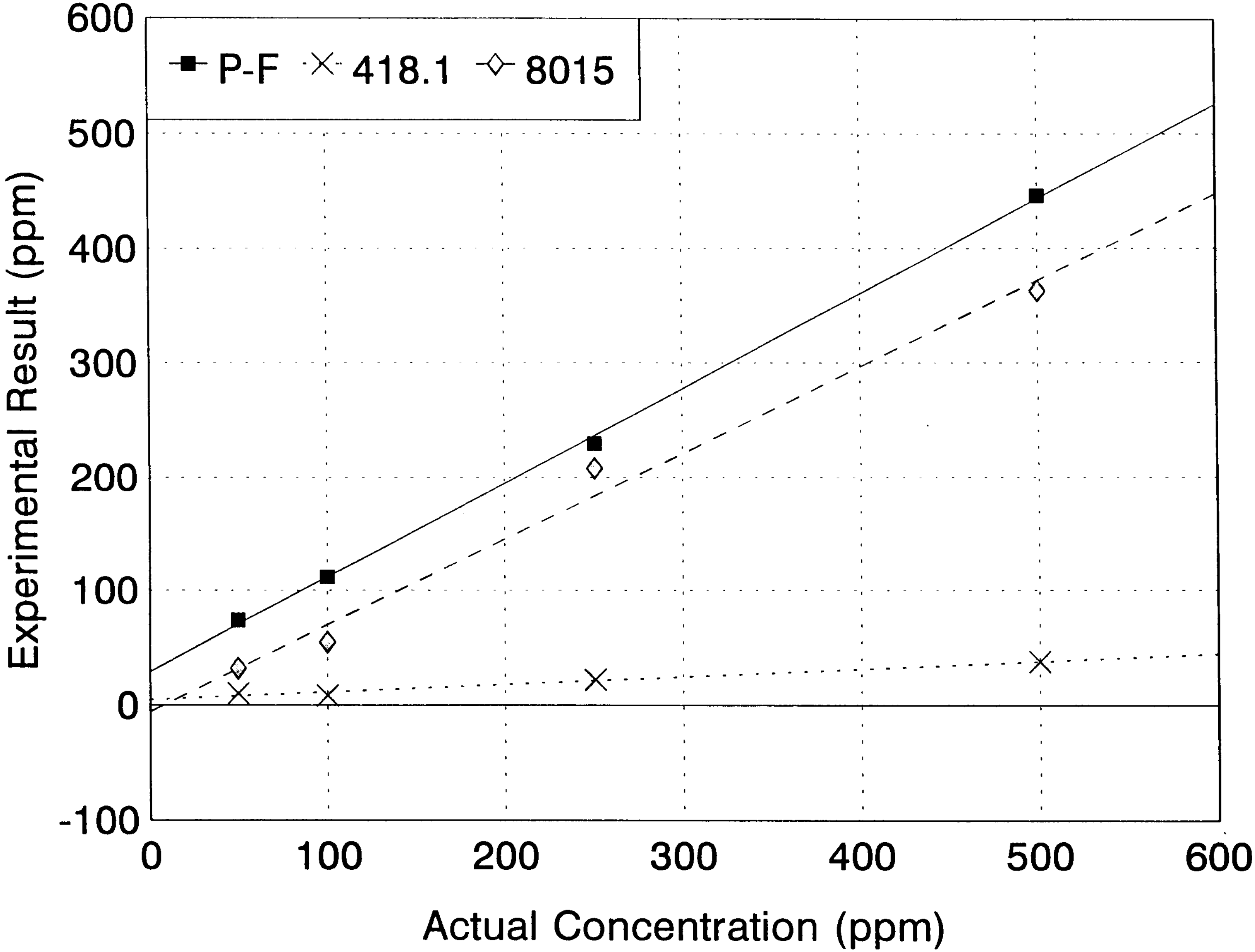

Figure 4: Petro-Flag Comarison With EPA Methods; #6 Fuel

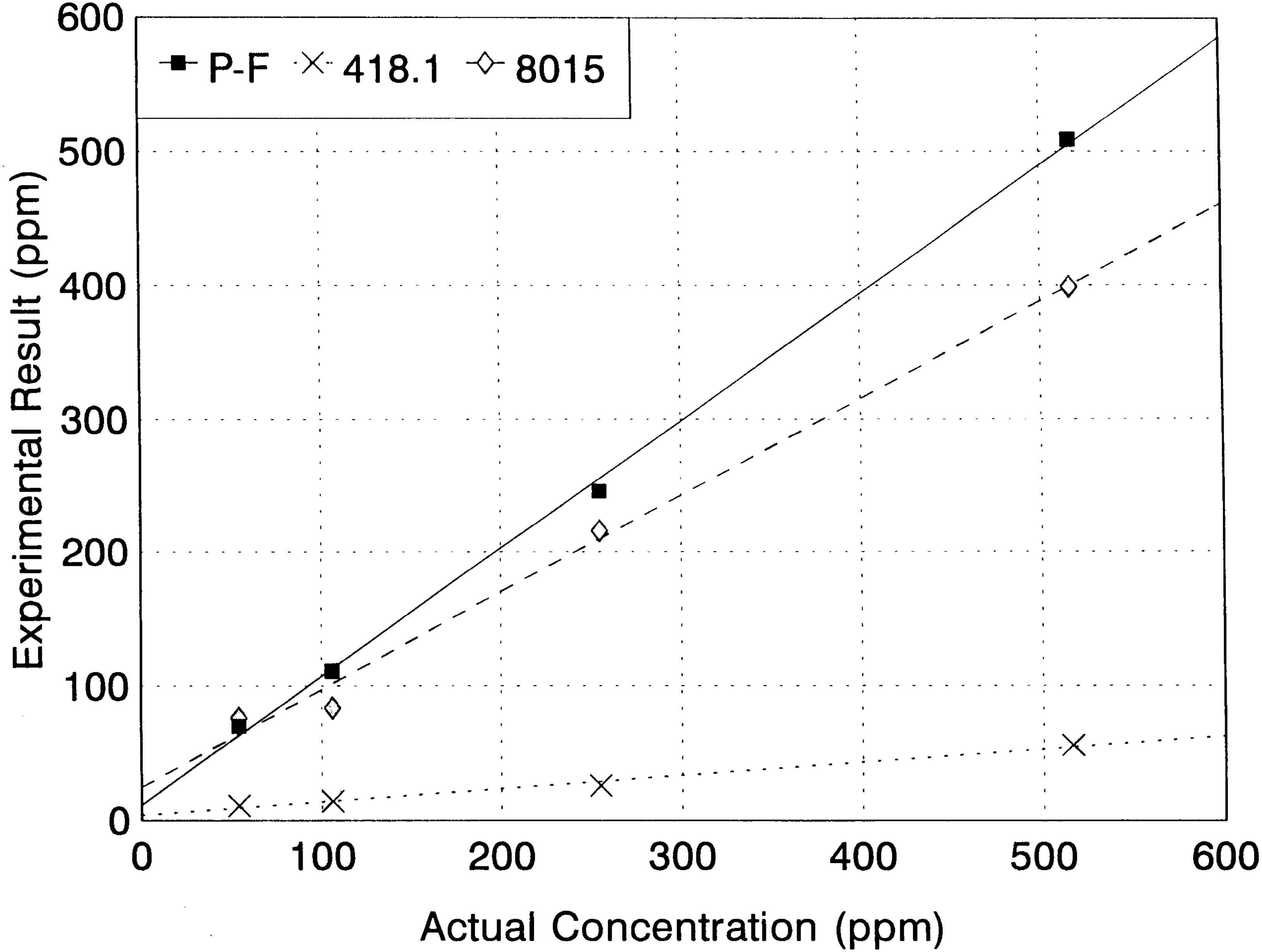

Figure 3: Petro-Flag Compared With EPA Methods; Diesel Fuel

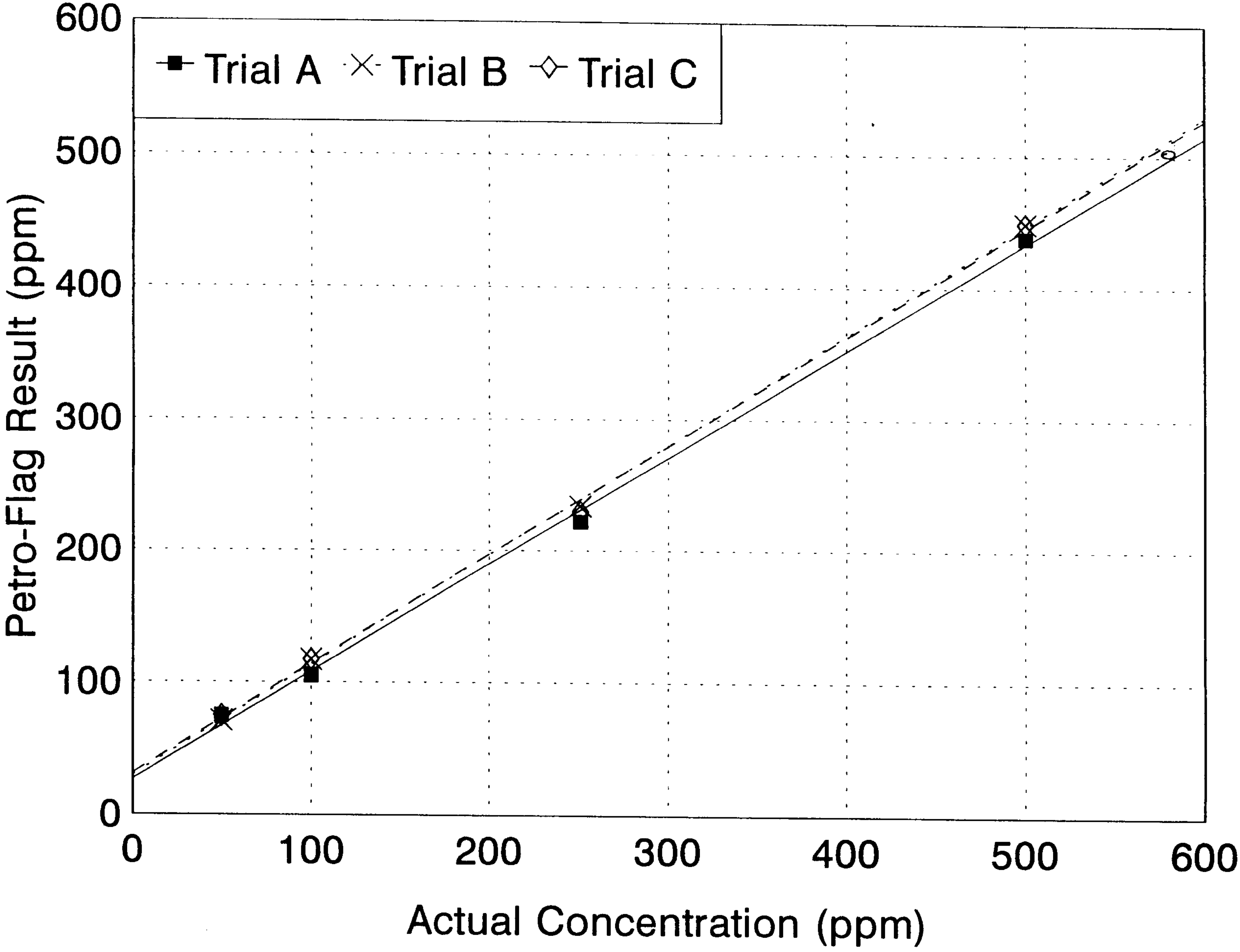

Figure 2: Petro-Flag Number 6 Fuel Results

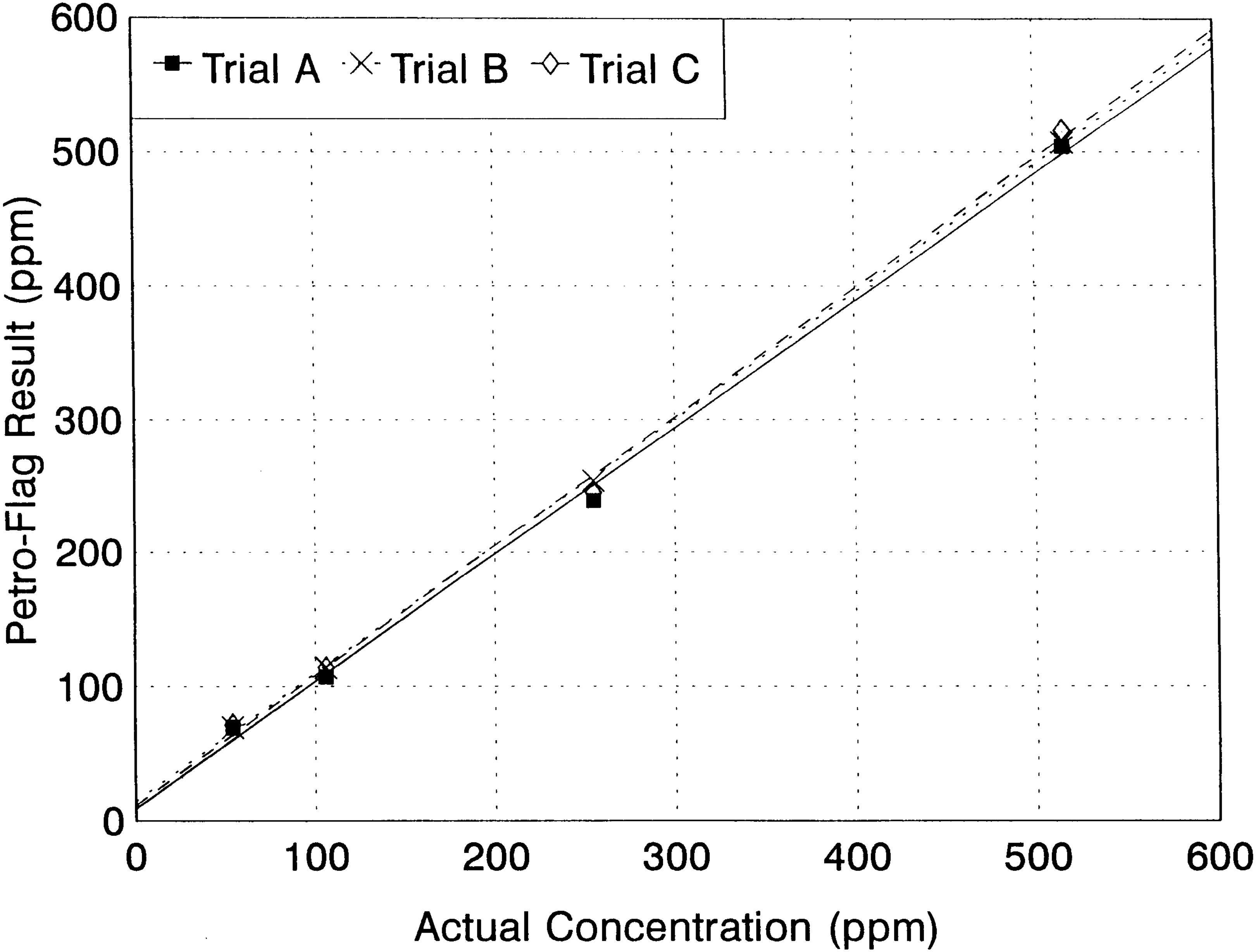

Figure 1: Petro-Flag Diesel Results