Mr. Russell D. Hart (HSRW-6J) Remedial Project Manager U. S. Environmental Protection Agency Region V 77 West Jackson Boulevard Chicago, IL 60604

WESTON Work Order No.: 02687.007.003 KMC Work Order No.: 40-50-01-AKW-B

Re: Immunoassay Testing of Floodplain Soil Moss-American Site, Milwaukee, Wisconsin

Dear Mr. Hart:

Weston Solutions, Inc. (WESTON_{SM}), is pleased to submit this report summarizing the results of the immunoassay testing of floodplain soil recently completed at the Moss-American Site in Milwaukee, Wisconsin for Kerr-McGee Chemical, (KMC), LLC. Contained within this report are:

- An outline of the purpose and objectives of the testing that was performed.
- Descriptions of the soil sampling activities and procedures employed during immunoassay testing.
- Summaries of both the field and laboratory analytical data, including a comparison of the two data sets.
- Conclusions.

1 PURPOSE AND OBJECTIVES

The purpose of this report is to present the evaluation of the effectiveness and implementability of field analysis of soil and sediment (supplemented with laboratory samples to confirm field analyses).

During implementation of the river remedy field analytical methods are preferred due to the potential impact to project schedule. Use of field methods to evaluate soil/sediment allows for timely data collection and enables decisions to be made in the field as work progresses. This is expected to increase construction efficiency and productivity since delays associated with obtaining analytical results from an off-site laboratory would not be timely and thus would be avoided. Analytical results of soil and sediment samples may be obtained within a few hours after sample collection when using field methods, whereas at least 1 day would be required to obtain analytical results from a local, off-site laboratory. If samples are shipped overnight to a remote laboratory, it may take several days to obtain analytical results. Although use of an on-

site laboratory may also provide rapid results for soil and sediment samples, due to the limited number of samples that would be collected each day the use of an on-site laboratory is not a realistic option.

The immunoassay soil testing was performed to accomplish two major objectives:

- To demonstrate that immunoassay testing is a valid and field-implementable method for determining total Carcinogenic Polycyclic Aromatic Hydrocarbon (CPAH) concentrations in environmental media (i.e., soil and sediment) during full-scale implementation of the river remedy for the Milwaukee Moss-American site.
- To complete Phase II of Predesign Task 7 (determine the extent of CPAH contamination in floodplain soil along the new river alignment) for Reach 1 of the Little Menomonee River (LMR), which extends from the railroad bridge south of Brown Deer Road to Bradley Road. The scope and objectives of Predesign Task 7 are more fully described in the 90% Design Report for Reroute of the Little Menomonee River (WESTON, June 2002).

2 SOIL SAMPLING ACTIVITIES AND ANALYTICAL PROCEDURES

Sample collection activities were completed on 30 May 2002, and field analysis and sample preparation for laboratory analysis was performed on 31 May 2002. Field work under the scope of this task was conducted in accordance with Addendum 4 of the Quality Assurance Project Plan (QAPP) for Installation of the Groundwater Remedial System, which is currently being prepared by WESTON. It is anticipated to be submitted in July 2002 for review by the United States Environmental Protection Agency (U.S. EPA) and the Wisconsin Department of Natural Resources (WDNR). Since the immunoassay testing is a U.S. EPA-approved analytical procedure (SW-846 Method 4035), and soil sampling, handling, and analysis for CPAHs have been previously addressed under the QAPP, discussions with U.S. EPA and WDNR confirmed that fieldwork may proceed without finalization of Addendum 4 of the QAPP. A copy of Method 4035 is included in Attachment A.

2.1 <u>Soil Sampling Locations</u>

Soil samples were collected from the 0 to 12-inch below ground surface (bgs) interval from stations located every 200 feet (ft) along the new alignment for Reach 1 of the LMR proposed in the 90% Design Report. Figure 1 depicts the soil sampling stations along Reach 1 of the LMR. Bernklau Surveying, Inc., of Sussex Wisconsin, surveyed each sampling station location prior to sampling.

Based on the 90% Design Report, the total length of the proposed new alignment for the LMR along Reach 1 is approximately 6,600 ft; however, for the lower 700 ft of Reach 1, sediment in I:\WO\MOSSAMER\31580.DOC

the existing channel will be removed to facilitate reuse of the existing LMR channel. Since excavation of a new channel along the lower 700 ft of Reach 1 is unnecessary, floodplain soil along this portion of Reach 1 was not sampled under the scope of this task. A total of 30 stations were surveyed and sampled along the upper 5,900 ft of Reach 1.

The surveyed stakes were identified using a temporary coordinate system consisting of stations beginning at Stake 100 (located approximately 70 ft south of the railroad bridge south of Brown Deer Road) to Stake 158, located 5,800 ft south of Stake 100. Subsequent to the fieldwork, a new coordinate system has been developed to identify stations along the new LMR alignment, with each station beginning with a "R" to signify that it is a "reroute" station. This coordinate system will be fully described in the Final (100%) Design Report. Table 1 correlates each of the survey stakes to the existing and rerouted river stations.

2.2 Soil Sampling, Handling, and Analysis

Sample collection activities were completed on 30 May 2002. Field conditions during sampling activities consisted of partially cloudy skies, with a high temperature of 86 °F. Although only trace precipitation was recorded on 28 and 29 May 2002 and the most recent substantive rainfall was 5 days previous (0.52 inches on 25 May 2002), the ground at most sampling locations was moist to wet.

Each soil sample was collected using a 4-inch diameter, bucket auger. The auger was decontaminated between sampling locations using an Alconox wash followed by a distilled water rinse. The sample media removed from the auger was placed into plastic bags and stored in an iced cooler until all soil samples were collected. Strict chain-of-custody procedures were employed during storage of the samples.

Samples were analyzed for moisture content and CPAHs using both field and laboratory methods. A total of 30 investigative and 3 duplicate samples were analyzed in the field for total CPAHs using the RaPID Assay® immunoassay test, which is distributed by Strategic Diagnostics, Inc. (SDI), of Newark, Delaware. A total of 30 investigative and 6 duplicate samples were analyzed in the field for moisture content using a Large Speedy Moisture Meter, manufactured by Ashworth Instrumentation of Lancashire, England. In addition, to confirm the data obtained by field analyses, ten investigative, one duplicate, and one matrix spike/matrix spike duplicate samples were submitted to Lancaster Laboratories of Lancaster, Pennsylvania, for analysis of PAHs by SW-846 Method 8310 and moisture content per EPA 160.3 modified.

All samples were field analyzed and prepared for shipment to Lancaster Laboratories (as applicable) on 31 May 2002. All samples that were submitted to Lancaster Laboratories for analysis were selected prior to having measured the total CPAH concentration with the RaPID Assay, so the total CPAH concentrations were unknown at the time of sample packaging. The following subsections provide a brief overview of the RaPID Assay and Speedy Moisture Meter I:\WO\MOSSAMER\31580.DOC

procedures employed during the field analysis, as well as a the procedures for sample preparation for field and laboratory analyses.

2.2.1 Sample Preparation

Each sample was homogenized using decontaminated, stainless-steel spoons and mixing bowls until the sample appearance (i.e., color and texture) was consistent. For those samples submitted for laboratory analysis, the sample media was placed into 16-ounce glass jars with Teflon-coated lids immediately following homogenization. The samples submitted for laboratory analysis were labeled with identifiers as indicated in Table 1, placed in an iced cooler for shipment, and delivered under chain-of-custody via overnight courier to Lancaster Laboratories.

Upon collection of the soil media required to perform the laboratory analyses, an aliquot was immediately collected from the same media for analysis using the RaPID Assay and the Speedy Moisture Meter.

SDI recommends that samples analyzed using the RaPID Assay not contain free liquid (i.e., water), and that a coffee filter may be used to apply pressure to the sample media to remove free water as necessary. As a general guideline, SDI indicate that approximately 30% moisture content may represent conditions where free water would be present in the sample media. When performing the moisture content analysis (as described in the following subsection), numerous samples were identified to have moisture content above 30 % and were dewatered using the coffee filter method. Although the coffee filter appeared moistened after the dewatering procedure, free liquid could not be removed from the samples using this technique, presumably due to the small particle size of the clayey silty soils. After reviewing the data from both the immunoassay and laboratory testing, and based on SDI's understanding of the site conditions, SDI concluded that the high moisture content of the samples did not adversely affect the accuracy or implementability of the RaPID Assay for soil testing at the Moss-American site. In addition, it appeared that the coffee filter dewatering procedure was minimally effective, if at all, at removing moisture from site soils that were predominantly silts and clays.

2.2.2 Moisture Content Analysis (Speedy Moisture Meter)

Moisture content of the soil samples was measured in the field using a Speedy Moisture Meter. All 30 soil samples were analyzed using the meter, and an additional 6 duplicate measurements were performed to evaluate the accuracy of the instrument. Analyses were performed in accordance with manufacturers specifications (see Attachment B).

All moisture content data was recorded in the project logbook as it was obtained. Copies of the logbook pages associated with sample collection, preparation, and analysis activities for the immunoassay pilot testing are provided as Attachment C.

Upon completion of the field work the Speedy Moisture Meter was sent for inspection and calibration to ELE International, Inc. (ELE), of Alabaster, Alabama. ELE reported that all readings should be corrected by subtracting 1.5 % from the moisture measurements.

2.2.3 Total CPAH Analysis (RaPID Assay)

All 30 flood plain and 3 duplicate soil samples were analyzed for total CPAHs using the RaPID Assay immunoassay. The standard detection range for the RaPID Assay test is 0.01 to 0.5 milligrams per kilogram (mg/kg). The standard test was modified to include a 50-fold serial dilution to increase the detection range from 0.5 to 25 mg/kg. Analyses were performed in accordance with manufacturers specifications (see Attachment D).

The spectrophotometer printout provides both the calibration curve information and prints out the concentration of each sample in micrograms per kilogram ($\mu g/kg$). Copies of the spectrophotometer printouts are included as Attachment E. The samples were extracted and analyzed in two batches, each with their own set of CPAH standards, with the first batch containing 10 investigative and one duplicate sample and the second batch containing the remaining 20 investigative and 2 duplicate samples. The correlation of the standardized curve for the first batch of data was 99.40 %, and was 99.63 % for the second batch.

3 <u>ANALYTICAL RESULTS</u>

Analytical results obtained through field and laboratory analysis of the soil samples are presented and discussed in the following subsections. Total CPAH and moisture content data for the soil samples are presented in Table 1, and Attachment F contains a copy of the laboratory analytical data package.

3.1 <u>Moisture Content: Comparison of Field and Laboratory Methods</u>

Based on all moisture content data (field and laboratory), the moisture content of the soil samples ranged from 17 to 57 %. The median moisture content was 33 % and the mean moisture content was 36 %. Table 2 provides a comparison of the field- versus laboratory-measured moisture content data.

In the duplicate sample sent to the laboratory (MA6-SSRR-309 and -309DP), a difference of 3 % moisture was observed between the two samples, representing approximately 10 % difference from the average moisture content (31 % moisture). When evaluating the difference in moisture contents of duplicate samples measured using the Speedy Moisture Meter, the % moisture varied from 0 to 8 % moisture, with only one duplicate sample varying by more than 3 % moisture.

Overall, excellent correlation was observed between the moisture content measured in the field and laboratory. The difference in moisture contents measured in the field and laboratory varied I/WO/MOSSAMER/31580 DOC

from 1 to 8 % moisture; however, only one sample had a difference of greater than 4 % moisture. Based upon these results we recommend that this method be used for field analysis of moisture content during river remediation.

These results indicate that the field method for moisture content measurement is suitably accurate for use during river remediation. As compared to the laboratory measurement of moisture content, the field method tends to overestimate the moisture by only a few percent. When combined with the results from the field method for total CPAHs, the slight overestimation of moisture content will act to overestimate the total CPAH concentration (dryweight basis) results by only 0.1 to 0.3 mg/kg.

3.2 Total CPAHs: Comparison of Field and Laboratory Methods

The total CPAH concentrations of the soil samples ranged from 0.2 to 20 mg/kg. Soil samples collected from two locations (rerouted river Stations R899+30 and R873+25) exceeded the 15 mg/kg criterion, and soil samples collected from these and one additional location (rerouted river Station R841+10) exceeded the 6.1 mg/kg criterion. Soil samples collected from the other 27 sampling stations contained total CPAH concentrations below 6.1 mg/kg. Figure 1 indicates the sampling locations as well as the total CPAH concentrations measured at each station. Table 2 provides a comparison of the field- versus laboratory-measured total CPAH concentrations.

All three of the sample locations, where soils exceeded 6.1 mg/kg total CPAHs, are in proximity to non-site-related sources of CPAHs. Station R899+30 is located approximately 70 ft south of the Union Pacific Railway that runs parallel to the northern site boundary, and is also located immediately opposite of the drainage swale that runs parallel to the northern site boundary west of the LMR. Station R873+25 is located approximately 20 ft north of the stormwater drainage channel that serves as a tributary to the LMR, and Station R841+10 was located atop the west LMR bank, approximately 50 ft east of the railway bridge north of Bradley Road.

As with the moisture content data, there was good correlation between the field- and laboratory-measured total CPAH concentrations. When considering the 6.1 and 15 mg/kg criteria for total CPAHs, all samples that were determined to contain total CPAHs above 15 mg/kg and below 6.1 mg/kg using the immunoassay (that were also sent for laboratory analysis) were determined to have total CPAH levels that fell within the same concentration "categories." Although only one of the two samples that had a total CPAH concentration exceeding 15 mg/kg total CPAH was submitted to the laboratory for analysis, this sample was submitted in duplicate to the laboratory, and the average of the total CPAH concentrations for the investigative and duplicate sample was calculated to be 17 mg/kg. All other samples submitted to the laboratory were determined by both the immunoassay and the laboratory to contain less than 6.1 mg/kg total CPAHs. Consequently, the accuracy and implementability of the RaPID Assay meet acceptable criteria to evaluate total CPAH concentrations in the soil/sediment sampled during implementation of the river remedy.

One trend that was identified in evaluating the RaPID Assay versus laboratory-determined total CPAH concentration data was that at lower total CPAH concentrations, the RaPID Assay typically indicated that the total CPAH concentrations were in the range of 1 to 3 mg/kg, whereas much the laboratory data indicated these concentrations to be less than 1 mg/kg.

Upon receipt of the laboratory data, SDI was consulted to evaluate the correlation between the laboratory and field data, assess the appropriateness of using RaPID Assay to determine total CPAH concentrations in the soil at the Moss-American site, and provide explanation of the deviation in laboratory versus field data for samples that contained low levels of CPAHs. After review of the data, SDI concluded that correlation between the laboratory and immunoassay data was good, the range of error within the data is not unusual, and that the RaPID Assay would be a viable technology for determining total CPAH concentrations in site soil/sediment. SDI indicated that the two most likely factors contributing to the overestimation of CPAHs by the RaPID Assay were (1) the serial dilution performed to increase the immunoassay's range of detection and (2) potential interference of noncarcinogenic PAHs. Our findings indicate that the total CPAH concentration reported by the RaPID Assay may be higher than the actual concentration by approximately 1 to 2 mg/kg.

4 <u>CONCLUSIONS</u>

Based on the soil sampling and analyses performed under the scope of this effort, the following conclusions were reached:

- KMC/WESTON recommends the use of RaPID Assay/Speedy Moisture method for the measurement of CPAHs/moisture content, respectively. This combination will allow for the rapid determination of compliance with applicable cleanup standards for CPAHs that are defined on a dry weight basis.
- Site conditions are such that soil within the LMR floodplain typically consists of silts and clays, and in many instances these soils have relatively high moisture content (i.e., >30% moisture).
- Use of the Speedy Moisture Meter is an accurate and implementable method to determine moisture content of soil during construction activities, as good correlation between laboratory- and field-measured moisture content was observed. Although there is a limited difference in the field- versus laboratory-measured moisture content (i.e., a few percent), based on the total CPAH levels observed in the LMR floodplain soil, the effect that this variability has in determining the total CPAH concentration of a given soil sample is relatively negligible (i.e., 0.1 to 0.3 mg/kg).

- The RaPID Assay is an accurate and implementable technology for determining total CPAH concentrations. Although error associated with evaluation of samples containing low levels (i.e., <3 mg/kg) of CPAHs is noticeable, it appears that this limitation would have minimal influence with respect to making soil management decisions during construction of the river remedy.
- The majority of floodplain soil along the path of the re-routed river (27 of 30 samples = 90%) is well below the 6.1 mg/kg cleanup standard.

If you have any questions or require additional information, please do not hesitate to call me at (847) 918-4142.

Very Truly Yours,

Weston Solutions, Inc.

Thomas P. Graan, Ph.D. Principal Project Manager

Attachments

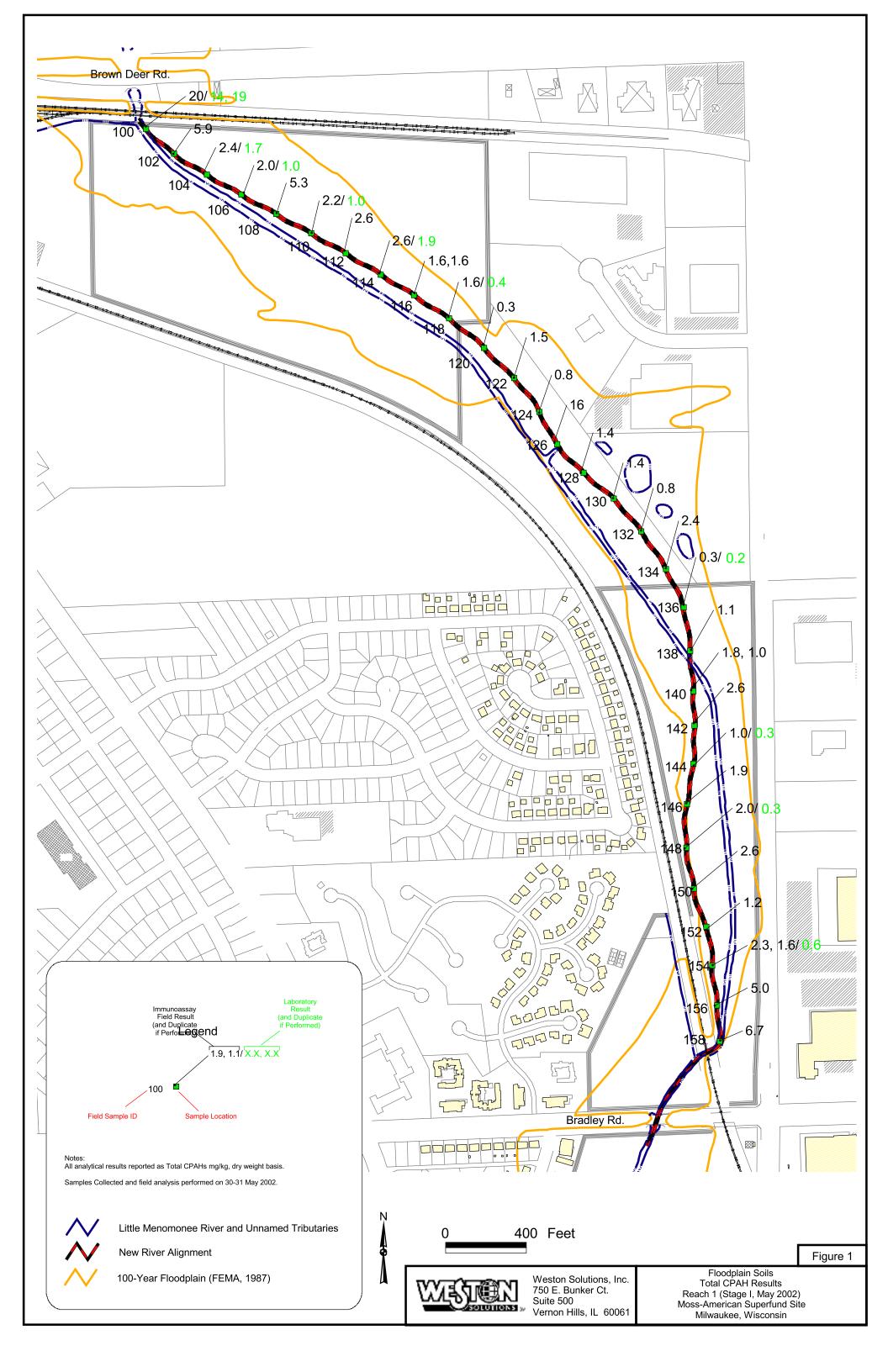


Table 1

Soil Analytical Results for Field- and Laboratory-Measured Moisture Content and Total CPAHs Moss-American Site Milwaukee, Wisconsin

Sa	mple Information		F	ield Analysis Resul	lts	Laboratory Results	
Field Sample Identifier (Survey Stake)	Lab Sample Identifier	Rerouted LMR Station	Total CPAHs (RaPID Assay) (wet weight), mg/kg	% Moisture (Speedy Moisture Meter)	Total CPAHs ¹ (RaPID Assay) (dry weight), mg/kg	% Moisture	Total CPAHs ¹ (dry weight), mg/kg
100	MA6-SSRR-309	R899+30	14	27	20	29	14
100-DUP	MA6-SSRR-309DP	R899+30				32	19
102		R897+45	3.7	37	5.9		
104	MA6-SSRR-305	R895+50	1.4	40	2.4	40	1.7
104-DUP		R895+50		43			
106	MA6-SSRR-303	R893+55	1.3	33	2.0	30	1.0
106-DUP		R893+55		32			
108		R891+55	2.7	49 *	5.3		
110	MA6-SSRR-299	R889+55	1.2	45	2.2	43	1.0
112		R887+60	1.4	48 *	2.6		
114	MA6-SSRR-295	R885+55	1.3	48	2.6	47	1.9
116		R883+60	0.9	43	1.6		
116-DUP		R883+60	0.9	43	1.6		
118	MA6-SSRR-291	R881+55	1.0	40	1.6	37	0.4
120		R879+25	0.5 U	17	0.3		
122		R877+15	0.9	38	1.5		
124		R875+05	0.5	32	0.8		
126		R873+25	12	21	16		
128		R871+30	1.0	26	1.4		
130		R869+35	0.9	34	1.4		
132		R867+20	0.5	38	0.8		
134		R864+95	1.1	53 *	2.4		
136	MA6-SSRR-273	R862+90	0.4 U	30	0.3	29	0.2
138		R860+70	0.9	21	1.1		-1
140		R858+70	1.3	31	1.8		
140-DUP		R858+70	0.7	32	1.0		
142		R857+00	1.4	46	2.6		
144	MA6-SSRR-265	R855+10	0.7	32	1.0	31	0.3
146		R853+05	1.2	37	1.9		
148	MA6-SSRR-261	R850+90	1.4	30	2.0	26	0.3
148-DUP		R850+90		38			-
150		R848+80	1.1	57 *	2.6		
152		R846+85	0.8	38	1.2		
154	MA6-SSRR-255	R844+90	1.5	33	2.3	31	0.6
154-DUP		R844+90	1.1	33	1.6		
156		R842+85	3.7	27	5.0		
158		R841+10	4.8	29	6.7		-

^{1 -} One half of the detection limit was used to calculate the total CPAH concentration for constituents reported as nondetect.

BOLD values exceed 6.1 mg/kg total CPAHs.

Shaded values exceed 15 mg/kg total CPAHs.

U- Constituent not detected. Detecion limit indicated.

^{* -} High-moisture content method used for Speedy Moisture Meter analysis.

Comparison of Field- and Laboratory-Measured Soil Data for Moisture Content and Total CPAHs

Moss-American Site

Milwaukee, Wisconsin

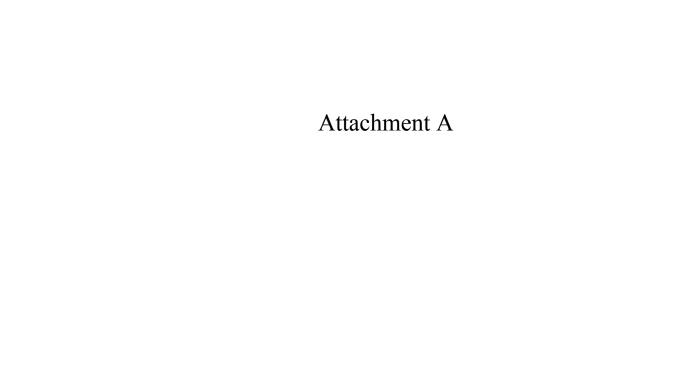
Table 2

	Sample Information		Mo	isture Content D	ata	Total CPAH Data		
Field Sample Identifier (Survey Stake)	Lab Sample Identifier	Rerouted LMR Station	Field-Measured Moisture Content ¹ , %	Laboratory- Measured Moisture Content ¹ , %	Difference in Moisture Content ² , %	Field-Measured CPAH Concentration ^{1,3} , mg/kg	Laboratory- Measured CPAH Concentration ^{1,3} , mg/kg	Difference in CPAH Concentration ² , mg/kg
100, 100-DUP	MA6-SSRR-309, MA6-SSRR-309DP	R899+30	27	31	-4.0	20	17	3
104, 104-DUP	MA6-SSRR-305	R895+50	41	40	1.0	2.4	1.7	0.7
106, 106-DUP	MA6-SSRR-303	R893+55	33	30	3.0	2.0	1.0	1.0
110	MA6-SSRR-299	R889+55	45	43	2.0	2.2	1.0	1.2
114	MA6-SSRR-295	R885+55	48	47	1.0	2.5	1.9	0.6
118	MA6-SSRR-291	R881+55	40	37	3.0	1.6	0.4	1.2
136	MA6-SSRR-273	R862+90	30	29	1.0	0.3	0.2	0.1
144	MA6-SSRR-265	R855+10	32	31	1.0	1.0	0.3	0.7
148, 148-DUP	MA6-SSRR-261	R850+90	34	26	8.0	2.0	0.3	1.7
154, 154-DUP	MA6-SSRR-255	R844+90	33	31	2.0	1.9	0.6	1.3

^{1 -} Results for duplicate samples were averaged.

^{2 -} Deviation as measured from laboratory result.

^{3 -} One-half the detection limit was used to calculate the total CPAH concentration for constituents reported as nondetect, as consistent with U.S. EPA.



METHOD 4035

SOIL SCREENING FOR POLYNUCLEAR AROMATIC HYDROCARBONS BY IMMUNOASSAY

1.0 SCOPE AND APPLICATION

- 1.1 Method 4035 is a procedure for screening soils to determine when total polynuclear aromatic hydrocarbons (PAHs) are present at concentrations above 1 mg/kg. Method 4035 provides an estimate for the concentration of PAHs by comparison with a PAH standard.
- 1.2 Using the test kit from which this method was developed, ≥95% of samples confirmed to have concentrations of PAHs below detection limits will produce a negative result in the 1 ppm test configuration.
- 1.3 The sensitivity of the test is influenced by the binding of the target analyte to the antibodies used in the kit. The commercial PAH kit used for evaluation of this method is most sensitive to the three (i.e., phenanthrene, anthracene, fluorene) and four (i.e., benzo(a)anthracene, chrysene, fluoranthene, pyrene) ring PAH compounds listed in Method 8310, and also recognizes most of the five and six ring compounds listed.
- 1.4 The sensitivity of the test is influenced by the nature of the PAH contamination and any degradation processes operating at a site. Although the action level of the test may vary from site to site, the test should produce internally consistent results at any given site.
- 1.5 In cases where the exact concentration of PAHs are required, quantitative techniques (i.e., Methods 8310, 8270, or 8100) should be used).
- 1.6 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

- 2.1 An accurately weighed sample is first extracted and the extract filtered using a commercially available test kit. The sample extract and an enzyme conjugate reagent are added to immobilized antibody. The enzyme conjugate "competes" with the PAHs present in the sample for binding to the immobilized anti-PAH antibody. The test is interpreted by comparing the response produced by testing a sample to the response produced by testing standard(s) simultaneously.
- 2.2 A portion of all samples in each analytical batch should be confirmed using quantitative techniques.

3.0 INTERFERENCES

- 3.1 Chemically similar compounds and compounds which might be expected to be found in conjunction with PAH contamination were tested to determine the concentration required to produce a positive result. These data are shown in Tables 1 and 2.
- 3.2 The kit was optimized to respond to three and four ring PAHs. The sensitivity of the test to individual PAHs is highly variable. Naphthalene, dibenzo(a,h)anthracene, and

benzo(g,h,i)perylene have 0.5 percent or less than the reactivity of phenanthrene with the enzyme conjugate.

3.3 The alkyl-substituted PAHs, chlorinated aromatic compounds, and other aromatic hydrocarbons, such as dibenzofuran, have been demonstrated to be cross-reactive with the immobilized anti-PAH antibody. The presence of these compounds in the sample may contribute to false positives.

4.0 APPARATUS AND MATERIALS

PAH RIScTM Soil Test (EnSys, Inc.), or equivalent. Each commercially available test kit will supply or specify the apparatus and materials necessary for successful completion of the test.

5.0 REAGENTS

Each commercially available test kit will supply or specify the reagents necessary for successful completion of the test.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.
- 6.2 Soil samples may be contaminated, and should therefore be considered hazardous and handled accordingly.

7.0 PROCEDURE

- 7.1 Method 4035 is intended for field or laboratory use.
- 7.2 Follow the manufacturer's instructions for the test being used. Those test kits used must meet or exceed the performance indicated in Tables 3-7.
 - 7.3 The action limit for each application must be within the operating range of the kit used.

8.0 QUALITY CONTROL

- 8.1 Follow the manufacturer's instructions for the test kit being used for quality control procedures specific to the test kit used. Additionally, guidance provided in Chapter One should be followed.
- 8.2 Use of replicate analyses, particularly when results indicate concentrations near the action level, is recommended to refine information gathered with the kit.
 - 8.3 Do not use test kits past their expiration date.
 - 8.4 Do not use tubes or reagents designated for use with other kits.

8.5 Use the test kits within the specified storage temperature and operating temperature limits.

9.0 METHOD PERFORMANCE

- 9.1 The extraction efficiency of a commercially available test kit was tested (PAH RIScTM Test, EnSys Inc.) by spiking phenanthrene, benzo(a)anthracene and benzo(a)pyrene into PAH negative soil matrices (PAH-116 and PAH-141 are field samples). The soils were spiked using detection limits established for each compound (see Table 1), extracted and determined by immunoassay. The results for these 3-, 4- and 5-ring PAHs (Table 4) demonstrated that they were extracted with good recovery and yielded the correct assay interpretation.
- 9.2 A single laboratory study was conducted with a commercially available test kit (PAH RISc[™] Test, EnSys Inc.), using 25 contaminated soil samples. Four replicate determinations were made on each test sample and the data compared with values obtained using HPLC Method 8310. Several analysts performed the immunoassay analyses. The immunoassay data agreed in all cases with the external HPLC data obtained (Table 5).
- 9.3 An additional single laboratory validation study on 30 randomly selected, PAH-contaminated field samples from multiple sites was run by the USEPA Region X Laboratory. Results are reported in Table 6 on an as found basis, and reported in Table 7 normalized to phenanthrene, based on cross-reactivity data (from Table 1). The false positive rate at the 1 ppm action level was 13% for unnormalized results and 19% for normalized results based on 31 analyses. The false negative rate at 1 ppm was 0 in both cases. At the 10 ppm action level, the false positive rate was 19% unnormalized and 26% normalized. False negative rates at 10 ppm were 6% unnormalized and 3% normalized.
- 9.4 The probabilities of generating false positive and false negative results at an action level of 1 ppm are listed in Table 3.

10.0 REFERENCES

- 1. PAH-RISc[™] Users Guide, EnSys Inc.
- 2. P. P. McDonald, R. E. Almond, J. P. Mapes, and S. B. Friedman, "PAH-RISc™ Soil Test A Rapid, On-Site Screening Test for Polynuclear Aromatic Hydrocarbons in Soil", J. of AOAC International (accepted for publication document #92263)
- 3. R. P. Swift, J. R. Leavell, and C. W. Brandenburg, "Evaluation of the EnSys PAH-RISc[™] Test Kit", Proceedings, USEPA Ninth Annual Waste Testing and Quality Assurance Symposium, 1993.

TABLE 1
Cross-reactivity of Method 8310 PAHs

Compound	Concentration Giving a Positive Result (ppm Soil Equivalent)	Percent Cross-Reactivity
2 <u>Rings</u> Naphthalene	200	0.5
3 Rings Acenaphthene Acenaphthylene Phenanthrene Anthracene Fluorene	8.1 7.5 1.0 0.81 1.5	12 13 100 123 67
4 Rings Benzo(a)anthracene Chrysene Fluoranthene Pyrene	1.6 1.2 1.4 3.5	64 84 73 29
5 Rings Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Dibenzo(a,h)anthracene	4.6 9.4 8.3 >200	22 11 12 <0.5
6 Rings Indeno(1,2,3-c,d)pyrene Benzo(g,h,i)perylene	11 >200	9.4 <0.5

TABLE 2

Cross Reactivity of Other PAHs and Related Compounds

Compound	Concentration Giving a Positive Result (ppm, Soil Equivalent)	Percent Cross-Reactivity
Other PAHs		
1-Methylnaphthylene	54	1.8
2-Methylnaphthylene	58	1.7
1-Chloronaphthylene	59	1.7
Haiowax 1013	18	5.7
Haiowax 1051	>200	<0.5
Dibenzofuran	14	7.2
Other Compounds		
Benzene	>200	<0.5
Toluene	>200	<0.5
CCA	>200	<0.5
Phenoi	>200	<0.5
Creosote	5.4	18.5
2,4,6-Trichlorobenzene	>200	<0.5
2,3,5,6-Tetrachlorobenzene	>200	<0.5
Pentachlorobenzene	>200	<0.5
Pentachiorophenoi	>200	<0.5
Bis(2-ethylhexyl) phthalate	>200	<0.5
Aroclor 1254	>200	<0.5
Aroclor 1260	>200	<0.5

TABLE 3

Probability of False Negative and False Positive Results for PAHs at A 1 ppm Action Level

Spike Concentration Phenanthrene (ppm)	Probability of False Positive (Mean ± SD)	Probability of False Negative (Mean ± SD)
0	0% ± 0%	N/A
0.4	23% ± 17%	N/A
0.8	94% ± 13%	N/A
1.0	N/A	0% ± 0%

Results were obtained from spiking four different validation lots, using 3 operators, 12 matrices for a total of 201 determinations at each concentration of phenanthrene.

N/A = No false positive or negative possible above action limit.

TABLE 4

Spike Recovery of Phenanthrene, Benzo(a)anthracene and Benzo(a)pyrene

Compound	Spike (ppm)	Soil	PAH RISc™ Results
Blank	0	Wake	<1
Blank	0	PAH-116	<1
Phenanthrene	1	Wake	1-10
Phenanthrene	1	PAH-116	1-10
Phenanthrene	1	PAH-141	1-10
Phenanthrene	10	Wake	>10
Phenanthrene	10	PAH-116	>10
Phenanthrene	10	PAH-141	>10
Benzo(a)anthracene	1.6	Wake	1-10
Benzo(a)anthracene	1.6	PAH-116	1-10
Benzo(a)anthracene	16	Wake	>10
Benzo(a)anthracene	16	PAH-116	>10
Benzo(a)pyrene	8.3	Wake	1-10
Benzo(a)pyrene	8.3	PAH-116	1-10
Benzo(a)pyrene	83	PAH-116	>10

TABLE 5
Powerplant Field Samples (Soil) Evaluated by Immunoassay

Field Sample Number	EnSys Method Immunoassay (ppm)	Method 8310 HPLC (ppm)
PAH-137	>10	<21
PAH-141	<1	<21
PAH-118	1-10	<26
PAH-136	>10	26
PAH-139	>10	<28
PAH-126	1-10, >10	<32
PAH-127	>10	<33
PAH-122	>10	<33
PAH-138	>10	33
PAH-131	>10	<34
PAH-128	>10	<35
PAH-132	>10	<43
PAH-112	>10	<48
PAH-140	>10	50
PAH-130	>10	54
PAH-116	<1	<61
PAH-135	>10	71
PAH-133	>10	<91
PAH-119	>10	<100
PAH-120	>10	<161
PAH-124	>10	<167
PAH-134	>10	182
PAH-114	>10	<247
PAH-113	>10	<294
PAH-115	>10	<343

TABLE 6

Total PAH Content of Region X Field Samples Using EnSys
PAH RISc™ Immunoassay Test Kit

	1 ppm Test		10 ppi	n Test	GC/MS	False +/-		
Sample ID	<1	>1	<10	>10	Lab Result (ppm ⁾¹	Eval @ 1 ppm	Eval @ 10 ppm	
PAH-1		•		•	0.2	+	+	
PAH-2		<u> </u>		•	12.2			
PAH-3				•	16.0			
PAH-4	•				0.00		<u></u>	
PAH-5					0.5		<u> </u>	
PAH-6		•		•	8.7		+	
<u>PAH-7</u>					148			
PAH-8				•	182			
PAH-9		•		•	4.4		+	
PAH-10		•		•	0.2	+	+	
PAH-11	•				0.00			
PAH-12				•	85.4			
PAH- 12Dup				•	85.4			
PAH-13				. •	28.5			
PAH-14	•		•		0.3			
PAH-15		•			0.6	+		
PAH-16	•		•		0.00			
PAH-17		•		9	1.8		+	
PAH-18		•	•		3.4			
PAH-19		•			6.7			
PAH-20	•		•		0.9			
PAH-21				•	43.2			

¹ Sum of all PAHs detected.

	1 ppm Test		10 ppm Test		GC/MS	Faise +/-	
Sample ID	<1	>1	<10	>10	Lab Result (ppm)¹	Eval @ 1 ppm	Eval @ 10 ppm
PAH-22				•	72.8		
PAH-23		•		•	1.3		+
PAH-24		•	•		0.3	+	
PAH-25	•		•		0.4		
PAH-26				ı	27.9	-	
PAH-27					0.00		
PAH-28			•		16.4		-
PAH-29	•		•		0.4		
PAH-30		•	•		9.6		

TABLE 7

Total PAH Content of Region X Field Samples Using EnSys
PAH RIScTM Immunoassay Test Kit Normalized to Cross-reactivity

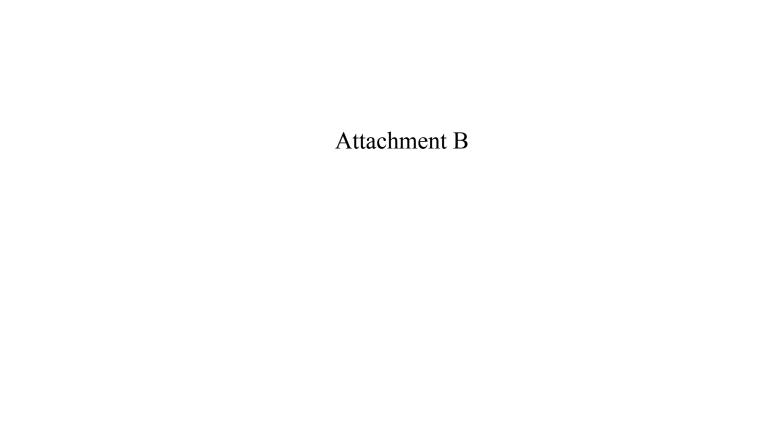
	1 ppm Test		10 ppm Test		GC/MS	False +/-	
Sample ID	<1	>1,	<10	>10	Lab Result (ppm) ¹	Eval @ 1 ppm	Eval @ 10 ppm
PAH-1	·	•		•	0.1	+	+
PAH-2				•	8.1		+
PAH-3				•	9.0		+
PAH-4	•				0.00		
PAH-5	•				0.2		
PAH-6		•		•	5.2		+
PAH-7				•	56.9		
PAH-8		<u> </u>		•	73.2		

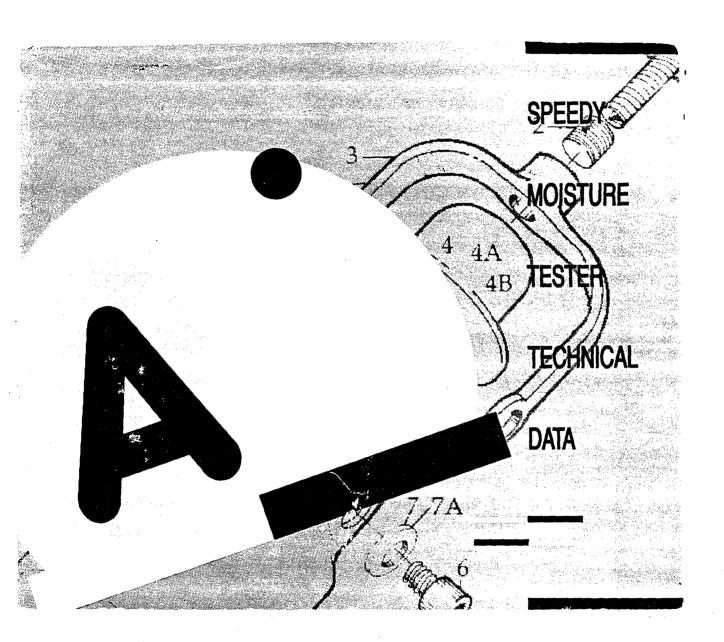
¹ Sum of all PAHs detected.

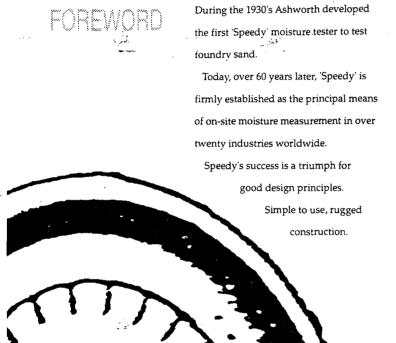
TABLE 7 (cont.)

	1 ppm Test		10 pp	m Test	GC/MS	False +/-	
Sample ID	<1	>1	<10	>10	Lab Result (ppm) ¹	Eval @ 1 ppm	Eval @ 10 ppm
PAH-9		•		•	0.1	+	+
PAH-10		•		•	0.00	+	+
PAH-11	•			<u> </u>	0.00		
PAH-12				•	47.3		
PAH-12Dup	·			•	47.3		
PAH-13				•	11.5		
PAH-14	•		•		0.2		
PAH-15		•			0.5	+	
PAH-16	•		*		0.00		
PAH-17		•		•	1.2		+
PAH-18		•	•		1.7		
PAH-19		•	*		3.6		
PAH-20	•		•		0.6		
PAH-21				•	27.5		
PAH-22				*	49.2		
PAH-23		•		•	0.8	+	+
PAH-24		•			0.1	+	
PAH-25	•		*		0.2		
PAH-26			•		13.5		
PAH-27	*		•		0.00		
PAH-28			•		6.4		
PAH-29	•		•		0.2		
PAH-30		•	•		2.8		

¹ Sum of all PAHs detected.







consistently accurate results - all of these factors combine to make 'Speedy' the only choice for many industries.

'Speedy' will provide many years of trouble-free service, this booklet will explain how to get the best from the range of 'Speedy' moisture testers.

For those industries requiring electronic based moisture

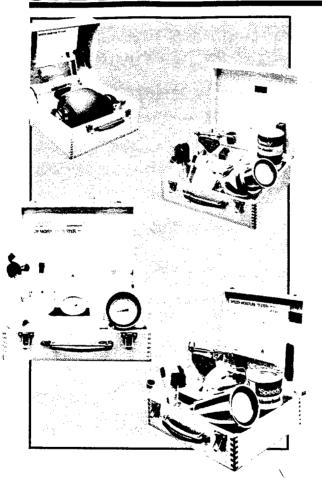
measurement, such as food and pharmaceuticals, Ashworth
Instrumentation's parent company produce a complete range of sophisticated, but user friendly, electronic moisture meters.

For more detailed information on electronic products, contact our sales office on 01282 426554.

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SPECIFICATION



3

Super 'Speedy'

For testing aggregates, wet concrete and lean mixes, and similar materials, where a large sample size is required (up to 40mm particle size).

Types	Moisture	Gauge	Sample
	Range	Divisions	Weight
Super 2	0-11% (WET WEIGHT) 1-12% (DRY WEIGHT)	0.1%	142.86

Large 'Speedy'

For testing sands, soils, clays, fine and coarse aggregate, coal, mineral ores and concentrates, sludge and many other materials of a similar consistency (up to 20mm particle size).

Types	Moisture Range	Gauge Divisions	Sample Weight
Large C.2	0-10%	0.1%	40 grm
Large D.2	0-20%	0.2%	20 grm
Large G.2	0-50%	0.5%	8 grm

Oil 'Speedy'

For testing marine lubricating and fuel oils, waste oil, mineral and vegetable oils and most other liquids

Types	Moisture Range	Gauge Divisions	Sample
Oil A.2	0-2%	0.05%	10ml
Oil B.2	0-5%	0.1%	10ml
Oil C.2	0-10%	0.1%	12ml
Oil D.2	0-20%	0.2%	6mi

Standard 'Speedy'

For testing sand, clays, powders, chemicals, brickwork, plaster and mortar, grain and food stuffs and most material with a granular consistency (up to 10mm particle size).

TOMETH PURITUR	conce,		
Types	Moisture	Gauge	Sample
	Range	Divisions	Weight
Standard A.2	0-2%	0.05%	10 grm
Standard B.2	0-5%	0.1%	10 grm
Standard C.2	0-10%	0.1%	12 grm
Standard D.2	0-20%	0.2%	6 grm
Standard G.2	0-50%	0.5%	2.4 grm

The 'SPEEDY' Test Procedure

The Speedy moisture test gives consistently accurate results in approximately 3 minutes. To carry our your own moisture testing just follow the illustrated instructions below and you will be surprised how easily Speedy works.



1. Clean the 'SPEEDY'

Make sure that the insides of Speedy body and cap are clean and free from residues of any previous test.



Prepare the material for test as follows: Sands and fine powders - No preparation

Sands and fine powders - No preparation necessary.

Coals, coke, ones and mineral concentrates Grind or pulverise before weighing or use steel ball method (see below).

Clays, soils and other coarse materials -

Use steel ball method as shown below. Use steet ball method as shown below.

Aggregates - No preparation necessary.

Lubricating oils, other oils and liquids - Using the oil Speedy take a volume sample with calibrated syringe provided and mix with five scoops of dry silica sand when placing sample in tester.

(instruction No.4).

Any other material - Ask our advice



4. Put material in

Place the sample of material in the body of the Speedy



5. Add 'SPEEDY' absorbent/reagent

Take two full measures of Speedy absorbent/reagent and place in cap. For bulky materials use 3-5 to ensure adequate coverage.

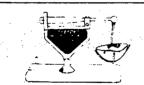


7. Mix

8. Take reading

Once more shake the Speedy and with Speedy horizontal (with gauge dial facing you at eye level) take the reading

With dial facing (illustration 'A') shake the



3. Weigh the material

Lift the balance into upright position. Weigh material, correct weight is shown when red markings on balance and beam coincide.



6. Seal the 'SPEEDY'

Hold the Speedy horizontal to prevent mixing of Speedy sample and absorbent/reagent before the instrument is sealed. Place cap in position. Bring stirrup round and tighten top screw.



9. Finally

Skowly release the pressure holding Speedy away from body contact, with the directional release arrow pointing away from the operator and empty contents. Clean out Speedy thoroughly with brush provided.

SPEEDY DATA

Five simple rules before Testing Make sure that:-

- The Body or Cap, which ever is being used for the material, is perfectly clean and contains no active absorbent from a previous test.
- The material is truly representative of the bulk and carefully weighed.
- 3 The material and the Absorbent are kept separate until the cap is tightly secured to the body.
- 4 The material has been thoroughly prepared ground or pulverised or mixed with sand (if necessary) so that the absorbent can act freely on the material.
- 5 Make sure the **Steel Ball Pulverisers** are used when testing Clays, Soils, etc. (See "Speedy" Steel Ball Pulverisers).

You will note that importance is attached to the necessity for accurate weighing, yet a slight error will not affect the result so seriously as it would in the Standard Oven Test. For example: If you were testing 10 grammes of material by the Oven Method, and weighed it two-tenths of a gramme too little, that shortage would be equivalent to 2% moisture content, but, using the "Speedy" method a shortage of two-tenths of a gramme, due to careless weighing, is only equivalent in error to the moisture content actually contained in those two-tenths of a gramme. With the "Speedy," testing 10 grammes of material with the gauge indicating 5% moisture, an error in

weighing of two-tenths of a gramme would be one-fiftieth of 5%:equivalent to 0.1% – one twentieth of the error you would have made in the Oven Test.

Preparing the Material for Testing "Speedy" Steel Ball Pulverisers
For use in the "Speedy" when testing clays, soils and some coarse materials. Put two 1½" dia. steel balls ("Speedy" Pulverisers) into the body of the "Speedy" with the Absorbent. In place of the normal mixing and shaking instructions, mix as follows:—Hold "Speedy" vertically, so that the material to be tested, which is in the cap falls into the "Speedy" body. Then holding "Speedy" horizontally, rotate it approximately 10 seconds so that the pulverisers are "put into orbit" round the inside circumference. Rest for approximately 20 seconds. Repeat the rotate-rest cycle until the result is constant (usually within 3 minutes).

Correction Factor

With the present range of thirteen models we can fulfil the requirements of any industry. It is necessary to emphasise that "Speedy" will give consistent results on any material and can easily be calibrated against any other method of moisture determination. Many materials contain other volatile matter in addition to water so that "Speedy" results may be lower than oven drying figures. If the difference is carefully noted in a series of comparative tests the correction factor required can easily be established

SPEEDY DATA

Points to keep in mind when Testing The chemical action of the "Speedy" Absorbent produces gas. The amount of gas depends on the amount of water in the material.

To prevent loss of the gas, material and the absorbent must not come into contact with one another until the Tester has been sealed by tightening the top screw on the cap. (It is important that this should be done with the "Speedy" horizontal as "Seal the Speedy" shown on Directions for Use Sheets inside of the lid of the "Speedy" case.

Proportional Method

High Moisture Contents

To obtain a higher reading than indicated on the gauge, we have supplied a small brass weight (half the Standard Weight). Hook this brass weight through the link holding the scale pan cradle, or hang on edge of scale pan, and weigh in the normal manner. Proceed to test the material (which is now only half the Standard Weight) in the usual way.

BUT

When reading the gauge, DOUBLE the percentage indicated, e.g. if the gauge reads 18% the moisture content is 18% multiplied by 2 equalling 36%.

A smaller proportion can be tested if your material is either very wet or of high value.

Low Moisture Contents

Two or more complete weighings can be tested at once, and the result divided by the

number of weighings, e.g. 2 weighings: result on gauge 1.8% moisture content equals 1.8 divided by 2 equalling 0.9%. N.B. The Proportional method cannot be used with the Super 2 (0.12%) because of its special calibration.

Temperature

"Speedy" should be used in a room temperature approximately 70 deg. F (20 deg. C). If used below 60 deg. F. ignore the first and possibly second tests. If the instrument is hot from previous testing, careful brushing out will allow it to cool.

Warm Materials

Sometimes materials have to be tested when warm – as from Drying Plants. These can be tested immediately while still warm, or placed in a sealed container for testing when convenient.

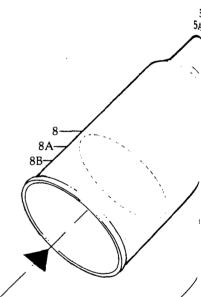
Liquids and Pastes

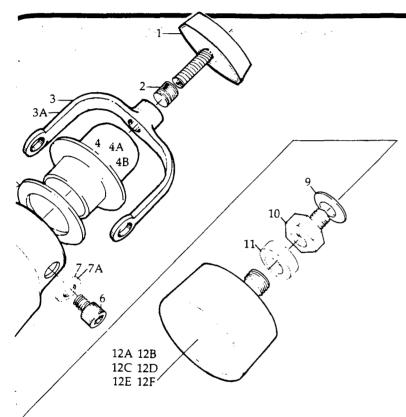
- 1 First measure the sample to be tested.
- 2 Have DRY sand available.
- 3 Add the dry sand to the liquid or paste in the scale pan and mix thoroughly so that the sand "absorbs" the liquid or the paste.
- paste.
 4 Quantity of sand used within reason does not affect the result. Usually three to four absorbent scoops of sand are sufficient, but use a little more if necessary.
- 5 The sand must be quite dry. Test it in the "Speedy" if there is any doubt.

Clays, Soils and Some Coarse Materials Use the "Speedy" Pulveriser Method

PARTS LIST: BODY

	ption	Description		Drwg No.
		Top Screw		1
		Stirrup	S03	3
		Stirrup for Super 2	S04	3A
		Standard Cap	S05	4
		Cap for Super 2	S07	4B
		Rubber Washer	S08	5
	per 2	Rubber Washer for Su	S09	5A
		Side Screw	S10	6
		Nylon Washer	S11	7
	er 2	Nylon Washer for Sup	S12	7A
	lo Gauge or	Standard Body only (N Stirrup)	S13	8
	Gauge or Stirrup)	Large Body Only (No	S14	8A
	No Gauge or	Body only for Super (N Stirrup)	S15	8B
		Adaptor Washer	S16	9
	г	Adaptor Nut and Filte	S17	10
		Gauge Washer	S18	11
		Gauge Type "A" 2	519	12A
	0-2%	Gauge Type "B" 2	S20	12B
	0-5%	Gauge Type "C" 2	S21	12C
	0-10%	Gauge Type D.2	. S22	12D
	0-20% 0-12% and	Gauge Type Super 2	S23	12E
(1	0-11%	Gauge Type "G"2	S25	12F
//		Complete Standard Bo	S26	_
//	•	Complete Large Body		
		Complete Super Body	S27 S28	
	top screw)	Complete Stirrup (incl. top screw)		
Complete Stirrup for Super (incl. top screw)		S29 S30		





Construction of the "Speedy"
DIE-CAST ALUMINIUM, making it light in weight, very sturdy, and easy to handle.
THE BALANCE is purposely made for handling by unskilled persons. It is very robust yet extremely sensitive, and is carefully checked for accuracy before leaving our works.

A SPECIAL NUMBER is allotted to each instrument on our records. This number is stamped on the Body of the Tester, on the Balance Stand, Balance Beam and Balance Pan.

GAUGE Bourdon Type with Beryllium Copper Tube, for robust and accurate operation. This is specially made to "Test Gauge" accuracy. Beryllium copper is used to maintain extreme accuracy, even under adverse conditions.

We should be failing in our duty if we implied that this instrument never goes wrong. It can go wrong, just as an excellent motor car can fail, through damage due to careless handling or "collisions" from outside.

In Britain

Please return the "Speedy" to us, and we will put it right and return it to your client very quickly. Please pack the instrument in a stout case or strong carton to avoid damage in transit.

Overseas

Owing to the time taken up in transit to and from England, we advise customers to replace damaged parts from a small stock of spares that can be supplied by our agents on request.

PARTS LIST: CASE & BALANCE

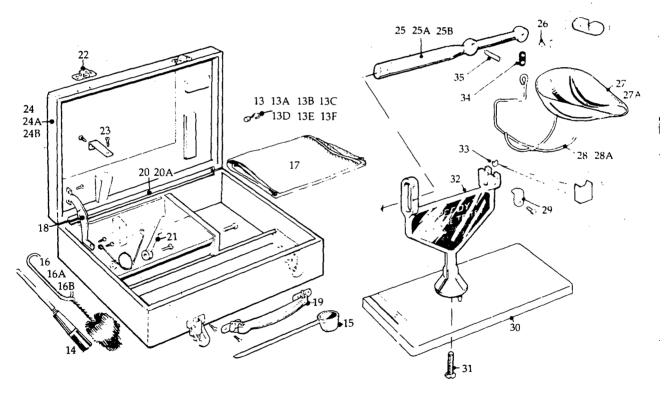
Case

Balance

Drwg No.	Part No.	Description	Drwg No.	Part No.	Description
13	S58	Brass Weight 1.2g	25	531	Standard Balance Beam®
13A	S57	Brass Weight 3 g.	25A	S31	Large Balance Beam
13B	S55	Brass Weight 5 g.	25B	S33	Balance Beam for Super●
13C	S56	Brass Weight 6g	26	S34	Knife Edge Square
13D	S59	Brass Weight 10g	27	S35	Scale Pan®
13É	560	Brass Weight 20g	27A	S36	Scale Pan for Super ●
13F	S62	Brass Weight 71.439	28	S37	Cradle•
14	S63	Scale Pan Brush	28A	538	Cradle for Super●
15	S64	Absorbent Scoop	29	539	Side Plate with screw
16	S67	Brush for Super	30	S40	Wood Platform
17	S68	Cleaning Cloth	31	541	Platform Screw
18	S69	Lid Stay*	32	S42	Scale Stand (Complete with
19	S70	Leather Handle*			agates and side plates)
20	S71	Hinge*	3 3	S43	Agate
20A	S72	Hinge for Super Case	34	S44	Scale Link
21	S73	Scale Platform Stay	35	S45	Pear Shaped Knife Edge
		with Spring*		546	Complete Balance Standard
22	S74	Locking Catch*			(on wood platform)
23 24	S75	Platform Support Plate Standard Case	· -	S47	Complete Balance Large (or wood platform)
24A	S76 S77		-	S48	Complete Balance for Supe
24B	577 S78	Large Case			(on wooden platform)
240		Case for Super Std Brush		S49	Complete Beam Assembly
	S65				(including cradle and pan).
	S66	Lg Brush		CF.	Please specify type
	S79	Case for Oil		S51	Complete Beam Assembly for Super
All these part	s are suppli	ed complete with screw	S.	S50	Complete Beam Assy. (Lg.
				S52	Pulverisers (pair)
				553	Gauge Removal Tool
				S54	Calibrated Syringe (for oil tester)

These parts must be individually calibrated to suit the complete balance. Details on request.

CASE & BALANCE



FAULT FINDING If the Gauge Reading is in doubt

A "Negative" Reading
If the "needle" does not move on the gauge, then proceed as follows:—

- 1 Make sure that the Absorbent has been put into the tester.
- 2 Make sure there is moisture in the material.
- 3 Try another Test with three times the Standard Weight of material.
- 4 If all the above fail, obtain a sample of the material, together with full details and send it to us.

A "Low" Reading
If your result is below yo

If your result is below your sure knowledge of your normal percentages please consider the following possibilities:—

- Excessive shaking on materials with moisture contained in cellular structures.
- 2 Material not correctly weighed and lighter than the Standard Weight.
- 3 "Speedy" too cold, will "Warm Up" with further tests.
- 4 Absorbent and material have been in contact before sealing the instrument, thereby allowing the gas to leak away and not work on the gauge. (See "Seal the Speedy" on Directions for Use.)
- 5 Rubber ring may be worn, or cap is not securely closed.
- 6 Material not correctly prepared or "Speedy" Absorbent not mixing fully with the material. Pour out on to a sheet

- of paper, and examine. If required use "Speedy" Pulverisers (Steel Ball method).
- 7 The "Speedy" Absorbent may be only semi-active, due to long storage or exposure to the atmosphere. This sometimes happens through failing to replace the lid on the tin after each test. Check for lighter colour of Absorbent at the top of the tin, and dispose of this non-active Absorbent. Try using 2 or 3 scoops of Absorbent.
- 8 Not cleaning the cap or body after each test. The material would commence working with any active Absorbent not removed after a previous test.
- 9 If using the "Proportional Method" see page 6, forgetting to multiply the reading on the gauge.
- 10 SPECIAL CASE: Volatile Matter in the material is generally recognised by the odour when testing in the laboratory oven. This is indicated as moisture by the Standard Oven Method. The "Speedy" Moisture Tester only reacts with water and if compared with the Oven Test will read lower if Volatile Matter is present. You can establish a correction factor after a series of tests as mentioned on page 5 (Correction Factor).
- 11 Very bulky material may require more than 1 scoop of Absorbent to give a full coverage.

FAULT FINDING

A "High" Reading

If your test shows a higher reading than your sure knowledge of your percentages, please consider the following possibilities:—

- 1 Material not accurately weighed.
- 2 Reading the gauge when this is not in the horizontal position. (See "Read the Dial" on the Directions for Use Sheet.)
- 3 Frequent and repeated tests without allowing the instrument to cool between tests. Careful brushing out will allow the instrument to cool sufficiently.
- 4 Does the "needle" return to zero after releasing the gas? If not the instrument must be re-calibrated.
- 5 If using the "Proportional Method" (see page 6) forgetting to divide the reading on the gauge.
- 6 The "Speedy" has not been shaken long enough to cool the gases before reading the results.

A "Very Slow" Reading

This sometimes happens with "difficult" materials. The material itself may give a naturally slow chemical reaction, and the "needle" rises only gradually. Proceed thus:

- Grind or pulverise the material more finely, or otherwise improve the method of preparation.
- Use "Speedy" Pulverisers (Steel Ball Method).
- 3 If still unsatisfactory, obtain a sample of the material, together with full details, and send it to us.

Replaceable parts that may be required Gauges

It is recommended that customers overseas purchasing twenty or more "Speedy" Moisture Testers of one type, should buy one spare gauge of that type. Normally, replacements will only be required through letting the "Speedy" fall.

This is the most sensitive part of the "Speedy". Dropping the "Speedy", may result in the "needle" not returning to zero when the pressure is released. Replace with a new gauge, using our special gauge-removing tool. If you replace a gauge with another type of gauge, be sure that the balance is adjusted for weight.

After a few years' time, if you have replaced several gauges, the damaged ones can be returned for repair.

Rubber Washers

As a spare is supplied with each "Speedy" Moisture Tester outfit, replacements should not be necessary for years.

Cleaning Brush

Replacements not usually necessary for several years.

Balance

No replacements normally required. Repairs are rarely needed. On one or two occasions, we have been asked to replace broken Agates which were damaged through negligence.

CONVERSION CHART

_Conversion Chart ____

Civil Engineering and certain other trades sometimes require Moisture Content expressed as a percentage of Dry weight. Use this chart for conversion.

SPEEDY READING	SPEEDY READING	SPEEDY READING
READING W.W. D.W. 1.0% 2.1% 2.0% 2.1% 3.0% 3.2% 4.0% 4.3% 5.0% 5.4% 6.0% 6.5% 7.0% 7.6% 8.0% 8.7% 9.0% 11.0% 10.0% 11.7% 11.0% 12.3% 11.0% 12.3% 11.5% 13.0% 12.5% 14.2% 13.0% 14.2% 13.0% 14.2% 13.0% 14.5% 15.6% 19.0% 15.5% 19.0% 16.0% 19.0% 16.5% 19.7% 17.0% 20.4% 17.5% 21.2%	## SPEEDY READING W. W.	W.W. D.W. 35.5°a 55.0°a 36.0°a 56.2°a 36.5°a 57.4°a 37.0°a 58.7°a 38.0°a 61.2°a 38.0°a 61.2°a 38.5°a 62.6°a 39.0°a 63.4°a 39.5°a 65.2°a 40.5°a 66.6°a 41.0°a 64.4°a 41.5°a 70.9°a 42.0°a 72.4°a 43.0°a 75.4°a 43.5°a 76.9°a 44.0°a 78.5°a 44.5°a 80.1°a 45.0°a 81.8°a 45.5°a 83.4°a
18.0% 21.9% 18.5% 22.7% 19.0% 23.4% 19.5% 24.2% 20.0% 25.0%	33.0% 49.2% 33.5% 50.3% 34.0% 51.5% 34.5% 52.6% 35.0% 53.8%	48.0% 92.3% 48.5% 94.1% 49.0% 96.0% 49.5% 98.0% 50.0% 100.0%

W.W.=Wet Weight D.W.=Dry Weight

Safe disposal of the contents of your Speedy after testing.

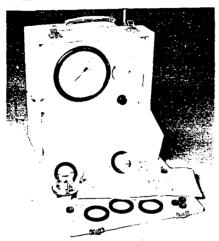
The reaction which occurs in your 'Speedy' tester, during the test procedure produces acetylene gas (the pressure of which provides the reading) and a mild alkali calcium hydroxide (hydrated or slaked lime).

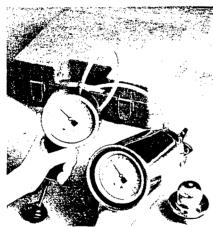
After testing, empty the contents of the tester into a disposable container or bag and when convenient empty the container onto open ground.

Spread the remnants thinly and allow any unreacted carbide to decompose (oxidise) on exposure to the air. This decomposition can be accelerated by the addition of water sprayed onto the powder, provided this is done well away from buildings, or inflammable substances.

Please do not empty the contents into a waste bin.

CALIBRATION KIT





SPEEDY PRESSURE TESTER

Under quality standards, such as BS5750, all quality control test equipment should be regularly checked and calibrated. Two calibrated devices are available to check the correct operation and accuracy of the Speedy Moisture Testers.

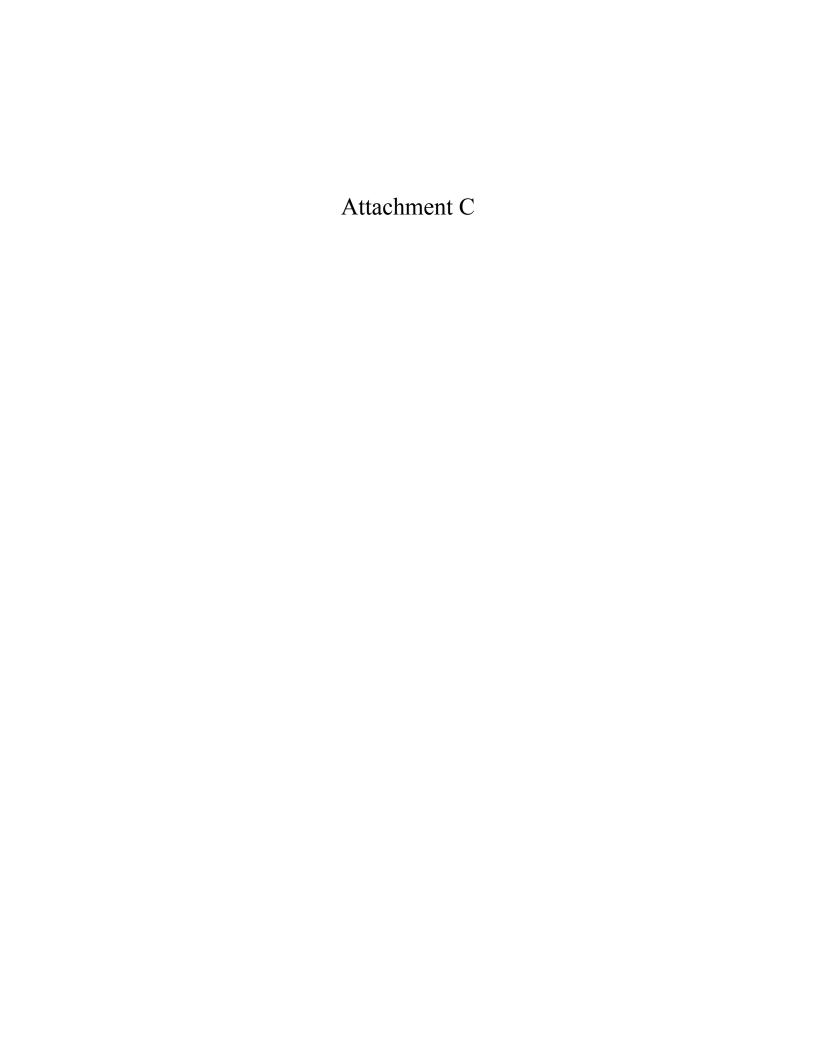
CALIBRATION KIT

This kit will be of particular interest to overseas agents. It enables individual pressure gauges and complete testers to be checked against a 6" diameter Master Gauge.

SPEEDY PRESSURE TESTER

The "Speedy Pressure Tester" enables the Speedy to be quickly pressured to check for accuracy and correct operation. This is particularly useful for on-site checks.

CALIBRATION KIT



Attachment D

& Industrial Testing



RaPID Assay®

Carcinogenic PAHs RaPID Assay

Features

- rapid field testing procedure for analysis of soil and water samples
- quantitative data results with excellent analytical precision
- test up to 50 samples at one time
- results in approximately 60 minutes

training recommended

magnetic particle immunoassay

EPA SW-846 Method # 4035 with increased sensitivity to Carcinogenic PAHs



Test Result Type

Quantitative, semi-quantitative or qualitative data.

Samples per Kit

- ° Two kit sizes available:
 - 30 Test Kit (tests up to 20 + samples)
 - 100 Test Kit (tests up to 80 + samples)

Assay Range

- Soil:10 to 500 ppbas Benzo[a]pyrene
- Water:
 0.2 to 10.0 ppb
 as Benzo[a]pyrene
- Range can be extended via additional dilutions
- Site specific calibration recommended to meet total PAH / C-PAH analysis requirements

Sample Preparation

- Soil samples
 require prior extraction
 using the SDI Sample
 Extraction Kit (sold separately)
- The Sample Extraction Kit provides materials for 12 soil sample extractions with methanol
 Water samples must be diluted 3 parts sample to 1 part water stabilities.
 - lizer to prevent adsorptive loss

Sampling Time

Soil extraction time is typically 2 minutes per sample plus assay run time of approximately 60 minutes







Specificity

The total Carcinogenic PAH concentrations (sum of seven Carcinogenic PAH compounds) of the indicated contaminant types in soil and water samples are expressed below, at each of the three kit calibrator (standard) levels, in units comparable to results from GC Method 8270 or HPLC Method 8310.

Carcinogenic PAHs RaPID Assay Total Carcinogenic PAHs in Soil (ppb)

	-			
Contaminant		Standard 2	Standard 3	
Creosote	0.5	10	100	
Coal Tar Oil	0.5	10	100	
Diesel Fuel	0.5	5	35	
Fuel Oil #4, 6	1.0	7.5	35	
Fuel Oil #5	0.5	5	25	

Carcinogenic PAHs RaPID Assay
Total Carcinogenic PAHs in Water (ppb)

		-· (F#+)_	
Standard 1	Standard 2	Standard 3	
0.01	0.2	2.0	
0.01	0.2	2.0	
0.01	0.1	0.7	
0.02	0.15	0.7	
0.01	0.1	0.5	
	Standard 1 0.01 0.01 0.01 0.01	Standard 1 Standard 2 0.01 0.2 0.01 0.2 0.01 0.1 0.1 0.02 0.15	0.01 0.2 2.0 0.01 0.1 0.7 0.02 0.15 0.7

The Carcinogenic PAHs RaPID Assay immunoassay test does not differentiate between Benzo[a]pyrene and other polyaromatic hydrocarbons. The table below shows compounds at the limit of quantitation (LOQ) - an approximate concentration required to yield a positive result at the lowest standard. The IC50 is the concentration required to inhibit one-half of the color produced by the negative control. It is also used to calculate cross-reactivity values to similar compounds.

Carcinogenic PAHs in Soil (ppb)			
Contaminant	LOQ	TC50	
Benzo(a)pyrene	10	160	
- Benzo[a]anthracene	3.0	48	
- Benzo[k]fluoranthene	3.9	63	
~Chrysene	4.3	69	
- Benzo[b]fluoranthene	8.1	130	
Indeno[1,2,3-c,d]pyrene	12.7	203	
Dibenzo[a,h]anthracene	15	241	
Anthracene	128	2050	
Phenanthrene	420	6720	
Fluoranthene	428	6850	
- Benzo[g,h,i]perylene	>625	>10,000	
Pyrene	1456	23,300	
Fluorene	2138	34,200	
Naphthalene	31250	500,000	
Acenaphthylene	36063	577,000	
Acenaphthene	>62500	>1,000,000	

.Test Kit Components

- Antibody coated magnetic particles, enzyme conjugate, color development, stop and wash solutions for analysis of either 100 or 30 test tubes
- Standards for 0.1, 1.0 and 5.0 ppb as Benzo(a)pyrene
- Kit control of 2.0 ppb as Benzo[a]pyrene
 - Disposable test tubes
- Test kit instructions

Storage & Precautions

- Shelf life is typically one year from date of manufacture, with specific kit expiration date information provided on product packaging.
- Reagents must be stored at 39° to 46°F (4° to 8°C) when not in use.
- * Kits must be brought to 64° to 81°F (18° to 27°C) before use.
- Do not expose color solution to direct sunlight.
- Soil sample must have <30% moisture content.

Required Test Materials CPAHs RaPID Assay: 100 test kit CPAHs RaPID Assay: 30 test kit SDI Sample Extraction Kit A00200 MO0204EA or (for soil only) Universal Range Extension Kit (as needed to extend range of detection)

A00203

Required Test Equipment

cPAHs Sample Diluent (100 mL)

(as needed to extend range of detection)

•	RaPID Assay Accessory Kit	605010 purchase
	which contains:	699701 rental
	RPA-I RaPID Analyzer	A00003
	Magnetic Separation Rack	A00004
	Repeator Pipet	A00008
	Adjustable Volume Pipet	A00176
	Vortex Mixer	A00014
	Portable Balance	A00131
	Digital Timer	A00015
•	Repeator Pipet Tips	A00009
•	Adjustable Pipet Tips	A00013

Other Recommended Materials

- Latex gloves
- Liquid and solid waste containers
- Calculator
- Absorbant paper for blotting
 - Marking pen



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STRATEGIC DIAGNOSTICS INC.

RaPID Assay® Carcinogenic PAH Test Kit A00200/A00201

Intended Use

The RaPID Assay® Carcinogenic PAH Test Kit can be used as a quantitative, semi-quantitative or qualitative enzyme immunoassay (EIA) for the analysis of Carcinogenic PAH (as benzo(a)pyrene) in water groundwater, surface water, well water). For applications in other matrices please contact our Technical Service department. The RaPID Assay® Carcinogenic PAH Test Kit allows reliable and rapid screening for Carcinogenic PAH (measured and reported as benzo(a)pyrene) and related compounds, with quantitation between 0.1 ppb and 5.0 ppb (as benzo(a)pyrene). The minimum detection level of the kit is 0.04 (as benzo(a)pyrene.)

Test Principles

The Carcinogenic PAH RaPID Assay® kit applies the principles of enzyme linked immunosorbent assay (ELISA) to the determination of Carcinogenic PAH and related compounds. The sample to be tested is added, along with an enzyme conjugate, to a disposable test tube, followed by paramagnetic particles with antibodies specific to Carcinogenic PAH attached. Both the Carcinogenic PAH (which may be in the sample) and the enzyme labeled Carcinogenic PAH (the enzyme conjugate) compete for antibody binding sites on the magnetic particles. At the end of an incubation period, a magnetic field is applied to hold the paramagnetic particles (with Carcinogenic PAH and labeled Carcinogenic PAH analog bound to the antibodies on the particles, in proportion to their original concentration) in the tube and allow the unbound reagents to be decanted. After decanting, the particles are washed with Washing Solution.

The presence of Carcinogenic PAH is detected by adding the enzyme substrate (hydrogen peroxide) and the chromogen (3,3',5,5' – tetramethylbenzidine). The enzyme labeled Carcinogenic PAH analog bound to the Carcinogenic PAH antibody catalyzes the conversion of the substrate/chromogen mixture to a colored product. After an incubation period, the reaction is stopped and stabilized by the addition of acid. Since the labeled Carcinogenic PAH (conjugate) was in competition with the unlabeled Carcinogenic PAH (sample) for the antibody sites, the color

developed is inversely proportional to the concentration of Carcinogenic PAH in the sample.

NOTE: Color development is inversely proportional to the Carcinogenic PAH concentration.

Darker color = lower concentration Lighter color = higher concentration

The determination of the Carcinogenic PAH level in an unknown sample is interpreted relative to the standard curve generated from kit standards after reading with a spectrophotometer.

Performance Characteristics

The Carcinogenic PAH RaPID Assay® will detect Carcinogenic PAH and related compounds to different degrees. Refer to the table below for data on several of these compounds. The Carcinogenic PAH RaPID Assay® kit provides screening results. As with any analytical technique (GC, HPLC, etc.) positive results requiring some action should be confirmed by an alternative method.

The Carcinogenic PAH RaPID Assay® immunoassay test does not differentiate between Carcinogenic PAH and other related compounds. The table below shows compounds at the method detection limit (MDL) which is the lowest concentration of the compound that can be picked up in the assay. The limit of quantitation (LOQ) is an approximate concentration required to yield a positive result at the lowest standard, this is the lowest concentration of the compound that can be quantified in the assay. The IC50 is the concentration required to inhibit one half of the color produced by the negative control. It is also used to calculate cross-reactivity values to similar compounds.

Compound	MDL (ppb)	LOQ (ppb)	IC50 (ppb)
Benzo(a)pyrene	0.08	0.2	3.2
Benzo(a)anthracene	0.02	0.06	0.96
Benzo(k)fluoranthe			
ne	0.02	0.08	1.26
Chrysene	0.04	0.09	1.38
Benzo(b)fluoranthe			
ne	0.04	0.16	2.60

Indeno(1,2			
,3-			
c,d)pyrene	0.02	0.254	4.06
Dibenzo(a,			
h)anthrace			ĺ
ne	0.14	0.30	4.82
Anthracen			
e	0.44	2.56	41
Phenanthr			
ene	2.70	8.4	134.4
Fluoranth			
ene	2.00	8.56	137
Benzo(g,h,	_		
i)perylene	0.30	> 12.5	> 200
Pyrene	2.00	29.1	466
Fluorene	37.0	42.8	684
Naphthale	,	_	
ne	376	625	10000
Acenaphth			
ylene	148	721	11540
Acenaphth			
alene	1078	> 1250	> 20000
Heating			
Oil	20.0	81.6	1306
Gasoline	200	> 1250	> 20000
Kerosene	2800	> 2800	> 20000
Jet A Fuel	> 20000	> 20000	> 20000

The total Carcinogenic PAH concentrations (sum of seven Carcinogenic PAH compounds) of the indicated contaminant types in soil and water samples are expressed below, at each of the three kit calibrator (standard) levels.

Carcinogenic PAH RaPID Assay® Total Carcinogenic PAH in Water (in ppb)

Contaminant	S1 Equivalent	S2 Equivalent	S3 Equivalent
Creosote	0.01	0.2	2
Coal Tar Oil	0.01	0.2	2
Diesel	0.01	0.1	0.7
Fuel Oil #4,6	0.02	0.15	0.7
Fuel Oil #5	0.01	0.1	0.5

The presence of the following substances up to 250 ppm were found to have no significant effect on Carcinogenic PAH RaPID Assay® results: copper, manganese, magnesium, mercury, nickel, nitrate, peroxide, phosphate, sulfite, thiosulfate and zinc. In addition, calcium up to 500 ppm, sodium chloride up to 1.0M, sulfate to 10,000 ppm, and iron to 100 ppm, showed no significant effect on results.

Precautions

- Training is strongly recommended prior to using the RaPID Assay® test system. Contact Strategic Diagnostics for additional information.
- Treat Carcinogenic PAH solutions that contain Carcinogenic PAH, and potentially contaminated samples as hazardous materials.
- Use gloves, proper protective clothing, and methods to contain and handle hazardous material where appropriate.
- Reagents must be added in a consistent manner to the entire rack. A consistent technique is the key to optimal performance. Be sure to treat each tube in an identical manner.
- Water samples should be at a neutral pH prior to analysis. Samples containing gross particulate should be filtered (e.g. 0.2 um AnotopTM 25 Plus, Whatman, Inc.) to remove particles.
- Store all test kit components at 2°C to 8°C (36°F to 46°F). Storage at ambient temperature (18°C to 27°C or 64°F to 81°F) on the day of use is acceptable. Test tubes require no special storage and may be stored separately to conserve refrigerator space.
- Allow all reagents to reach ambient temperature (18°C to 27°C or 64°F to 81°F) before beginning the test. This typically requires at <u>least</u> 1 hour to warm from recommended storage conditions.
- Do not freeze test kit components or expose them to temperatures above 100°F (39°C).
- Do not use test kit components after the expiration date.
- Do not use reagents or test tubes from one test kit with reagents or test tubes from a different test kit.
- Do not mix reagents from kits of different lot numbers.
- Use approved methodologies to confirm any positive results.
- Do not under any circumstances attempt to disassemble the base of the magnetic rack. Magnets will be violently attracted to each other.
- Adequate sample number and distribution are the responsibility of the analyst.
- The photometer provided in the accessory kit requires electricity and comes with a 110V adapter. Adapters for 220V are available.

Do not expose color solution to direct sunlight.

Do not dilute or adulterate test reagents or use samples not called for in the test procedure; this may give inaccurate results.

Tightly recap the standard vials when not in use to prevent evaporative loss.

*Aaterials Provided

 Antibody Coupled Paramagnetic Particles in buffered saline containing preservative and stabilizers.

30 test kit: one 20 mL vial 100 test kit: one 65 mL vial

Enzyme Conjugate.

30 test kit: one 10 mL vial 100 test kit: one 35 mL vial

Standards

Three concentrations (0.1, 1.0, 5.0 ppb) of Carcinogenic PAH (as benzo(a)pyrene) standards in buffered saline containing preservative and stabilizers are supplied. Each vial contains 4 mL.

Control

A concentration (approximately 2.0 ppb) of Carcinogenic PAH (as benzo(a)pyrene) in buffered saline containing preservative and stabilizers. A 4 mL volume is supplied in one vial.

Diluent/Zero Standard

Buffered saline containing preservative and stabilizers without any detectable Carcinogenic PAH.

30 test kit: one 10 mL vial 100 test kit: one 35 mL vial

• Color Solution containing hydrogen peroxide and 3,3',5,5'-tetramethylbenzidine in an organic base.

30 test kit: one 20 mL vial 100 test kit: one 65 mL vial

 Stop Solution containing a solution of 2M sulfuric acid.

30 test kit: one 20 mL vial 100 test kit: one 60 mL vial

 Washing Solution containing preserved deionized water, with preservatives and stabilizers.

30 test kit: one 70 mL vial 100 test kit: one 250 mL vial

Polystyrene test tubes

30 test kit: one 36 tube box 100 test kit: three 36 tube boxes

User's Guide

Materials Required and Ordered Separately

See "Ordering Information" for the appropriate catalogue numbers.

Rapid Assay® Accessory Kit

Accessory equipment may be rented or purchased from Strategic Diagnostics. See "Ordering Information" for the appropriate catalogue numbers.

The accessory kit contains the following items:

- Adjustable Volume Pipet
- EppendorfTM Repeater® Pipettor
- Electronic timer
- Portable balance capable of weighing 10 g (for soil samples)
- Vortex mixer
- Magnetic separation rack
- RPA-I RaPID Analyzer (or equivalent spectrophotometer capable of reading 450 nm in a 1 mL sample size).

Other Items

- 12.5 mL Combitips[®] for the Repeater pipettor for 0.25 mL to 1.25 mL dispensing volumes (5)
- Pipet tips for adjustable volume pipet (100-1000 uL)

NOTE: Order replacement Combitips® and pipet tips separately. See the "Ordering Information section.

Materials Required but Not Provided

- Protective clothing (e.g., latex gloves)
- Absorbent paper for blotting test tubes
- Liquid and solid waste containers
- Marking pen
- Instructional video (optional)
- Methanol (HPLC Grade or equivalent) for water samples

Suggestions for Pipettor Use

- Practice using both pipettes (adjustable volume and Repeater pipettor) with water and extra tips before you analyze your samples.
- Use a new tip each time you use the Repeater pipettor to pipette a different reagent to avoid reagent cross-contamination. Tips can be rinsed thoroughly, dried completely and reused. By using the same tip to dispense the same reagent each time you can avoid cross contamination.

NOTE: Repeator tips should be changed periodically (after ~10 uses) since precision deteriorates with use.

- Draw the desired reagent volume into the Repeater pipettor and dispense one portion of the reagent back into the container to properly engage the ratchet mechanism. If you do not do this, the first volume delivered may be inaccurate.
- To add reagents using the Repeater pipettor, pipette down the side of the test tube just below the rim.
- When adding samples and standard using the positive displacement pipettor, always pipette into the bottom of the tube without touching the sides or bottom of the tube.
- Use a new adjustable volume pipet tip each time you pipette a new unknown.

Assay Procedure

Prior to performing your first Rapid Assay®, please take time to read the package inserts in their entirety and review the videotape if available. On site training is strongly recommended for new users of this test system. Please contact your account manager for further information.

Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

1. Water samples should be collected in glass vessels with teflon cap liners. Immediately upon collection, water samples should be diluted with and equal volume (1:1) of methanol (HPLC grade) to prevent adsorptive losses to the glass containers. This is a 2x dilution, which must be accounted for when interpreting the results. See "Results Interpretation", Section 3a for further details. Use this diluted sample as "sample" in "Perform the Test".

NOTE: This 2x dilution is <u>not</u> required for soil samples.

- Samples should be collected in appropriately sized and labeled containers.
- 3. If testing soil samples, follow the SDI Sample Extraction Kit User's Guide or the appropriate technical bulletin to properly collect and store your sample.
- 4. Samples should be tested as soon as possible after collection. If this is not possible, storage at 4°C (39°F) is recommended to minimize evaporative losses.

Set Up

- 1. Remove kits from refrigerator. All reagents must be allowed to come to room temperature prior to analysis. Remove reagents from packaging and place at room temperature at least 1 hour prior to testing.
- 2. Turn on the RPA-1 or other spectrophotometer. The RPA-1 should be warmed up for at least 30 minutes prior to the run.
- 3. Label five 12.5 mL Combitips "Conjugate", "Particles", "Wash", "Color" and "Stop". In addition, add the name of the compound you are testing for to each Combitip.
- 4. Remove nine clean blank test tubes for standards and control and one test tube for each sample (if testing in singlicate). Label the test tubes according to contents as follows.

Contents
Negative control(replicate 1)
Negative control (replicate 2)
Standard 1 (replicate 1)
Standard 1 (replicate 2)
Standard 2 (replicate 1)
Standard 2 (replicate 2)
Standard 3 (replicate 1)
Standard 3 (replicate 2)
Control
Sample 1
Etc.

*Label at top of tubes to avoid interference with reading of tubes in photometer

Sample Extraction and Dilution

Filtration may be necessary to remove gross particulate from the sample. If testing at levels higher than standard kit levels is desired, contact SDI for special instructions. Please follow the instructions from the SDI Sample Extraction Kit to prepare and dilute the soil extract prior to running the assay. Dilute water samples as described in "Collect/Store the Sample."

Perform the Test

- Separate the upper rack from the magnetic base. Place labeled test tubes into the rack.
- .. Add 200 uL of standards, control or samples to the appropriate tubes using the adjustable volume pipet with the dial set on 0200. The negative control, standards and control must be run with each batch of samples.

NOTE: Sample should be added to the bottom of the tube by inserting the pipet tip into the tube without touching the sides or the bottom of the tube. Take care not to contact sample with pipette tip once dispensed into bottom of the tube.

- 3. Using the Repeater Pipettor with the "Conjugate" tip attached and the dial set on "1", add 250 uL of Enzyme conjugate down the inside wall of each tube. (Aim the pipet tip ¼" to ½" below the tube rim or tube wall; deliver liquid gently to avoid splashback.)
- 4. Thoroughly mix the magnetic particles by swirling (avoid vigorous shaking) and attach the "Particles" tip to the Repeater Pipettor. With the dial set on "2" add 500 uL of magnetic particles to each tube, aiming down the side of the tube as described above. Vortex, mixing each tube 1 to 2 seconds at low speed to minimize foaming. Pipetting of magnetic particles should be kept to 2 minutes or less.
- 5. Incubate 20 minutes at room temperature.
- 6. After the incubation, combine the upper rack with the magnetic base and press all tubes into the base; allow 2 minutes for the particles to separate.
- 7. With the upper rack and magnetic base combined, use a smooth motion to invert the combined rack assembly over a sink and pour out the tube contents.

NOTE: If the rack assembly inadvertently comes apart when lifting to pour out tube contents, recombine and wait an additional 2 minutes to allow particles to separate.

8. Keep the rack inverted and gently blot the test tube rims on several layers of paper towels. It is important to remove as much liquid as possible but do not bang the rack or you may dislodge the magnetic particles and affect the results.

9. Set the Repeater Pipettor dial to "4" and put on the tip labeled "Wash". Add 1 mL of Washing Solution down the inside wall of each tube by using the technique described earlier. Wait 2 minutes and pour out the tube contents as described previously. Repeat this step one more time.

NOTE: The number of washes and wash volume are important in ensuring accurate results.

- 10. Remove the upper rack (with its tubes) from the magnetic base. With the "Color" tip attached to the Repeater Pipet and the dial set to "2" add 500 uL of Color Reagent down the inside wall of each tube as described previously. Vortex 1 to 2 seconds (at low speed).
- 11. Incubate 20 minutes at room temperature. During this period, add approximately 1 mL of Washing solution to a clean tube for use as an instrument blank for "Results Interpretation".
- 12. After the incubation, position the Repeater pipettor at Setting "2" and use the "Stop" tip to add 500 uL of Stop solution to all test tubes.
- 13. Proceed with results interpretation.

WARNING: Stop solution contains 2M sulfuric acid. Handle carefully.

Results Interpretation

- 1. After addition of Stop Solution to the test tubes, results should be read within 15 minutes.
- 2. Wipe the outside of all antibody coated tubes prior to photometric analysis to remove fingerprints and smudges.

Photometric Interpretation Using the RPA-I

1. The RPA-I photometer (provided in the Rapid Assay® Accessory kit) can be used to calculate and store calibration curves. It is preprogrammed with various RaPID Assay® protocols. For the Carcinogenic PAH Rapid Assay® test kit parameter settings are as follows:

Data Reduct : Lin. Regression

Xformation : Ln/LogitB
Read Mode : Absorbance

Wavelength : 450 nm

Units : PPB # Rgt Blk : 0

Calibrators:

of Cals : 4 # of Reps : 2

Concentrations:

#1: 0.00 ppb #2: 0.10 ppb #3: 1.00 ppb #4: 5.00 ppb

Range : 0.04 - 5.00 Correlation : 0.990 Rep. %CV : 10%

NOTE: Prior to analysis the RPA-I User's Manual should be thoroughly reviewed for more detailed operation instructions.

2. Follow the instrument prompts to read the absorbance of all tubes:

instrument Display Operator Respons	Instrument Display	Operator Response
-------------------------------------	--------------------	-------------------

SELECT COMMAND Press RUN RUN PROTOCOL Scroll using or NO [] kg

Scroll using the YES [] or NO [] keys until the desired protocol appears. Then press ENTER

SPL. REPLICATES (1-5) Press 1 (for analysis of samples in singlicate.)

Press ENTER
Insert blank tube

BLANK TUBE, INSERT TUBE, EVALUATING TUBE, REMOVE TUBE (Beep)

containing 1mL wash solution.

Remove tube

CAL #1, REP. #1, INSERT TUBE, EVALUATING TUBE, REMOVE TUBE (Beep) Insert Tube #1

Remove tube

Follow prompts to read tubes.

NOTE: Tube order is important. The RPA-I expects to see the standards in ascending order, in duplicate, starting with the negative control.

Following evaluation of all standards, the instrument will display:

PRINTING DATA, Data will print

PRINTING CURVE Curve will print only if

programmed to print (See RPA1 User's

Manual).

CTRL #1 REP #1, INSERT TUBE, Insert Control Tube

EVALUATING TUBE, REMOVE TUBE (Beep)

Remove Tube

EDIT CALIBRATORS YES/NO

Press NO (if editing is necessary press YES and refer to the RPA1

User's Manual).

SPL #1 REP#1 INSERT TUBE EVALUATING TUBE Insert first sample tube

REMOVE TUBE (Beep) Remove tube

Continue to follow prompts. After all samples have been read, press STOP.

Expected Results:

- %CV (coefficient of variation) between standard duplicates of 10% or less.
- Absorbance reading for the 0 ppb standard should be between 0.8 and 2.0 for all assays.
- Correlation (r) of 0.990 or greater for all assays.
- Kit control within range specified on vial.
- Absorbance of negative control and standards should be as follows:

Negative Control > Std. 1 > Std. 2 > Std. 3.

- 3. Concentrations will be indicated for all samples on the RPA-I printout.
 - a) The concentration indicated on the printout, is multiplied by the appropriate dilution factor (if applicable) introduced in the procedure. The quantitation range of the kit is also multiplied by this factor.

EXAMPLE: Water samples were diluted 2-fold with methanol upon collection (see "Collect/Store the Sample" in this User's Guide). As a result, the concentrations listed on the printout should be multiplied by 2 to determine the sample concentration.

The standard concentrations are alsomultiplied by 2 to give a quantitation range in water for this test kit of 0.2 to 10 ppb.

- b) Samples with an "nd" and no concentration listed have an absorbance greater than the negative control; therefore, no concentration can be computed for these samples. Results must be reported as <0.2 ppb (or Standard 1 multiplied by the dilution factor).
- c) Samples with an "nd" next to a listed concentration have an estimated concentration below the minimum detection level of the test kit. Results must be reported as <0.2 ppb (or Standard 1 multiplied by the dilution factor).

NOTE: Any samples with concentrations determined to be lower than Standard 1 (the limit of quantitation) must be reported as <0.2 ppb (or Standard 1 multiplied by the dilution factor.) Quantitation is not possible below this standard as this is outside the linear range of the assay.

d) Similarly, samples with a "hi" next to a listed concentration have an estimated concentration higher than Standard 3 and must be reported as > 10.0 ppb (or Standard 3 multiplied by the dilution factor.)

NOTE: In order to determine the concentration of samples with concentrations greater than Standard 3, they must be subjected to repeat testing using a diluted sample. A ten-fold or greater dilution of the sample is recommended with an appropriate amount of Carcinogenic PAH diluent. This additional dilution must then be taken into account when calculating the concentration.

Photometric Interpretation Using Other Photometers

Other photometers may also be used to interpret results obtained from the RPA-I photometer. It is important that the photometer be able to read absorbance at 450nm and that the instrument can read

at a 1 mL fill volume. Absorbances obtained from other spectrophotometers (reading at 450 nm) may be used to manually calculate sample concentrations as outlined below.

- 1. Calculate the mean absorbance for each of the three standards and the negative control.
- Determine the standard deviation and %CV (coefficient of variation) of each standard and ensure %CV is less than 10% for each.
- 3. Calculate the %B/Bo for each standard by dividing the mean absorbance value for the standard by the mean absorbance value for the negative control and multiplying the results by 100.
- 4. Construct a standard curve by plotting the %B/Bo for each standard, on the vertical logit (y) axis versus the corresponding analyte concentration on the horizontal logarithmic (x) axis on the graph paper provided in the test kit. Graph papers are specific for each method. Use only the graph paper supplied with each kit.
- 5. Draw the best straight line through all points. Using the %B/Bo of the sample, the concentration can be interpolated from the standard curve.
- 6. Multiply results by the appropriate dilution factor (if applicable) introduced in the procedure. For example, if the sample was diluted 10-fold to increase the detection levels of the kit then the results, must be multiplied by 10. This dilution also changes the range of the assay (standards) by the same factor.

NOTE: Do not forget to account for the 2x dilution introduced in the "Collect/Store the Sample" procedure for the water samples.

Limitations of the Procedure

The Rapid Assay® Carcinogenic PAH Test Kit is a screening test only. Sampling error may significantly affect testing reliability. Adequate sample number and distribution are the responsibility of the analyst.

Ordering Information

Description	Catalogue Number	
Rapid Assay® Carcinogenic PAH Kit	A00200/A00201	
Rapid Assay® Accessory Kit**	6050100	
Adjustable Volume Pipet Tips (100-1000 uL)	A00013	
12.5 mL Combitip for Repeating Pipette (1 each)	A00009	
CarcinogenicPAH Diluent	A00203	
Rapid Assay® Accessory Kit Rental	6997010	
** To obtain part numbers and pricing for individual items in the Accessory Kit contact SDI at the number below.		

Ordering/Technical Assistance

Should you have any questions regarding this procedure prior to analysis contact Technical Service to avoid costly mistakes.

To Place an Order or Receive Technical Assistance, please call Strategic Diagnostics Inc. at:

Call toll-free 800-544-8881

Or 302-456-6789 Phone 302-456-6782 Fax

Web site: www.sdix.com E-mail: techservice@sdix.com

General Limited Warranty

SDI's products are manufactured under strict quality control guidelines and are warranted to be free from defects in materials and workmanship. New instruments and related non-expendable items are warranted for one year from date of shipment against defective materials or workmanship under normal use and service.

Warranty obligation is limited to repair or replacement of the defective product or to refund of the purchase price, at the discretion of SDI. Other warranties, express or implied, are disclaimed. SDI's liability under any warranty claim shall not exceed the refund of the purchase price paid by the customer. Under no circumstances shall SDI be liable for special, indirect or consequential damages.

Safety

To receive an MSDS for this product, visit our web site at www.sdix.com.

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Z00361.1, Rev 4/5/00

)peration of the Repeater Pipet

'o Set or Adjust Volume

To determine the pipetting olume, the dial setting (1-5) is nultiplied by the minimum pipetting volume of the tip 'indicated on the side of the Combitip, e.g. 1_100 uL.)

To Assemble Pipet Tip

ilide filling lever down until it stops. Then raise the locking clamp and insert the tip until it tlicks into position. Be sure he tip plunger is fully inserted into the barrel before lowering the locking clamp to affix the tip in place.

To Fill Tip

With tip mounted in position on pipet, immerse end of tip into solution. Slide filling lever upward slowly. Combitip will fill with liquid.

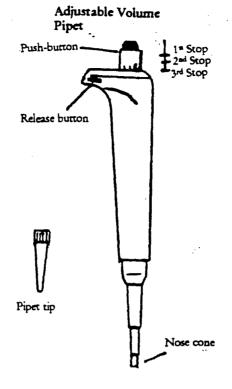
To Dispense Sample

Check the volume selection dial to ensure pipetting volume. Place tip inside test tube so that tip touches the inner wall of tube.

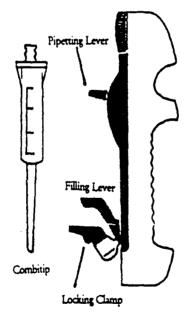
Completely depress the pipetting lever to deliver sample. NOTE: Dispense one portion of reagent back into the container to engage the ratchet mechanism and ensure accuracy.

To Eject Tip

Empty tip of any remaining solution into appropriate container by pushing filling lever down. Raise locking clamp upward, and remove the Combitip.



Repeater Pipet



Operation of the Adjustable Volume Pipet

To Set or Adjust Volume

Press release button on side of pipette and turn the pushbutton to adjust volume up or down. Volume setting is displayed on top of pipet. See kit instructions for appropriate setting. Pipet will accurately dispense volumes between 100 and 1000 uL.

To Assemble Pipet Tip

Gently push nose cone of pipet firmly into a pipet tip contained in the pipet tip rack.

To Withdraw Sample

Keep pipet almost vertical. With tip mounted in position on pipet, press push-button to 1st stop and hold it. Place tip at bottom of liquid sample and slowly release push-button to withdraw measured sample. Ensure that no air bubbles exist in the pipette tip. If bubbles exist, dispense sample and re-withdraw. Slide tip out along the inside of the vessel.

To Dispense Sample

Wipe any liquid from outside of tip taking care not to touch orifice. Place tip into tube, almost to the bottom, and slowly press push-button to 2nd stop. Hold push-button at 2nd stop when removing tip from tube.

To Eject Tip

Press push-button to 3rd stop. Tip is ejected.

STRATEGIC DIAGNOSTICS INC.

Sample Extraction Kit User's Guide

Intended Use

This extraction kit is for use with the appropriate mmunoassay test kit. Each Sample Extraction kit contains the materials necessary to process twelve (12) soil or wipe samples.

Test Principles

The reagents contained in the Sample Extraction kit have sen optimized for fast, efficient removal of compounds on soil or surfaces and convenient preparation of the sample for immunoassay testing at levels of interest to the investigator. The system allows for reliable, convenient and cost effective determinations at the field testing or remediation site.

Performance Characteristics

Precautions

- Treat potentially contaminated samples as hazardous materials.
- Use gloves, proper protective clothing, and methods to contain and handle hazardous material where appropriate.
- Store all kit components at ambient temperature (18°C to 27°C or 64°F to 81°F).
- Do not mix reagents from kits of different lot numbers.
- When testing soil samples, samples obtained from areas adjacent to standing water, surface soils collected during or immediately after rain or snow, or any soils with relatively high amounts of water (≥ 30% by weight) should be dried prior to extraction. Contact technical service for recommended methods.
- Adequate sample number and distribution are the responsibility of the analyst.
- Do not dilute or adulterate test reagents; this may give inaccurate results.

 Cloudy or dark sample extracts may indicate the presence of interference in your sample. Please contact Technical Support if this occurs.

Materials Provided

Filter unit bottoms: 12 per kit

• Filter unit tops: 12 per kit

• Wooden spatulas: 12 per kit

• Plastic Weigh Boats: 12 per kit

Bulb Pipettes: 12 per kit

Ampule crackers: 12 per kit

Extraction jars: 12 per kit
 (Jars for soil extraction contain 3 ball bearings)

 10 cm x 10 cm Plastic Wipe Templates: 12 per kit (PCB Wipe Kit only)

 Gauze Wipes: 12 per kit (PCB Wipe Kit only)

 Protective gloves: 24 per kit (PCB Wipe Kit only)

User's Guide

• Kit Specific Extraction Solution: 12 per kit as described below:

20 mL of 100% Methanol for use with:

Ensys PCB Soil/Wipe (Part #7020301/7021301) *
Ensys Petro Soil (Part #7042301)
Ensys PAH Soil (Part #7061301) *
Ensys Penta Soil (Part #7000301) *

RaPID Assay PCB (Part # A00133/A00134) *

RaPID Assay PAH (Part # A00156/A00157)*

RaPID Assay CaPAH (Part # A00200/A00201)*

RaPID Assay TNT (Part # A00186) *

10 mL of 100% Methanol for use with:

Envirogard PAH in Soil (Part #7060600) *
Envirogard BTEX in Soil (Part #7004000)
Envirogard TPH in Soil (Part #7042000)
Envirogard DDT in Soil (Part #7310000) *
Envirogard PCB Soil/Wipe *
(Part #7020800/7021600) or #7021500/7021600)

20 mL of 90% Methanol for use in:

Envirogard Chlordane in Soil (Part #7311000) Envirogard Toxaphene in Soil (Part #7420000) Envirogard Lindane in Soil (Part #7630000)

10 mL of 75% Methanol for use in:

Rapid Assay BTEX (Part # A00161/A0)162)

20 mL of 75% Methanol with Sodium Hydroxide for use in:

Rapid Assay PCP (Part # A00110/A00111)

 $20\ mL$ of 100% Methanol with Surfactant for use in:

Rapid Assay Cyclodienes (Part # A00216)

* Indicates extraction solution is also available in bulk (i.e. two 125 mL bottles per kit)

- Kit specific dilution material (for Ensys and RaPID Assay test kits)
 - For Ensys test kits dilution ampules will be provided based on customer specified detection levels.
 - For Rapid Assay test kits, 12 dilution vials with the appropriate volume of assay diluent (see "Dilute the Sample" section of this User's Guide) will be provided. The kit may also contain fixed volume disposable pipets and tips as appropriate.

Materials Required and Ordered Separately

- 50 mL Combitips® for the Repeater pipettor for 1.0 mL to 5.0 mL dispensing volumes (if using bulk extraction solution)
- Eppendorf Repeater pipettor (if using bulk extraction solution)
- Portable balance capable of weighing 10 g (for soil samples)
- Electronic timer

NOTE: Order replacement Combitips® separately. See the "Ordering Information" section.

Materials Required but Not Provided

- Protective clothing (e.g., latex gloves)
- Liquid and solid waste containers
- Marking pen

Soil Procedure

Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

- 1. Collect soil in appropriately sized and labeled containers.
- Take care to remove excess twigs, organic matter, and rocks or pebbles from the soil sample to be tested.
- Soils obtained from areas adjacent to standing water, surface soils collected during or immediately after rain or snow, or any soils with relatively high amounts of water (≥ 30% by weight) should be dried before testing. Contact Technical Services for recommended methods.
- 4. When comparing data from fields and laboratory methods it is important that split samples are obtained from thoroughly homogenized samples.
- Store soil samples at 4°C (39°F), staying within the EPA recommended holding times for your analyte of interest.

Weigh the Sample

- Verify digital balance is calibrated correctly by pressing the ON/MEMORY button on the instrument and placing the 100 g weight (in the pocket of the instrument cover) onto the balance. If the instrument does not read 100±0.1 g, you must recalibrate the instrument as per the manufacturer's instructions provided with your accessory kit.
- 2. Place an unused plastic weigh boat on the digital balance (provided in the Field Accessory Kit).
- 3. Press the ON/MEMORY button on the digital balance. The balance will beep and display 0.0.
- 4. Weigh out 10 ± 0.1 grams of sample into the weigh boat on the balance using a wooden spatula.

NOTE: If the balance turns off prior to completing the weighing of the sample, use an empty weigh boat o re-tare the instrument and then continue.

 Repeat Steps 1-3 for each sample to be tested, using a new weigh boat and wooden spatula for each sample.

Extract the Soil

- . Uncap an extraction jar (containing ball bearings) and place it on a flat surface. Label the extraction jar with the sample identification. Transfer 10 grams of sample from the weigh boat into the appropriately labeled extraction jar, using the same wooden spatula used to weigh the sample. Be careful to get your entire sample into the extraction jar.
- 2. Open the solvent ampule using the ampule cracker provided in your extraction kit by placing the ampule cracker over the scored neck of the ampule. The ampule cracker is designed to protect your hands from broken glass.
- Pour the entire contents of one solvent ampule into the extraction jar and immediately recap the extraction jar. Do not leave the jar open or the solvent will evaporate and affect results.
- 4. Shake the jar vigorously for one full minute.
- 5. Allow the sample to settle for one minute or until a liquid solvent layer is observed above the sample.

NOTE: If the solvent layer is not observed within 15 minutes, contact Technical Support for assistance. Clay samples are often difficult to extract because they absorb the solvent. In this case, Technical Support may recommend decreasing the soil to solvent ratio. This will affect detection levels and should be discussed in advance

Repeat Steps 1-5 for each sample to be tested.

Filter the Extract

- Insert the bulb pipet into the top (liquid) layer in the extraction jar (being careful not to disturb the lower, solid layer) and draw up some of the sample.
 Transfer at least ½ bulb capacity into the bottom portion of the filtration unit. Do not use more than one full bulb.
- 2. Press the top portion of the filtration unit (which is the piece with the cap and filter) into the bottom portion (containing the sample) until it snaps

- together or until the majority of the liquid has passed upward through the filter. Place on a flat surface.
- 3. Repeat Steps 1-2 for each sample to be tested.

NOTE: Do not store sample in the filtration unit for extended periods of time. The seal on the unit will not sufficiently prevent evaporative losses of the solvent. Evaporation of the solvent will affect results.

Dilute the Sample

- I. Envirogard and Ensys test kits Use the filtered extract as "SAMPLE" in the test kit User's Guide procedure. The Ensys User's Guide describes a sample dilution method based on your individual testing needs.
- II. Rapid Assay Dilute the filtered extract into the appropriate sample diluent as described in the following table:

Kit	Extract Vol. (uL)	Diluent (mL)	*Total Dil. Factor	Test Range (ppm)
PCB	25	25	2000	0.5 to 10 (Aroclor 1254)
PAH	250	12.25	100	0.2 to 5 (Phenanthrene)
CaPAH	200	9.8	100	0.01 to 0.5 (Benzo(a)pyrene)
BTEX/ TPH	500	4.5	10	0.9 to 30 (total BTEX)
PCP	50	25	1000	0.1 to 10 (PCP)
TNT	50	25	1000	0.25 to 5 (TNT)
Cyclo- dienes	250	12.25	100	0.1 to 2 (Dieldrin)

- *Note: "Total dilution factor" takes the extraction dilution into account as well as the kit dilution (i.e. 10 g soil to 20 mL solvent is a 2x dilution).
- a. Remove a pre-measured diluent vial from your extraction kit for each sample to be tested. Label vials with the appropriate sample identification. Vials contain the volume of diluent specified in the preceding table corresponding to your test kit.
- b. Using the adjustable volume pipet (for volumes between 100 and 1000 uL) or the tan fixed volume pipet provided in the extraction kit (for 25 or 50 uL volumes) pipet the volume of filtered extract specified

in the table above directly into the liquid in the corresponding diluent vial.

- c. Screw the cap tightly onto the diluent vial and mix by inverting several times. Repeat steps 1 and 2 for each sample being tested using a new, clean piper tip for each one.
- d. The diluted extract should be used as "Sample" in the test kit User's guide procedure.

Wipe Procedure

Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

- 1. Collect sample in appropriately sized and labeled containers.
- 2. Wearing a clean pair of protective gloves provided in the extraction kit, uncap an extraction jar.
- Open the solvent ampule using the ampule cracker provided in your extraction kit by placing the ampule cracker over the scored neck of the ampule. The ampule cracker is designed to protect your hands from broken glass.
- 4. Pour the entire contents of one solvent ampule into the extraction jar and immediately recap the extraction jar. Do not leave the jar open or the solvent will evaporate and affect results.
- 5. Soak a gauze pad in the extraction jar containing solvent. Remove the gauze wipe from the extraction jar carefully squeezing the excess solvent from the pad back into the extraction jar.
- 6. Hold a clean 10 x 10 plastic template on the surface to be wiped. Wipe the entire exposed area according to proper wipe sampling techniques. The wipe should be damp when finished.
- 7. Place the wipe back into the same extraction jar used in Step 4 and cap tightly.
- 8. Remove and discard the gloves.
- 9. Repeat Steps 1-7 for each sample to be tested.
- Store samples at 4°C (39°F), staying within the EPA recommended holding times for your analyte of interest.

Extract the Sample

- 1. Shake the jar vigorously for one full minute.
- 2. Repeat for each sample to be tested.

Filter the Extract

- Insert the bulb piper into the top (liquid) layer in the extraction jar and draw up some of the sample.
 Transfer at least ½ bulb capacity into the bottom portion of the filtration unit. Do not use more than one full bulb.
- 2. Press the top portion of the filtration unit (which is the piece with the cap and filter) into the bottom portion (containing the sample) until it snaps together. Place on a flat surface.
- 3. Repeat Steps 1-2 for each sample to be tested.

NOTE: Do not store sample in the filtration unit for extended periods of time. The seal on the unit will not sufficiently prevent evaporative losses of the solvent. Evaporation of the solvent will affect results.

Dilute the Sample

- I. Envirogard and Ensys test kits Use the filtered extract as "SAMPLE" in the test kit User's Guide procedure. The Ensys User's Guide describes a sample dilution method based on your individual testing needs.
- *II.* Rapid Assay Dilute the filtered extract into the appropriate sample diluent as described below.

Kit	Extract Vol. (uL)	Diluent (mL)	Total Dil. Factor	Test Range (ppm)
PCB	25	25	2000	5 to 100 ug/100 cm2 (Aroclor 1254)

*Note: "Total dilution factor" takes the extraction dilution into account as well as the kit dilution (i.e. 10 g soil to 20 mL solvent is a 2x dilution).

- a. Remove a pre-measured diluent vial from your extraction kit for each sample to be tested. Label vials with the appropriate sample identification. Vials contain the volume of diluent specified in the table above corresponding to your test kit.
- b. Using the adjustable volume pipet (for volumes between 100 and 1000 uL) or the tan fixed volume pipet provided in the extraction kit (for 25 or 50 uL)

volumes) pipet the volume of filtered extract specified in the table above directly *into* the liquid in the corresponding diluent vial.

- c. Screw the cap tightly onto the diluent vial and mix by inverting several times. Repeat steps 1 and 2 for each sample being tested using a new, clean pipet tip for each.
- 1. The diluted extract should be used as "Sample" in the test kit User's guide procedure.

Limitations of the Procedure.

Sampling error may significantly affect testing reliability. The distribution of contaminants in soils can be extremely neterogeneous. Adequate sample number and distribution are the responsibility of the analyst.

Ordering Information

Description	Catalogue Number Contact Customer Support	
SDI Sample Extraction Kit (with methanol in ampules or bulk)		
50 mL Combitip for Repeating Pipette (1 each)	6005600	
Portable balance**	A00131	
Eppendorf Repeater Pipettor**	A00008	
Electronic Timer**	A00015	
** These items are also included in our field accessory kits which are	available for rent or purchase.	

Ordering/Technical Assistance

Should you have any questions regarding this procedure prior to analysis contact Technical Service to avoid costly mistakes.

To Place an Order or Receive Technical Assistance, please call Strategic Diagnostics Inc. at:

Call toll-free 800-544-8881

Or 302-456-6789 Phone 302-456-6782 Fax

web site: www.sdix.com
e-mail: techservice@sdix.com

General Limited Warranty

SDI's products are manufactured under strict quality control guidelines and are warranted to be free from defects in materials and workmanship. New instruments and related non-expendable items are warranted for one year from date of shipment against defective materials or workmanship under normal use and service.

Warranty obligation is limited to repair or replacement of the defective product or to refund of the purchase price, at the discretion of SDI. Other warranties, express or implied, are disclaimed. SDI's liability under any warranty claim shall not exceed the refund of the purchase price paid by the customer. Under no circumstances shall SDI be liable for special, indirect or consequential damages.

Safety

To receive an MSDS for this product, visit our web site at www.sdix.com.

3090103.1, Rev 7/27/00

STRATEGIC DIAGNOSTICS INC.

RaPID Assay® Carcinogenic PAHs In Soil Application

Intended Use

For detection of Carcinogenic PAHs (as benzo(a)pyrene) in soil. For testing in other matrices, please contact our technical support department at 1-800-544-8881.

Materials Required but Not Provided

SDI Sample Extraction Kit (Part Number: A00204EA/A00204EB)

Procedural Notes and Precautions

- Prepare soil samples for analysis according to the procedure in the SDI Sample Extraction Kit Users Guide.
- After extraction and dilution of samples, follow the immunoassay procedure as described in the Rapid Assay ® Carcinogenic PAHs Test Kit User's Guide.
- The initial 2x dilution described for water samples in Step 1 of "Collect/Store the Sample" does not need to be performed for soil samples.

Quality Control

A control solution at approximately 2.0 ppb (as benzo(a)pyrene) is provided with the Carcinogenic PAHs RaPID Assay® Kit. It is recommended that it be included in every run and treated in the same manner as unknown samples. If running standard soil procedures an acceptable result should be 100 times the value stated on the control vial (i.e. 200 + or - 40 ppb) when the control results are corrected for the dilution factors (see Results section below).

Results Interpretation

Interpret soil sample results as described in the RaPID Assay® Carcinogenic PAHs Test Kit procedure, accounting for the total dilution factor indicated in the table of the SDI Sample Extraction Kit Users Guide. Alternatively, program the RPA-1 Analyzer as listed below to automatically correct for this dilution factor.

1. The RPA-I photometer (provided in the Rapid Assay® Accessory kit) can be used to calculate and store calibration curves. To obtain soil results from the Carcinogenic PAHs Rapid Assay® test

kit on the RPA-I the following parameter settings are recommended:

Data Reduct

Lin. Regression

Xformation Read Mode Ln/LogitB

Wavelength

Absorbance

Units

450 nm PPB

Rgt Blk

^

Calibrators:

of Cals :

4

of Reps:

2

Concentrations:

#1:

0.00 PPB

#2:

10.0 PPB

#3: #4: 100.0 PPB 500.0 PPB

Range

: 10 - 500

Correlation

0.990

Rep. %CV

10%

Performance Data

The Carcinogenic PAHs RaPID Assay® does not differentiate between Carcinogenic PAHs and other related compounds. The table below shows compounds at the method detection limit (MDL) which is the lowest concentration of the compound in soil that can be picked up in the assay. The limit of quantitation (LOQ) is an approximate concentration required to yield a positive result at the lowest standard, this is the lowest concentration of the compound in soil that can be quantified in the assay. The IC50 is the concentration in soil required to inhibit one half of the color produced by the negative control. It is also used to calculate cross-reactivity values to similar compounds.

Compound	MDL (ppb)	LOQ (ppb)	IC50 (ppb)
Benzo(a)pyrene	8.0	20.0	320

Benzo(a)anthracene	2.0	6.0	96
Benzo(k)fluoranthen			
e jí	2.0	8.0	126
Chrysene	4.0	8.0	138
Benzo(b)fluoranthen			
e	4.0	16.0	260
Indeno(1,2,3-			
_ c,d)pvrene	2.0	24.6	406
Dibenzo(a,h)anthrac			
ene	14.0	30.0	482
Anthracene	44.0	256	44.0
Phenanthrene	270	840	13440
Fluoranthene	200	856	46600
Benzo(g,h,i)perylene	30.0	> 1250	> 20000
Pyrene	200	2920	46600
Fluorene	3700	4280	68400
Naphthalene	18800	615,00	10000000
Acenaphthylene	14800	<i>722</i> 00	1154000
Acenaphthalene	107800	> 125000	> 2000000

The total Carcinogenic PAH concentrations (sum of seven Carcinogenic PAH compounds) of the indicated contaminant types in soil and water samples are expressed below, at each of the three kit calibrator (standard) levels, in units comparable to results from GC Method 8270 or HPLC Method 8310.

Carcinogenic PAHs RaPID Assay® Total Carcinogenic PAHs in Soil (in ppb)

	S1	S2	S3
Contaminant	equivalent	equivalent	equivalent
Creosote	0.5	10	100
Coal Tar Oil	0.5	10	100
Diesel	0.5	5	35
Fuel Oil #4,6	1.0	7.5	35
Fuel Oil #5	0.5	5	25

Range of Detection

The Carcinogenic PAHs RaPID Assay® has a range of detection in soil of 10.0 to 500 ppb (as Benzo(a)pyrene) when used in conjunction with the SDI Sample Extraction Kit.

Recovery

Carcinogenic PAH recoveries will vary depending on soil type, retention mechanism, solvent and extraction apparatus used, length of extraction period and levels of potentially interfering substances in the soil.

Z00366.1, Revised 4/4/00

Start-Up
Manual for
the RaPID
Assay®
System

Start-Up Manual for the RaPID Assay® System

Strategic Diagnostics Inc.

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·		

INTRODUCTION

This manual is intended to act as a guide for first time users of the RaPID Assay R kits and the associated equipment and as a reference for experienced users. It contains information on how to set-up the required equipment and run SDI's RaPID Assays. For more detailed explanation, refer to the operating manual for individual pieces of equipment and to the package insert for each assay kit.

Before running the first assay, read thoroughly those sections referring to each piece of equipment to be used (Sections 1 - 8). Next proceed to Sections 9 and 10 to run the assay. Section 11 is provided to assist the operator in resolving problems which might be encountered.

If any of the material contained in this manual is unclear or if problems are encountered, please feel free to call SDI's Technical Support at (800) 544-8881.

SECTION 1 - RPA-I™ ANALYZER

The RPA-I Analyzer is a laboratory benchtop-based, single wavelength, dual beam, microprocessor-controlled analyzer. It can read the absorbances of calibrators and samples, perform mathematical computations, and report raw absorbances and sample concentrations with statistics. For a complete and detailed description of the RPA-I, please refer to the RPA-I RaPID Analyzer Operations Manual (Part No. A00046).

ENVIRONMENT

- 5° C to 33° C
- 10% to 85% humidity
- Flat, level surface away from strong sources of electromagnetic interference.
- No direct sunlight or drafts.
- Removed from sources of direct heat and moisture.
- Ventilation space at least 6 inches on sides and back.

UNPACKING AND INSTALLATION

- 1. Inspect the carton for visible signs of damage and note the condition of the SHOCK-WATCH indicator on the side of the carton. If damage has occurred, or a part is missing, immediately contact SDI.
- 2. Open the carton and remove the brown rectangular box from the grey packing material. (Save all boxes.) This box contains the power transformer, roll of paper, and Program Cartridge. Refer to

Figure 1 for identification of shipping carton contents.

- 3. Lift off the gray packing material to reveal the photometer. Remove it from the carton.
- 4. Insert the Program Cartridge (with the white label facing up) into the Program Cartridge Holder found on the rear panel of the instrument. Push in until the white label is no longer visible (Refer to Figure 2).
- 5. With the power OFF to the instrument, (bottom of the white toggle power switch should be depressed) insert the round end of the Power Transformer (notched end facing up) into the AC Power Connector found on the rear panel of the instrument. Plug the square end of the power cable into a grounded AC outlet.
- 6. The instrument is then activated by depressing the top of the white toggle power switch. The instrument will perform a "Self Test." During this short test, the various electronic components of the RPA-I are automatically analyzed. This includes checks of EPROM and RAM memory. If there are any abnormalities in these areas, the RPA-I will alert the operator with an "ERROR" message. If all the parameters are satisfactory, the "Select Command" prompt will appear and the operator may continue.

SHIPPING CARTON CONTENTS

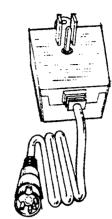
The shipping carton should contain the following items:

RPA-I Analyzer with a 450/600nm filter block.

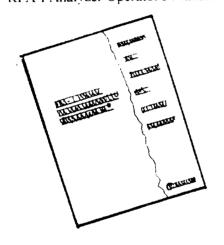
Domestic Power Cord/Mains Transformer

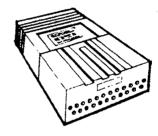


RPA-I Analyzer Operator's Manual



Program Cartridge





Printer Paper

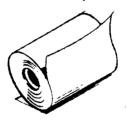


Figure 1. Shipping Carton Contents

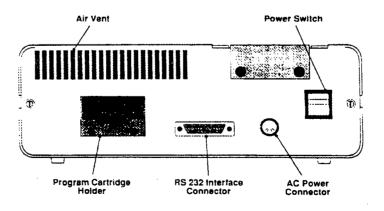


Figure 2. Rear Panel

SHORT OPERATING PROCEDURE FOR THE RPA-I

ALLOW THE RPA-I TO WARM UP FOR 30 MINUTES PRIOR TO USE. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter. The RPA-I performs a self-test first. If all parameters are satisfactory, the "Select Command" prompt will appear. If there are abnormalities, an "Error" message will appear.

The RPA-I reports all results on a thermal paper printout. The unit is turned off by switching the power switch in the rear of the unit to the off position.

INSTRUMEN'	T DISPLAY
------------	-----------

SELECT COMMAND

RUN PROTOCOL. Aldicarb. Atrazine. Alachlor, etc.

SPL. REPLICATES (1-5)

BLANK TUBE INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)

CAL. #1 REP. #1 **INSERT TUBE EVALUATING TUBE** REMOVE TUBE (Beep)

Follow the prompts on the instrument display.

OPERATOR RESPONSE

Press RUN

Scroll using the YES [s] or NO [>] until the desired protocol appears.

Press ENTER.

Press 1 (Press 2 if analyzing samples in duplicate, etc.). Press ENTER.

Insert tube with 1 mL of washing solution

Remove tube

Insert first standard replicate (0 ppb calibrator/tube #1).

Remove Tube

Note: Tube order is important here. The RPA-I expects to see the standards/calibrators in ascending order in duplicate, starting with 0 ppb.

After all the standards (calibrators) have been evaluated, the instrument will display:

PRINTING DATA LISTING XFORM DATA

PRINTING CURVE

CTRL, #1 REP, #1 **INSERT TUBE EVALUATING TUBE** REMOVE TUBE (Beep)

EDIT CALIBRATORS YES/NO

SPL #1 REP. #1 **INSERT TUBE EVALUATING TUBE** REMOVE TUBE (Beep) Data will print.

Curve will print only if programmed to print (See Section 3 Special Functions -Instrument Functions: Print Curve).

Insert Control Tube.

Remove Tube.

Press NO if it is not necessary to edit the calibrators, press YES to edit (See Section 3 Run).

Insert first Sample Tube.

Remove Tube.

Follow the prompts on the instrument display. After all the samples have been evaluated, press STOP.

EXPLANATION OF DATA

Bolded areas are explained in the right hand column.

	1 12:36	:38		
	* SDI **		•	
	L : ATF			
	:			
EMP DAT	E:			
Data Re	dust:Lir	Regr	ession n/LgtBA	
Read Mo	dustili. ion: de : gth :	Absc	rbance	
Wavelen Units	gtn :		450 nm PPB	
EQUATIO	N OF LI	Έ:		
 Slope	=		.2 À	
Interce Corr (r	pt =	-Ø.10	16 A	
	rmed Dat	- -		
Conc			_	
-2.3Ø Ø.ØØ		926 334	À À À	
1.61	-1.3	327	À	
cailbra 	tor Data	: 		
_		~··	5	
Cons	Abs	CA	Predic Diff	
	0155		Predic Diff	
	0155		Predic Diff	
3.30 Mean	1.032 1.024 1.028		0111	
C.ØØ Mean	0.55 1.032 1.024 1.028 Ø.889	2.5	Ø.1ØÀ -2.4À	
3.30 Mean 3.10	0.55 1.032 1.024 1.028 Ø.889	2.5	Ø.1ØÀ -2.4À 0.0Ø8	
3.30 Mean	0.554 1.024 1.028 Ø.889 -Ø.ØØ2 0.9Ø6 -0.019	2.5	Ø.1ØÀ -2.4À	
0.00 Mean 0.10 Mean	0.528 0.889 -Ø.0028 0.906 -0.906 -0.901	2.5	Ø.1ØÀ -2.4À Ø.08 -20.7 Ø.09À -11.9	
0.00 Mean 0.10 Mean	0.528 0.889 -Ø.0028 0.906 -0.906 -0.901	2.5	Ø.1ØÀ -2.4À Ø.08 -20.7 Ø.09À -11.9	
c.co Mean c.lo Mean	0.528 0.889 -Ø.0028 0.906 -0.906 -0.901	2.5	Ø.1ØÀ -2.4À Ø.0Ø8 -20.7 Ø.09À -11.9	
0.00 Mean 0.10 Mean	0.889 -0.000 0.889 -0.000 0.000 0.000 0.000 0.000 0.000	2.5	Ø.1ØÀ -2.4Å Ø.Ø8 -20.7 Ø.Ø9Å -11.9	
c.co Mean c.lo Mean	0.528 0.889 -Ø.0028 0.906 -0.906 -0.901	2.5	Ø.1ØÀ -2.4À Ø.0Ø8 -20.7 Ø.09À -11.9	
C.30 Mean C.10 Mean 1.00	######################################	2.5	Ø.1ØÀ -2.4À Ø.0Ø8 -20.7 Ø.09À -11.9 -11.9 -11.9 -11.9 -11.9	
C.30 Mean C.10 Mean 1.00	0.528 0.889 -Ø.0028 0.906 -0.906 -0.901	2.5	Ø.1ØÀ -2.4À Ø.0Ø8 -20.7 Ø.09À -11.9	

Data Reduction

Method of transformation for data. Example, Ln refers to the natural log of the concentration and LgtB refers to the logit function of the absorbance divided by the absorbance at zero concentration.

Equation of Line

These values are the coefficients which describe a "best fit" or linear regression straight line where Logit (B/B_0) = slope x Loge (conc. in ppb) + intercept. The Corr(r) is the correlation coefficient which indicates "goodness of fit" of the data to the best fit line. The square of this value represents the proportion of variance (on the y axis) that is explained by the linear regression.

Transformed Data

This section shows the average "transformed data" for each standard point. For example, $Log_{\rm C}$ (0.1 ppb) = -2.30 $Log_{\rm C}$ (0.897 or \underline{B}) = 1.026 \underline{B} 0

Calibrator Data

0.889 = observed absorbance
0.10 = observed concentration
-0.002 = known conc.(0.10) observed conc.(0.10)*
-2.4 = concentration diff (-0.002) '
observed conc. (0.10) x 100*
1.4 = standard deviation of observed
absorbances ' mean (0.897) x
100
4.2 = coefficient of variation (%CV) is
calculated using absorbances

^{*}For accuracy, the data reduction software of the RPA-I utilizes seven significant digit numbers although only three are displayed or printed.

Control lata :		•
Ctrl# Abs	Conc	
. 1 2.274	2.93	
ID:		
 Samples Data : 		
Spl# Abs	Conc CV	
1 0.492 0.460 Mean 0.471	1.02 1.13 1.08 7.3Ã	
ID:		
2 Ø.36Ø	1.54	
0.368 Mean 0.364	1.54 1.76 1.3Ø 3.Ø	
ID:		
3 0.925 Ø.93Ø Mean 0.929	თ.თ7 თ.თ6	
Mean Ø.929	3.26 4.7	
ID:		
4 Ø.991 Ø.998 Mean Ø.995	Ø.Ø2 nd À Ø.Ø1nd	
Mean Ø.995	Ø.Ø2nd 17 .7*À	
ID:		
3 0.407 0.711 Mean 0.904	ວ.ສ6 ວ.ສາ ສ.ສ7 5.4	
Mean 0.924	5.4 5.97 5.4	
ID:		
6 Ø.23Ø Ø.233 Mean Ø.232	3.86 3.78 3.92 1.4	
Mean 3.232	3.92 1.4	
[ID:		
7 1.239 1.236 Mean 1.237	nd À nd	
Mean 1.237	nd	
TD: END OF RUN		
Ø4-12-91 12:39	9:24	

Control Data

Displays absorbance and concentration of control sample. This concentration should be compared to the reported range located on the control yial label to assure the quality of the run.

Sample Data

7.3 = this %CV is calculated using the sample concentrations

"nd" indicates concentration below the "Normal Range Low" value entered during the protocol setup. This value is the least detectable dose (LDD) for RaPID Assay protocols.

"*" indicates the %CV exceeds the parameter setting limit.

An "nd" without a concentration indicates the absorbance measured is greater than the absorbance of the 0 ppb standard therefore a concentration cannot be calculated.

SECTION 2 - RPA-III" ANALYZER

The RPA-III RaPID Analyzer is a hand-held microprocessor based unit with fully interchangeable filters for wavelength selection. The instrument features a liquid crystal display and is powered through a cable connected to an electrical outlet. By itself, the RPA-III does not provide a printout of results, nor does it perform mathematical functions. For a complete and detailed description of the RPA-III, please refer to the RPA-III RaPID Analyzer Operations Manual (Part No. A00101).

UNPACKING AND INSTALLATION

- 1. Inspect the package for visible signs of damage and note the condition of the carton. If damage has occurred, or a part is missing, immediately contact SDI.
- 2. Open the carton and remove the photometer and power cord from the packing material.
- 3. Plug the square end of the power cord into a grounded 110 v AC outlet. Insert the other end into the back of the RPA-III.
- 4. The unit is turned off by unplugging the power cable from the unit.

SHORT OPERATING PROCEDURE FOR THE RPA-III

Before reading tubes, allow five minutes after powering the RPA-III for warm up. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter.

•	INSTRUMENT DISPLAY	OPERATOR RESPONSE
	STANDARDIZE? Y/N	Press the Zero/No button.
	ZERO BLANK	Insert the blank tube containing at least 1 mL of Washing Solution and press the Zero/No button.
	READ	The RPA-III reads the blank tube and zeros the instrument (NOTE: for optimum performance, re-zero the unit after every ten readings. To re-zero, insert the blank and press the Zero/No button twice.)
	00 0.000 ABS.	The unit displays the reading number and the absorbance. Remove tube.
	READ SAMPLE	Insert the first tube and press the Read/Yes button.
	01 X.XXX ABS.	The tube is read and the absorbance is displayed. RECORD THE ABSORBANCE VALUE.
	02 X.XXX ABS.	Repeat for all standards, control, and samples.

CALCULATIONS

Using the graph paper provided with the RaPID Assay Kit, draw the standard curve by plotting the B/B_O ratios versus concentrations using the absorbance data obtained above. Graph papers are specific for each method. Use only the graph paper supplied with each kit.

The mean absorbance value for the 0 standard is the $B_{\rm O}$. The mean absorbance value for the other calibrators is the B value. Divide the absorbance of the standard, control or sample by the zero absorbance and multiply by 100 to obtain the % B/B_O. Draw the best straight line through all standard %B/B_O points (%B/B_O - y-axis, concentration - x-axis). Using the 9 %B/B_O of the sample interpolate the concentration using the standard curve.

SECTION 3 - RPA-III" FIELD KIT

The RPA-III RaPID Field Kit consists of the RPA-III Analyzer: a hand-held microprocessor based unit with liquid crystal display, and a portable case which contains a rechargeable battery, printer and tube cover. The Field kit also contains a power cord for battery charging and optional AC operation. The RPA-III Field Kit is intended for use outside of the laboratory environment.

For a complete and detailed description of the RPA-III. please refer to the RPA-III RaPID Analyzer Operations Manual (Part No. A00101).

INSTRUMENT DISPLAY

UNPACKING AND INSTALLATION

- 1. Seat the RPA-III firmly in the case making sure the battery/printer connection is inserted into the photometer.
- 2. Assuming the battery is charged, turn on both the printer and the RPA-III via the two power toggle switches.

Refer to the RPA-III Operating Manual for complete and detailed descriptions of operations.

OPERATOR RESPONSE

SHORT OPERATING PROCEDURE FOR THE RPA-III FIELD SYSTEM

Allow the RPA-III to warm up for five minutes after switching it on before reading tubes. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter.

OPERATOR RESPONSE
Press the Zero/No button.
Insert the blank tube containing at least 1 mL of Washing Solution. Place the tube cover over the tube and seat it firmly on the reader. Press the Zero/No button.
The RPA-III reads the blank tube and zeros the instrument (NOTE: for optimum performance, re-zero the unit after every ten readings. To re-zero, insert the blank and press the Zero/No button twice.)
The unit displays the reading number and the absorbance. Remove tube.
Insert the first tube. Place the tube cover over the tube. Press the Read/Yes button. The absorbance is displayed and printed.
The tube is read and the absorbance is displayed. RECORD THE ABSORBANCE VALUE.
Insert the next tube. Place the tube cover over the tube and press the Read/Yes button. The absorbance is displayed and printed.

CALCULATIONS

Refer to the appropriate RPA-III Field Protocol found in the RPA-III RaPID Analyzer Operations Manual for directions on

Calculations and Interpretation of Results for qualitative or semi-quantitative results.

SECTION 4 - OTHER SPECTROPHOTOMETERS

REQUIREMENTS

This section describes the requirements for use of RaPID Assays with tube readers other than the RPA series photometers. These requirements are:

Absorbance Range: 0 - 2.0 AU

-Wavelength: 450 nm.

Detection volume: 1 mL in cuvettes.

Drift: 9 0.005 AU per hour at 0 AU.

Linearity: 9 0.005 A or 2% difference from calculated regression line. Correlation coefficient, r = 0.9995 or better.

Results reporting: to a display or printer.

Sipper cell: the operator must first establish pump cycle times that allow for 1 mL to be drawn up from the tube and provide delivery to the flow cell to be read. Once this has been established, a return cycle of two times the pick-up volume

should be returned to avoid cross-over contamination.

Throughput: ability to read the tubes within 15 minutes after the addition of stop solution to the assay tube.

CALCULATIONS

Using the graph paper provided with the RaPID Assay Kit. draw the standard curve by plotting the B/B_O ratios versus concentrations using the absorbance data obtained above. Graph papers are specific for each method. Use only the graph paper supplied with each kit.

The mean absorbance value for the 0 standard is the B_O. The mean absorbance value for the other calibrators is the B value. Divide the absorbance of the standard, control or sample by the zero absorbance and multiply by 100 to obtain the % B/B_O. Draw the best straight line through all standard %B/B_O points (%B/B_O - y-axis, concentration - x-axis). Using the %B/B_O of the sample interpolate the concentration using the standard curve

SECTION 5 - FIXED VOLUME PIPETTES

ASSEMBLY

Secure a tip on the pipette nose by pressing the nose cone **firmly** into a tip contained in the pipette tip rack.

USE

General

The piston stroke is divided into three sections: measuring, blow-out, and tip ejection.

First stop: required volume is defined and dispensed

Second stop: dispense any residual liquid

Third stop: tip ejection

Practice several pipette transfers with water using the following procedure:

Filling

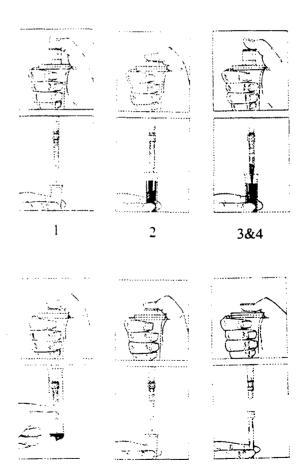
- 1. Keep pipette almost vertical.
- 2. Press button down to the first stop. Immerse tip 2-3 mm into the liquid.
- 3. **Slowly** allow the button to glide back (never let it snap back).
- 4. Slide tip out along the inside of the vessel.

Dispensing

- 5. Insert pipette almost to the bottom of the test tube. Touch the tip to the side of the test tube about 5 mm above the dispensed liquid. Press button down to the second stop.
- 6. Remove pipette from the test tube.
- Press button down to the third stop and discard the tip.

IMPORTANT: A new tip should be used for <u>each</u> standard or sample.





5

SECTION 6 - TRI-VOLUME PIPETTES

ASSEMBLY

Secure a tip on the pipette nose by pressing the nose cone **firmly** into a tip contained in the pipette tip rack.

USE

The tri-volume pipette will deliver the volume displayed opposite the red mark.

To select the volume to be delivered, press the button down to the horizontal mark (middle of the numbers) in the volume ring (e.g. 100--200--250) and turn to the right or left. The button must be returned to the fully extended position (numbers visible) to lock the required volume into place.

General

The piston stroke is divided into three sections: measuring, blow-out, and tip ejection.

First stop: required volume is defined and dispensed.

Second stop: dispense any residual liquid.

Third stop: tip ejection

Practice several pipette transfers with water using the following procedure:

Filling

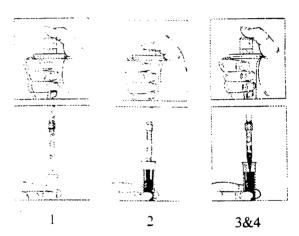
- 1. Keep pipette almost vertical.
- 2. Press button down to the first stop. Immerse tip 2-3 mm into the liquid.
- 3. **Slowly** allow the button to glide back (never let it snap back)
- 4. Slide tip out along the inside of the vessel.

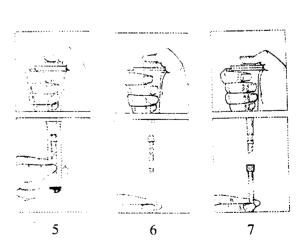
Dispensing

- 5. Insert pipette almost to the bottom of the test tube. Press button down to the second stop.
- 6. Remove pipette from the test tube.
- 7. Press button down to the third stop and discard the tip.

IMPORTANT: A new tip should be used for each standard or sample.





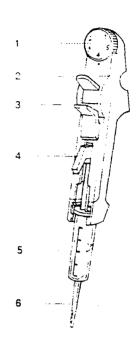


SECTION 7 - REPEATER PIPETTES

ASSEMBLY

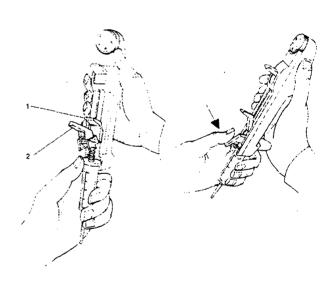
DESIGN PRINCIPLE:

- 1. Volume Selection Dial: To determine the pipette volume, the dial setting (1-5) is multiplied by the minimum pipette volume of the reservoir. See example under Reservoir/Use of the Repeater Pipette.
- 2. Pipette Lever: To deliver the selected volume, press this lever down until it stops.
- 3. Filling Lever: To fill the reservoir, slide this lever upward.
- 4. Locking Clamp: The locking clamp serves to firmly clamp the reservoir into the pipette.
- 5. Reservoir
- 6. Reservoir Cone



INSERTING A RESERVOIR:

- 1. Slide the filling lever (1) down until it stops.
- 2. Raise the locking clamp (2) upward.
- 3. Insert the reservoir <u>until it clicks</u> into position. Be sure the reservoir plunger is fully inserted into the barrel and the filling lever is completely down before attaching the reservoir to the pipette.
- 4 Lower the locking clamp to secure the reservoir in place.



USE

RESERVOIR:

The minimum pipetting volume and the maximum filling capacity are shown on each reservoir. The volume to be pipetted is obtained by multiplying the set number located on the volume selection dial by the minimum pipetting volume of the reservoir.

Example: 12.5 mL Reservoir: Maximum fill volume = 12.5 mL Minimum pipetting volume. Dial set to 1 = 250 μ L At dial setting 4: 4 x 250 μ L = 1000 μ L Pipetted volume: 1000 μ L

Practice several pipette transfers with water using the following procedure:

FILLING:

- 1. Immerse the reservoir cone into the liquid.
- 2. Fill by **slowly** sliding the filling lever upward.
- 3 Discard the first pipetting step by completely depressing the pipetting lever with a smooth, continuous motion until it stops.
- 4. Allow the pipetting lever to return to its starting position.

The repeater pipette is now ready for operation.

ADDITIONAL FILLING PRECAUTIONS:

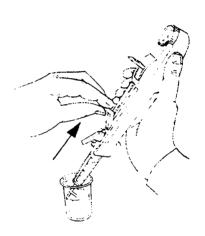
Sliding the filling lever too quickly can cause excessive vacuum. This can cause tiny air bubbles to accumulate in the liquid which may lead to pipetting inaccuracies. If this occurs, empty and refill the reservoir.

It is important to discard the first pipetting step in order to release any residual pressure from the pipetting system after filling and to prepare the system for precise pipetting.

Small bubbles in the reservoir beneath the piston do not affect pipetting accuracy due to an incorporated residual stroke lock which prevents the pipetting of any residual material after the last dispensing.

It is not necessary to completely fill the reservoir. Partially filling reservoirs does not affect pipetting accuracy.





PIPETTING:

A set of five labeled 12.5 mL (1=250 μ L) reservoirs is needed for each RaPID Assay. They should be labeled with the assay name and the following: "conjugate 250 μ L", "particles 500 μ L", "wash 1 mL", "color 500 μ L", and "stop 500 μ L".

1. Check the volume selection dial for the appropriate setting.

Enzyme conjugate A set dial at 1

Magnetic particles A set dial at 2

Wash solution A set dial at 4

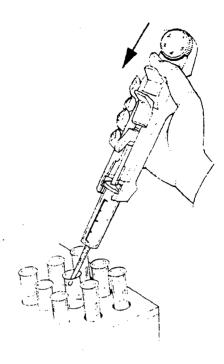
Color reagent A set dial at 2

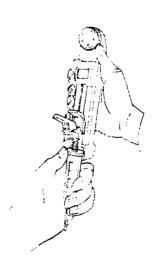
Stop reagent A set dial at 2

- 2. Hold the repeater at an angle so the tip is about one half inch below the tube rim without touching the rim or tube wall with the reservoir cone and aim the cone to deliver the liquid down the inside wall of the test tube.
- 3. Dispense the liquid by completely depressing the pipetting lever with a smooth, continuous motion until it stops.
- 4. Allow the pipetting lever to return to its starting position and repeat delivery into the next tube.
- 5. After the pipetting is completed, return the unused reagent to its container by holding the repeater unit over the container and pressing the filling lever down until it stops.
- 6 Prior to storage of the reservoirs, rinse the dedicated reservoirs twice with distilled water (keeping the reservoir in the pipette) by filling with 12.5 mL of distilled water each time and discarding the contents into a sink. Store syringes assembled (plunger inserted into barrel). Keep washed assembled syringes separated from each other. (Hint: An empty tray from the fixed volume pipette tips makes a handy storage rack). Reservoirs should be changed periodically (after 5-10 uses) since precision deteriorates with use

REMOVING THE RESERVOIR:

Once the filling lever (1) is completely down, raise the locking clamp (2) upward and remove the reservoir.





SECTION 8 - MAGNETIC RACK

USE

The magnetic rack is composed of two parts: the top rack that firmly holds the test tubes in place and the bottom base which contains the magnets.

ASSEMBLY

Place the rack on top of the base making sure they fit together securely and the test tubes are pushed down as far as they will go into the base.

USE OF THE ASSEMBLED RACK

For separation steps (washing and decanting) - to pull the magnetic particles to the sides of the tubes allowing the unbound components to be discarded.

DISASSEMBLY

Separate the top rack from the bottom base.

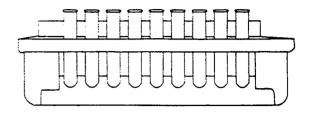
USE OF THE DISASSEMBLED RACK

For incubation steps - to allow the magnetic particles to remain suspended throughout the solution.

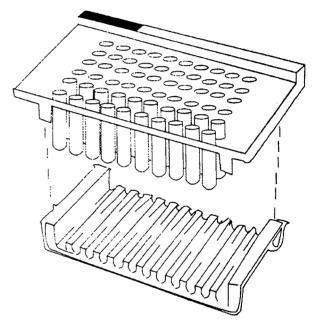
CARE

Do not submerge base in water. Clean with warm water and mild detergents.

CAUTION: Exposure to extreme heat or organic solvents could effect product's performance. Do not under any circumstances attempt to disassemble base to observe magnets. Magnets will be violently attracted to each other.



Assembled View



Disassembled View

SECTION 9 - START-UP

MATERIALS NECESSARY TO PERFORM A RAPID ASSAY

- 1. RaPID Assay test kit with assay protocol. flowchart and package insert.
- 2. RPA-I, RPA-III with operating instructions (or equivalent spectrophotometer capable of reading 450 nm in a 1 mL sample size).
- 3. Magnetic rack separation unit.
- Precision pipettes and appropriate tips, capable of delivering 100 μL, 200 μL, or 250 μL depending on method.

- 5. Repeating pipette, with appropriate reservoirs, capable of delivering 250 μ L, 500 μ L, and 1000 μ L.
- 6. Thermolyne Maxi MixTM, Vortex GenieTM (or equivalent vortex mixer).
- 7 Blotting paper for decanting steps (usually use a stack of 5 kitchen type paper towels).
- 8. Permanent marking pens for labeling tubes.
- 9 Instructional video
- 10. Laboratory timer or wrist watch.

ASSAY TIPS

Gently blot. Avoid banging the rack during the decanting steps.

Decant with the magnetic base joined to the top rack.

Wait a full two minutes for each separation step.

Bring samples and reagents fully to room temperature.

Vortex when specified without foaming.

Pay attention to the number of washes (2) and the amount of wash solution added (1 mL).

Add color reagent without the magnetic base joined to the top rack.

Remove as much liquid as possible from the test tubes when decanting.

Assure precise pipetting techniques.

Swirl the magnetic particles, without foaming, prior to addition.

PREPARATION AND RUNNING

Prior to performing your first RaPID Assay please do the following:

- 1. Read the <u>entire</u> package insert (found inside the box of reagents).
- 2. View the videotape (if available).
- Gather together all the materials necessary to perform a RaPID Assay in a work space.
- 4 Remove the RaPID Assay kits from the refrigerator.
- 5. Remove the bottles from the kit box and place them on a countertop.
- Allow the reagent bottles to come to room temperature.
- 7. Turn on the RPA-I or other spectrophotometer. The RPA-I should be warmed-up for 30 minutes.
- 8. Practice using the fixed volume and repeater pipettes with distilled water.
- 9. Label the top portion of the test tubes, with a permanent marker, in the following manner starting with the standard curve (in duplicate) followed by a control tube and sample tubes (in singlet).

Tube#	Contents of Tube
1,2	Zero Standard
3.4	Standard 1
5.6	Standard 2
7,8	Standard 3
9	Control
10	Sample #1
11	Sample #2
12	Etc.

10. Label 5 - 12.5 mL (1 = 250ml) reservoirs, with a permanent marker, with the following: the first reservoir = conjugate 250μL the second reservoir = particles 500μL the third reservoir = wash 1ml the fourth reservoir = color 500μL the fifth reservoir = stop 500μL

In addition, add the name of the pesticide you are testing for to each syringe.

11. Continue with the **Assay Procedure** section of the package insert or flowchart.

Expected Results for RaPID Assays

- %CVs between standard duplicates of 10% or less.
- Absorbance readings for the 0 ppb standard greater than or equal to 0.800 for all assays.
- Corr (r) of 0.990 or greater for all assays.

SECTION 10 - ASSAY WORKSHEET

PURPOSE OF WORKSHEET

To aid in your evaluation of the assay kit, this familiarization run (a typical calibration curve plus proficiency samples) should be performed. Operators should repeat this run until the %CV of absorbances are consistently better than 10%. Experienced operators will obtain %CV's approaching 5% or better. The concentration values obtained on the control and the proficiency samples should be within the stated ranges.

FAMILIARIZATION RUN

Include in the run each standard in duplicate, a single control, and each proficiency sample in duplicate. Following the instructions in the kit package insert and found elsewhere in this manual, perform the assay.

RESULTS

RPA-I:

- Observe the absorbance, the absorbance %CV, and the predicted result for each standard from the RPA-I printout (see Section 1 for a description of the printout).
- 2. Calculate the mean, SD, and %CV for the results from the standards and proficiency samples using a statistical calculator or the formulas given below the worksheet. Record in the following worksheet. Note that the %CV on the results may be significantly different than the absorbance %CV.

3. Compare to the above guidelines.

RPA-III or other photometers:

- 1 Record the absorbance value for each standard or sample in the worksheet which follows
- 2. Calculate the mean, standard deviation (SD), and %CV on the absorbances for each standard and sample using a statistical calculator or the formulas given below the worksheet.
- 3 Using the graph paper provided with the kit, prepare a standard curve as described in the "Results" section of the kit package insert under "Manual Calculations". Read all samples and calibrators as individual points from the standard curve. Use the worksheet which follows to record the results.
- 4. Calculate and record the mean, SD, and %CV for the results using a statistical calculator or the formulas given below the worksheet.
- 5. Compare to the above guidelines.

INTERPRETATION

If you run two curves and do not achieve acceptable %CV's on the absorbances call SDI's Technical Support (1-800-544-8881) to discuss your results.

NAME:	<u> </u>
DATE:	
METHOD:	
LOT NUMBER:	
EXPIRATION DATE:	

Tube #	Contents	Absorbance	%CV of Absorbance	Result [ppb]	% CV of Result
Ē				·	
Ī	() ppb Standard				
2	() ppb Standard				
3	Standard #1				
4	Standard #1				
5	Standard #2				
6	Standard #2				
7	Standard #3				
8	Standard =3				
9	Control				٠.
10	Sample A				
11	Sample A				
12	Sample B				
13	Sample B				
14	Sample C				
15	Sample C				

Calculations -

 $n = \pi$ of samples Mean: $\overline{X} = \sum X/n$

Standard Deviation (S.D.): = $\sqrt{(\Sigma(x - \overline{x})^2/(n - 1))}$

Percent Coefficient of Variance (%CV): = $(S.D./\bar{x}) \times 100$

SECTION 11 - TROUBLESHOOTING

Symptom	Cause	Corrective Action
Increased Absorbance	Long incubation.	Adhere to incubation times.
	Washed tubes only once.	Be sure to wash tubes twice.
	Washed tubes with less than 1 mL of Washing Solution.	Check pipette for delivery of 1 mL of Washing Solution.
	Warm reagents.	Bring reagents to room temperature.
Decreased Absorbance	Banging rack during decanting.	Gently blot tubes, don't bang.
	Decanting without top rack joined to magnetic base.	Join rack and base prior to decanting.
1	Short incubation. Adhere to incubation times.	
	Did not wait 2 minutes between washings.	Allow particles to separate between washings.
	Cold reagents.	Bring reagents to room temperature.
	Did not vortex after addition of color reagent or particles. Vortex tubes after the addition reagent and particles.	
	Addition of color reagent while top rack is joined to magnetic base.	Separate rack from base before adding color reagent.
Higher than expected results	Presence of cross reactants, particulate matter, or other interferences in the sample.	For particulate matter, filter samples with a 0.2 µm filter and re-assay. For cross reactants and possible interfering substances, dilute sample and reassay.
	Inaccurate standard curve.	Re-run standard curve.
:	Expired reagents and/or kit.	Discard and replace with a fresh kit.
	Drift in sample results from beginning to end of run.	Add all reagents in a consistent manner to entire rack within one minute.
Lower than expected results	Standards contaminated with analyte.	Discard and replace with a fresh kit.
I	Inaccurate standard curve.	Re-run standard curve.
	Expired kit.	Discard and replace with a fresh kit.

Symptom	Cause	Corrective Action
Increased % CV's	Banging rack during decanting.	Blot, don't bang.
	Decanting without top rack joined to magnetic base.	Join rack and base prior to decanting.
	Did not vortex after addition of color reagent or particles.	Vortex tubes after the addition of color reagent and particles.
	Addition of 2 mL of wash solution instead of 1 mL	Check pipette for delivery of 1 mL of liquid.
	Forgot to wait 2 minutes for separation.	Allow particles to separate between washings.
	Excessive wash solution remaining in tubes after decanting (may appear as bubbles).	After decanting, while holding top rack and bottom base together, allow tubes to drain in an inverted position for a few minutes. Also obtain more absorbent toweling.
	Imprecise addition of reagents.	Replace reservoir. Pipettes may need maintenance.
	Neglecting to vortex during the wash steps in the PCB procedure.	In the PCB RaPID Assay after adding the Washing Solution to each tube, vortex each tube for 1-2 seconds.

Attachment E

05-31-02 18:14:33	

PROTOCOL : CARC PAH	Samples Data :
TECH ID :	Spl# Abs Conc
Anta Daductilin Pagracsian	1 1.083 9.81nd
Xformation: Ln/L9tB Read Mode : Absorbance	ID:
Xformation: Ln/L9t8 Read Mode : Absorbance Wavelen9th : 450 nm Units : PPB	2 1.049 18.33
	ID:
EQUATION OF LINE:	3 1.080 10.47
Slope = -0.944 Intercept = 5.332 Corr (r) = 0.9963	ID:
Corr (r) = 0.9963	4 0.599 248.53
Transformed Data :	ID:
Conc Abs	5 1.040 20.73
	••-
7.01	6 1.048 18.69
į	ID:
Calibrator Data:	
Conc Abs XCV Predic	7 1.081 10.31
Conc Abs ZCU Predic Diff %Diff	ID:
0.00 1.108 1.148	8 1.034 22.42
Mean 1.128 2.6	ID:
10.00 1.085 9.31 -0.686 -7.4 1.085 9.15	9 1.088 8.50nd
11000	ID:
Mean 1.085 0.1 9.23 -0.768 -8.3	10 1.052 17.60
189.89 8.794 113.16 -	ID:
13.161 9.763 130.09	11 1.023 25.51
30.093 23.1 Mean 0.779 2.9 121.44	ID:
21.439	12 1.065 14.22
500.00 0.449 4 39. 98 -60.023 -13.6 0.442 4 52. 07	ID:
_47 931 -18.6	13 1.014 28.07
Mean 0.445 1.1 445.98 -54.025 -12.1	ID:
	14 1.065 14.22
Control Data:	ID:
Ctrl# Abs Conc	15 1.028 23.95
1 9.650 295.22	
	ID:

	1.012		-
17	1.034		
	1.061	15.28	
	1.004	30.91	
_	1.037	21.48	•
	 0.882	73.35	-
ID:-			-
			-
END 05-	OF RUN 31-02 18	:19:41	

		•				
****** 5 D	I ***	*****	·	•		
PROTOCOL: CA	RC PAI	H				
TECH ID : LOT # : EXP DATE:					amples Data	
Data Reduct:Lir Xformation: Read Mode : Wavelength : Units :	.Regr	ession	-	10	1 0.626	285.98
EQUATION OF LIM				*	2 0. 976	
Slope = Intercept = Corr (r) =	-1.02 5.86 0.994	3 9 0	·		3 1.105	
Transformed Dat					4 1.111	26.71
2.30 3.6 4.61 0.9 6.21 -0.3				·	: 5 1.030 :	53.92
Calibrator Data					6 1.119	
Conc Abs Diff	2CV	Predic %Diff			7 1.110	27.14
0.00 1.225 1.177 Mean 1.201	2.8	_			:B 1.111	26.71
10.00 1.168 -0.472 1.172 -1.468 Mean 1.170 -0.970	0.2	9.53 -5.0 8.53 -17.2 9.03		•	1.140	
100.00 0.846 32.942		132.94 24.8			1.138	
0.865 23.468 Mean 0.855 28.156		123.47 19.0 128.16 22.0			1.135	
500.00 0.513 -85.947 0.488 -49.001 Mean 0.500 -67.935	3.6	414.05 -20.8 451.00 -10.9 432.06 -15.7		END) OF RUN -31-02 16:5	
Control Data :						
Ctrl# Abs	Conc					

1 0.704 221.10

ID:_____

Attachment F

Kerr McGee Moss American KMA14

PAHs - SW846-8310

Lab Sample No.	Sample Location MA6-SSRR	Date Collected	Date Analyzed
3829105	255	5/31/02	6/14/02
3829108	309	5/31/02	6/14/02
3829109	305	5/31/02	6/14/02
3829110	309DP	5/31/02	6/14/02
3829111	261	5/31/02	6/14/02
3829112	273	5/31/02	6/14/02
3829113	265	5/31/02	6/14/02
3829114	299	5/31/02	6/14/02
3829115	295	5/31/02	6/14/02
3829116	291	5/31/02	6/14/02
3829117	303	5/31/02	6/14/02

1. Holding Time:

All samples were extracted on 6/4 and analyzed on 6/14/02. All holding times were acceptable.

2. Method Blanks:

The was one method blank associated with the samples. Results were free of contamination.

3. Surrogate Recovery:

All surrogate recoveries were within control limits.

4. Laboratory Control Sample:

All LCS recoveries and RPDs were within control limits.

5. Matrix Spike/Matrix Spike Duplicate:

Sample 255 was used as the MS/MSD. The benzo(b)fluoranthene RPD was high (71) outside control limits. Based on an acceptable MS/MSD and LCS/LCSD recovery, no qualifications are required.

6. Calibration:

Calibration data showed acceptable recoveries.

Date Reviewed by: T. Balla

Date: 7/1/02



Lancaster Laboratories Sample No. SW 3829105

Collected: 05/31/2002 13:35 by V/D Account Number: 07802

Submitted: 06/01/2002 10:25 Reported: 06/17/2002 at 15:35

Discard: 05/17/2002 at 15:39

MA6-SSRR-255 Unspiked Grab Soil Sample

Moss American /WI

Kerr-McGee Corporation
P.O. Box 25861

Oklahoma City OK 73125

MA255 SDG#: KMA14-01BKG

					Dry		
CAT			Dry		Method		Dilution
No.	Analysis Name	CAS Number	Result		Detection Limit	Units	Factor
00111	Moisture	n.a.	30.7		0.50	% by wt.	1
	"Moisture" represents the loss	in weight of the	ne sample	e after o	ven drying at		
	103 - 105 degrees Celsius. The	result reported	d above :	is on an	as-received		
	basis.						
				•			
00941	PAH's in Solids by HPLC						
00942	Naphthalene	91-20-3	N.D.		300.	ug/kg	5
00974	Fluorene	86-73-7	N.D.		30.	ug/kg	5
01014	Benzo(a)anthracene	56-55-3	50.		10.	ug/kg	5
01016	Chrysene	218-01-9	70.	J	10.	ug/kg	5
01017	Benzo(b) fluoranthene	205-99-2	90.	J	20.	ug/kg	5
01018	Benzo(k) fluoranthene	207-08-9	40.	J	10.	ug/kg.	5
01019	Benzo(a)pyrene	50-32-8	70.	J	10.	ug/kg	5
01022	Dibenz(a,h)anthracene	53-70-3	N.D.		10.	ug/kg	5
01025	Indeno(1,2,3-cd)pyrene	193-39-5	90.	J	20.	ug/kg	5
01026	Benzo(g,h,i)perylene	191-24-2	160.		20.	ug/kg	5
	Due to the high concentration of	f non-target co	mpounds,	, a dilut:	ion was		
	necessary to perform the PAH by	HPLC analysis	There	fore, the	reporting		
	limits for the HPLC PAH compound	ds were raised	•				

Laboratory Chronicle

CAT	CAT Analysis					Dilution
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:26	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 01:56	Mark A Clark	5
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1

-7-



Questions? Contact your Client Services Representative Carrie A Fleming at (717) 656-2300.

Respectfully Submitted,

Rachel R. Cochis Rachel R. Cochis Sr. Chemist/Coordinator





Lancaster Laboratories Sample No. SW 3829107

Collected: 05/31/2002 13:35 by V/D Account Number: 07802

 Submitted: 06/01/2002 10:25
 Kerr-McGee Corporation

 Reported: 06/17/2002 at 15:35
 P.O. Box 25861

Discard: 07/18/2002 Oklahoma City OK 73125

MA6-SSRR-255 Matrix Spike Dup. Grab Soil Sample

Moss American /WI

MA255 SDG#: KMA14-01MSD

				Dry		
CAT			Dry	Method		Dilution
No.	Analysis Name	CAS Number	Result	Detection Limit	Units	Factor
00118	Moisture	n.a.	30.7	0.50	% by wt.	1
00121	Moisture Duplicate	n.a.	30.4	0.50	% by wt.	1
	The duplicate moisture value is moisture test. For comparabilidetermination is the value used	ty purposes, t	he initial moistu	ıre		
00941	PAH's in Solids by HPLC					
00942	Naphthalene	91-20-3	9,000.	300.	ug/kg	5
00974	Fluorene	86-73-7	900.	30.	ug/kg	5
01014	Benzo(a) anthracene	56-55-3	110.	10.	ug/kg	5
01016	Chrysene	218-01-9	320.	10.	ug/kg	5
01017	Benzo(b)fluoranthene	205-99-2	120.	20.	ug/kg.	5
01018	Benzo(k)fluoranthene	207-08-9	90.	10.	ug/kg	5
01019	Benzo(a)pyrene	50-32-8	120.	10.	ug/kg	5
01022	Dibenz(a,h)anthracene	53-70-3	150.	10.	ug/kg	5
01025	Indeno(1,2,3-cd)pyrene	193-39-5	320.	20.	ug/kg	5
01026	Benzo(g,h,i)perylene	191-24-2	650.	20.	ug/kg	5
	Due to the high concentration on necessary to perform the PAH by	_	-			

Laboratory Chronicle

CAT	CAT			Analysis			
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor	
00118	Moisture	EPA 160.3 modified	1	06/05/2002 19:26	Justin M Bowers	1	
00121	Moisture Duplicate	EPA 160.3 modified	1	06/05/2002 19:26	Justin M Bowers	1	
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 03:20	Mark A Clark	5	
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1	



Lancaster Laboratories Sample No. SW 3829106

Collected: 05/31/2002 13:35 by V/D

Submitted: 06/01/2002 10:25 Reported: 06/17/2002 at 15:35

Discard: 07/18/2002

MA6-SSRR-255 Matrix Spike Grab Soil Sample

Moss American /WI

MA255 SDG#: KMA14-01MS

Account Number: 07802

Kerr-McGee Corporation

P.O. Box 25861

Oklahoma City OK 73125

				Dry		
CAT			Dry	Method		Dilution
No.	Analysis Name	CAS Number	Result	Detection Limit	Units	Factor
00118	Moisture	n.a.	30.7	0.50	% by wt.	1
00941	PAH's in Solids by HPLC					
00942	Naphthalene	91-20-3	9,000.	300.	ug/kg	5
00974	Fluorene	86 - 73-7	900.	30.	ug/kg	5
01014	Benzo(a)anthracene	56-55-3	90.	10.	ug/kg	5
01016	Chrysene	218-01-9	300.	10.	ug/kg	5
01017	Benzo(b)fluoranthene	205-99-2	100.	20.	ug/kg	5
01018	Benzo(k)fluoranthene	207-08-9	80.	10.	ug/kg	5
01019	Benzo(a)pyrene	50-32-8	100.	10.	ug/kg	5
01022	Dibenz(a,h)anthracene	53-70-3	140.	10.	ug/kg	5
01025	Indeno(1,2,3-cd)pyrene	193-39-5	300.	20.	ug/kg	5
01026	Benzo(g,h,i)perylene	191-24-2	610.	20.	ug/kg	5
	Due to the high concentration of	non-target o	compounds, a	dilution was	- -	

Laboratory Chronicle

necessary to perform the PAH by HPLC analysis. Therefore, the reporting

			CITE	111010		
CAT				Analysis		Dilution
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00118	Moisture	EPA 160.3 modified	1	06/05/2002 19:26	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 02:38	Mark A Clark	5
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1



Lancaster Laboratories Sample No. SW 3829109

Collected: 05/31/2002 13:30 by V/D Account Number: 07802

Submitted: 06/01/2002 10:25 Reported: 06/17/2002 at 15:35

Discard: 07/18/2002

MA6-SSRR-305 Grab Soil Sample

Moss American /WI

Kerr-McGee Corporation

P.O. Box 25861

Oklahoma City OK 73125

MA305 SDG#: KMA14-03

					Dry		
CAT			Dry		Method		Dilution
No.	Analysis Name	CAS Number	Result		Detection Limit	Units	Factor
00111	Moisture	n.a.	39.6		0.50	% by wt.	1
	"Moisture" represents the loss	in weight of t	he sample	after o	ven drying at		
	103 - 105 degrees Celsius. The	result reporte	d above i	s on an a	as-received		
	basis.						
00941	PAH's in Solids by HPLC						
00942	Naphthalene	91-20 - 3	N.D.		700.	ug/kg	10
00974	Fluorene	86-73-7	N.D.		70.	ug/kg	10
01014	Benzo(a) anthracene	56-55-3	200.		20.	ug/kg	10
01016	Chrysene	218-01-9	170.	J	30.	ug/kg	10
01017	Benzo(b)fluoranthene	205-99-2	200.		40.	ug/kg	10
01018	Benzo(k)fluoranthene	207-08-9	100.		20.	ug/kg	10
01019	Benzo(a)pyrene	50-32-8	200.		30.	ug/kg	10
01022	Dibenz(a,h)anthracene	53 - 70-3	20.	J	20.	ug/kg	10
01025	Indeno(1,2,3-cd)pyrene	193-39-5	300.		40.	ug/kg	10
01026	Benzo(g,h,i)perylene	191-24-2	500.		40.	ug/kg	10
	Due to the high concentration o	f non-target co	ompounds,	a diluti	on was		
	necessary to perform the PAH by						
	limits for the HPLC PAH compound	ds were raised					
	•						

CAT			•	Dilution		
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:26	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 04:44	Mark A Clark	10
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1



Lancaster Laboratories Sample No. SW 3829108

by V/D Account Number: 07802 Collected: 05/31/2002 13:30

Submitted: 06/01/2002 10:25 Kerr-McGee Corporation Reported: 06/17/2002 at 15:35 P.O. Box 25861 Oklahoma City OK 73125

Discard: 07/18/2002

MA6-SSRR-309 Grab Soil Sample

Moss American /WI

MA309 SDG#: KMA14-02

				Dry		
CAT			Dry	Method		Dilution
No.	Analysis Name	CAS Number	Result	Detection Limit	Units	Factor
00111	Moisture	n.a.	29.4	0.50	% by wt.	1
	"Moisture" represents the loss	in weight of t	he sample after o	ven drying at		
	103 - 105 degrees Celsius. The basis.	result reporte	d above is on an	as-received		
00941	PAH's in Solids by HPLC					
00942	Naphthalene	91-20-3	N.D.	1,000.	· ug/kg	20
00974	Fluorene	86 - 73-7	N.D.	100.	ug/kg	20
01014	Benzo(a)anthracene	56-55-3	1,800.	40.	ug/kg	20
01016	Chrysene	218-01-9	1,800.	60.	ug/kg	20
01017	Benzo(b)fluoranthene	205-99-2	2,000.	80.	ug/kg	20
01018	Benzo(k)fluoranthene	207-08-9	1,100.	40.	ug/kg	20
01019	Benzo(a)pyrene	50-32 - 8	2,000.	60.	ug/kg	20
01022	Dibenz(a,h)anthracene	53-70-3	200.	40.	ug/kg	20
01025	Indeno(1,2,3-cd)pyrene	193-39-5	2,000.	80.	ug/kg	20
01026	Benzo(g,h,i)perylene	191-24-2	2,900.	80.	ug/kg	20
	The surrogate data is outside t	he QC limits d	ue to unresolvabl	e matrix		

problems evident in the sample extraction.

Due to the high concentration of non-target compounds, a dilution was necessary to perform the PAH by HPLC analysis. Therefore, the reporting limits for the HPLC PAH compounds were raised.

CAT			•	Dilution		
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:26	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 04:02	Mark A Clark	20
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1





Lancaster Laboratories Sample No. SW 3829111

Collected: 05/31/2002 13:40 by V/D Account Number: 07802

Submitted: 06/01/2002 10:25 Kerr-McGee Corporation

Reported: 06/17/2002 at 15:35 P.O. Box 25861

Discard: 07/18/2002 Oklahoma City OK 73125 MA6-SSRR-261 Grab Soil Sample

Moss American /WI

MA261 SDG#: KMA14-05

					Dry		
CAT			Dry		Method		Dilution
No.	Analysis Name	CAS Number	Result		Detection Limit	Units	Factor
00111	Moisture	n.a.	26.1		0.50	% by wt.	1
	"Moisture" represents the loss :	in weight of t	he sample	e after o	ven drying at		
	103 - 105 degrees Celsius. The	result reporte	d above :	is on an	as-received		
	basis.						
00941	PAH's in Solids by HPLC						
	-						
00942	Naphthalene	91-20-3	N.D.		300.	ug/kg	5
00974	Fluorene	86-73-7	N.D.		30.	ug/kg	5
01014	Benzo(a)anthracene	56-55-3	22.	J	9.	ug/kg	5
01016	Chrysene	218-01-9	20.	J	10.	ug/kg	5
01017	Benzo(b) fluoranthene	205-99-2	30.	J	20.	ug/kg	5
01018	Benzo(k) fluoranthene	207-08-9	16.	J	9.	ug/kg .	5
01019	Benzo(a)pyrene	50-32 - 8	30.	J	10.	ug/kg	5
01022	Dibenz(a,h)anthracene	53 - 70-3	N.D.		9.	ug/kg	5
01025	Indeno(1,2,3-cd)pyrene	193-39-5	50.	J	20.	ug/kg	5
01026	Benzo(g,h,i)perylene	191-24-2	80.	Ĵ	20.	ug/kg	5
	Due to the high concentration of		ompounds.	. a dilut		J. J	
	•	3					

necessary to perform the PAH by HPLC analysis. Therefore, the reporting limits for the HPLC PAH compounds were raised.

CAT		_		Analysis		Dilution
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:26	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 06:09	Mark A Clark	5
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1

Kerr-McGee Corporation

Oklahoma City OK 73125

P.O. Box 25861



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Lancaster Laboratories Sample No. SW 3829110

Collected: 05/31/2002 13:30 by V/D Account Number: 07802

Submitted: 06/01/2002 10:25 Reported: 06/17/2002 at 15:35

problems evident in the sample extraction.

Discard: 07/18/2002

MA6-SSRR-309DP Grab Soil Sample

Moss American /WI

309DP SDG#: KMA14-04FD

				Dry		
CAT			Dry	Method		Dilution
No.	Analysis Name	CAS Number	Result	Detection Limit	Units	Factor
00111	Moisture	n.a.	31.5	0.50	% by wt.	1
	"Moisture" represents the loss	in weight of t	he sample aft	er oven drying at		
	103 - 105 degrees Celsius. The	result reporte	ed above is on	an as-received		
	basis.					
00941	PAH's in Solids by HPLC					
00942	Naphthalene	91-20-3	N.D.	1,000.	ug/kg	20
00974	Fluorene	86-73-7	N.D.	100.	ug/kg	20
01014	Benzo(a)anthracene	56-55-3	2,300.	40.	ug/kg	20
01016	Chrysene	218-01-9	2,400.	60.	ug/kg	20
01017	Benzo(b)fluoranthene	205-99-2	2,800.	80.	ug/kg	20
01018	Benzo(k)fluoranthene	207-08-9	1,400.	40.	ug/kg	20
01019	Benzo(a)pyrene	50-32-8	2,700.	60.	ug/kg	20
01022	Dibenz(a,h)anthracene	53-70-3	300.	40.	ug/kg	20
01025	Indeno(1,2,3-cd)pyrene	193-39-5	2,800.	80.	ug/kg	20
01026	Benzo(g,h,i)perylene	191-24-2	4,200.	80.	ug/kg	20
	The surrogate data is outside	the QC limits d	ue to unresol	able matrix	- 5,5	
				·		

Due to the high concentration of non-target compounds, a dilution was necessary to perform the PAH by HPLC analysis. Therefore, the reporting limits for the HPLC PAH compounds were raised.

Laboratory Chronicle

an m	1 2						
CAT				Dilution			
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor	
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:26	Justin M Bowers	1	
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 05:27	Mark A Clark	20	
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	20 1	



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Kerr-McGee Corporation



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Lancaster Laboratories Sample No. SW 3829113

Collected: 05/31/2002 13:40 by V/D Account Number: 07802

Submitted: 06/01/2002 10:25 Reported: 06/17/2002 at 15:35

Discard: 07/18/2002

MA6-SSRR-265 Grab Soil Sample

Moss American /WI

/17/2002 at 15:35 P.O. Box 25861 18/2002 Oklahoma City OK 73125

MA265 SDG#: KMA14-07

					Dry		
CAT			Dry		Method		Dilution
No.	Analysis Name	CAS Number	Result	<u> </u>	Detection Limit	Units	Factor
00111	Moisture	n.a.	31.2		0.50	% by wt.	1
	"Moisture" represents the loss	in weight of t	he sampl	le after d	oven drying at		
	103 - 105 degrees Celsius. The	result reporte	d above	is on an	as-received		
	basis.						
00941	PAH's in Solids by HPLC						
						4-	
00942	Naphthalene	91-20-3	N.D.		300.	ug/kg	5
00974	Fluorene	86-73-7	N.D.		30.	ug/kg	5
01014	Benzo(a)anthracene	56-55-3	20.	J	10.	ug/kg	5
01016	Chrysene	218-01-9	50.	J	10.	ug/kg	5
01017	Benzo(b)fluoranthene	205-99-2	40.	J	20.	ug/kg	5
01018	Benzo(k)fluoranthene	207-08-9	20.	J	10.	ug/kg	5
01019	Benzo(a)pyrene	50-32-8	30.	J	10.	ug/kg	5
01022	Dibenz(a,h)anthracene	53-70-3	N.D.		10.	ug/kg	5
01025	Indeno(1,2,3-cd)pyrene	193-39-5	40.	J	20.	ug/kg	5
01026	Benzo(g,h,i)perylene	191-24-2	70.	J	20.	ug/kg	5
	Due to the high concentration of	of non-target c	ompounds	, a dilut	ion was		
	necessary to perform the PAH by	HPLC analysis	. There	fore, the	reporting		

Laboratory Chronicle

CAT				Dilution		
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:44	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 08:11	Mark A Clark	5
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1



Lancaster Laboratories Sample No. SW 3829112

Collected:05/31/2002 13:45 by V/D Account Number: 07802

 Submitted:
 06/01/2002 10:25
 Kerr-McGee Corporation

 Reported:
 06/17/2002 at 15:35
 P.O. Box 25861

 Discard:
 07/18/2002
 Oklahoma City OK 73125

MA6-SSRR-273 Grab Soil Sample

Moss American /WI

MA273 SDG#: KMA14-06

					Dry		
CAT			Dry		Method		Dilution
No.	Analysis Name	CAS Number	Result	:	Detection Limit	Units	Factor
00111	Moisture	n.a.	29.3		0.50	% by wt.	1
	"Moisture" represents the loss	in weight of t	he sampl	e after	oven drying at		
	103 - 105 degrees Celsius. The basis.	result reporte	ed above	is on ar	n as-received		
00941	PAH's in Solids by HPLC						
00942	Naphthalene	91-20-3	N.D.		300.	ug/kg	5
00974	Fluorene	86-73-7	N.D.		30.	ug/kg	5
01014	Benzo(a)anthracene	56-55-3	19.	J	10.	ug/kg	5
01016	Chrysene	218-01-9	40.	J	10.	ug/kg	5
01017	Benzo(b)fluoranthene	205-99-2	30.	J	20.	ug/kg	5
01018	Benzo(k)fluoranthene	207-08-9	14.	J	10.	ug/kg	5
01019	Benzo(a)pyrene	50-32-8	30.	J	10.	ug/kg	5
01022	Dibenz(a,h)anthracene	53-70-3	N.D.		10.	ug/kg	5
01025	Indeno(1,2,3-cd)pyrene	193-39-5	40.	J	20.	ug/kg	5
01026	Benzo(g,h,i)perylene	191-24-2	60.	J	20.	ug/kg	5
	Due to the high concentration	of non-target c	ompounds	, a dilu		-3, 113	ŭ
	necessary to perform the PAH by	y HPLC analysis	. There	fore, th	e reporting		
				,	F		

	Laboratory Chronicle							
CAT No.	Analysis Name	Analysis Name Method Trial# Date and Time Analyst						
00111	Moisture		III TAI#		-	Factor		
		EPA 160.3 modified	1	06/05/2002 19:44	Justin M Bowers	1		
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 06:51	Mark A Clark	5		
03338	PAH Solid Extraction	SW-846 3550B	,			3		
		24-040 3330B	1	06/04/2002 17:15	Desiree J Wann	1		



Oklahoma City OK 73125



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Lancaster Laboratories Sample No. SW 3829115

Collected: 05/31/2002 13:45 by V/D Account Number: 07802

Submitted: 06/01/2002 10:25 Kerr-McGee Corporation Reported: 06/17/2002 at 15:35 P.O. Box 25861

Discard: 07/18/2002

MA6-SSRR-295 Grab Soil Sample

Moss American /WI

MA295 SDG#: KMA14-09

					Dry					
CAT			Dry		Method		Dilution			
No.	Analysis Name	CAS Number	Result		Detection Limit	Units	Factor			
00111	Moisture	n.a.	46.5		0.50	% by wt.	1			
	"Moisture" represents the loss in weight of the sample after oven drying at									
	103 - 105 degrees Celsius. The	result reporte	ed above i	is on an	as-received					
	basis.									
00941	PAH's in Solids by HPLC									
00942	Naphthalene	91-20-3	N.D.		1,000.	ug/kg	20			
00974	Fluorene	86-73-7	N.D.		100.	ug/kg	20			
01014	Benzo(a) anthracene	56-55-3	N.D.		50.	ug/kg	20			
01016	Chrysene	218-01-9	N.D.		70.	ug/kg	20			
01017	Benzo(b) fluoranthene	205-99-2	110.	J	100.	ug/kg	20			
01018	Benzo(k) fluoranthene	207-08-9	50.	J	50.	ug/kg	20			
01019	Benzo(a)pyrene	50-32-8	130.	J	70.	ug/kg	20			
01022	Dibenz(a,h)anthracene	53-70 - 3	N.D.		50.	ug/kg	20			
01025	Indeno(1,2,3-cd)pyrene	193~39-5	340.	J	100.	ug/kg	20			
01026	Benzo(g,h,i)perylene	191-24-2	1,200.		100.	ug/kg	20			
	Due to the high concentration of	of non-target o	ompounds,	a dilu	tion was					
	necessary to perform the PAH by	y HPLC analysis	. Theref	ore, the	e reporting					

Laboratory Chronicle

CAT			•	Dilution		
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:44	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 09:36	Mark A Clark	20
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1



Lancaster Laboratories Sample No. SW 3829114

Collected: 05/31/2002 13:45 by V/D Account Number: 07802

 Submitted:
 06/01/2002 10:25
 Kerr-McGee Corporation

 Reported:
 06/17/2002 at 15:35
 P.O. Box 25861

 Discard:
 07/18/2002
 Oklahoma City OK 73125

necessary to perform the PAH by HPLC analysis. Therefore, the reporting

Discard: 07/18/2002 Oklahoma (MA6-SSRR-299 Grab Soil Sample

Moss American /WI

MA299 SDG#: KMA14-08

					Dry		•
CAT			Dry		Method		Dilution
No.	Analysis Name	CAS Number	Result		Detection Limit	Units	Factor
00111	Moisture	n.a.	43.0		0.50	% by wt.	1
	"Moisture" represents the loss	in weight of th	ne sample	e after o	oven drying at		
	103 - 105 degrees Celsius. The	result reported	d above :	is on an	as-received		
	basis.						
00941	PAH's in Solids by HPLC						
00942	Naphthalene	91-20-3	N.D.		700.	ug/kg	10
00974	Fluorene	86-73-7	N.D.		70.	ug/kg	10
01014	Benzo(a)anthracene	56-55-3	80.	J	20.	ug/kg	10
01016	Chrysene	218-01-9	90.	J	40.	ug/kg	10
01017	Benzo(b)fluoranthene	205-99 - 2	150.	J	50.	ug/kg	10
01018	Benzo(k)fluoranthene	207-08-9	70.	J	20.	ug/kg	10
01019	Benzo(a)pyrene	50 - 32-8	110.	J	40.	ug/kg	10
01022	Dibenz(a,h)anthracene	53-70-3	N.D.		20.	ug/kg	10
01025	Indeno(1,2,3-cd)pyrene	193-39-5	150.	J	50.	ug/kg	10
01026	Benzo(g,h,i)perylene	191-24-2	300.		50.	ug/kg	10
	Due to the high concentration o	f non-target co	mpounds,	a dilut	ion was		

Laboratory Chro	ronic	le
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CAT				Analysis		Dilution
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:44	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 08:53	Mark A Clark	10
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1



Oklahoma City OK 73125



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Lancaster Laboratories Sample No. SW 3829117

Collected: 05/31/2002 13:40 by V/D Account Number: 07802

 Submitted:
 06/01/2002 10:25
 Kerr-McGee Corporation

 Reported:
 06/17/2002 at 15:36
 P.O. Box 25861

necessary to perform the PAH by HPLC analysis. Therefore, the reporting

Discard: 07/18/2002

MA6-SSRR-303 Grab Soil Sample

Moss American /WI

MA303 SDG#: KMA14-11

					Dry		
CAT			Dry		Method		Dilution
No.	Analysis Name	CAS Number	Result		Detection Limit	Units	Factor
00111	Moisture	n.a.	30.2		0.50	% by wt.	1
	"Moisture" represents the loss	in weight of the	he sample	after o	ven drying at		
	103 - 105 degrees Celsius. The	result reporte	d above i	s on an	as-received		
	basis.						
00941	PAH's in Solids by HPLC						
00942	Naphthalene	91-20-3	N.D.		600.	ug/kg	10
00974	Fluorene	86-73-7	N.D.		60.	ug/kg	10
01014	Benzo(a) anthracene	56-55 - 3	90.	J	20.	ug/kg	10
01016	Chrysene	218-01-9	120.	J	30.	ug/kg	10
01017	Benzo(b) fluoranthene	205-99-2	150.	J	40.	ug/kg	10
01018	Benzo(k) fluoranthene	207-08 - 9	70.	J	20.	ug/kg	10
01019	Benzo(a)pyrene	50-32-8	120.	J	30.	ug/kg	10
01022	Dibenz(a,h)anthracene	53-70-3	N.D.		20.	ug/kg	10
01025	Indeno(1,2,3-cd)pyrene	193-39-5	140.	J	40.	ug/kg	10
01026	Benzo(g,h,i)perylene	191-24-2	300.		40.	ug/kg	10
	Due to the high concentration o	f non-target co	mpounds,	a diluti	ion was		

Laboratory Chronicle

CAT				Dilution		
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:44	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 11:00	Mark A Clark	10
03338	PAH Solid Extraction	SW-846 3550B	1	06/04/2002 17:15	Desiree J Wann	1





Lancaster Laboratories Sample No. SW 3829116

Collected: 05/31/2002 13:40 by V/D Account Number: 07802

Submitted: 06/01/2002 10:25 Reported: 06/17/2002 at 15:36

Discard: 07/18/2002

MA6-SSRR-291 Grab Soil Sample

Moss American /WI

Kerr-McGee Corporation P.O. Box 25861

Oklahoma City OK 73125

MA291 SDG#: KMA14-10

					Dry					
CAT			Dry		Method		Dilution			
No.	Analysis Name	CAS Number	Result	.	Detection Limit	Units	Factor			
00111	Moisture	n.a.	36.8		0.50	% by wt.	1			
	"Moisture" represents the loss in weight of the sample after oven drying at									
	103 - 105 degrees Celsius. The result reported above is on an as-received									
	basis.									
00941	PAH's in Solids by HPLC									
00942	Naphthalene	91-20-3	N.D.		600.	ug/kg	10			
00974	Fluorene	86-73 - 7	N.D.		60.	ug/kg	10			
01014	Benzo(a)anthracene	56-55-3	40.	Ĵ	20.	ug/kg	10			
01016	Chrysene	218-01-9	40.	J	30.	ug/kg	10			
01017	Benzo(b)fluoranthene	205-99-2	60.	J	40.	ug/kg	10			
01018	Benzo(k)fluoranthene	207-08-9	30.	J	20.	ug/kg	10			
01019	Benzo(a)pyrene	50-32-8	50.	J	30.	ug/kg	10			
01022	Dibenz(a,h)anthracene	53-70-3	N.D.		20.	ug/kg	10			
01025	Indeno(1,2,3-cd)pyrene	193-39-5	60.	J	40.	ug/kg	10			
01026	Benzo(g,h,i)perylene	191-24-2	130.	J	40.	ug/kg	10			
	Due to the high concentration o	f non-target co	ompounds	, a dilut:	ion was	-5/5				
	necessary to perform the PAH by	HPLC analysis	. There	fore, the	reporting					
	limits for the HPLC PAH compoun	ds were raised		,0						
	-									

CAT		-		Analysis		Dilution
No.	Analysis Name	Method	Trial#	Date and Time	Analyst	Factor
00111	Moisture	EPA 160.3 modified	1	06/05/2002 19:44	Justin M Bowers	1
00941	PAH's in Solids by HPLC	SW-846 8310	1	06/14/2002 10:18	Mark A Clark	10
03338	PAH Solid Extraction	SW-846 3550B	-	06/04/2002 17:15		10
		5. 010 3330B	_	06/04/2002 17:15	Desiree J Wann	1







Page 2 of 2

Client Name: Kerr-McGee Corporation

Reported: 06/17/02 at 03:36 PM

Group Number: 809613

		Surrogate	Quality	Control
85	100		_	
83	101			
9.0	102			

3829111 3829112 3829113 3829114 3829115 3829116 3829117 Blank LCS MS	85 83 89 85 90 90 86 92 86	100 101 102 120 122 108 129 102 97
MSD	88	110

Limits: 54-126 38-187

^{• (2)} The background result was more than four times the spike added.



^{*-} Outside of specification

⁽¹⁾ The result for one or both determinations was less than five times the LOQ.



Client Name: Kerr-McGee Corporation Reported: 06/17/02 at 03:36 PM

Group Number: 809613

Laboratory Compliance Quality Control

Analysis Name	Blank <u>Result</u>	Blank MDL	Report <u>Units</u>	LCS <u>%REC</u>	LCSD %REC	LCS/LCSD <u>Limits</u>	RPD	RPD Max
Batch number: 02155SLB026	Sample nu	umber(s):	3829105-38	29117				
Naphthalene	N.D.	40.	ug/kg	94		67-117		
Fluorene	N.D.	4.	ug/kg	94		78-115		
Benzo(a)anthracene	N.D.	1.	ug/kg	97		81-125		
Chrysene	N.D.	2.	ug/kg	96		80-120		
Benzo(b) fluoranthene	N.D.	3.	ug/kg	102		83-126		
Benzo(k)fluoranthene	N.D.	1.	ug/kg	100		84-125		
Benzo(a)pyrene	N.D.	2.	ug/kg	91 .		68-126		
Dibenz(a,h)anthracene	N.D.	1.	ug/kg	108		91-129		
Indeno(1,2,3-cd)pyrene	N.D.	3.	ug/kg	100		85-125		
Benzo(g,h,i)perylene	N.D.	3.	ug/kg	99		85-126		
Batch number: 02156820003B	Sample nu	mber(s):	3829105-38	29111				
Moisture	_			100		99-101		
Batch number: 02156820004A	Sample nu	mber(s):	3829112-38	29117				
Moisture	•			100		99-101		

Sample Matrix Quality Control

	MS	MSD	ms/msd		RPD	BKG	DUP	DUP	Dup
Analysis Name	%REC	%REC	Limits	RPD	MAX	Conc	Conc	RPD	RPD Max
Batch number: 02155SLB026	Sample	number	(s): 382910	5-38291	L 1 7		•		
Naphthalene	93 -	94	41-141	1	50				
Fluorene	90	92	30-154	2	50				
Benzo(a)anthracene	31	40	2-120	13	50				
Chrysene	79	88	17-170	8	50				
Benzo(b)fluoranthene	33	67	4-190	17	50				
Benzo(k)fluoranthene	63	81	23-172	12	50				
Benzo(a)pyrene	44	68	34-165	15	50				
Dibenz(a,h)anthracene	100	101	14-181	1	50				
Indeno(1,2,3-cd)pyrene	73	80	25-165	7	50				
Benzo(g,h,i)perylene	78	85	29-160	6	50				
Batch number: 02156820003B	Sample	number	(s): 382910	5-38201	11				
Moisture			(5). 302310	5 50251		30.7	30.4	1	15
Batch number: 02156820004A	Sample number(s): 3829112-3829117								
Moisture	p#c		157. 502511.	2-30291	1,	4.65	4.62	1	15

Surrogate Quality Control

Analysis Name: PAH's in Solids by HPLC Batch number: 02155SLB026

Dacon nam	Nitrobenzene	Triphenylene	
3829105	91	116	
3829106	85	102	
3829107	88	110	
3829108	94	498*	
3829109	86	135	
3829110	93	630*	

*- Outside of specification

- (1) The result for one or both determinations was less than five times the LOQ.
- (2) The background result was more than four times the spike added.



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Velote Property Color of the Carlo Color of the Car

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2102 Rev 3/7/0

Acct. # 6149 Sample # 3829119 36 (3) (10Up) 7802 3829105-147 Please print. Instructions on reverse side correspond with circled numbers. Client: Kerr McGee **Analyses Requested** Matrix (4) (5)Acct. #: SCR#://66258 Project Name/#: Moss American PWSID #: ______

Project Manager: Tom Graan P.O.#

Sampler: Voss Duddy Quote #: _____ **Fotal # of Containers** Date Collected C Name of state where samples were collected: Wisconsin Water Soil Sample Identification Remarks 5/31/02 13:40 MAG-SSER-303 Relinquished by: Time (Turnaround Time Requested (TAT) (please circle): Normal (Rush TAT is subject to Lancaster Laboratories approval and surcharge.)

Date results are needed: 6 14 0 Z

Rush results requested by (please circle): Phone Fax 10:00 Time Date Fax #: 847 918 4055 Phone #: ___ Time Date Time 8 Data Package Options (please circle if requested) SDG-Complete? Type VI (Raw Data) **QC Summary** Relinguished by: Date Time Received by: Date Time GLP Type I (Tier I) Site-specific QC required? Yes (If yes, indicate QC sample and submit triplicate volume.) Type II (Tier II) Other Relinguished by: Received by: Date Time Date Type III (NJ Red. Del.) Internal Chain of Custody required? Yes No Type IV (CLP) Lancaster Laboratories, Inc., 2425 New Holland Pike, PO Box 12425, Lancaster, PA 17605-2425 (717) 656 2300

Copies: White and yellow should accompany samples to Lancaster Laboratories. The pink copy should be retained by the client



JUN 2 3 2007

For Lancaster Laboratories use only

Acct. # 6149 Sample # 3829119-36

2102 Rev 3/7/01

	Please pri	nt. Instru	tions o	n rev	erse si	de co	rresp	ond with	circled n	umbers.		, ,	307		36 47	102-11	- T
Client: Kerr-McGee	Acct. #: _			Ma	trix (1)		(5)		F	Analyse	es Requ	uested		FSC-	For lab use o	٠. ٨
Project Name/#: MUSS Pm.,,					(Check if applicable)		İ		0		/ /	/ /			SCR#	11660	388
Project Manager: Tom Graan							2	0	'b/ b/ /	/ /				/ /			3 6
Sampler: Voss/Duddy					table DES		Containers		Z /			/ /	/ . /		9.10	15V	sampl
Name of state where samples were collected: $\stackrel{\checkmark}{\mathbb{W}}$			(3)		r □ Potable □ NPDES			\x'\			/ /	/ /			γ, ι		rature of eceipt (if
Sample Identification		Time Collected	Grab	Soil	Water	Other		4						/ Remark:	5		Temper upon n
MA6-SSRR-255 MS/MSD	5/31/02	13:35	X	X		13	2 >	X						Perform	. Ms/	MSD	
MA6-SSRR - 309		13:30	$\perp \downarrow \perp$			_ _	1	(
MA6-SSRR - 305	<u> </u>	13:30															
MAG- SSRR - 309 DP		13:30															
MAG- SSRR- 261		13:40					1										
MAG - SSRR - 273		13:45														•	
MA6-SSRR-265		13:40					۱										
MA6-SSRR-299		13:45															
MA6-SSRR-295		13:45															
MAG- SSRR - 291	+	13:40	4	4			1	+ /									
Turnaround Time Requested (TAT) (please circle): (Rush TAT is subject to Lancaster Laboratories approval and Date results are needed:	surcharge.)	Rush	Relinqu		1	e v	/	13	Date	02,23	Receive	red by:	ni-	Un		5/31/ve	Time (
Date results are needed: 6/14/02 Rush results requested by (please circle): Phone Fax Phone #: Fax #:	28479	184055	Reling	Si	a)	la	7		Date 5/31/	14:0	_ 1		Fea	Uss 1 Ex		Date	Time
)Data Package Options (please circle if requested)		Complete?	Relingu	ished I	by:				Date	Time	Receiv	ved by:				Date	Time
QC Summary Type VI (Raw Data) Type I (Tier I) GLP	(Ye	s) No	Relinqu	ished l	by:				Date	Time	Receiv	ved by:				Date	Time
Type I (Tier I) GLP Site-specific QC requ (If yes, indicate QC sample	ired? Yes	No /				_	<u></u>										
Type III (NJ Red. Del.)	•		Relingu	iished l	by:				Date	Time	Renei	ved by:	`	_	/.	Date:	Time
Type IV (CLP)			<u>/</u>								100	<u> </u>	50 K			11/00/	

Copies: White and yellow should accompany samples to Lancaster Laboratories. The pink copy should be recained by the client (



CASE NARRATIVE

Client: Kerr-McGee Corporation

SDG #: KMA14

R/Latrix

LANCASTER LABORATORIES PAH BY HPLC

SAMPLE NUMBER(S):

•		Matrix	
LL #'s	Sample Code	<u>Soil</u>	<u>Comments</u>
3829105	MA255	Χ	Unspiked 5X Dilution
3829106	MA255MS	X	Matrix Spike 5X Dilution
3829107	MA255MSD	X	Matrix Spike Dup 5X Dilution
3829108	-MA309	Χ	20X Dilution
3829109	MA305	Χ	10X Dilution
3829110	309DP	Χ	20X Dilution
3829111	MA261	Χ	5X Dilution
3829112	MA273	X	5X Dilution
3829113	MA265	X	5X Dilution
	MA299	X	10X Dilution
3829114	MA295	X	20X Dilution
3829115	•••	X	10X Dilution
3829116	MA291	X	10X Dilution
3829117	MA303	^	IOV Dilution

LABORATORY SUBMITTED QC:

SBLKLB155	SBLKLB1552	X	Method Blank
155LBLCS	155LBLCS2	Χ	Lab Control Sample

SAMPLE PREPARATION:

No problems were encountered during the extraction of these samples.

ANALYSIS:

The method used for analysis was SW-846 8310.

Lancaster Laboratories ◆ 2425 New Holland Pike, PO Box 12425, Lancaster, PA 17605-2425 ◆ Phone: 717-656-2300 ◆ Fax: 717-656-2681 ◆ http://www.LancasterLabs.com



Case Narrative SDG #: KMA14 continued

All samples were analyzed for the client specified list of compounds by HPLC.

The following samples were analyzed at initial dilutions due to high concentrations of non-target compounds.

Sample Code	<u>Dilution</u>
MA255, MA255MS, MA255MSD	5X
MA309	20X
MA305	10X
309DP	20X
MA261	5X
MA273	5X
MA265	5X
MA299	10X
MA295	20X
MA291	10X
MA303	10X

No other problems were encountered during the analysis of these samples.

QUALITY CONTROL AND NONCONFORMANCE SUMMARY:

The relative percent difference (RPD) for benzo(b)fluoranthene between MA255MS and MA255MSD was greater than 30 percent.

All other QC was within specifications.

DATA INTERPRETATION:

Only non-conformances for client requested compounds are addressed in this case narrative.

No further interpretation is necessary for the data submitted.



Case Narrative SDG #: KMA14 continued

Case Narrative Reviewed and Approved by:

Christine M. Ratchell for CJN Date: 6-20/02

Group Leader, GC/MS Semivolatiles