ANALYTICAL METHOD VERIFICATION FOR THE DETERMINATION OF WATER SOLUBLE COMPONENTS OF PETROLEUM COKE IN FRESHWATER USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 472C-104

GUIDELINE:
European Commission Working Document SANCO/3029/99 rev.4

AUTHORS:

[redacted]

STUDY INITIATION DATE: April 28, 2004

STUDY COMPLETION DATE: February 1, 2006

AMENDED REPORT DATE: April 10, 2007

Submitted to

American Petroleum Institute
1220 L Street, N.W.
Washington, DC 20005

Wildlife International, Ltd.

8598 Commerce Drive
Easton, Maryland 21601
(410) 822-8600

AMENDED

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

SPONSOR: American Petroleum Institute

TITLE: Analytical Method Verification for the Determination of Water Soluble Components of Petroleum Coke in Freshwater Using High Performance Liquid Chromatography (HPLC)

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 472C-104

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This study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Parts 160 and 792, 17 August 1989 and OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17), with the following exceptions:

Periodic analyses of well water for potential contaminants were performed using a certified laboratory and standard U.S. EPA analytical methods, but not under Good Laboratory Practice Standards.

The characterization of the test substance was not determined in accordance with Good Laboratory Practice Standards.

The stability of the test substance under storage conditions at the test site was not determined in accordance with Good Laboratory Practice Standards.

STUDY DIRECTOR:

[redacted]

Wildlife International, Ltd.

4-10-07
DATE

SPONSOR:
American Petroleum Institute, by:

[redacted]

4/26/2007
DATE

AMENDED

QUALITY ASSURANCE STATEMENT

This study was examined for compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Parts 160 and 792, 17 August 1989 and OECD Principles of Good Laboratory Practice, (ENV/MC/CHEM (98) 17). The dates of all inspections and audits and the dates that any findings were reported to the Study Director and Laboratory Management were as follows:

		DATE REPORTED TO:	
ACTIVITY:	DATE CONDUCTED:	STUDY DIRECTOR:	MANAGEMENT:
Protocol	April 22, 2004	April 22, 2004	December 13, 2004
Matrix Fortification	September 17, 2004	September 17, 2004	September 27, 2004
Test Substance Preparation	October 29, 2004	October 29, 2004	November 9, 2004
Data and Draft Report	December 2, 3, 6, 7 and 8, 2004	December 8, 2004	December 17, 2004
Final Report	February 1, 2006	February 1, 2006	February 1, 2006
Amended Report	April 9, 2007	April 9, 2007	April 9, 2007

All inspections were study-based unless otherwise noted.

[redacted]

4/10/2007
DATE

AMENDED

AMENDED REPORT APPROVAL

SPONSOR: American Petroleum Institute

TITLE: Analytical Method Verification for the Determination of Water Soluble Components of Petroleum Coke in Freshwater Using High Performance Liquid Chromatography (HPLC)

WILDLIFE INTERNATIONAL, LTD. PROJECT NO.: 472C-104

This report was reviewed by the individuals involved in the conduct and management of the study, and was found to be an accurate reflection of the methods used, data collected and results of the study.

STUDY DIRECTOR:

[redacted]

Wildlife International, Ltd.

4-10-07
DATE

MANAGEMENT:

[redacted]

Wildlife International, Ltd.

4/10/07
DATE

AMENDED

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SUMMARY

SPONSOR:	American Petroleum Institute
SPONSOR'S REPRESENTATIVE:	[redacted]
LOCATION OF STUDY, RAW DATA AND A COPY OF THE FINAL REPORT:	Wildlife International, Ltd. Easton, Maryland 21601

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER:	472C-104
TEST SUBSTANCE: Petroleum Coke	
STUDY:	Analytical Method Verification for the Determination of Water Soluble Components of Petroleum Coke in Freshwater Using High Performance Liquid Chromatography (HPLC)
FORTIFIED TEST CONCENTRATIONS:	10.0, 40.0 and 100 µg/L
TEST DATES:	Experimental Start (OECD) – August 4, 2004 Experimental Start (EPA) – September 20, 2004 Experimental Termination – November 5, 2004.

TEST SYSTEM:	Freshwater

SUMMARY

(Continued)

SUMMARY: Method verification samples containing nineteen polyaromatic hydrocarbons (PAHs) were fortified in freshwater. Samples were either further diluted in freshwater or analyzed directly against PAH external standards also prepared in freshwater. Recovery samples and standards were analyzed by reverse phase, gradient elution high performance liquid chromatography with on-line sequential UV and fluorescence detection. Ultraviolet detection at 220 nm was used to quantify naphthalene, acenaphthylene, 1-methylnaphthalene, 2-methylnaphthalene, fluorene, acenaphthene, phenanthrene, chrysene and indeno(1,2,3-cd)pyrene. Fluorescence detection at an excitation wavelength of 340 nm and an emission wavelength of 425 nm was used to quantify anthracene, fluoranthene, pyrene, benz(a)anthracene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, dibenz(a,h)anthracene, benzo(g,h,i)perylene and dibenzo(a,e)pyrene. Each analyte was quantified against external standards via linear regression analysis. Recoveries of each of the PAH analytes from freshwater are presented in tables 2-20. Linear regression analyses of each of PAH analytes are presented in figures 2-20.

Water accommodated fractions (WAFs) were analyzed for the presence of PAHs after mixing for 24, 48, 72 and 96 hours. No PAHs were detected in any WAF samples. A blank and three quality control (QC) samples were prepared and analyzed at each interval. All QC samples were quantitative for the nineteen PAHs; see tables 21-39.

INTRODUCTION

Analytical trials were conducted to verify analytical methods for the determination of polyaromatic hydrocarbons (PAH) in water accommodated fraction (WAF) solutions made with petroleum coke and freshwater. The study was conducted by Wildlife International, Ltd. and identified as Project Number 472C-104. The study was performed based on procedures in *Residues: Guidance for Generating and Reporting Methods of Analysis in Support of Pre-registration Data Requirements for Annex II (Part A, Section 4) and Annex III (Part A, Section 5) of Directive 91/414* (1). Petroleum coke was defined as the product formed by subjecting the heavy tar-like residue remaining following oil refining to high temperatures and pressures. It consists of primarily elemental carbon with considerably smaller amounts of hydrocarbons, sulfur and trace amounts of heavy metals. The method was verified by fortifying freshwater with the test substance and recoveries were determined. Limits of quantitation (LOQ) for the method also were established. All raw data generated by Wildlife International, Ltd. and a copy of the final report are filed under Project Number 472C-104 in archives located on the Wildlife International, Ltd. site. The protocol and amendments are presented in Appendix 3 and personnel involved in the study are listed in Appendix 5.

PURPOSE

The objectives of this study were to; 1) verify a High Performance Liquid Chromatography (HPLC) method for determination of polycyclic aromatic hydrocarbons (PAH) in water accommodated fraction (WAF) solutions of petroleum coke; 2) employ the method to determine the optimum WAF mixing time to achieve maximum leaching of PAHs from the test substance matrix into freshwater.

EXPERIMENTAL DESIGN

Wildlife International, Ltd. well water was fortified at three different concentrations and analyzed using HPLC methods developed by Wildlife International, Ltd. Reagent and matrix blanks were analyzed concurrently to evaluate potential analytical interferences. Calibration curves were prepared and analyzed with each series of matrix fortification samples.

An equilibration trial was run for 96 hours to determine an appropriate mixing time for preparing WAF solutions of green petroleum coke. Compounds of interest in WAF solutions were 20 PAHs. Analytical samples were taken approximately 24, 48, 72 and 96 hours after initiation of stirring and analyzed for PAHs. Matrix blanks were analyzed concurrently to evaluate potential analytical interferences. Calibration curves were prepared and analyzed with each series of matrix fortification samples.

MATERIALS AND METHODS

Test Substance

The test substance was green petroleum coke (CAS Number 64741-79-3). Petroleum coke was defined as the product formed by subjecting the heavy tar-like residue remaining following oil refining to high temperatures and pressures. It consists of primarily elemental carbon with considerably smaller amounts of hydrocarbons, sulfur and trace amounts of heavy metals. Analyses of selected components in petroleum coke are provided in Appendix 4. The test substance was received from EPL on October 7, 2003 and was assigned Wildlife International, Ltd. identification number 6485A. The test substance appeared as black pellets and was identified as 2 mm Particle Size Petroleum Coke (aka Milled Pellets). The test substance was stored under ambient conditions. An expiration date was not provided.

The identity, strength, purity, composition and method of synthesis, fabrication and/or derivation of each batch of the test substance and the maintenance of these records was the responsibility of the Sponsor.

Reference Standards

Purified PAH reference standard was made up of components received from three manufacturers. The following standards were received from Accu Standard Inc. and were stored under ambient conditions:

Component	Test Substance Number	Lot/Batch	CAS Number	Date Received	Expiration Date	Description
Benzo(a)pyrene	6705	052803MT-AC	50-32-8	6/07/04	6/03/07	green powder
Anthracene	6706	A33783	120-12-7	6/07/04	6/03/07	white powder
Benz(a)anthracene	6707	19587	56-55-3	6/07/07	6/03/07	colorless plates
Acenaphthylene	6708	011504MS-AC	208-96-8	6/07/04	6/03/07	yellow powder
Acenaphthene	6709	01915EQ	83-32-9	6/07/04	6/03/07	white crystal
Benzo(b)fluoranthene	6710	020402AG-AC	205-99-2	6/07/04	6/03/07	white flakes
Benzo(g,h,i)perylene	6711	122 500MT-AC	191-24-2	6/07/04	6/03/07	green powder
Benzo(k)fluoranthene	6712	112603AG-AC	207-08-9	6/08/04	6/03/07	yellow powder
Chrysene	6713	13103	218-01-9	6/08/04	6/03/07	white powder
Dibenz(a,h)anthracene	6714	13246	53-70-3	6/08/04	6/03/07	green powder
Fluoranthene	6715	19762	206-44-0	6/08/04	6/03/07	white powder
Fluorene	6716	19675	86-73-7	6/08/04	6/03/07	white powder
Indeno(1,2,3-cd)pyrene	6717	19641	193-39-5	6/08/04	6/03/07	yellow powder
Naphthalene	6718	167A-A	91-20-3	6/08/04	6/03/07	white flakes
Phenanthrene	6719	090903AG-AC-1	85-01-8	6/08/04	6/03/07	white powder
Pyrene	6720	09617LR	129-00-0	6/08/04	6/03/07	green crystal

The following standards were received from Cambridge-Isotope Labs and was stored under ambient conditions:

Component	Test Substance Number	Lot/Batch	CAS Number	Date Received	Expiration Date	Description
Dibenzo(a,e)pyrene	6518	I1-7628	192-65-4	10/22/03	Not given	Solids
Perylene	6493	20330PO	198-55-0	10/09/03	Not given	Cystalline solid

The following standards were received from ChemService and were stored under ambient conditions:

Component	Test Substance Number	Lot/Batch Number	CAS Number	Date Received	Expiration Date	Description
2-Methylnaphthalene	6765	310-43C	91-57-6	8/03/04	9/01/08	Solid
1-Methylnaphthalene	6766	325-31A	90-12-0	8/03/04	5/01/09	Liquid

All certificates of analysis for the components are presented in Appendix 4.

Reagents and Solvents

All solvents used in the methods were of HPLC grade or equivalent.

Freshwater

The freshwater used to prepare the method verification studies was obtained from a well approximately 40 meters deep located on the Wildlife International, Ltd. site. The well water is characterized as moderately-hard water. The means and ranges of specific conductance, hardness, alkalinity and pH measurements of the well waterduring the four-week period immediately preceding the test are presented in Appendix 1.

The well water was passed through a sand filter toremove particles greater than approximately 25 µm, and pumped into a 37,800-L storage tankand aerated with spray nozzles. Prior to use, the water was filtered (0.45 µm) again to remove microorganisms and particles. The results of periodic analyses performed to measure the concentrations of selectedorganic and inorganic constituents in the well water are presented in Appendix 2.

Stocks Preparation by HPLC Analysis

For all compounds received from AccuStandard, with the exception of Benzo(g,h,i)perylene, Benzo(k)fluoranthene and Fluoranthene, the mass received was quantitatively transferred to a 100-mL class A volumetric flask using methanol. These primary stock solution concentrations were 0.1 mg/mL.

Benzo(g,h,i)perylene, Benzo(k)fluoranthene and Fluoranthene were quantitatively transferred to a 200-mL class A volumetric flask using methanol. These primary stock solution concentrations were 0.05 mg/mL.

A stock of Dibenzo(a.e)pyrene (received from Cambridge Isotope Labs) was prepared by weighing 0.01000 g on an analytical balance, transferred to a 100-mL class A volumetric flask and brought to volume using tetrahydrofuran. This primary stock solution concentration was 0.1 mg/mL.

Stocks of 2-methylnaphthylene and 1-methynaphthylene (received from ChemService) were prepared by weighing 0.1004 g and 0.01003 g, respectively, on an analytical balance. The test materials were transferred to 100-mL class A volumetric flasks and brought to volume using methanol. These primary stock solutions contained 1.00 mg/mL of the test material and were diluted in methanol to prepare 0.100 mg/mL secondary stock solutions.

Aliquots (1 mL) of the 0.1 mg/mL primary stocks and 2 mL of the 0.05 mg/mL primary stocks, were added to a 100-mL class A volumetric flask and brought to volume with methanol to produce a 0.1 mg/L combined stock solution. The following shows the dilution scheme for the set of calibration standards prepared in freshwater:

Stock Concentration mg/L	Aliquot (µL)	Final Volume (mL)	Standard Concentration (µg/L)
1.00	50.0	10.0	5.00
1.00	150	10.0	15.0
1.00	250	10.0	25.0
1.00	350	10.0	35.0
1.00	500	10.0	50.0

Analytical Method by HPLC

The method used for the analysis of the method verification samples was based upon methodology developed by Wildlife International, Ltd. The analytical method consisted of diluting the samples in freshwater, as necessary, and analyzing by direct injection high performance liquid chromatography (HPLC) with either UV detection at 220 nm or fluorescence detection at 340 nm to 425 nm. It was necessary to utilize two detection systems (UV and fluorescence) to measure the PAH compounds

because some compounds lacked sufficient fluorescence activity for detection. The detectors used for the individual components are noted in the tabulated data.

During evaluation of the instrument settings, the chromatographic retention times for perylene and dibenzo (a,e) pyrene overlapped such that no distinction of the two could be made. The decision was made not to expend further effort to resolve these two minor components in coke. Therefore, only dibenzo (a,e) pyrene was reported.

Concentrations of each PAH compound in the fortified samples were determined using an Agilent Model 1100 High Performance Liquid Chromatograph, equipped with either an Agilent Series 1100 Variable Wavelength Detector or a Jasco Model FP-1520 Fluorescence Detector. Chromatographic separations were achieved using a YMC Pack ODS-AM column (150 mm x 4.6 mm, 3 µm particle size). Instrumental parameters for the analysis of PAH components are summarized in Table 1 and a method flowchart is provided in Figure 1.

Calibration Curve and Limit of Quantitation (LOQ)

Calibration standards of the components, ranging in concentration from 5.00 to 50.0 µg/L, were analyzed with the freshwater verification sample set. Linear regression equations were generated using the peak area responses versus the respective concentrations of the calibration standards. Representative calibration curves are presented in Figures 2 through 20. The concentrations of the components in the samples was determined by substituting the peak area responses of the samples into the applicable linear regression equation. Representative chromatograms of low and high-level calibration standards are presented in Figures 21 and 22, respectively.

The method limit of quantitation (LOQ) for the method verification analysis of the components in freshwater was set at 5.00 µg/L, calculated as the product of the lowest calibration standard (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

Reagent and Matrix Blank Samples

Concurrent with the series of matrix fortification samples, two reagent blanks and two matrix blanks for each component were analyzed to determine possible interferences. No interferences were observed at or above the LOQ during the sample analyses (Tables 2 through 20). A representative chromatogram of a reagent blank is presented in Figure 23. A representative chromatogram of a matrix blank is presented in Figure 24.

Freshwater Method Verification Samples

Freshwater was fortified at 10.0, 40.0 and 100 µg/L using stock solutions containing PAH components in methanol. Results are presented in Tables 21 through 39. Representative chromatograms of low and high-level freshwater fortifications are presented in Figures 25 and 26, respectively.

Example Calculations

The analytical result and percent recovery for sample number 472C-104-VMAS-21 for 1-Methylnaphthalene, nominal concentration of 40.0 µg/L in freshwater, were calculated using the following equations:

$$\text{1-Methylnaphthalene (µg/L) in sample} = \frac{\text{Peak area - (Y-intercept)}}{\text{Slope}} \text{ X Dilution factor}$$

Peak area = 96.84348
Y-intercept = -0.2359
Slope = 2.4290
Dilution Factor = 1.00

$$\text{Concentration of 1-Methylnaphthalene (µg/L) in sample} = \frac{96.84348 + 0.2359}{2.4290} \text{ X } 1.00$$

Concentration of 1-Methylnaphthalene in sample (µg/L) = 39.97

$$\text{Percent of nominal concentration} = \frac{39.97 \text{ (µg/L)}}{40.0 \text{ (µg/L)}} \text{ X } 100$$

Percent of nominal concentration = 99.9%

Preparation of Test Concentration for the WAF Trial

Petroleum coke was mixed directly with dilution water (well water) on a weight:volume basis. A WAF was prepared at a single high concentration of 1000 mg/L in two different size Pyrex® aspirator bottles with tubulation. For the first WAF, 12.0 grams of the test substance were transferred into 12,000 ml of dilution water contained in a 13.2 L vessel. For the second WAF, 4.00 grams of test substance were transferred into 4000 mLs of dilution water contained in a 4 L vessel. Solutions were prepared by mixing the test solutions with Teflon®-coated stir bars to create a vortex depth of approximately 30% of the test solution height. After mixing, the WAFs were allowed to settle for 30 minutes to one hour. The test solutions were sampled following approximately 24, 48, 72 and 96 hours of mixing. Samples of each test solution were taken from mid-depth of the mixing vessels using graduated pipettes. Samples were

centrifuged at 14,000 rpm for approximately 5 minutes and submitted for HPLC analysis (Tables 21 through 39).

Calibration Curve and Limit of Quantitation (LOQ) for the WAF Trial

Calibration standards of the components, ranging in concentration from 5.00 to 50.0 µg/L, were analyzed with each sample set. Linear regression equations were generated using the peak area responses versus the respective concentrations of the calibration standards. The concentration of the components in the samples was determined by substituting the peak area responses of the samples into the applicable linear regression equation. Representative chromatograms of low and high-level calibration standards are presented in Figures 50 and 51, respectively.

The method limit of quantitation (LOQ) for the WAF trial was set at 5.00 µg/L, calculated as the product of the lowest calibration standard (5.00 µg/L) and the dilution factor of the matrix blank (1.00). Representative chromatograms of a matrix blank and matrix fortification are presented in Figures 27 and 28, respectively. A representative chromatogram of a test sample is presented in Figure 31.

RESULTS OF WAF EQUILIBRATION TRIAL

Water accommodated fractions (WAFs) were analyzed for the presence of the 19 polyaromatic hydrocarbons (PAHs) after mixing for 24, 48, 72 and 96 hours. No PAHs were detected in any WAF samples. Representative chromatograms for a WAF sample are presented in Figures 50-54. A blank and three quality control (QC) samples were prepared and analyzed at each interval. The QC samples were quantitative for all 19 PAHs (Tables 21-39).

CONCLUSIONS

Freshwater validation samples containing components of PAH were prepared and analyzed by direct injection high performance liquid chromatography with either UV or fluorescence detection and fortified at nominal concentrations of 10.0, 40.0 and 100 µg/L. Recoveries of Naphthalene in freshwater yielded mean percent recoveries of 100, 101 and 88.8%, respectively, with an overall mean recovery of 96.4 ± 5.82% (RSD = 6.04%) (Table 2). Recoveries of Acenaphthylene in freshwater yielded mean percent recoveries of 99.1, 99.8 and 96.4%, respectively, with an overall mean recovery of 98.5 ± 1.67% (RSD = 1.70%) (Table 3). Recoveries of 1-Methylnaphthalene in freshwater yielded mean percent recoveries of 100, 101 and 89.2%, respectively, with an overall mean recovery of 96.6 ± 5.62% (RSD =

5.82%) (Table 4). Recoveries of 2-Methylnaphthalene in freshwater yielded mean percent recoveries of 100, 100 and 86.6%, respectively, with an overall mean recovery of 95.6 ± 6.84% (RSD = 7.15%) (Table 5). Recoveries of Fluorene in freshwater yielded mean percent recoveries of 98.0, 100 and 95.7%, respectively, with an overall mean recovery of 97.9 ± 2.04% (RSD = 2.08%) (Table 6). Recoveries of Acenaphthene in freshwater yielded mean percent recoveries of 98.4, 99.6 and 93.0%, respectively, with an overall mean recovery of 97.0 ± 3.14% (RSD = 3.24%) (Table 7). Recoveries of Phenanthrene in freshwater yielded mean percent recoveries of 98.8, 99.6 and 95.8%, respectively, with an overall mean recovery of 98.1 ± 1.96% (RSD = 2.00%) (Table 8). Recoveries of Anthracene in freshwater yielded mean percent recoveries of 99.1, 99.8 and 92.1%, respectively, with an overall mean recovery of 97.0 ± 3.68% (RSD = 3.79%) (Table 9). Recoveries of Fluoranthene in freshwater yielded mean percent recoveries of 98.5, 98.7 and 92.4%, respectively, with an overall mean recovery of 96.6 ± 3.40% (RSD = 3.52%) (Table 10). Recoveries of Pyrene in freshwater yielded mean percent recoveries of 93.5, 95.6 and 91.2%, respectively, with an overall mean recovery of 93.5 ± 2.39% (RSD = 2.55%) (Table 11). Recoveries of Chrysene in freshwater yielded mean percent recoveries of 94.6, 98.6 and 101%, respectively, with an overall mean recovery of 98.1 ± 2.94% (RSD = 3.00%) (Table 12). Recoveries of Benz(a)anthracene in freshwater yielded mean percent recoveries of 94.5, 96.0 and 93.7%, respectively, with an overall mean recovery of 94.7 ± 2.04% (RSD = 2.15%) (Table 13). Recoveries of Benzo(b)fluoranthene in freshwater yielded mean percent recoveries of 91.4, 96.0 and 95.5%, respectively, with an overall mean recovery of 94.3 ± 3.29% (RSD = 3.49%) (Table 14). Recoveries of Benzo(k)fluoranthene in freshwater yielded mean percent recoveries of 92.7, 97.7 and 99.9%, respectively, with an overall mean recovery of 96.8 ± 3.34% (RSD = 3.45%) (Table 15). Recoveries of Benzo(a)pyrene in freshwater yielded mean percent recoveries of 91.9, 97.0 and 98.1%, respectively, with an overall mean recovery of 95.7 ± 3.14% (RSD = 3.28%) (Table 16). Recoveries of Dibenz(a,h,)anthracene in freshwater yielded mean percent recoveries of 92.1, 97.3 and 100%, respectively, with an overall mean recovery of 96.5 ± 3.74% (RSD = 3.88%) (Table 17). Recoveries of Indeno(1,2,3-cd)pyrene in freshwater yielded mean percent recoveries of 94.1, 97.6 and 98.6%, respectively, with an overall mean recovery of 96.8 ± 2.44% (RSD = 2.52%) (Table 18). Recoveries of Benzo(g,h,i)perylene in freshwater yielded mean percent recoveries of 95.9, 96.3 and 97.1%, respectively, with an overall mean recovery of 96.4 ± 1.49% (RSD = 1.55%) (Table 19). Recoveries of Dibenzo(a,e)pyrene in freshwater yielded mean percent recoveries of 93.3, 96.3 and 99.1%, respectively, with an overall mean recovery of 96.2 ± 2.95% (RSD = 3.07%) (Table 20).

REFERENCES

1 **European Commission.** 2002. *Residues: Guidance for Generating and Reporting M ethods of Analysis in Support of Pre-registration Data Requirements for Annex II (Part A, Section 4) and Annex III (Part A, Section 5) of Directive 91/414.* SANCO/3029/99 rev. 4, 11/07/00.

Table 1

Typical HPLC Operational Parameters

INSTRUMENT:	Agilent Model 1100 High Perform ance Liquid Chrom atograph (HPLC) with a either an Agilent Series 1100 Variable Wavelength Detector or a Jasco Model FP-1520 Fluorescence Detector
ANALYTICAL COLUMN:	YMC-Pack ODS AM (150 mm x 4.6 mm, 3 µm particle size)
STOP TIME:	35 minutes
FLOW RATE:	1.00 mL/minute
OVEN TEMPERATURE:	40°C
MOBILE PHASE:	SOLVENT A: 0.1% H_3PO_4 SOLVENT B: CH_3CN

GRADIENT:

Time (min)	%A	%B (m	Flow L/min)
0.01	40.0	60.0	1.000
1.00	40.0	60.0	1.000
30.00	0.0	100.0	1.000
30.10	40.0	60.0	1.000
35.00	40.0	60.0	1.000

INJECTION VOLUME:	100 µL	
APPROXIMATE RETENTION TIMES:	Naphthalene = 6.7 min. Acenaphthylene = 7.7 min. 1-Methylnaphthalene = 8.8 min. 2-Methylnaphthalene = 9.1 min. Fluorene = 9.7 min. Acenaphthene = 9.9 min. Phenanthrene = 10.6 min. Anthracene = 11.3 min. Fluoranthene = 12.9 min. Pyrene = 13.7 min.	Chrysene = 16.0 min. Benz(a)anthracene = 16.3 min. Benzo(b)fluoranthene = 19.0 min. Benzo(k)fluoranthene = 19.4 min. Benzo(a)pyrene = 20.1 min. Dibenz(a,h,)anthracene = 21.7 min. Indeno(1,2,3-cd)pyrene = 23 min. Benzo(g,h,i)perylene = 23.2 min. Dibenzo(a,e)pyrene = 25 min.
PRIMARY ANALYTICAL WAVELENGTHS	UV = 220 nm; Fluorescence = 340 nm to 425nm	

Table 2

Method Verification Recoveries of Naphthalene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	10.1	101	10.0	Mean = 100
VMAS-17	Matrix Fortification	10.0	10.0	100		Std. Dev. = 0.581
VMAS-18	Matrix Fortification	10.0	9.99	99.9		RSD = 0.581%
VMAS-19	Matrix Fortification	10.0	10.0	100		
VMAS-20	Matrix Fortification	10.0	9.94	99.4		
VMAS-21	Matrix Fortification	40.0	40.0	99.9	40.1	Mean = 101
VMAS-22	Matrix Fortification	40.0	40.2	101		Std. Dev. = 0.576
VMAS-23	Matrix Fortification	40.0	40.3	101		RSD = 0.570%
VMAS-24	Matrix Fortification	40.0	40.2	101		
VMAS-25	Matrix Fortification	40.0	40.0	100		
VMAS-26	Matrix Fortification	100	90.9	90.9	88.8	Mean = 88.8
VMAS-27	Matrix Fortification	100	92.9	92.9		Std. Dev. = 3.01
VMAS-28	Matrix Fortification	100	86.7	86.7		RSD = 3.39%
VMAS-29	Matrix Fortification	100	87.5	87.5		
VMAS-30	Matrix Fortification	100	85.8	85.8		
			Mean =	96.4		
			Std. Dev.=	5.82		
			RSD =	6.04%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 3

Method Verification Recoveries of Acenaphthylene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.96	99.6	9.92	Mean = 99.1
VMAS-17	Matrix Fortification	10.0	9.89	98.9		Std. Dev. = 0.647
VMAS-18	Matrix Fortification	10.0	9.88	98.8		RSD = 0.652%
VMAS-19	Matrix Fortification	10.0	10.0	100		
VMAS-20	Matrix Fortification	10.0	9.84	98.4		
VMAS-21	Matrix Fortification	40.0	39.8	99.5	39.9	Mean = 99.8
VMAS-22	Matrix Fortification	40.0	40.0	99.9		Std. Dev. = 0.235
VMAS-23	Matrix Fortification	40.0	40.0	100		RSD = 0.235%
VMAS-24	Matrix Fortification	40.0	40.0	100		
VMAS-25	Matrix Fortification	40.0	39.8	99.6		
VMAS-26	Matrix Fortification	100	96.2	96.2	96.4	Mean = 96.4
VMAS-27	Matrix Fortification	100	98.0	98.0		Std. Dev. = 0.923
VMAS-28	Matrix Fortification	100	96.0	96.0		RSD = 0.957%
VMAS-29	Matrix Fortification	100	96.0	96.0		
VMAS-30	Matrix Fortification	100	95.7	95.7		
			Mean =	98.5		
			Std. Dev.=	1.67		
			RSD =	1.70%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 4

Method Verification Recoveries of 1-Methylnaphthalene in Freshwater
Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	10.1	101	10.0	Mean = 100
VMAS-17	Matrix Fortification	10.0	10.0	100		Std. Dev. = 0.683
VMAS-18	Matrix Fortification	10.0	9.99	99.9		RSD = 0.683%
VMAS-19	Matrix Fortification	10.0	10.1	101		
VMAS-20	Matrix Fortification	10.0	9.95	99.5		
VMAS-21	Matrix Fortification	40.0	40.0	99.9	40.1	Mean = 101
VMAS-22	Matrix Fortification	40.0	40.3	101		Std. Dev. = 0.631
VMAS-23	Matrix Fortification	40.0	40.2	101		RSD = 0.625%
VMAS-24	Matrix Fortification	40.0	40.2	101		
VMAS-25	Matrix Fortification	40.0	39.9	99.8		
VMAS-26	Matrix Fortification	100	90.9	90.9	89.2	Mean = 89.2
VMAS-27	Matrix Fortification	100	93.9	93.3		Std. Dev. = 2.83
VMAS-28	Matrix Fortification	100	87.5	87.5		RSD = 3.18%
VMAS-29	Matrix Fortification	100	87.7	87.7		
VMAS-30	Matrix Fortification	100	86.5	86.5		
			Mean =	96.6		
			Std. Dev.=	5.62		
			RSD =	5.82%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 5

Method Verification Recoveries of 2-Methylnaphthalene in Freshwater
Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	10.1	101	9.99	Mean = 100
VMAS-17	Matrix Fortification	10.0	10.0	100		Std. Dev. = 0.720
VMAS-18	Matrix Fortification	10.0	9.97	99.7		RSD = 0.720%
VMAS-19	Matrix Fortification	10.0	10.0	100		
VMAS-20	Matrix Fortification	10.0	9.90	99.0		
VMAS-21	Matrix Fortification	40.0	40.0	100	40.1	Mean = 100
VMAS-22	Matrix Fortification	40.0	40.3	101		Std. Dev. = 0.780
VMAS-23	Matrix Fortification	40.0	40.2	101		RSD = 0.780%
VMAS-24	Matrix Fortification	40.0	40.3	101		
VMAS-25	Matrix Fortification	40.0	39.7	99.3		
VMAS-26	Matrix Fortification	100	88.8	88.8	86.6	Mean = 86.6
VMAS-27	Matrix Fortification	100	91.4	91.4		Std. Dev. = 3.35
VMAS-28	Matrix Fortification	100	84.6	84.6		RSD = 3.87%
VMAS-29	Matrix Fortification	100	84.9	84.9		
VMAS-30	Matrix Fortification	100	83.4	83.4		
			Mean =	95.6		
			Std. Dev.=	6.84		
			RSD =	7.15%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 6

Method Verification Recoveries of Fluorene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.74	97.4	9.80	Mean = 98.0
VMAS-17	Matrix Fortification	10.0	9.87	98.7		Std. Dev. = 1.17
VMAS-18	Matrix Fortification	10.0	9.81	98.1		RSD = 1.19%
VMAS-19	Matrix Fortification	10.0	9.93	99.3		
VMAS-20	Matrix Fortification	10.0	9.63	96.3		
VMAS-21	Matrix Fortification	40.0	40.0	99.9	40.0	Mean = 100
VMAS-22	Matrix Fortification	40.0	40.2	101		Std. Dev. = 0.844
VMAS-23	Matrix Fortification	40.0	40.3	101		RSD = 0.843%
VMAS-24	Matrix Fortification	40.0	40.1	100		
VMAS-25	Matrix Fortification	40.0	39.6	99.0		
VMAS-26	Matrix Fortification	100	95.3	95.3	95.7	Mean = 95.7
VMAS-27	Matrix Fortification	100	97.4	97.4		Std. Dev. = 0.986
VMAS-28	Matrix Fortification	100	95.8	95.8		RSD = 1.03%
VMAS-29	Matrix Fortification	100	95.5	95.5		
VMAS-30	Matrix Fortification	100	94.8	94.8		
			Mean =	97.9		
			Std. Dev.=	2.04		
			RSD =	2.08%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 7

Method Verification Recoveries of Acenaphthene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.87	98.7	9.84	Mean = 98.4
VMAS-17	Matrix Fortification	10.0	9.76	97.6		Std. Dev. = 0.820
VMAS-18	Matrix Fortification	10.0	9.82	98.2		RSD = 0.834%
VMAS-19	Matrix Fortification	10.0	9.96	99.6		
VMAS-20	Matrix Fortification	10.0	9.77	97.7		
VMAS-21	Matrix Fortification	40.0	39.8	99.5	3.98	Mean = 99.6
VMAS-22	Matrix Fortification	40.0	39.9	99.8		Std. Dev. = 0.451
VMAS-23	Matrix Fortification	40.0	39.9	99.9		RSD = 0.452%
VMAS-24	Matrix Fortification	40.0	39.9	99.8		
VMAS-25	Matrix Fortification	40.0	39.5	98.8		
VMAS-26	Matrix Fortification	100	93.4	93.4	93.0	Mean = 93.0
VMAS-27	Matrix Fortification	100	95.6	95.6		Std. Dev. = 1.65
VMAS-28	Matrix Fortification	100	92.5	92.5		RSD = 1.77%
VMAS-29	Matrix Fortification	100	91.8	91.8		
VMAS-30	Matrix Fortification	100	91.5	91.5		
			Mean =	97.0		
			Std. Dev.=	3.14		
			RSD =	3.24%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 8

Method Verification Recoveries of Phenanthrene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.94	99.4	9.88	Mean = 98.8
VMAS-17	Matrix Fortification	10.0	9.84	98.4		Std. Dev. = 0.614
VMAS-18	Matrix Fortification	10.0	9.83	98.3		RSD = 0.621%
VMAS-19	Matrix Fortification	10.0	9.95	99.5		
VMAS-20	Matrix Fortification	10.0	9.83	98.3		
VMAS-21	Matrix Fortification	40.0	40.0	99.9	40.0	Mean = 99.6
VMAS-22	Matrix Fortification	40.0	40.2	100		Std. Dev. = 0.795
VMAS-23	Matrix Fortification	40.0	40.2	100		RSD = 0.798%
VMAS-24	Matrix Fortification	40.0	40.1	100		
VMAS-25	Matrix Fortification	40.0	39.3	98.2		
VMAS-26	Matrix Fortification	100	94.8	94.8	95.8	Mean = 95.8
VMAS-27	Matrix Fortification	100	97.4	97.4		Std. Dev. = 1.01
VMAS-28	Matrix Fortification	100	95.8	95.8		RSD = 1.05%
VMAS-29	Matrix Fortification	100	95.8	95.8		
VMAS-30	Matrix Fortification	100	95.1	95.1		
			Mean =	98.1		
			Std. Dev.=	1.96		
			RSD =	2.00%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 9

Method Verification Recoveries of Anthracene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.95	99.5	9.91	Mean = 99.1
VMAS-17	Matrix Fortification	10.0	9.90	99.0		Std. Dev. = 0.495
VMAS-18	Matrix Fortification	10.0	9.92	99.2		RSD = 0.499%
VMAS-19	Matrix Fortification	10.0	9.95	99.5		
VMAS-20	Matrix Fortification	10.0	9.83	98.3		
VMAS-21	Matrix Fortification	40.0	39.8	99.6	39.9	Mean = 99.8
VMAS-22	Matrix Fortification	40.0	40.3	101		Std. Dev. = 1.44
VMAS-23	Matrix Fortification	40.0	40.2	101		RSD = 1.44%
VMAS-24	Matrix Fortification	40.0	40.1	100		
VMAS-25	Matrix Fortification	40.0	39.0	97.5		
VMAS-26	Matrix Fortification	100	92.5	92.5	92.1	Mean = 92.1
VMAS-27	Matrix Fortification	100	93.0	93.0		Std. Dev. = 0.766
VMAS-28	Matrix Fortification	100	92.2	92.2		RSD = 0.832%
VMAS-29	Matrix Fortification	100	91.7	91.7		
VMAS-30	Matrix Fortification	100	91.0	91.0		
			Mean =	97.0		
			Std. Dev.=	3.68		
			RSD =	3.79%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 10

Method Verification Recoveries of Fluoranthene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	10.1	101	9.84	Mean = 98.5
VMAS-17	Matrix Fortification	10.0	9.73	97.3		Std. Dev. = 1.48
VMAS-18	Matrix Fortification	10.0	9.84	98.4		RSD = 1.50%
VMAS-19	Matrix Fortification	10.0	9.76	97.6		
VMAS-20	Matrix Fortification	10.0	9.80	98.0		
VMAS-21	Matrix Fortification	40.0	39.4	98.4	39.5	Mean = 98.7
VMAS-22	Matrix Fortification	40.0	40.0	100		Std. Dev. = 1.48
VMAS-23	Matrix Fortification	40.0	40.1	100		RSD = 1.50%
VMAS-24	Matrix Fortification	40.0	39.6	98.9		
VMAS-25	Matrix Fortification	40.0	38.6	96.4		
VMAS-26	Matrix Fortification	100	92.3	92.3	92.4	Mean = 92.4
VMAS-27	Matrix Fortification	100	95.5	95.5		Std. Dev. = 1.97
VMAS-28	Matrix Fortification	100	92.2	92.2		RSD = 2.13%
VMAS-29	Matrix Fortification	100	92.1	92.1		
VMAS-30	Matrix Fortification	100	90.0	90.0		
			Mean =	96.6		
			Std. Dev.=	3.40		
			RSD =	3.52%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 11

Method Verification Recoveries of Pyrene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.62	96.2	9.35	Mean = 93.5
VMAS-17	Matrix Fortification	10.0	9.22	92.2		Std. Dev. = 1.55
VMAS-18	Matrix Fortification	10.0	9.31	93.1		RSD = 1.66%
VMAS-19	Matrix Fortification	10.0	9.30	93.0		
VMAS-20	Matrix Fortification	10.0	9.30	93.0		
VMAS-21	Matrix Fortification	40.0	38.0	95.0	38.3	Mean = 95.6
VMAS-22	Matrix Fortification	40.0	38.6	96.5		Std. Dev. = 1.49
VMAS-23	Matrix Fortification	40.0	39.0	97.4		RSD = 1.56%
VMAS-24	Matrix Fortification	40.0	38.3	95.8		
VMAS-25	Matrix Fortification	40.0	37.4	93.5		
VMAS-26	Matrix Fortification	100	90.1	90.1	91.2	Mean = 91.2
VMAS-27	Matrix Fortification	100	94.1	94.1		Std. Dev. = 1.80
VMAS-28	Matrix Fortification	100	91.1	91.1		RSD = 1.97%
VMAS-29	Matrix Fortification	100	91.5	91.5		
VMAS-30	Matrix Fortification	100	89.4	89.4		
			Mean =	93.5		
			Std. Dev.=	2.39		
			RSD =	2.55%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 12

Method Verification Recoveries of Chrysene in Freshwater Analyzed by HPLC/UV

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.50	95.0	9.46	Mean = 94.6
VMAS-17	Matrix Fortification	10.0	9.31	93.1		Std. Dev. = 1.34
VMAS-18	Matrix Fortification	10.0	9.55	95.5		RSD = 1.41%
VMAS-19	Matrix Fortification	10.0	9.33	93.3		
VMAS-20	Matrix Fortification	10.0	9.61	96.1		
VMAS-21	Matrix Fortification	40.0	39.7	99.1	39.4	Mean = 98.6
VMAS-22	Matrix Fortification	40.0	39.9	99.7		Std. Dev. = 1.27
VMAS-23	Matrix Fortification	40.0	39.5	98.8		RSD = 1.29%
VMAS-24	Matrix Fortification	40.0	39.6	99.0		
VMAS-25	Matrix Fortification	40.0	38.5	96.4		
VMAS-26	Matrix Fortification	100	101	101	101	Mean = 101
VMAS-27	Matrix Fortification	100	102	102		Std. Dev. = 0.837
VMAS-28	Matrix Fortification	100	100	100		RSD = 0.828%
VMAS-29	Matrix Fortification	100	101	101		
VMAS-30	Matrix Fortification	100	100	100		
			Mean =	98.1		
			Std. Dev.=	2.94		
			RSD =	3.00%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 13

Method Verification Recoveries of Benz(a)anthracene in Freshwater
Analyzed by HPLC with Fluorescence Detection

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.68	96.8	9.45	Mean = 94.5
VMAS-17	Matrix Fortification	10.0	9.37	93.7		Std. Dev. = 1.77
VMAS-18	Matrix Fortification	10.0	9.54	95.4		RSD = 1.87%
VMAS-19	Matrix Fortification	10.0	9.21	92.1		
VMAS-20	Matrix Fortification	10.0	9.45	94.5		
VMAS-21	Matrix Fortification	40.0	38.1	95.2	38.4	Mean = 96.0
VMAS-22	Matrix Fortification	40.0	38.5	96.2		Std. Dev. = 2.32
VMAS-23	Matrix Fortification	40.0	38.9	97.2		RSD = 2.42%
VMAS-24	Matrix Fortification	40.0	39.5	98.8		
VMAS-25	Matrix Fortification	40.0	37.0	92.6		
VMAS-26	Matrix Fortification	100	95.2	95.2	93.7	Mean = 93.7
VMAS-27	Matrix Fortification	100	94.5	94.5		Std. Dev. = 1.61
VMAS-28	Matrix Fortification	100	93.9	93.9		RSD = 1.72%
VMAS-29	Matrix Fortification	100	94.1	94.1		
VMAS-30	Matrix Fortification	100	91.0	91.0		
			Mean =	94.7		
			Std. Dev.=	2.04		
			RSD =	2.15%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 14

Method Verification Recoveries of Benzo(b)fluoranthene in Freshwater
Analyzed by HPLC with Fluorescence Detection

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.29	92.9	9.14	Mean = 91.4
VMAS-17	Matrix Fortification	10.0	9.03	90.3		Std. Dev. = 1.63
VMAS-18	Matrix Fortification	10.0	9.16	91.6		RSD = 1.78%
VMAS-19	Matrix Fortification	10.0	8.92	89.2		
VMAS-20	Matrix Fortification	10.0	9.29	92.9		
VMAS-21	Matrix Fortification	40.0	38.4	96.1	38.4	Mean = 96.0
VMAS-22	Matrix Fortification	40.0	39.4	98.5		Std. Dev. = 2.31
VMAS-23	Matrix Fortification	40.0	39.2	98.1		RSD = 2.41%
VMAS-24	Matrix Fortification	40.0	37.3	93.2		
VMAS-25	Matrix Fortification	40.0	37.7	94.3		
VMAS-26	Matrix Fortification	100	96.9	96.9	95.5	Mean = 95.5
VMAS-27	Matrix Fortification	100	100	100		Std. Dev. = 3.58
VMAS-28	Matrix Fortification	100	93.0	93.0		RSD = 3.75%
VMAS-29	Matrix Fortification	100	96.8	96.8		
VMAS-30	Matrix Fortification	100	90.9	90.9		
			Mean =	94.3		
			Std. Dev.=	3.29		
			RSD =	3.49%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 15

Method Verification Recoveries of Benzo(k)fluoranthene in Freshwater
Analyzed by HPLC with Fluorescence Detection

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.31	93.1	9.27	Mean = 92.7
VMAS-17	Matrix Fortification	10.0	9.22	92.2		Std. Dev. = 1.30
VMAS-18	Matrix Fortification	10.0	9.41	94.1		RSD = 1.40%
VMAS-19	Matrix Fortification	10.0	9.07	90.7		
VMAS-20	Matrix Fortification	10.0	9.33	93.3		
VMAS-21	Matrix Fortification	40.0	39.2	98.0	39.1	Mean = 97.7
VMAS-22	Matrix Fortification	40.0	39.5	98.6		Std. Dev. = 1.27
VMAS-23	Matrix Fortification	40.0	39.3	98.4		RSD = 1.30%
VMAS-24	Matrix Fortification	40.0	39.3	98.2		
VMAS-25	Matrix Fortification	40.0	38.2	95.5		
VMAS-26	Matrix Fortification	100	100	100	99.9	Mean = 99.9
VMAS-27	Matrix Fortification	100	101	101		Std. Dev. = 0.787
VMAS-28	Matrix Fortification	100	99.7	99.7		RSD = 0.788%
VMAS-29	Matrix Fortification	100	100	100		
VMAS-30	Matrix Fortification	100	98.8	98.8		
			Mean =	96.8		
			Std. Dev.=	3.34		
			RSD =	3.45%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 16

Method Verification Recoveries of Benzo(a)pyrene in Freshwater
Analyzed by HPLC with Fluorescence Detection

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.36	93.6	9.19	Mean = 91.9
VMAS-17	Matrix Fortification	10.0	9.20	92.0		Std. Dev. = 2.15
VMAS-18	Matrix Fortification	10.0	9.35	93.5		RSD = 2.34%
VMAS-19	Matrix Fortification	10.0	8.83	88.3		
VMAS-20	Matrix Fortification	10.0	9.23	92.3		
VMAS-21	Matrix Fortification	40.0	38.8	96.9	38.9	Mean = 97.0
VMAS-22	Matrix Fortification	40.0	39.2	98.1		Std. Dev. = 1.27
VMAS-23	Matrix Fortification	40.0	39.1	97.9		RSD = 1.31%
VMAS-24	Matrix Fortification	40.0	38.8	97.0		
VMAS-25	Matrix Fortification	40.0	38.0	94.9		
VMAS-26	Matrix Fortification	100	98.5	98.5	98.1	Mean = 98.1
VMAS-27	Matrix Fortification	100	99.9	99.9		Std. Dev. = 1.28
VMAS-28	Matrix Fortification	100	97.6	97.6		RSD = 1.30%
VMAS-29	Matrix Fortification	100	98.2	98.2		
VMAS-30	Matrix Fortification	100	96.4	96.4		
			Mean =	95.7		
			Std. Dev.=	3.14		
			RSD =	3.28%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 17

Method Verification Recoveries of Dibenz(a,h)anthracene in Freshwater
Analyzed by HPLC with Fluorescence Detection

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.33	93.3	9.21	Mean = 92.1
VMAS-17	Matrix Fortification	10.0	9.29	92.9		Std. Dev. = 2.67
VMAS-18	Matrix Fortification	10.0	9.38	93.8		RSD = 2.90%
VMAS-19	Matrix Fortification	10.0	8.74	87.4		
VMAS-20	Matrix Fortification	10.0	9.33	93.3		
VMAS-21	Matrix Fortification	40.0	39.2	97.9	38.9	Mean = 97.3
VMAS-22	Matrix Fortification	40.0	39.1	97.8		Std. Dev. = 1.21
VMAS-23	Matrix Fortification	40.0	39.0	97.5		RSD = 1.24%
VMAS-24	Matrix Fortification	40.0	39.3	98.2		
VMAS-25	Matrix Fortification	40.0	38.1	95.2		
VMAS-26	Matrix Fortification	100	100	100	100	Mean = 100
VMAS-27	Matrix Fortification	100	100	100		Std. Dev. = 0.743
VMAS-28	Matrix Fortification	100	101	101		RSD = 0.743%
VMAS-29	Matrix Fortification	100	100	100		
VMAS-30	Matrix Fortification	100	98.9	98.9		
			Mean =	96.5		
			Std. Dev.=	3.74		
			RSD =	3.88%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 18

Method Verification Recoveries of Indeno(1,2,3-cd)pyrene in Freshwater
Analyzed by HPLC with Fluorescence Detection

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.45	94.5	9.41	Mean = 94.1
VMAS-17	Matrix Fortification	10.0	9.38	93.8		Std. Dev. = 1.63
VMAS-18	Matrix Fortification	10.0	9.56	95.6		RSD = 1.74%
VMAS-19	Matrix Fortification	10.0	9.15	91.5		
VMAS-20	Matrix Fortification	10.0	9.53	95.3		
VMAS-21	Matrix Fortification	40.0	39.2	98.0	39.1	Mean = 97.6
VMAS-22	Matrix Fortification	40.0	39.7	99.3		Std. Dev. = 1.42
VMAS-23	Matrix Fortification	40.0	39.4	98.4		RSD = 1.45%
VMAS-24	Matrix Fortification	40.0	38.4	95.9		
VMAS-25	Matrix Fortification	40.0	38.6	96.4		
VMAS-26	Matrix Fortification	100	99.0	99.0	98.6	Mean = 98.6
VMAS-27	Matrix Fortification	100	101	101		Std. Dev. = 1.62
VMAS-28	Matrix Fortification	100	97.2	97.2		RSD = 1.64%
VMAS-29	Matrix Fortification	100	98.7	98.7		
VMAS-30	Matrix Fortification	100	97.0	97.0		
			Mean =	96.8		
			Std. Dev.=	2.44		
			RSD =	2.52%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 19

Method Verification Recoveries of Benzo(g,h,i)perylene in Freshwater
Analyzed by HPLC with Fluorescence Detection

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.28	92.8	9.59	Mean = 95.9
VMAS-17	Matrix Fortification	10.0	9.63	96.3		Std. Dev. = 1.99
VMAS-18	Matrix Fortification	10.0	9.67	96.7		RSD = 2.08%
VMAS-19	Matrix Fortification	10.0	9.55	95.5		
VMAS-20	Matrix Fortification	10.0	9.82	98.2		
VMAS-21	Matrix Fortification	40.0	38.5	96.3	38.5	Mean = 96.3
VMAS-22	Matrix Fortification	40.0	39.1	97.7		Std. Dev. = 0.802
VMAS-23	Matrix Fortification	40.0	38.5	96.2		RSD = 0.833%
VMAS-24	Matrix Fortification	40.0	38.3	95.8		
VMAS-25	Matrix Fortification	40.0	38.3	95.7		
VMAS-26	Matrix Fortification	100	96.3	96.3	97.1	Mean = 97.1
VMAS-27	Matrix Fortification	100	98.5	98.5		Std. Dev. = 1.50
VMAS-28	Matrix Fortification	100	96.3	96.3		RSD = 1.55%
VMAS-29	Matrix Fortification	100	98.8	98.8		
VMAS-30	Matrix Fortification	100	95.4	95.4		
			Mean =	96.4		
			Std. Dev.=	1.49		
			RSD =	1.55%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 20

Method Verification Recoveries of Dibenzo(a,e)pyrene in Freshwater
Analyzed by HPLC with Fluorescence Detection

Sample		Concentration (µg/L)		Percent Recovery[2]	Mean Measured (µg/L)	Mean % Recovery Std. Dev. (RSD)
Number (472C-104-)	Type	Fortified	Measured[1,2]			
VREB-3	Reagent Blank	0.0	< LOQ	--	< LOQ	--
VREB-4	Reagent Blank	0.0	< LOQ	--		
VMAB-3	Matrix Blank	0.0	< LOQ	--	< LOQ	--
VMAB-4	Matrix Blank	0.0	< LOQ	--		
VMAS-16	Matrix Fortification	10.0	9.26	92.6	9.33	Mean = 93.3
VMAS-17	Matrix Fortification	10.0	9.28	92.8		Std. Dev. = 2.34
VMAS-18	Matrix Fortification	10.0	9.55	95.5		RSD = 2.51%
VMAS-19	Matrix Fortification	10.0	8.99	89.9		
VMAS-20	Matrix Fortification	10.0	9.55	95.5		
VMAS-21	Matrix Fortification	40.0	38.4	96.0	38.5	Mean = 96.3
VMAS-22	Matrix Fortification	40.0	39.5	98.7		Std. Dev. = 1.73
VMAS-23	Matrix Fortification	40.0	38.4	96.0		RSD = 1.79%
VMAS-24	Matrix Fortification	40.0	37.6	93.9		
VMAS-25	Matrix Fortification	40.0	38.7	96.8		
VMAS-26	Matrix Fortification	100	99.7	99.7	99.1	Mean = 99.1
VMAS-27	Matrix Fortification	100	101	101		Std. Dev. = 1.36
VMAS-28	Matrix Fortification	100	98.0	98.0		RSD = 1.38%
VMAS-29	Matrix Fortification	100	99.1	99.1		
VMAS-30	Matrix Fortification	100	97.6	97.6		
			Mean =	96.2		
			Std. Dev.=	2.95		
			RSD =	3.07%		
			N =	15		

[1] The limit of quantitation (LOQ) was 5.00 µg/L, calculated as the product of the concentration of the lowest standard (5.00 µg/L) and the dilution factor of the matrix blanks (1.00).

[2] Results were generated using Excel 2000 in the full precision mode. Manual calculations may differ slightly.

Table 21

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Naphthalene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC/UV

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0	MAB-1	24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0	MAS-1	24	9.97	99.7
	MAS-4	48	10.0	100
	MAS-7	72	9.98	99.8
	MAS-10	96	10.2	102
40.0	MAS-2	24	40.1	100
	MAS-5	48	40.3	101
	MAS-8	72	40.2	100
	MAS-11	96	40.9	102
100	MAS-3	24	84.2	84.2
	MAS-6	48	93.3	93.3
	MAS-9	72	85.8	85.8
	MAS-12	96	94.9	94.9
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 22

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Acenaphthylene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC/UV

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.0	100
	MAS-4	48	10.0	100
	MAS-7	72	10.1	101
	MAS-10	96	10.1	101
40.0 MAS-2		24	40.1	100
	MAS-5	48	40.1	100
	MAS-8	72	39.9	99.8
	MAS-11	96	40.3	101
100	MAS-3	24	93.5	93.5
	MAS-6	48	97.2	97.2
	MAS-9	72	94.9	94.9
	MAS-12	96	97.1	97.1
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 23

Matrix Blanks, Matrix Fortifications and Measured Concentrations of 1-Methylnaphthalene in Samples
Collected from a Water Accommodated Fraction (WAF)
Equilibration Trial Analyzed by HPLC/UV

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.0	100
	MAS-4	48	10.1	101
	MAS-7	72	9.99	99.9
	MAS-10	96	10.2	102
40.0 MAS-2		24	40.1	100
	MAS-5	48	40.2	100
	MAS-8	72	40.2	100
	MAS-11	96	40.8	102
100	MAS-3	24	84.8	84.8
	MAS-6	48	93.4	93.4
	MAS-9	72	86.3	86.3
	MAS-12	96	94.2	94.2
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).
[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 24

Matrix Blanks, Matrix Fortifications and Measured Concentrations of 2-Methylnaphthalene in Samples
Collected from a Water Accommodated Fraction (WAF)
Equilibration Trial Analyzed by HPLC/UV

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	9.99	99.9
	MAS-4	48	10.0	100
	MAS-7	72	10.0	100
	MAS-10	96	10.2	102
40.0 MAS-2		24	40.2	100
	MAS-5	48	40.2	100
	MAS-8	72	40.3	101
	MAS-11	96	40.9	102
100	MAS-3	24	81.6	81.6
	MAS-6	48	91.8	91.8
	MAS-9	72	84.2	84.2
	MAS-12	96	93.1	93.1
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).
[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 25

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Fluorene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC/UV

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.1	101
	MAS-4	48	9.62	96.2
	MAS-7	72	9.97	99.7
	MAS-10	96	9.96	99.6
40.0 MAS-2		24	40.3	101
	MAS-5	48	39.8	99.4
	MAS-8	72	40.0	100
	MAS-11	96	40.2	101
100	MAS-3	24	93.7	93.7
	MAS-6	48	97.2	97.2
	MAS-9	72	94.8	94.8
	MAS-12	96	95.5	95.5
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 26

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Acenaphthene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC/UV

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	9.91	99.1
	MAS-4	48	10.0	100
	MAS-7	72	10.1	101
	MAS-10	96	10.1	101
40.0 MAS-2		24	40.0	100
	MAS-5	48	40.0	100
	MAS-8	72	40.0	100
	MAS-11	96	40.5	101
100	MAS-3	24	91.4	91.4
	MAS-6	48	96.7	96.7
	MAS-9	72	92.4	92.4
	MAS-12	96	96.0	96.0
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 27

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Phenanthrene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC/UV

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.1	101
	MAS-4	48	9.99	99.9
	MAS-7	72	10.2	102
	MAS-10	96	10.0	100
40.0 MAS-2		24	40.1	100
	MAS-5	48	39.7	99.3
	MAS-8	72	39.7	99.3
	MAS-11	96	40.1	100
100	MAS-3	24	95.1	95.1
	MAS-6	48	97.2	97.2
	MAS-9	72	92.2	92.2
	MAS-12	96	95.0	95.0
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 28

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Anthracene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.1	101
	MAS-4	48	9.98	99.8
	MAS-7	72	10.1	101
	MAS-10	96	10.1	101
40.0 MAS-2		24	40.6	101
	MAS-5	48	39.9	99.6
	MAS-8	72	39.9	99.8
	MAS-11	96	40.2	101
100	MAS-3	24	94.2	94.2
	MAS-6	48	96.6	96.6
	MAS-9	72	90.3	90.3
	MAS-12	96	94.2	94.2
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 29

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Fluoranthene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.3	103
	MAS-4	48	9.98	99.8
	MAS-7	72		102
	MAS-10	96	10.2	103
			10.3	
40.0 MAS-2		24	40.0	100
	MAS-5	48	39.1	97.6
	MAS-8	72	39.4	98.4
	MAS-11	96	40.0	99.9
100	MAS-3	24	93.3	93.3
	MAS-6	48	96.2	96.2
	MAS-9	72	82.1	82.1
	MAS-12	96	95.4	95.4
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 30

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Pyrene in Samples

Collected from a Water Accommodated Fraction (WAF)
Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.2	102
	MAS-4	48	10.1	101
	MAS-7	72	9.92	99.2
	MAS-10	96	10.2	102
40.0 MAS-2		24	39.6	99.1
	MAS-5	48	39.5	98.8
	MAS-8	72	38.7	96.9
	MAS-11	96	39.6	99.1
100	MAS-3	24	91.6	91.6
	MAS-6	48	96.7	96.7
	MAS-9	72	80.0	80.0
	MAS-12	96	94.4	94.4
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 31

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Chrysene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0	MAB-1	24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0	MAS-1	24	10.2	102
	MAS-4	48	10.1	101
	MAS-7	72	10.2	102
	MAS-10	96	9.88	98.8
40.0	MAS-2	24	39.5	98.6
	MAS-5	48	39.0	97.5
	MAS-8	72	39.3	98.2
	MAS-11	96	39.9	99.7
100	MAS-3	24	100	100
	MAS-6	48	98.6	98.6
	MAS-9	72	95.8	95.8
	MAS-12	96	104	104
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 32

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Benz(a)anthracene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0	MAB-1	24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0	MAS-1	24	10.5	105
	MAS-4	48	10.1	101
	MAS-7	72	10.4	104
	MAS-10	96	10.2	102
40.0	MAS-2	24	39.1	97.7
	MAS-5	48	37.9	94.9
	MAS-8	72	38.2	95.5
	MAS-11	96	38.9	97.2
100	MAS-3	24	96.0	96.0
	MAS-6	48	95.3	95.3
	MAS-9	72	87.5	87.5
	MAS-12	96	98.2	98.2
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 33

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Benzo(b)fluoranthene in Samples
Collected from a Water Accommodated Fraction (WAF)
Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.5	105
	MAS-4	48	9.58	95.8
	MAS-7	72	10.3	103
	MAS-10	96	10.1	101
40.0 MAS-2		24	39.4	98.6
	MAS-5	48	37.3	93.3
	MAS-8	72	38.9	97.3
	MAS-11	96	38.5	96.3
100	MAS-3	24	93.6	93.6
	MAS-6	48	95.1	95.1
	MAS-9	72	85.2	85.2
	MAS-12	96	98.0	98.0
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).
[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 34

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Benzo(k)fluoranthene in Samples

Collected from a Water Accommodated Fraction (WAF)
Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.1	101
	MAS-4	48	9.91	99.1
	MAS-7	72	10.2	102
	MAS-10	96	9.82	98.2
40.0 MAS-2		24	39.5	98.7
	MAS-5	48	38.7	96.8
	MAS-8	72	39.1	97.7
	MAS-11	96	39.4	98.5
100	MAS-3	24	99.6	99.6
	MAS-6	48	98.1	98.1
	MAS-9	72	95.8	95.8
	MAS-12	96	102	102
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 35

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Benzo(a)pyrene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.3	103
	MAS-4	48	9.83	98.3
	MAS-7	72	10.3	103
	MAS-10	96	9.98	99.8
40.0 MAS-2		24	39.4	98.5
	MAS-5	48	38.6	96.5
	MAS-8	72	38.8	97.1
	MAS-11	96	39.2	98.0
100	MAS-3	24	98.2	98.2
	MAS-6	48	97.9	97.9
	MAS-9	72	93.3	93.3
	MAS-12	96	100	100
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 36

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Dibenz(a,h)anthracene in Samples
Collected from a Water Accommodated Fraction (WAF)

Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0	MAB-1	24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0	MAS-1	24	10.1	101
	MAS-4	48	10.1	101
	MAS-7	72	10.3	103
	MAS-10	96	9.88	98.8
40.0	MAS-2	24	39.5	98.7
	MAS-5	48	39.0	97.5
	MAS-8	72	39.2	97.9
	MAS-11	96	39.4	98.6
100	MAS-3	24	99.8	99.8
	MAS-6	48	99.4	99.4
	MAS-9	72	97.9	97.9
	MAS-12	96	103	103
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).
[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 37

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Indeno(1,2,3-cd)pyrene in Samples
Collected from a Water Accommodated Fraction (WAF)
Equilibration Trial Analyzed by HPLC/UV

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0	MAB-1	24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0	MAS-1	24	10.2	102
	MAS-4	48	9.67	96.7
	MAS-7	72	10.2	102
	MAS-10	96	10.0	100
40.0	MAS-2	24	39.5	98.7
	MAS-5	48	38.1	95.4
	MAS-8	72	39.2	98.0
	MAS-11	96	39.0	97.5
100	MAS-3	24	98.3	98.3
	MAS-6	48	97.2	97.2
	MAS-9	72	93.7	93.7
	MAS-12	96	100	100
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).
[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 38

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Benzo(g,h,i)perylene in Samples
Collected from a Water Accommodated Fraction (WAF)
Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.3	103
	MAS-4	48	9.95	99.5
	MAS-7	72	10.2	102
	MAS-10	96	10.2	102
40.0 MAS-2		24	39.3	98.2
	MAS-5	48	38.2	95.6
	MAS-8	72	38.9	97.2
	MAS-11	96	39.1	97.7
100	MAS-3	24	94.6	94.6
	MAS-6	48	96.2	96.2
	MAS-9	72	91.5	91.5
	MAS-12	96	98.9	98.9
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).
[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

Table 39

Matrix Blanks, Matrix Fortifications and Measured Concentrations of Dibenzo(a,e)pyrene in Samples Collected from a Water Accommodated Fraction (WAF) Equilibration Trial Analyzed by HPLC with Fluorescence Detection

Nominal Concentration (µg/L)	Sample Identification (472C-104-)	Sampling Interval (Hour)	Measured Concentration (µg/L)[1]	Percent of Nominal[2]
0.0 MAB-1		24	< LOQ	--
	MAB-2	48	< LOQ	--
	MAB-3	72	< LOQ	--
	MAB-4	96	< LOQ	--
10.0 MAS-1		24	10.2	102
	MAS-4	48	9.72	97.2
	MAS-7	72		102
	MAS-10	96	10.2	99.4
			9.94	
40.0 MAS-2		24	39.3	98.2
	MAS-5	48	38.4	95.9
	MAS-8	72	39.0	97.6
	MAS-11	96	39.3	98.3
100	MAS-3	24	98.5	98.5
	MAS-6	48	98.0	98.0
	MAS-9	72	94.6	94.6
	MAS-12	96	102	102
1000000	1 (13.2-L bottle)	24	< LOQ	--
	2 (4-L bottle)	24	< LOQ	--
	3 (13.2-L bottle)	48	< LOQ	--
	4 (4-L bottle)	48	< LOQ	--
	5 (13.2-L bottle)	72	< LOQ	--
	6 (4-L bottle)	72	< LOQ	--
	7 (13.2-L bottle)	96	< LOQ	--
	8 (4-L bottle)	96	< LOQ	--

[1] The limit of quantitation (LOQ) was 5.00µg/L, calculated as the product of the lowest standard concentration (5.00 µg/L) and the dilution factor of the matrix blank samples (1.00).

[2] Results were generated using Excel 2000 in full precision mode. Manual calculations may differ slightly.

FLOWCHART FOR THE ANALYSIS OF WATER SOLUBLE PAH COMPONENTS OF PETROLEUM COKE IN FRESHWATER

Prepare samples in freshwater using volumetric flasks, volumetric pipettes, gas tight syringes and culture tubes. Freshwater served as the matrix blanks and bottled water served as the reagent blanks.

↓

For the WAF trial, samples were centrifuged at 14,000 rpm for approximately 5 minutes.

↓

Dilute samples, as necessary, with freshwater such that the final sample concentrations fall within the calibration standard range.

↓

Transfer samples and standards to autosampler vials for analysis by either HPLC/UV or fluorescence detection.

Figure 1. Analytical method flowchart for the analysis of PAH components in freshwater by HPLC.

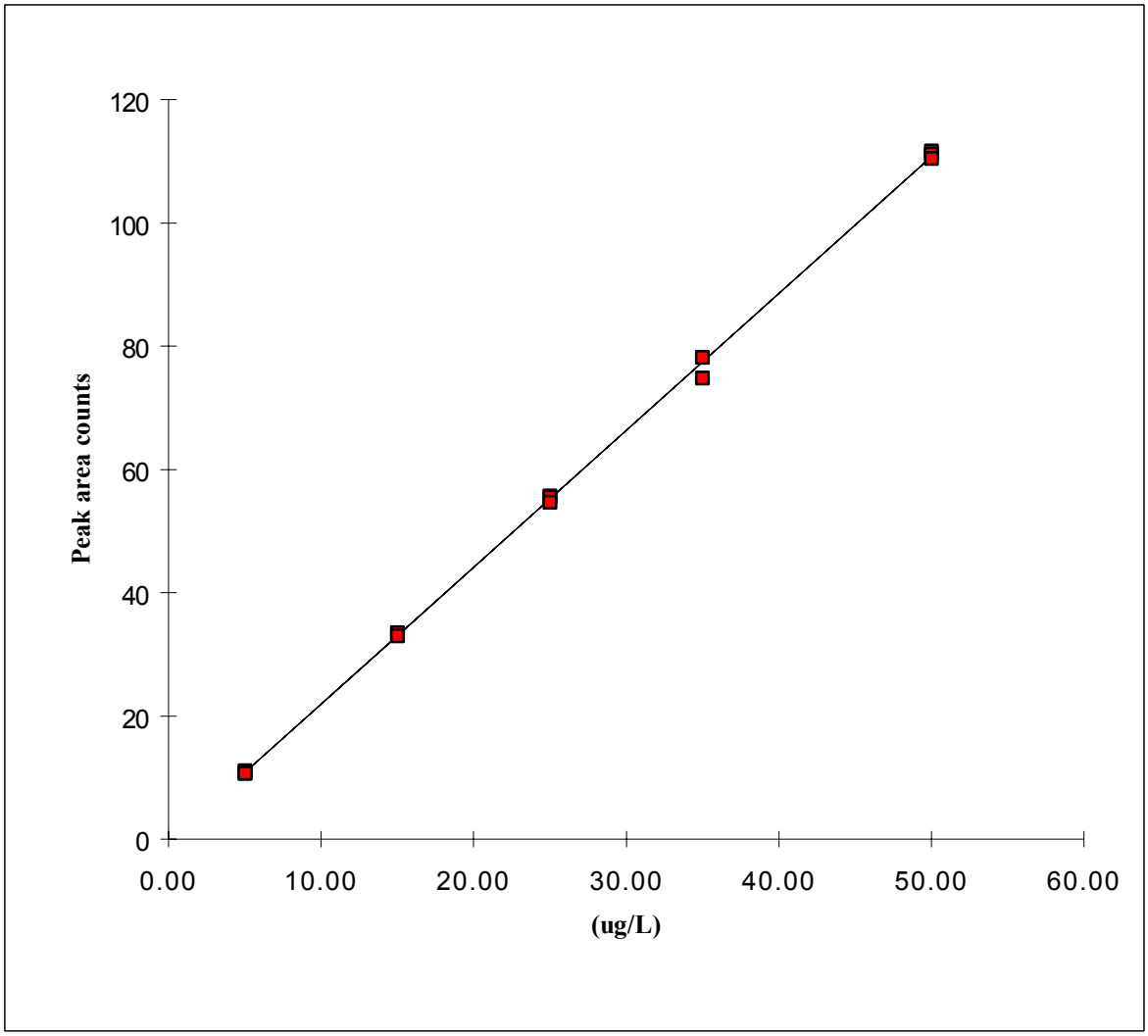

Figure 2. Calibration curve for Naphthalene analyzed by HPLC/UV. Slope = 2.2220; Intercept = -0.3549; R^2 = 0.9995.

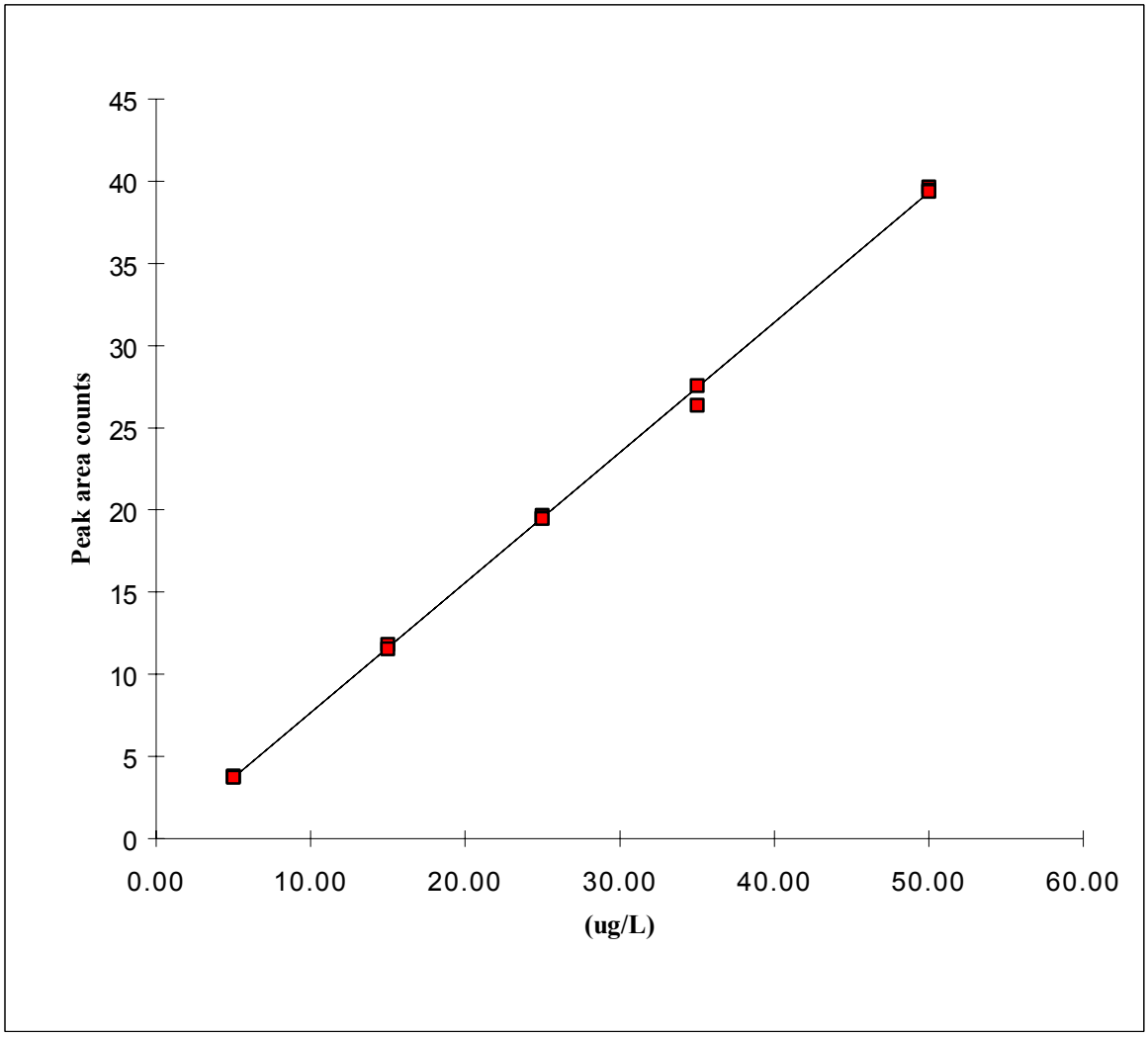

Figure 3. Calibration curve for Acenaphthylene analyzed by HPLC/UV. Slope = 0.7908; Intercept = -0.2349; $R^2 = 0.9994$.

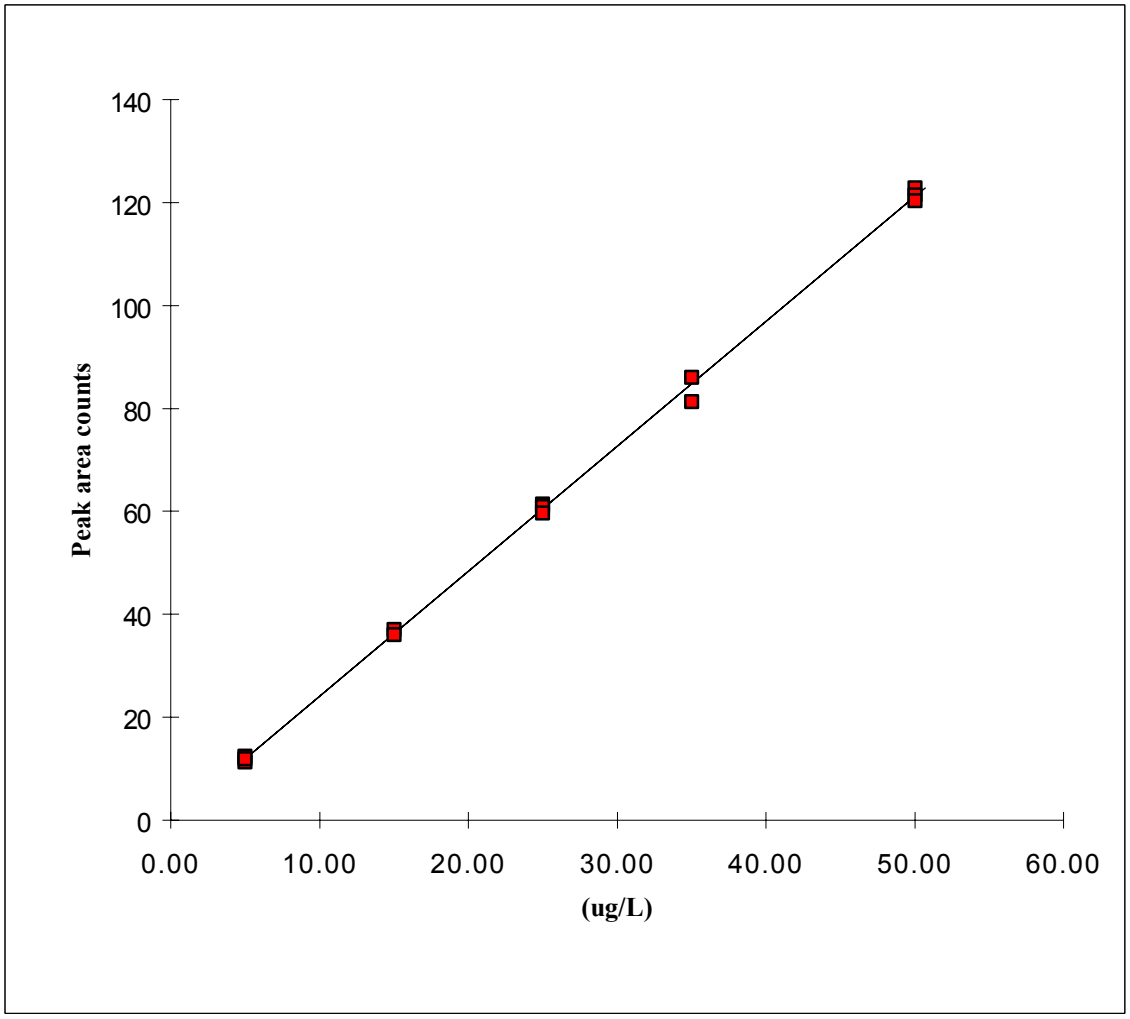

Figure 4. Calibration curve for 1-Methylnaphthalene analyzed by HPLC/UV. Slope = 2.4290; Intercept = -0.2359; R^2 = 0.9990.

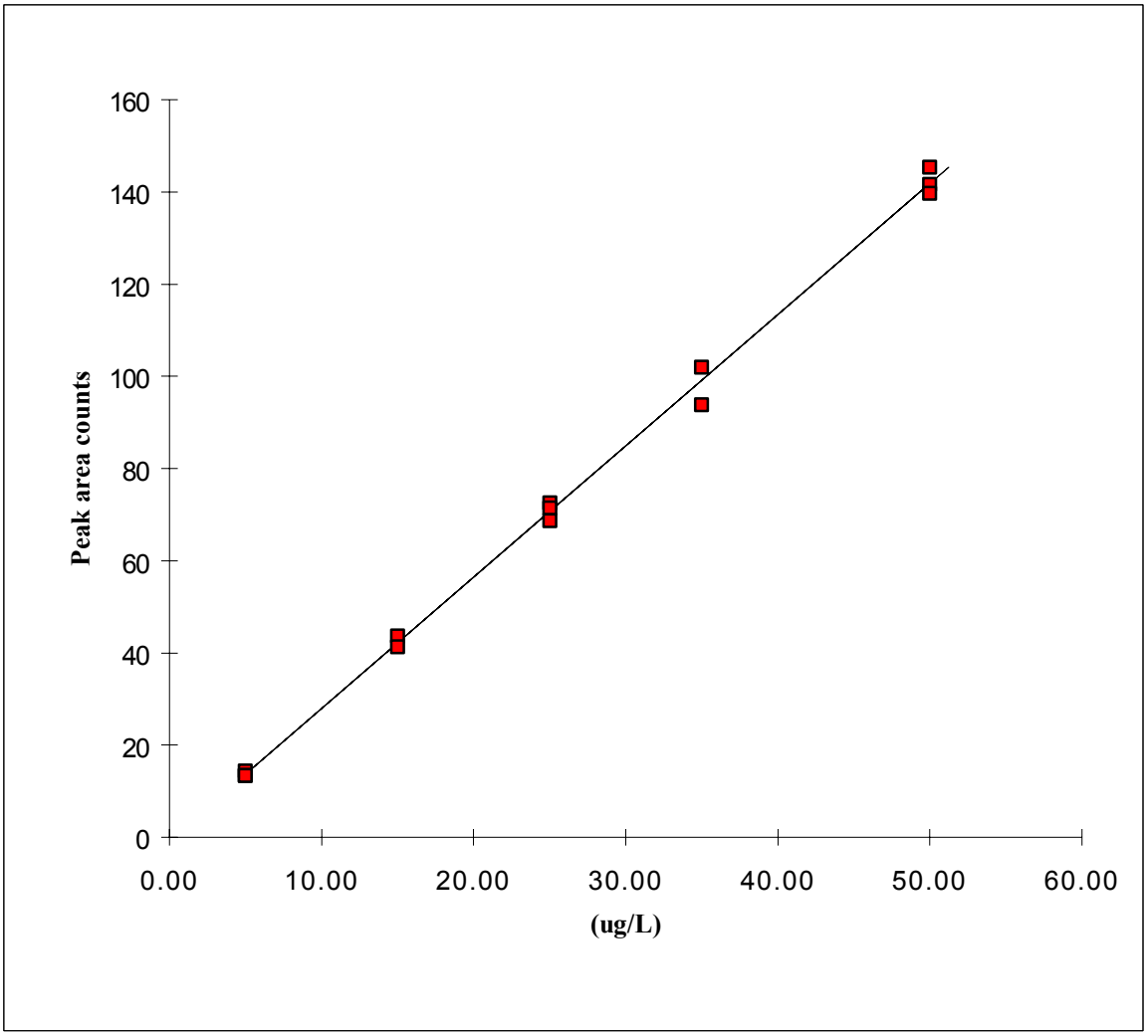

Figure 5. Calibration curve for 2-Methylnaphthalene analyzed by HPLC/UV. Slope = 2.8463; Intercept = -0.4854; $R^2 = 0.9977$.

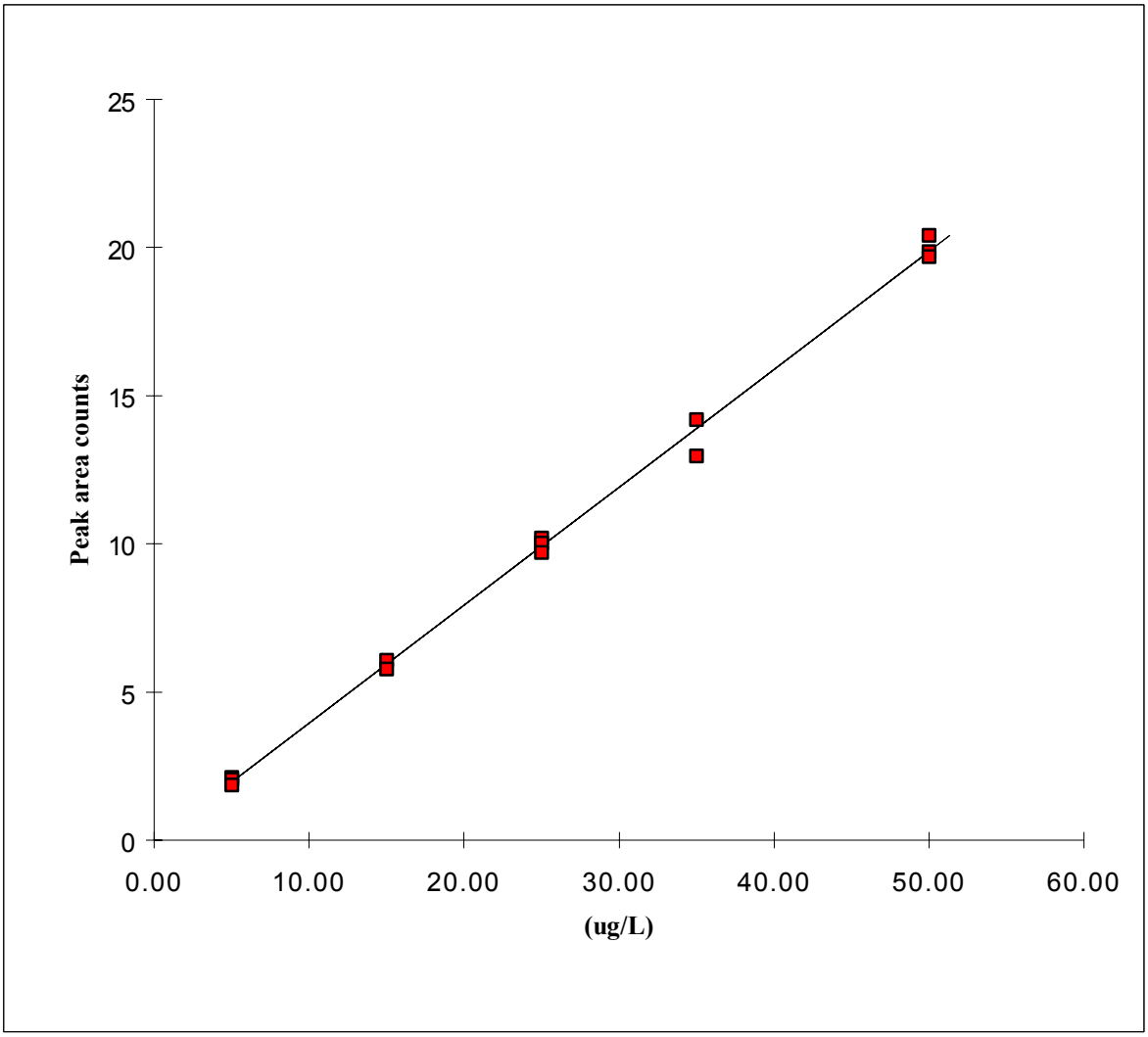

Figure 6. Calibration curve for Fluorene analyzed by HPLC/UV.
Slope = 0.3979; Intercept = -0.0264; R^2 = 0.9973.

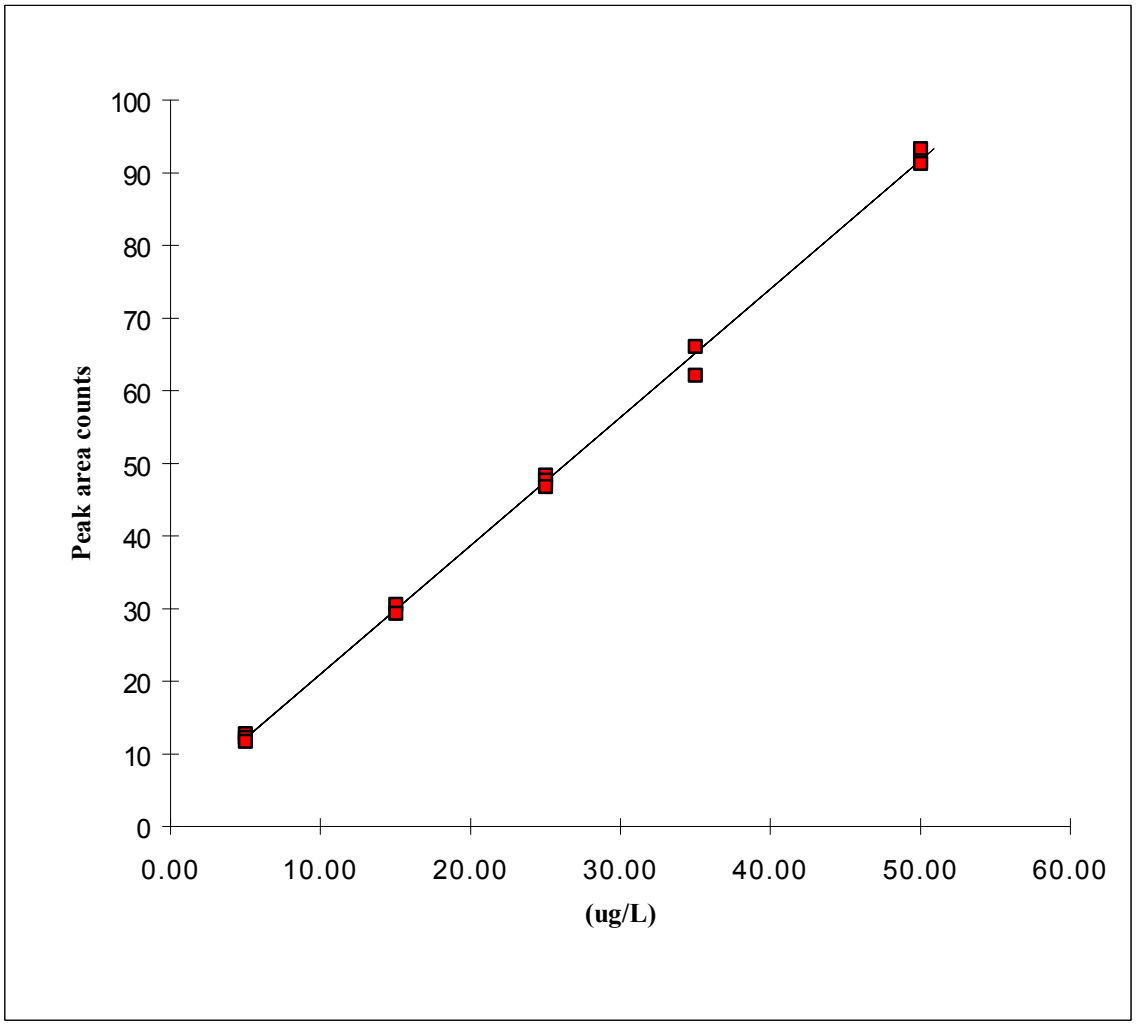

Figure 7. Calibration curve for Acenaphthene analyzed by HPLC/UV. Slope = 1.7666; Intercept = 3.3210; R^2 = 0.9986.

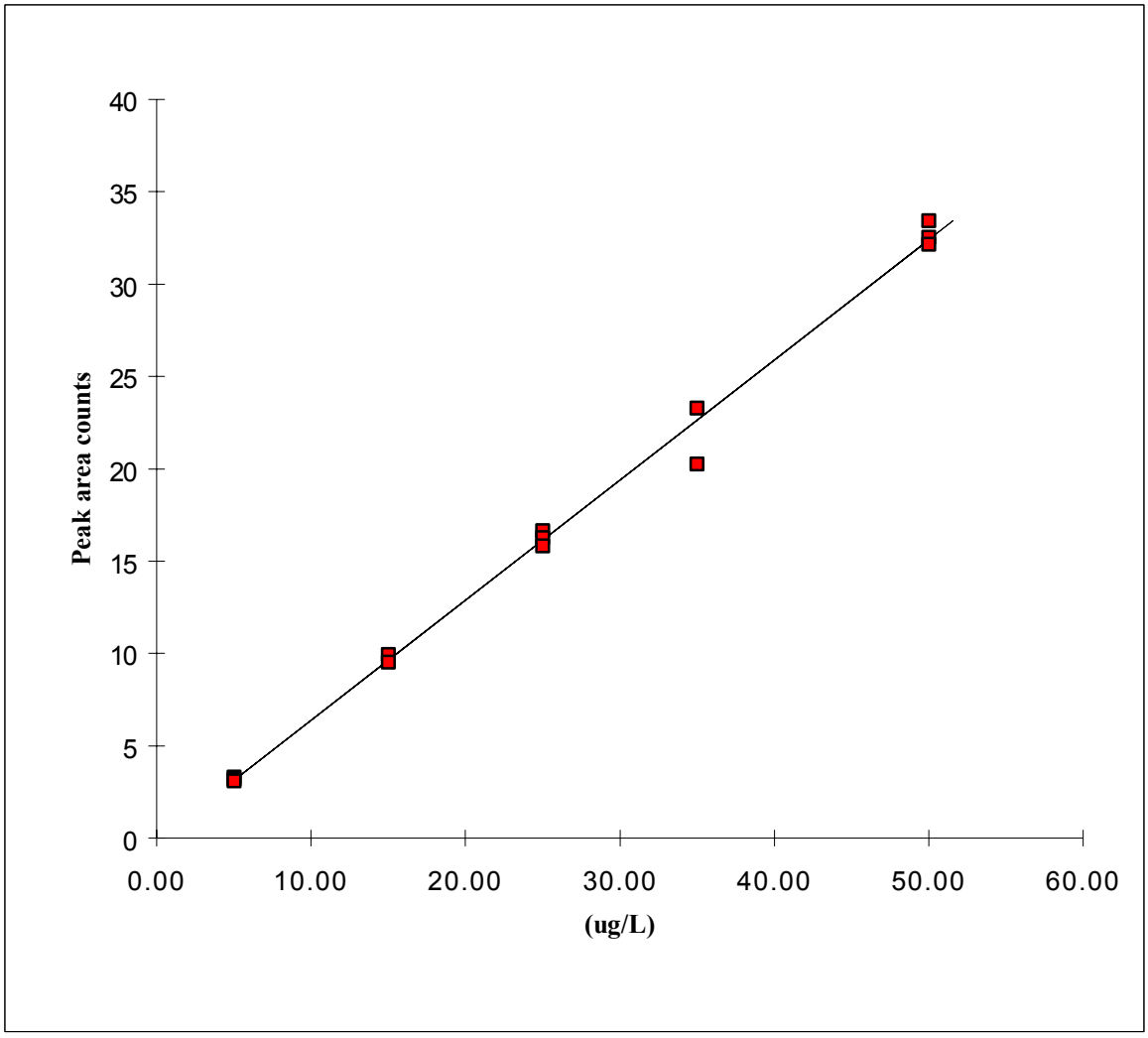

Figure 8. Calibration curve for Phenanthrene analyzed by HPLC/UV. Slope = 0.6505; Intercept = -0.1268; R^2 = 0.9947.

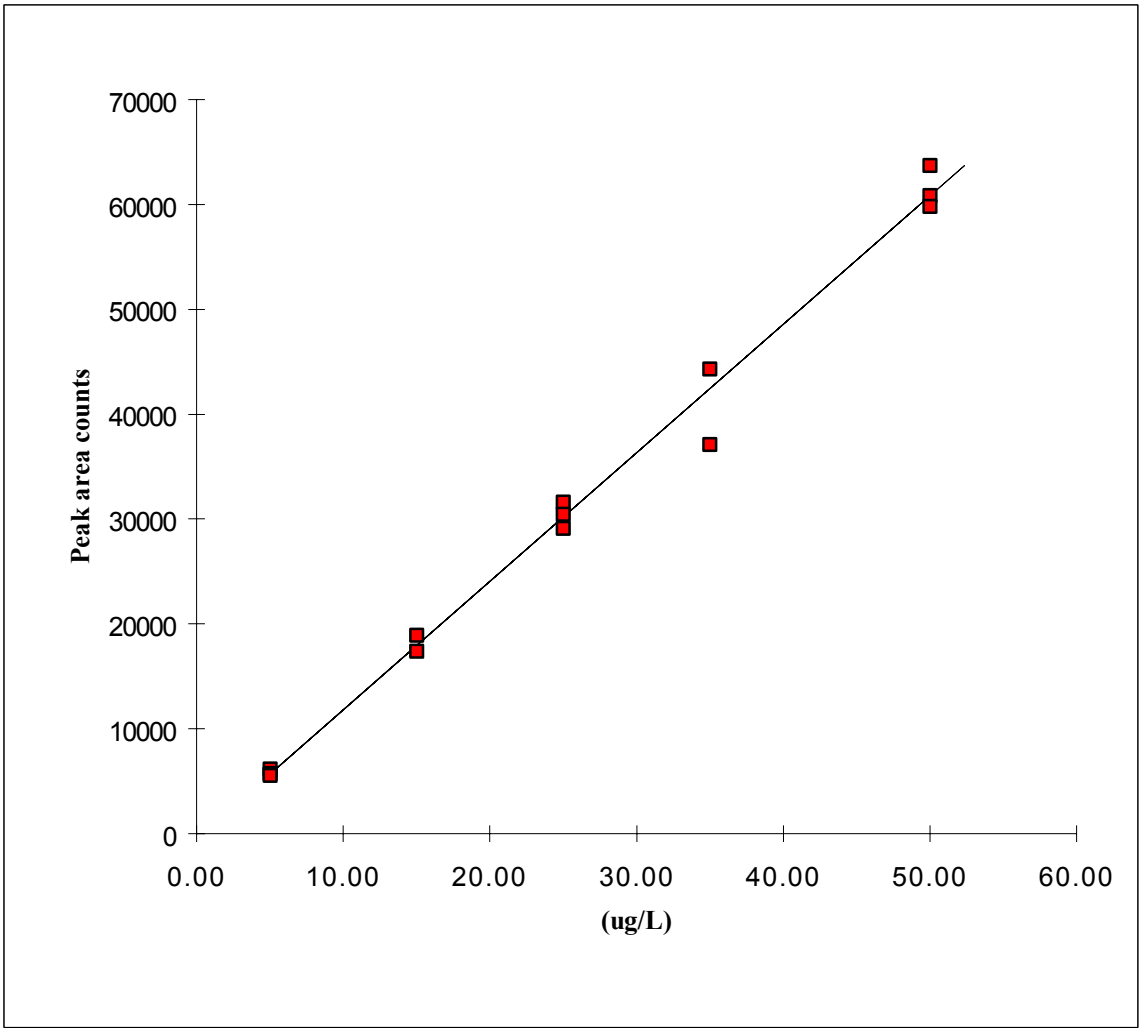

Figure 9. Calibration curve for Anthracene analyzed by HPLC/UV. Slope = 1226.1033; Intercept = -478.7707; R^2 = 0.9912.

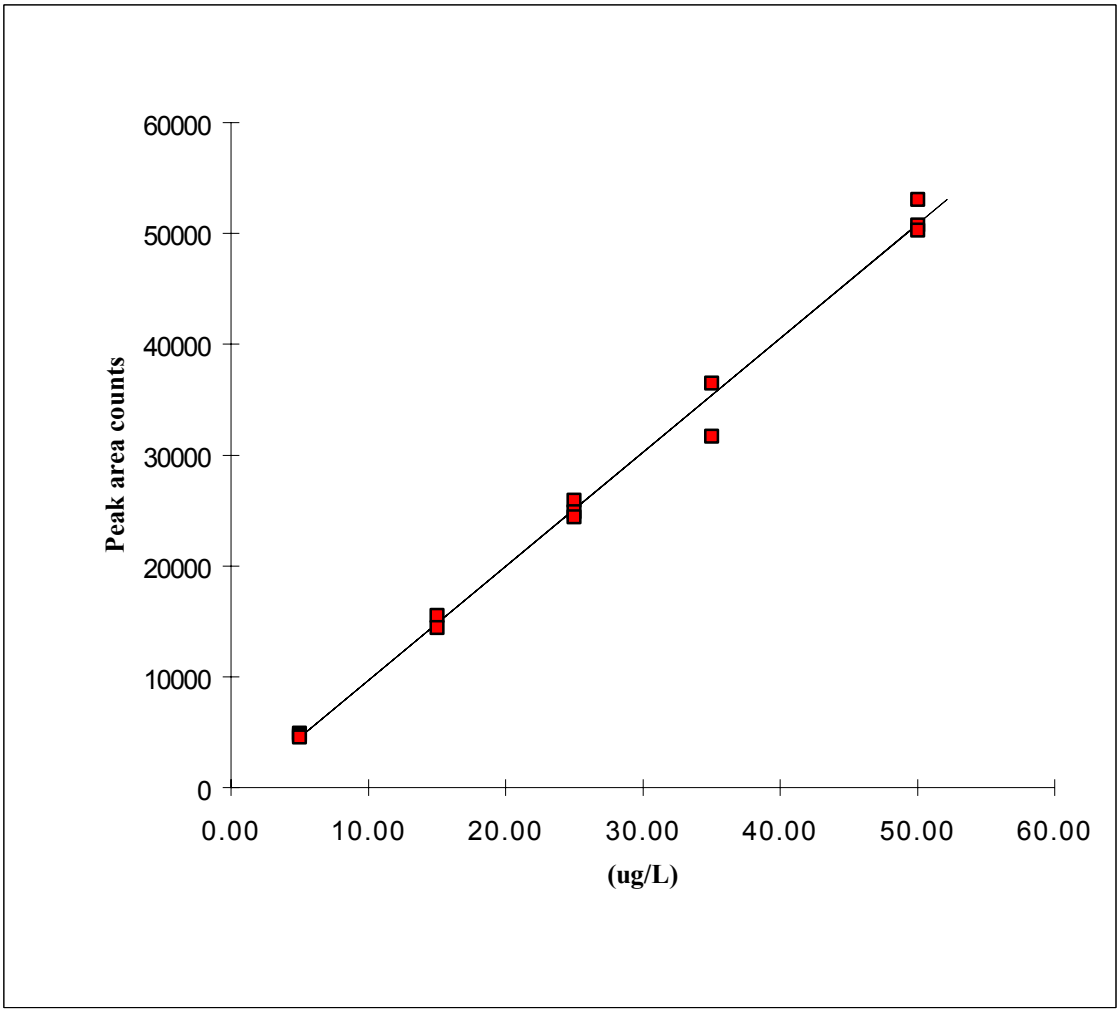

Figure 10. Calibration curve for Fluoranthene analyzed by HPLC with fluorescence detection. Slope = 1028.8562; Intercept = -619.9091; R^2 = 0.9940.

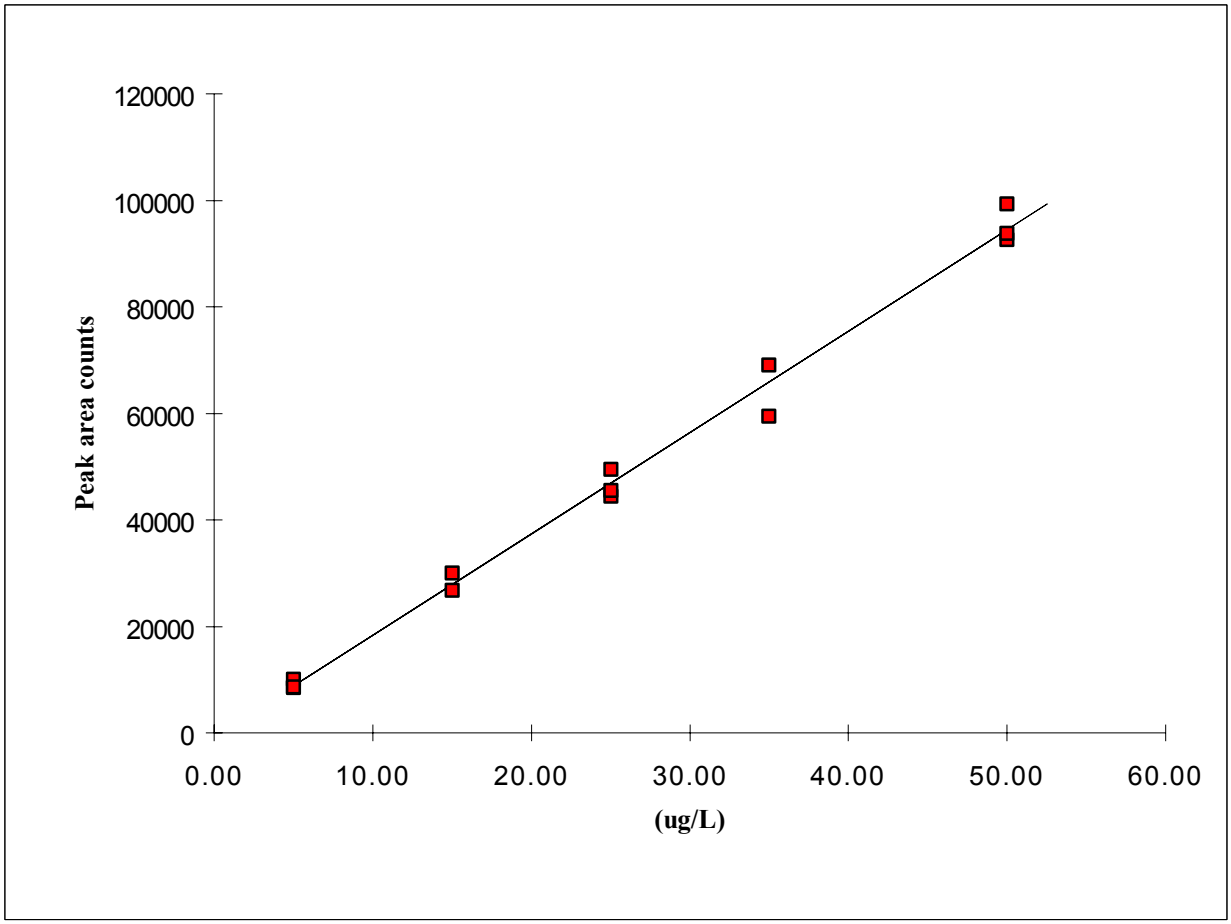

Figure 11. Calibration curve for Pyrene analyzed by HPLC with fluorescence detection. Slope = 1903.6239; Intercept = -731.9500; R^2 = 0.9920.

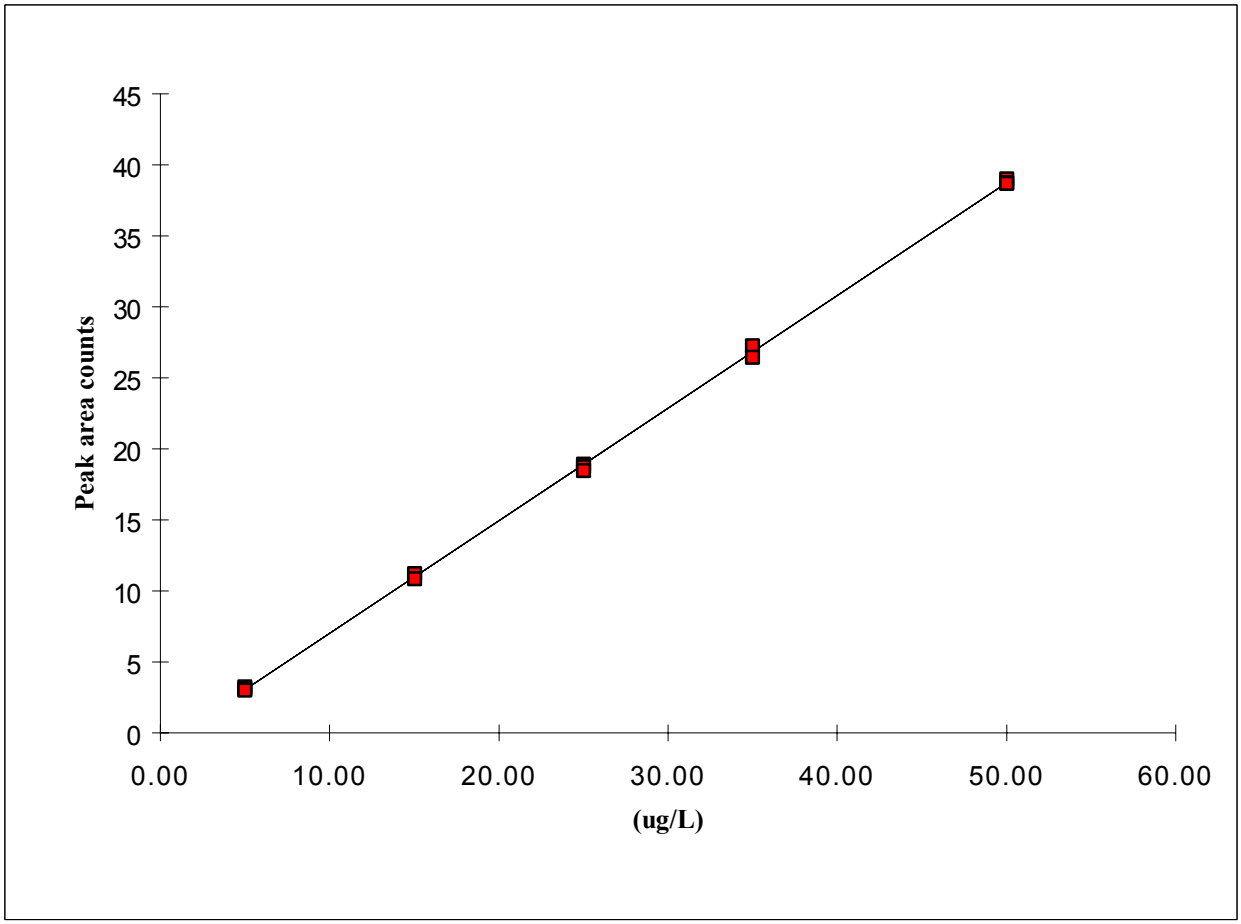

Figure 12. Calibration curve for Chrysene analyzed by HPLC/UV. Slope = 0.7935; Intercept = -0.9282; R^2 = 0.9997.

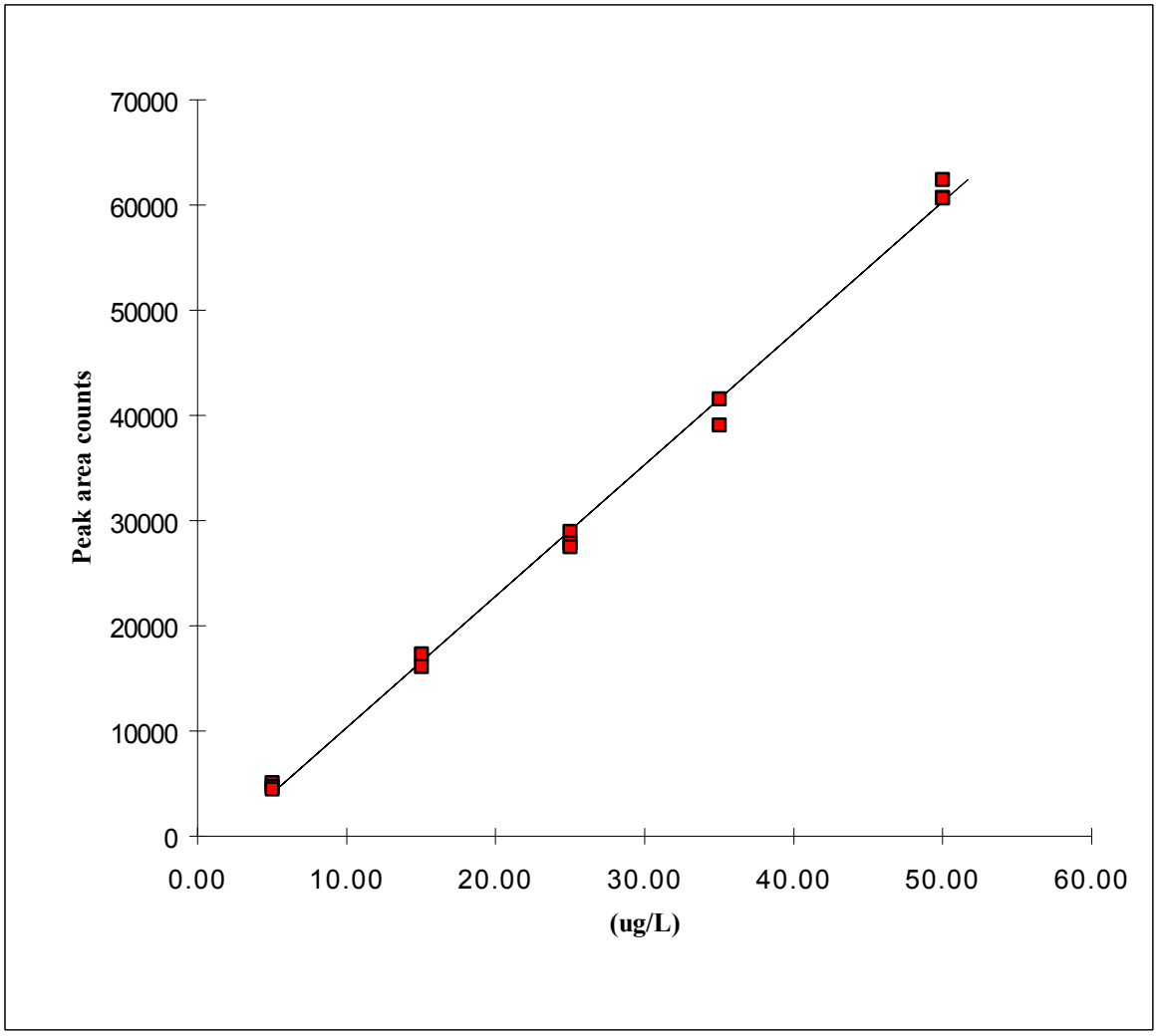

Figure 13. Calibration curve for Benz(a)anthracene analyzed by HPLC with fluorescence detection.

Slope = 1250.4635; Intercept = -2198.3155; R^2 = 0.9968.

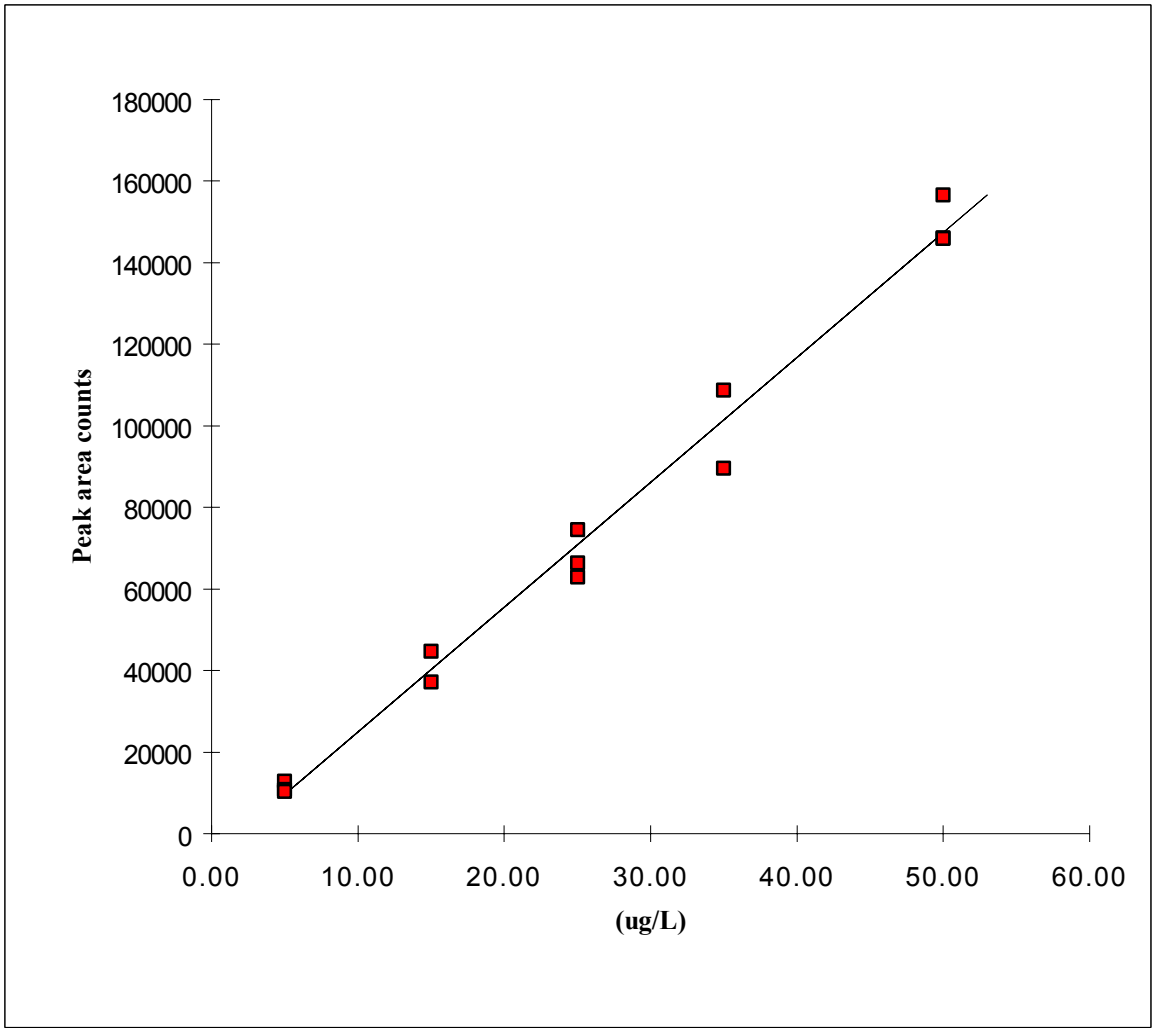

Figure 14. Calibration curve for Benzo(b)fluoranthene analyzed by HPLC with fluorescence detection.

Slope = 3060.8384; Intercept = -5672.1194; R^2 = 0.9871.

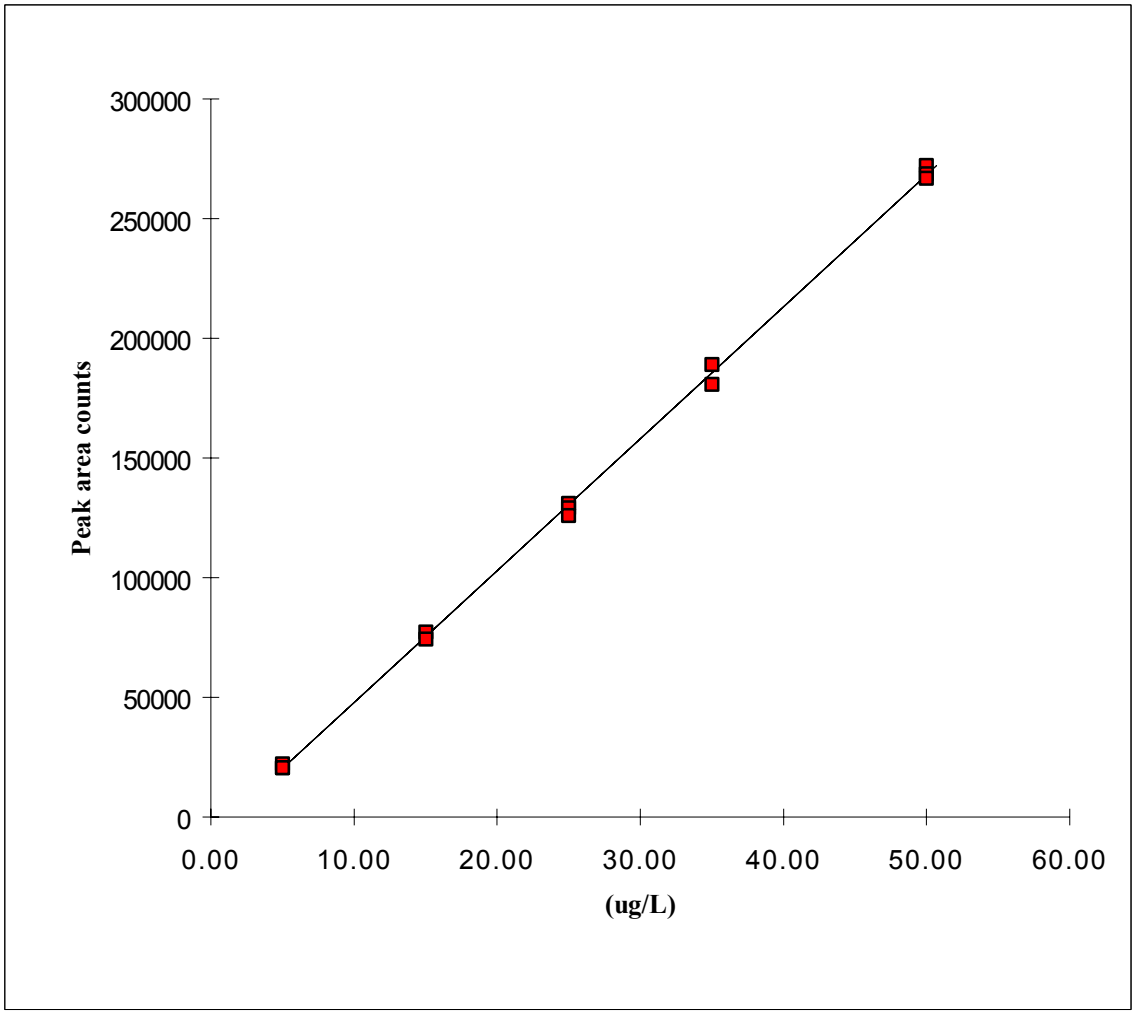

Figure 15. Calibration curve for Benzo(k)fluoroanthene analyzed by HPLC with fluorescence detection.

Slope = 5507.4417; Intercept = -7239.2283; R^2 = 0.9992.

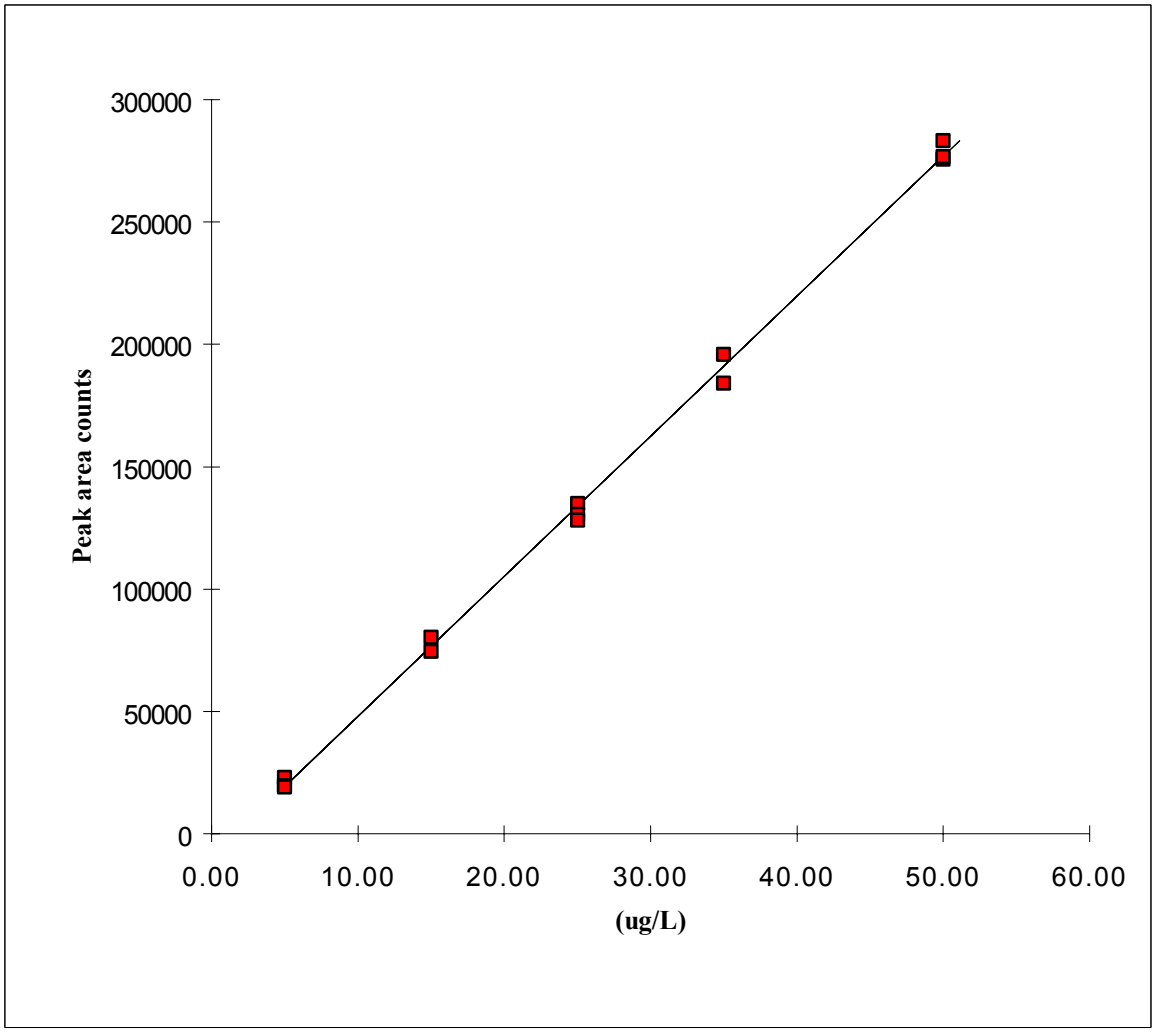

Figure 16. Calibration curve for Benzo(a)pyrene analyzed by HPLC with fluorescence detection. Slope = 5726.8197; Intercept = -9335.3924; $R^2 = 0.9983$.

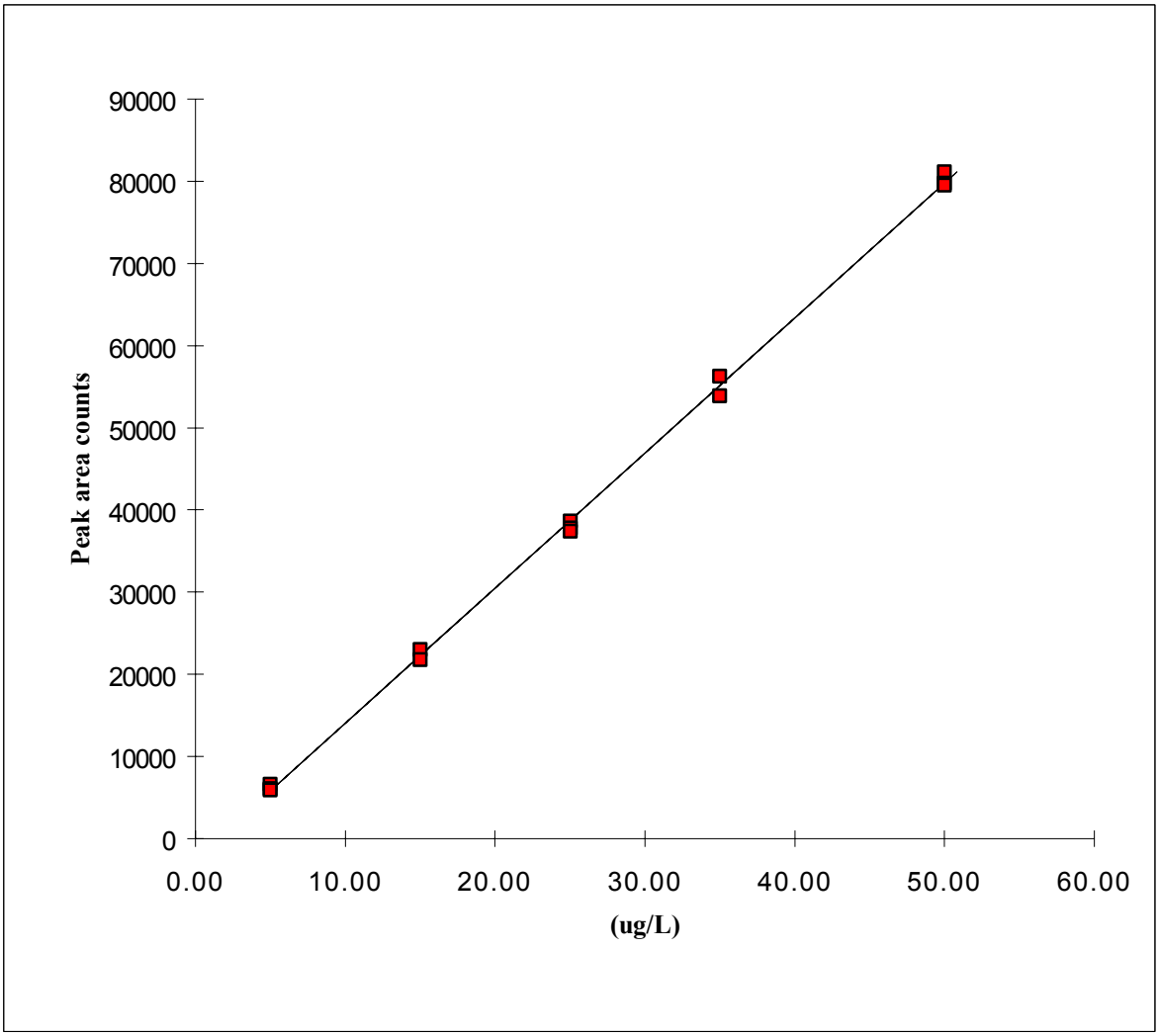

Figure 17. Calibration curve for Dibenz(a,h)anthracene analyzed by HPLC with fluorescence detection.

Slope = 1643.6430; Intercept = -2368.5919; R^2 = 0.9991.

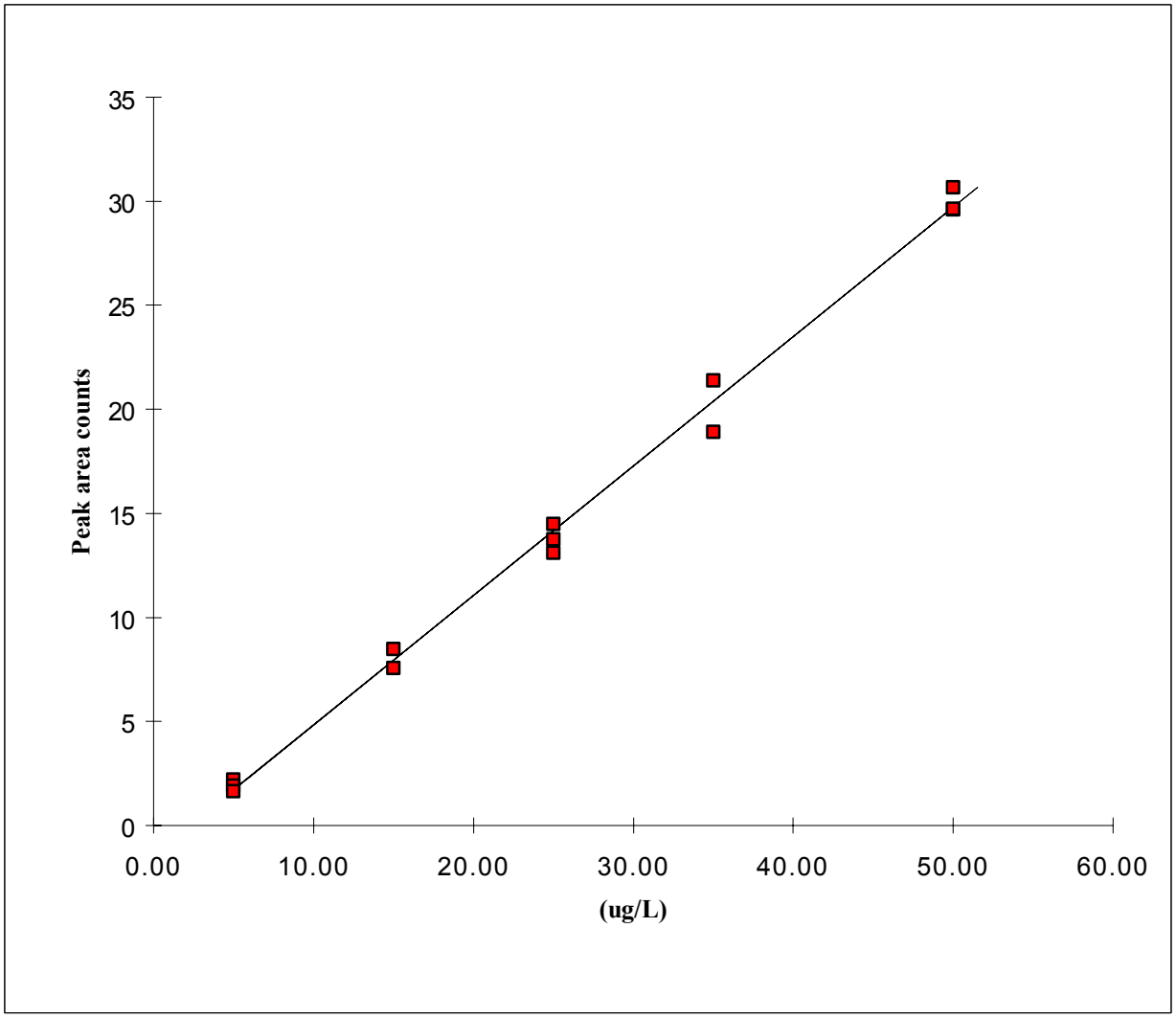

Figure 18. Calibration curve for Indeno(1,2,3-cd)pyrene analyzed by HPLC/UV. Slope = 0.6221; Intercept = -1.3937; R^2 = 0.9954.

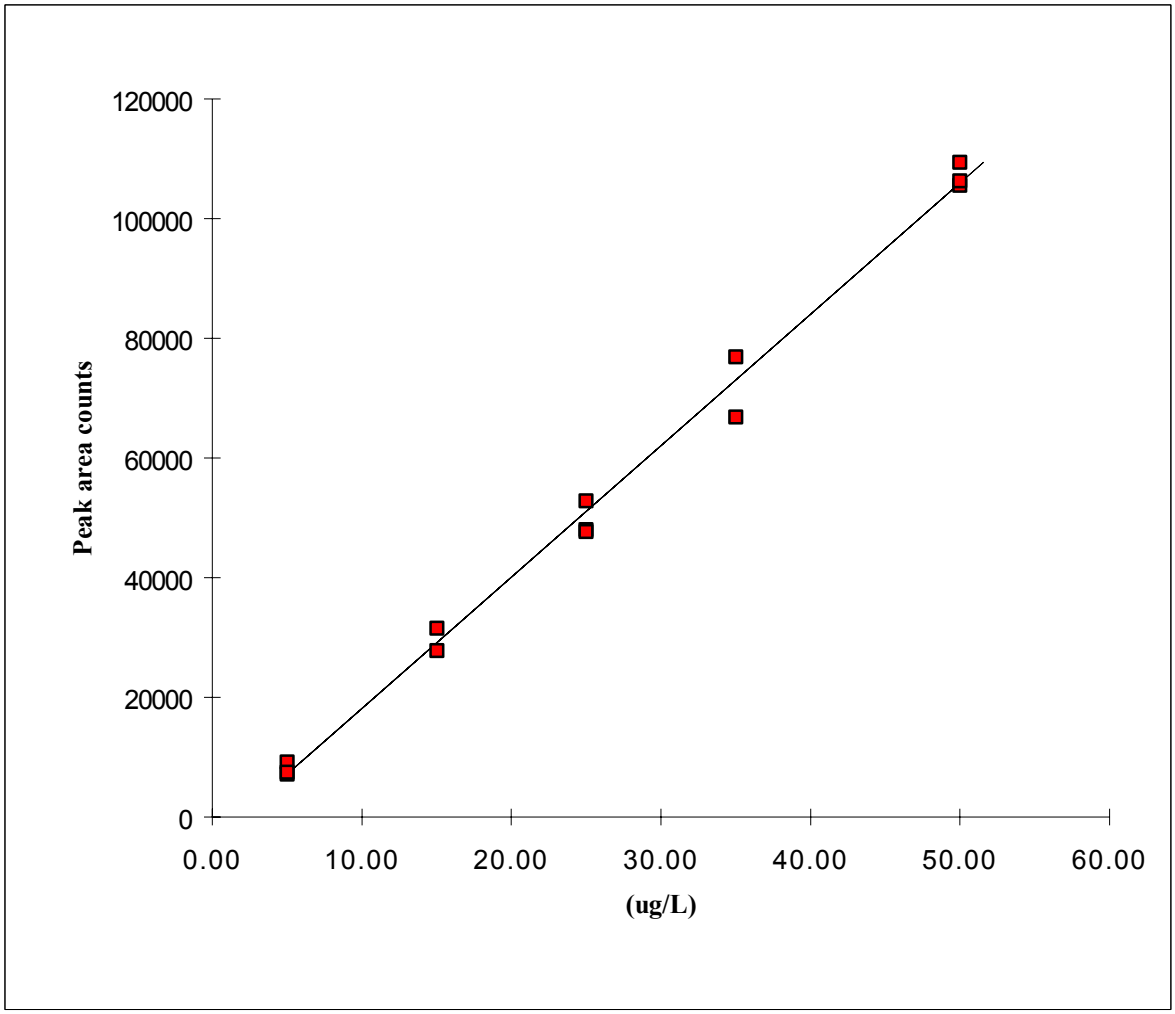

Figure 19. Calibration curve for Benzo(g,h,i)perylene analyzed by HPLC with fluorescence detection.

Slope = 2196.2936; Intercept = -3845.0455; R^2 = 0.9940.

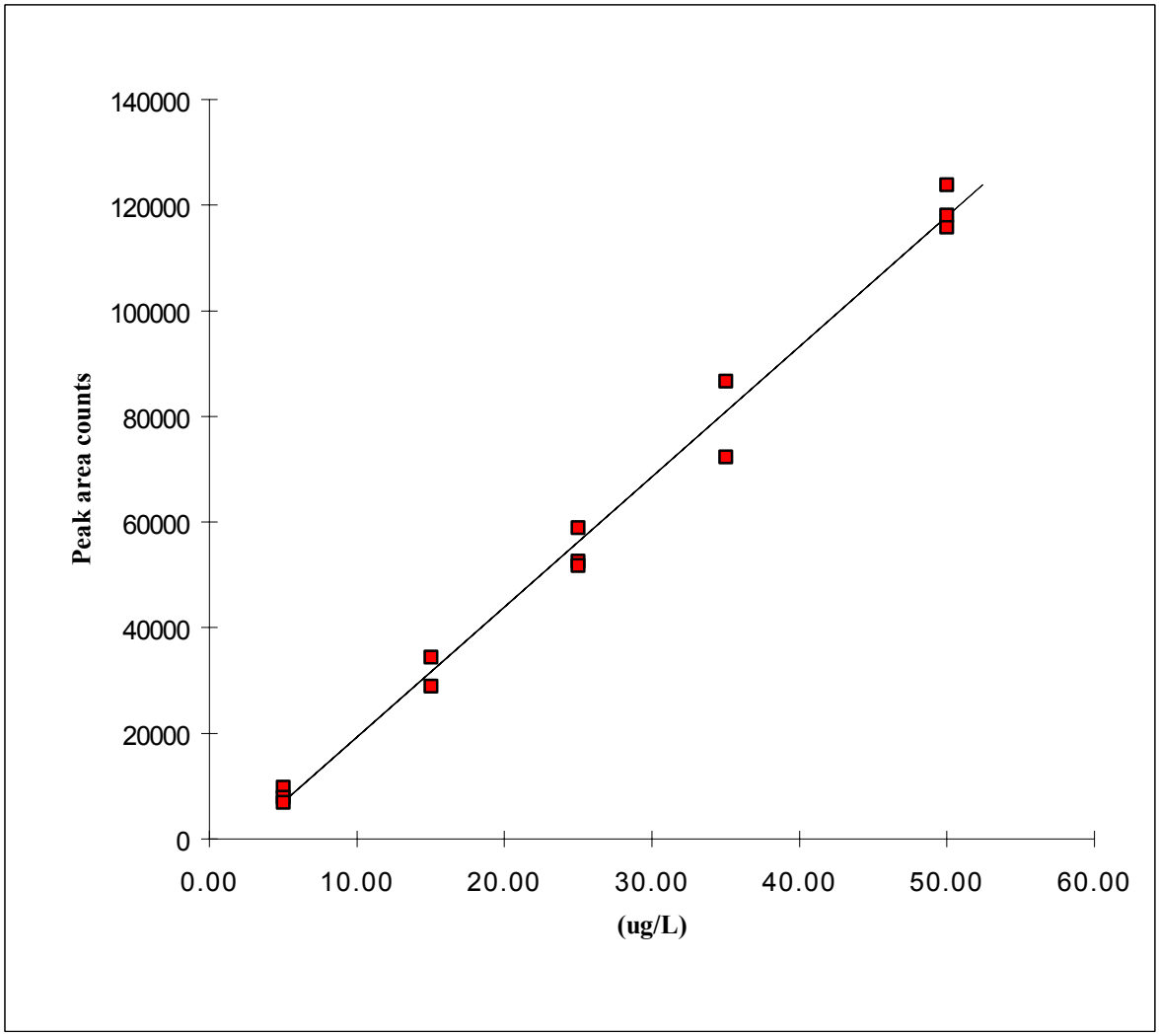

Figure 20. Calibration curve for Dibenzo(a,e)pyrene analyzed by HPLC with fluorescence detection.

Slope = 2464.2422; Intercept = -5373.3490; R^2 = 0.9900.

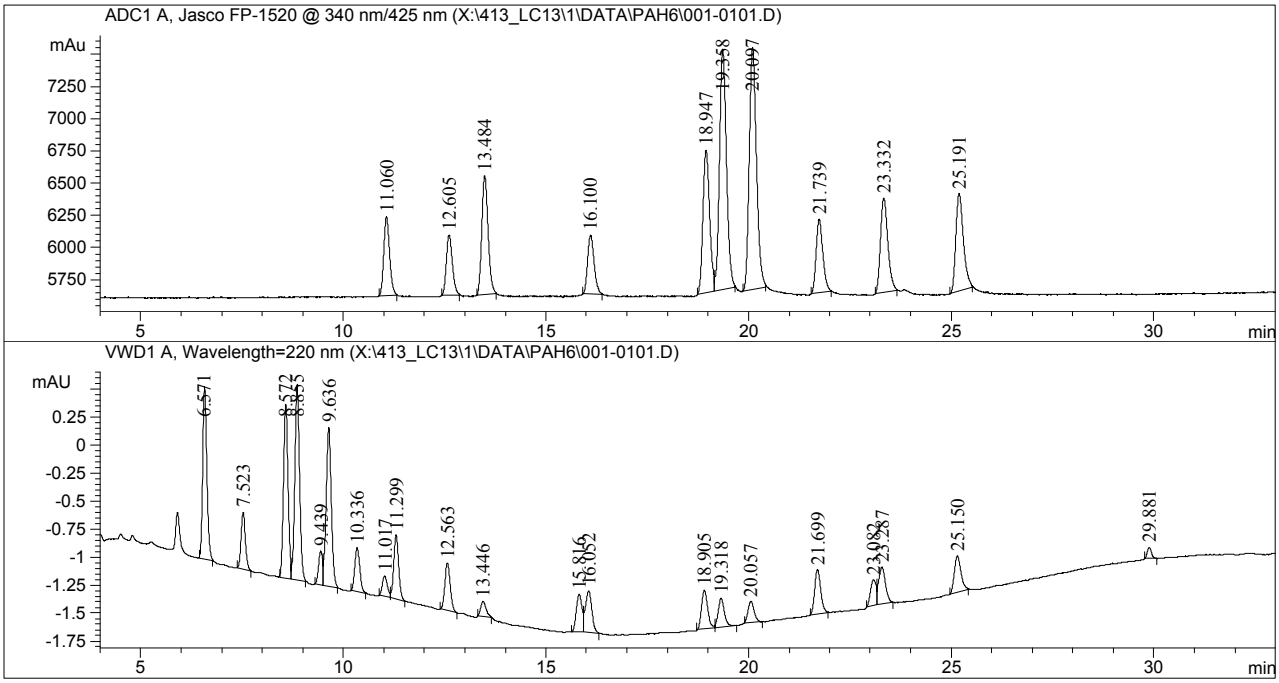

Figure 21. Representative chromatograms of a low-level (5.00 µg/L) calibration standard analyzed by HPLC/UV and fluorescence detection.

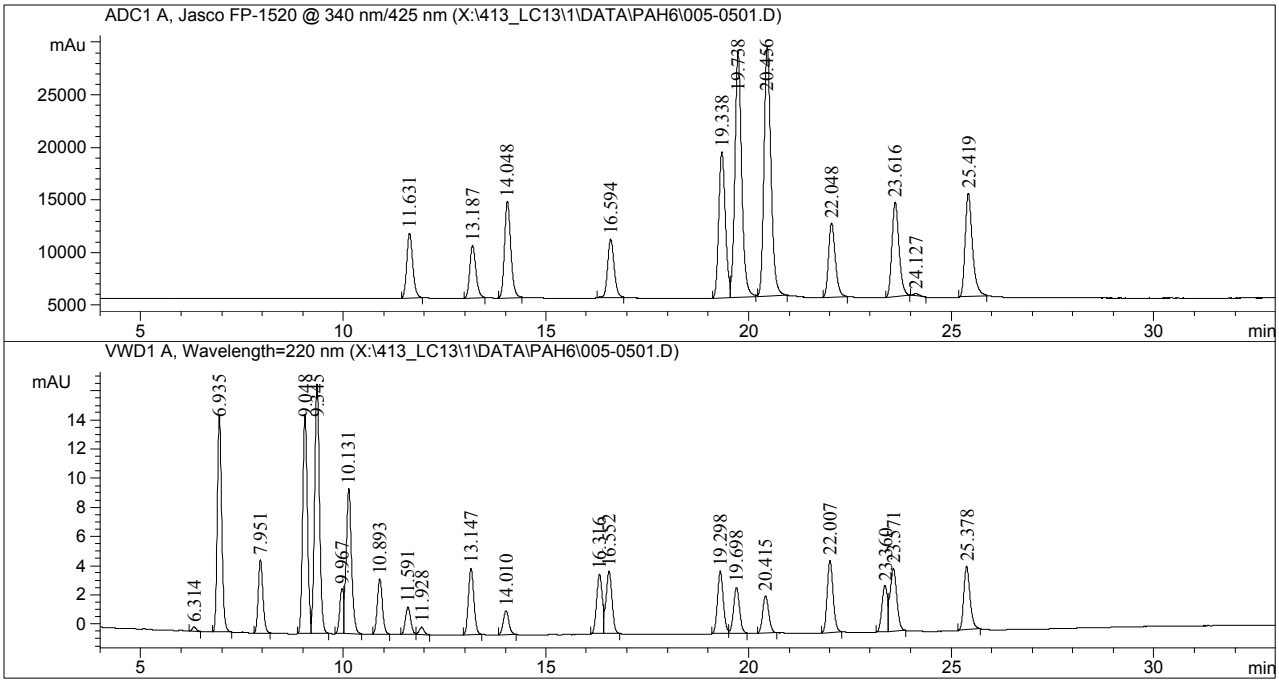

Figure 22. Representative chromatograms of a high-level (50.0 µg/L) calibration standard analyzed by HPLC/UV and fluorescence detection.

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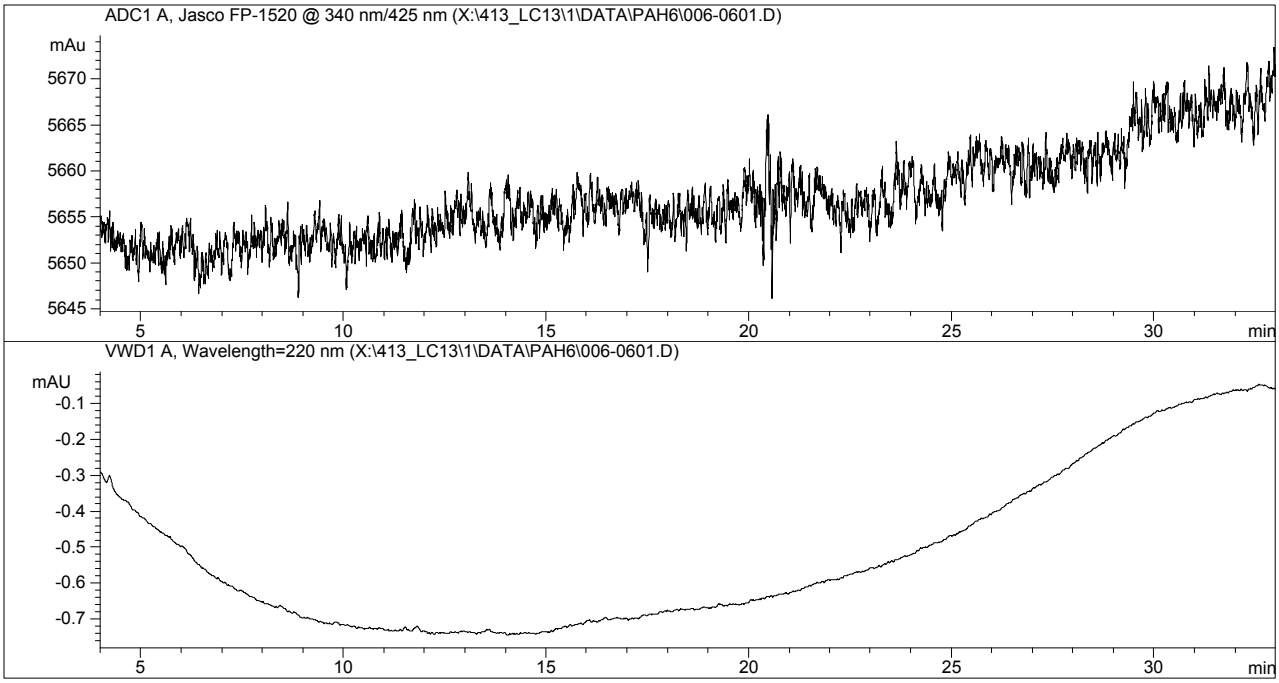

Figure 23. Representative chromatogram of a reagent blank, 472C-104-VREB-3 analyzed by HPLC/UV and fluorescence detection.

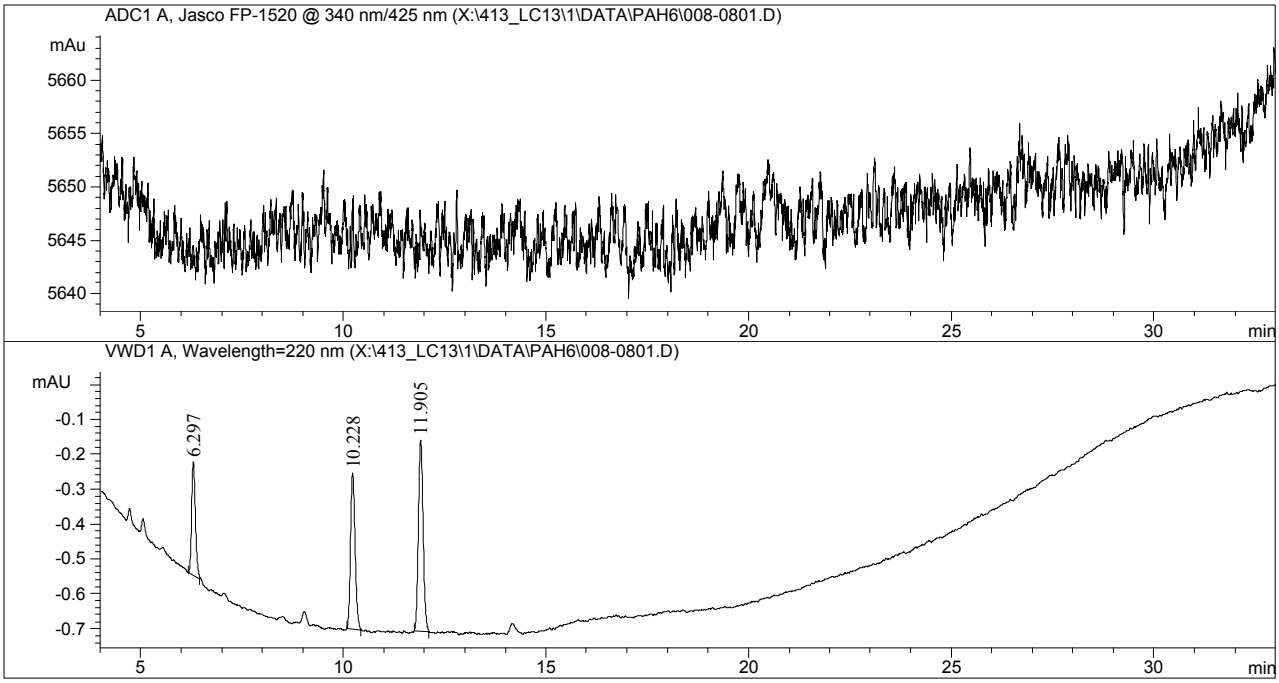

Figure 24. Representative chromatograms of a matrix blank, 472C-104-VMAB-3, analyzed by HPLC/UV and fluorescence detection.

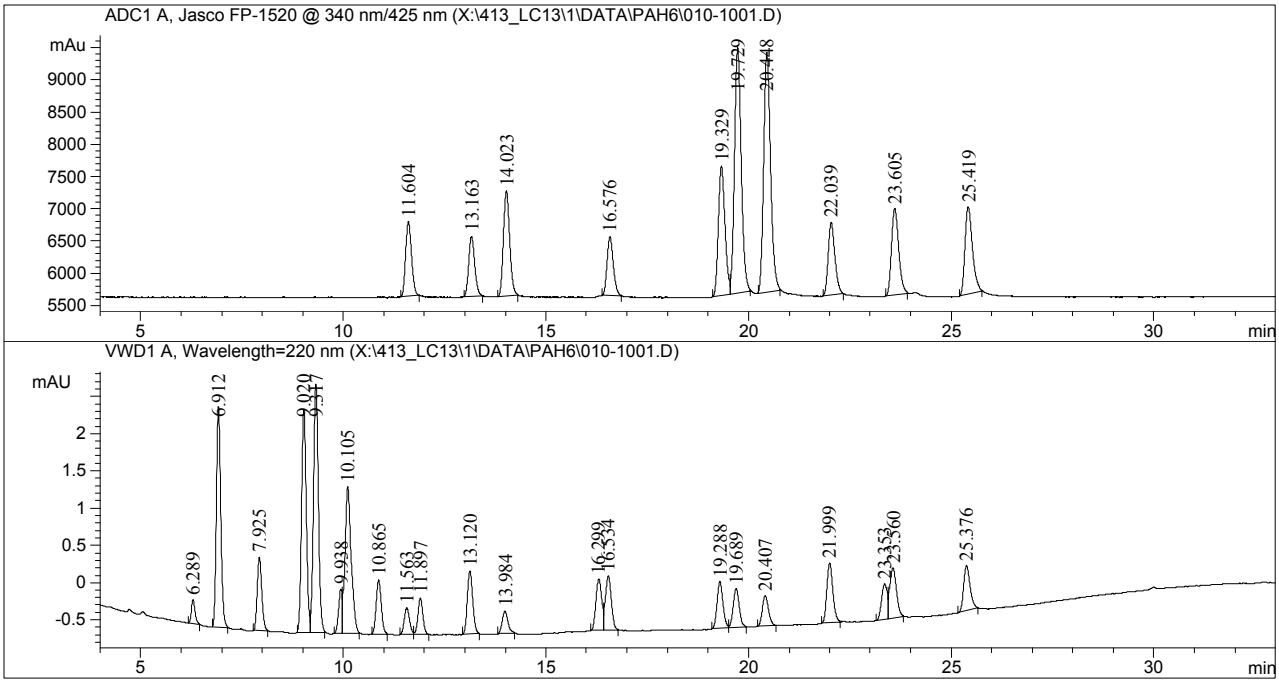

Figure 25. Representative chromatograms of a low-level m atrix fortification, 472C-104-VMAS-16 (10.0 µg/L, nominal concentration) analyzed by HPLC/UV and fluorescence detection.

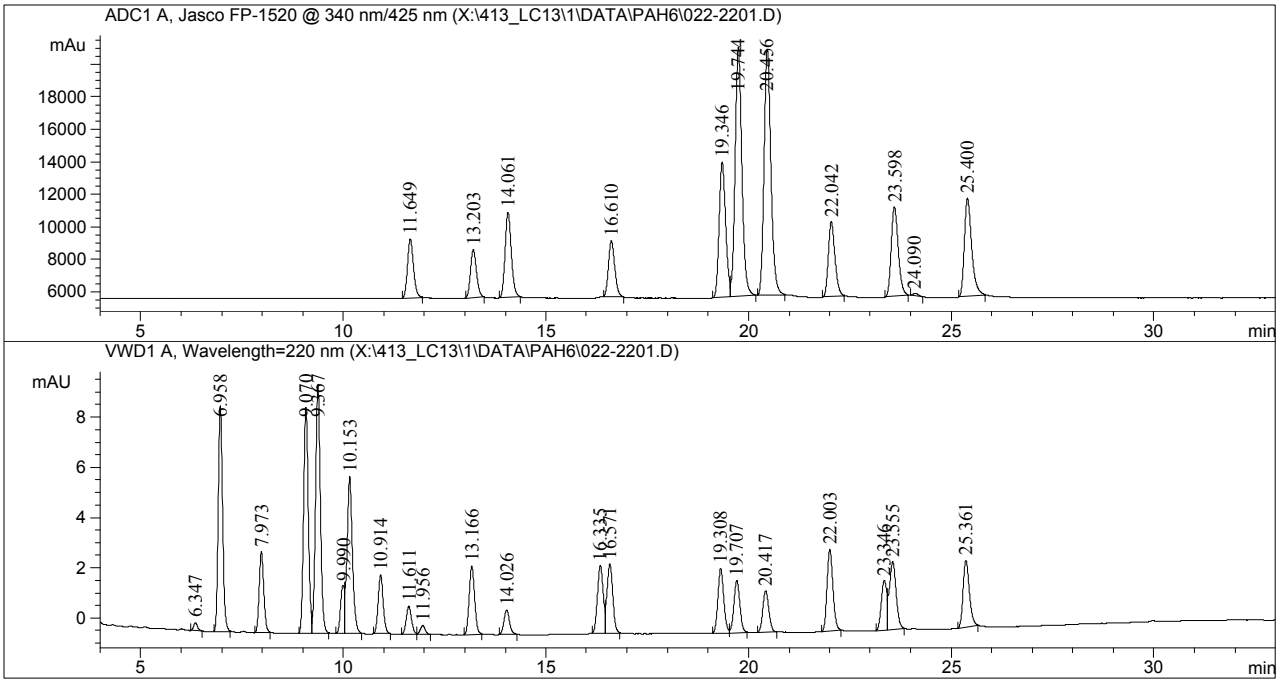

Figure 26. Representative chromatograms of a high-level matrix fortification, 472C-104-VMAS-26 (100 µg/L, nominal concentration) analyzed by HPLC/UV and fluorescence detection.

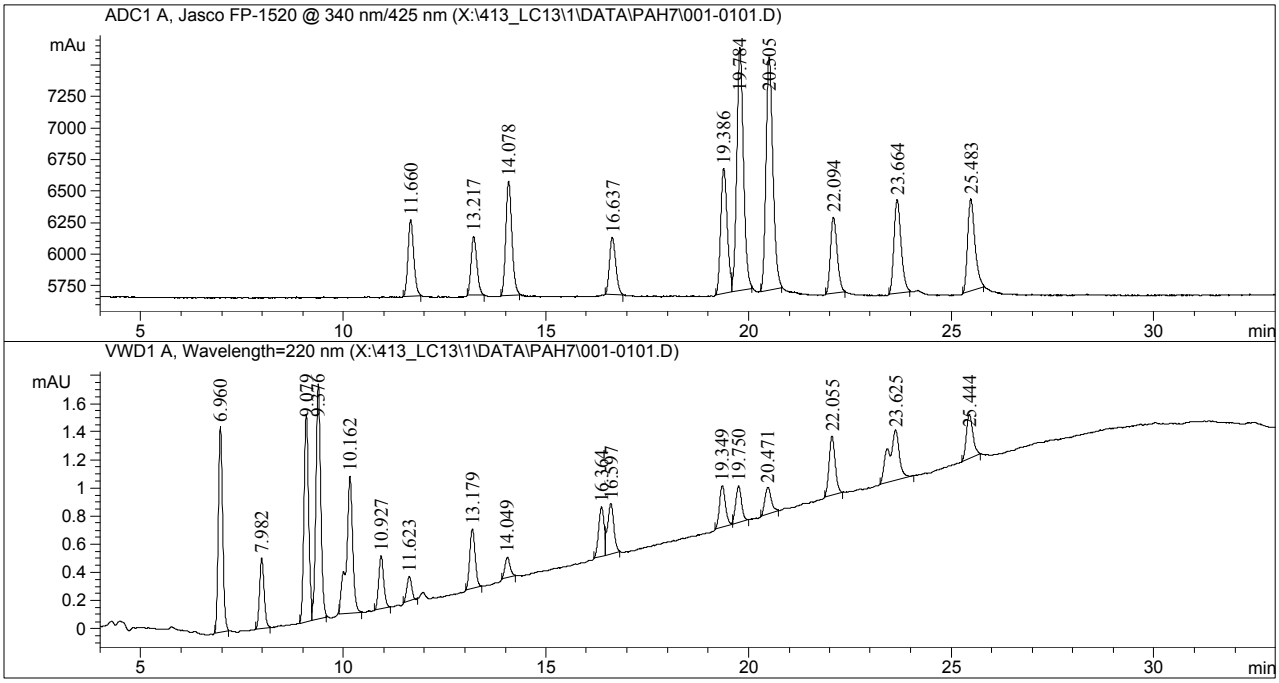

Figure 27. Representative chromatograms for the WAF trial of a low-level (5.00 µg/L) calibration standard analyzed by HPLC/UV and fluorescence detection.

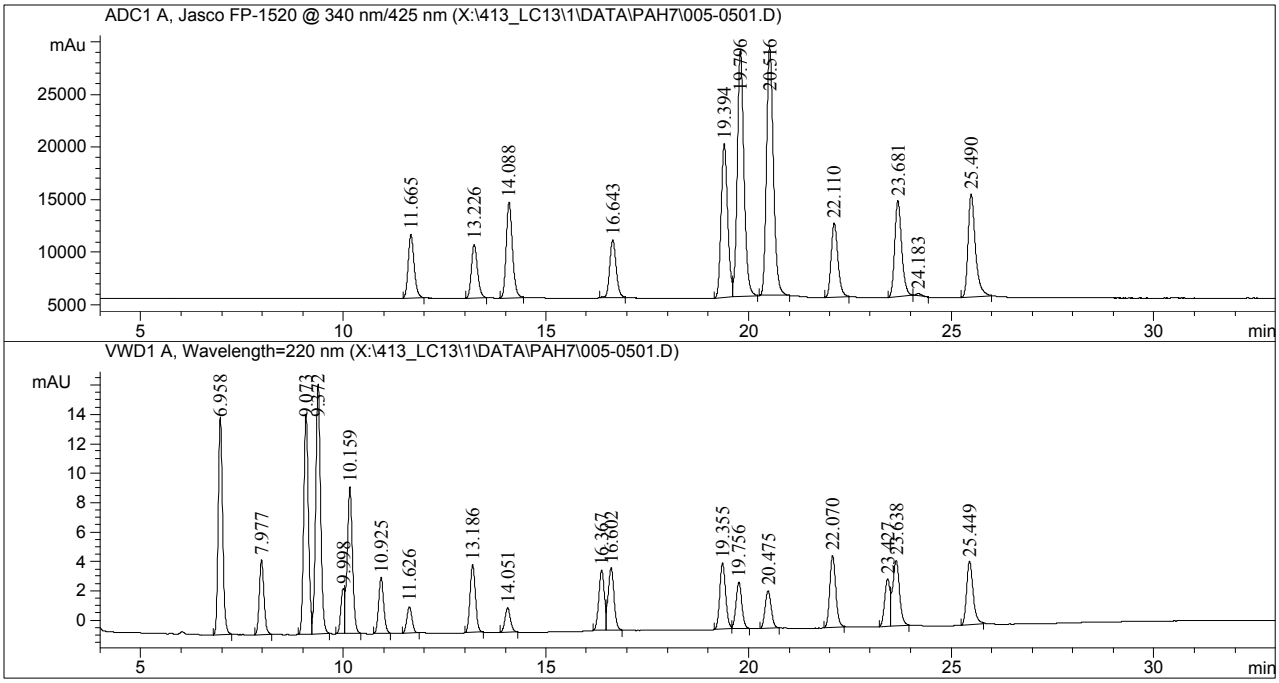

Figure 28. Representative chromatograms for the WAF trial of a high-level (50.0 µg/L) calibration standard analyzed by HPLC/UV and fluorescence detection.

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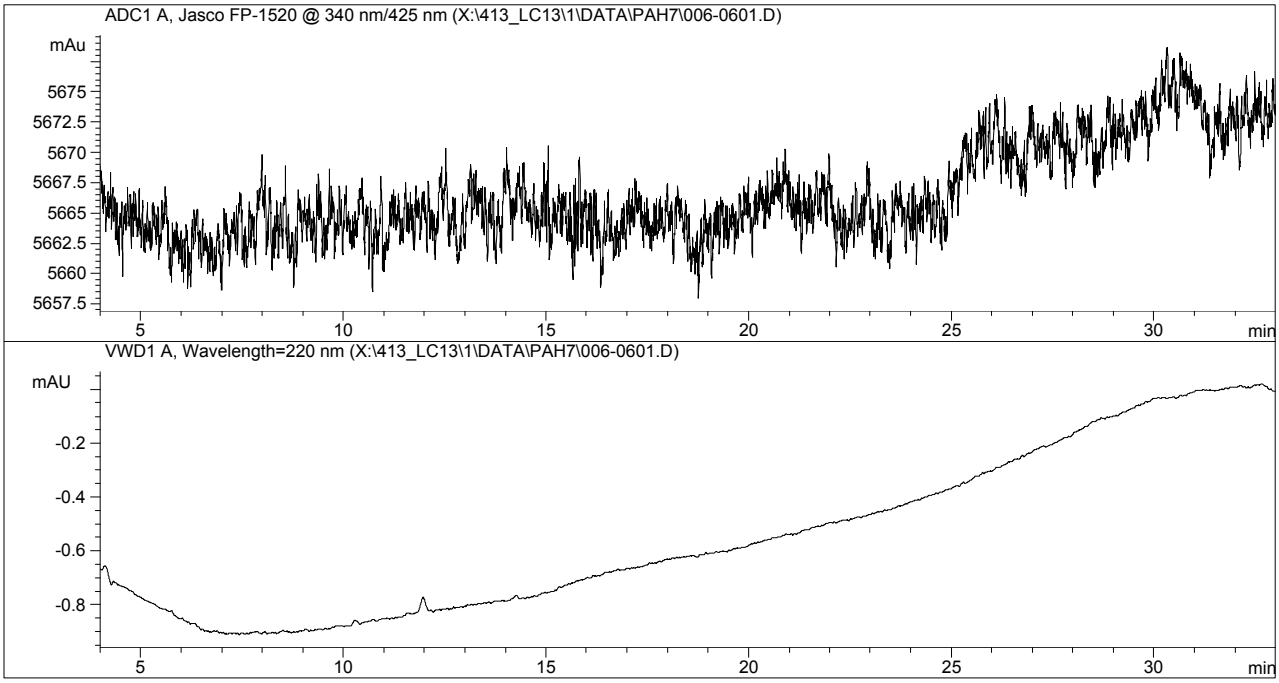

Figure 29. Representative chromatograms for the WAF trial of a m atrix blank, 472C-104-MAB-1, analyzed by HPLC/UV and fluorescence detection.

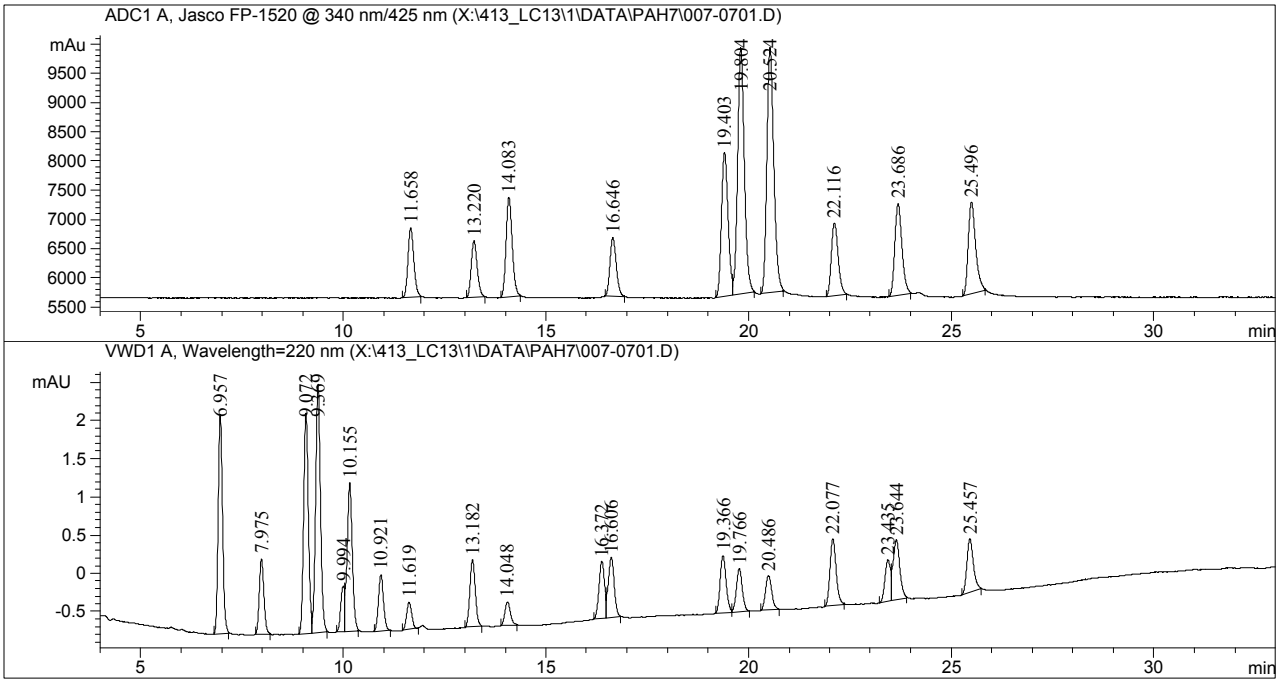

Figure 30. Representative chromatograms for the WAF trial of a matrix fortification, 472C-104-MAS-1, nominal concentration 10.0 µg/L, analyzed by HPLC/UV and fluorescence detection.

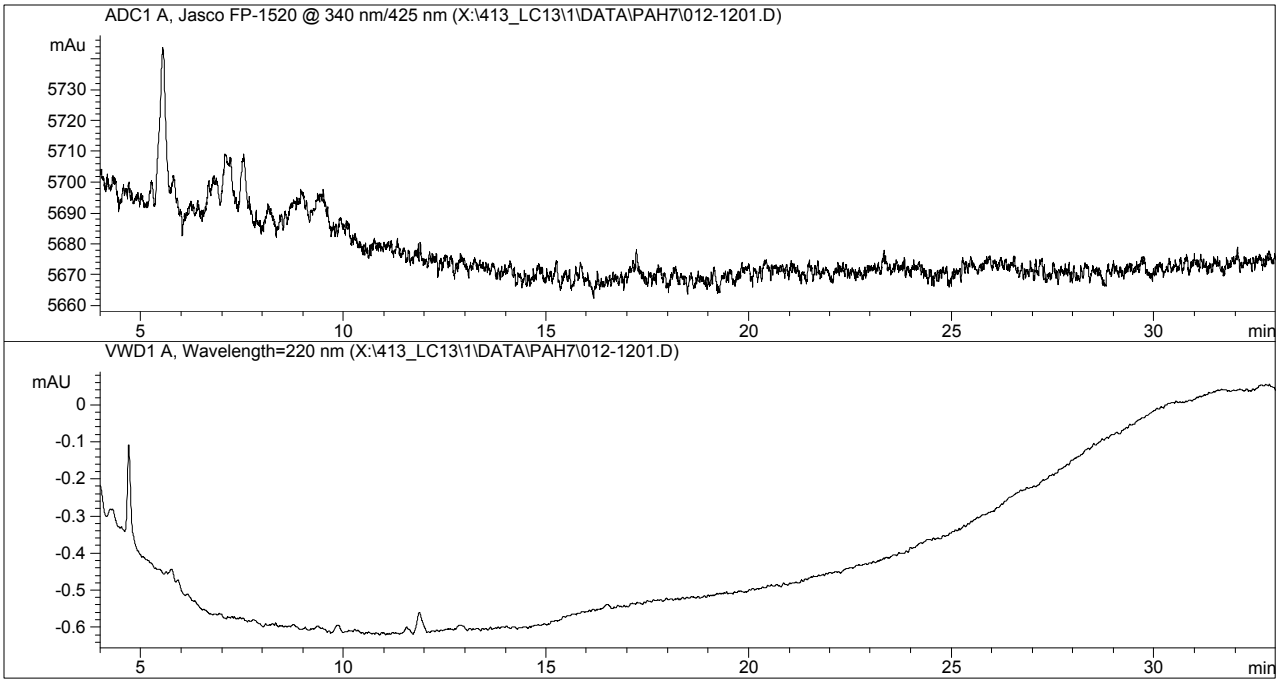

Figure 31. Representative chromatograms for the WAF trial of a 24 hour test sample, 472C-104-2 (1000 mg/L, nominal concentration) analyzed by HPLC/UV and fluorescence detection.

Appendix 1

Specific Conductance, Hardness, Alkalinity and pH of Well Water Measured During the 4-Week Period Immediately Preceding the Freshwater Verification Test

	Mean	Range
Specific Conductance (μmhos/cm)	321 (N = 4)	320 – 325
Hardness (mg/L as $CaC0_3$)	125 (N = 4)	120 – 132
Alkalinity (mg/L as $CaC0_3$)	179 (N = 4)	178 - 180
pH	8.3 (N = 4)	8.2 - 8.3

Appendix 2

Analyses of Pesticides, Organics and Metals in Wildlife International, Ltd. Well Water[1]

Pesticides and Organics			
Component	Measured Concentration (µg/L)	Component	Measured Concentration (µg/L)
Aldrin	< 0.0099	Heptachlor Epoxide	< 0.0099
Alpha BHC	< 0.0099	Malathion	< 2.0
Beta BHC	< 0.040	Merphos	< 2.0
Bolstar	< 2.0	Methoxychlor	< 0.099
Chlordane	< 0.50	Methyl Parathion	< 2.0
Coumaphos	< 3.0	Mevinphos	< 2.0
Delta BHC	< 0.0099	Mirex	< 0.050
Demeton-O	< 2.0	Naled	< 3.0
Demeton-S	< 2.0	o,p-DDD	< 0.020
Diazinon	< 2.0	o,p-DDE	< 0.020
Dichlorvos	< 2.0	o,p-DDT	< 0.020
Dieldrin	< 0.020	p,p-DDD	< 0.020
Disulfoton	< 2.0	p,p-DDE	< 0.020
Dursban (Chlorpyrifos)	< 2.0	p,p-DDT	< 0.025
Endosulfan I	< 0.0099	PCB-1016	< 0.50
Endosulfan II	< 0.042	PCB-1221	< 1.2
Endosulfan Sulfate	< 0.020	PCB-1232	< 0.89
Endrin	< 0.020	PCB-1242	< 0.50
EPN	< 4.0	PCB-1248	< 0.50
Ethion	< 2.0	PCB-1254	< 0.50
Ethoprop	< 2.0	PCB-1260	< 0.50
Ethyl Parathion	< 2.0	Phorate	< 2.0
Famphur	< 2.0	Ronnel	< 2.0
Fensulfothion	< 4.0	Stirophos	< 2.0
Fenthion	< 2.0	Telodrin	< 0.0099
Gamma BHC – Lindane	< 0.0099	Tokuthion	< 2.0
Guthion (Azinphos-methyl)	< 4.0	Toxaphene	< 0.99
HCB	< 0.099	Trichloronate	< 2.0
Heptachlor	< 0.0099	Trithion	< 2.0

[1]Analyses performed by Lancaster Laboratories on samples collected on December 22, 2004.

Appendix 2 (Continued)

Analyses of Pesticides, Organics and Metals in Wildlife International, Ltd. Well Water[1]

Metals			
Component	Measured Concentration (mg/L)	Component	Measured Concentration (mg/L)
Aluminum <	0.200	Magnesium	12.7
Antimony	< 0.0200	Manganese	< 0.0050
Arsenic	< 0.0100	Mercury	< 0.00020
Barium	< 0.0050	Nickel	< 0.0100
Beryllium	< 0.0050	Nitrate Nitrogen	< 0.50
Bromide	< 2.5	Nitrite Nitrogen	< 0.50
Cadmium <	0.0050	Potassium	6.64
Calcium 31.1		Selenium	< 0.0100
Chloride 6.9		Silver	< 0.0050
Chromium <	0.0050	Sodium	19.7
Cobalt <	0.0050	Sulfate	5.5
Copper	< 0.0100	Thallium	< 0.0200
Fluoride	< 0.50	Vanadium	< 0.0050
Iron	< 0.200	Zinc	< 0.0200
Lead <	0.0200		

[1]Analyses performed by Lancaster Laboratories on samples collected on December 22, 2004.

Appendix 3

Protocol and Protocol Amendments

PROTOCOL

ANALYTICAL METHOD VERIFICATION FOR THE DETERMINATION OF WATER SOLUBLE COMPONENTS OF PETROLEUM COKE IN FRESHWATER USING GAS CHROMATOGRAPHY (GC) OR HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

European Commission Working Document SANCO/3029/99 rev. 4

Submitted to

American Petroleum Institute
1220 L Street, N.W.
Washington, DC 20005

Wildlife International, Ltd.

8598 Commerce Drive
Easton, Maryland 21601
(410) 822-8600

March 30, 2004

Wildlife International, Ltd.

- 2 -

ANALYTICAL METHOD VERIFICATION FOR THE DETERMINATION OF WATER SOLUBLE COMPONENTS OF PETROLEUM COKE IN FRESHWATER USING GAS CHROMATOGRAPHY (GC) OR HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

SPONSOR: American Petroleum Institute
1220 L Street, N.W.
Washington, DC 20005

SPONSOR'S REPRESENTATIVE: [redacted]

SPONSOR'S TECHNICAL STUDY MONITOR: [redacted]

TESTING FACILITY: Wildlife International, Ltd.
8598 Commerce Drive
Easton, Maryland 21601

STUDY DIRECTOR: [redacted]
Wildlife International, Ltd.

LABORATORY MANAGEMENT: [redacted]
Wildlife International Ltd.

FOR LABORATORY USE ONLY

Proposed Dates:

Experimental Start Date: 4/28/04

Experimental Termination Date: 6/28/04

Project No.: 472C-104

Test Substance No.: 6485

PROTOCOL APPROVAL

[redacted]

4/28/04
DATE

4/28/04
DATE

01 April, 2004
DATE

PROTOCOL NO.: 472/033004/MVFW-GC-HPLC/SUB472

Wildlife International, Ltd.

INTRODUCTION

Wildlife International, Ltd. will conduct analytical trials to verify analytical methods for the determination of polyaromatic hydrocarbons (PAH) in water accommodated fraction (WAF) solutions made with petroleum coke and freshwater. The study will be performed at the Wildlife International, Ltd. analytical chemistry facility in Easton, Maryland. The study will be performed based on procedures in *Residues: Guidance for Generating and Reporting Methods of Analysis in Support of Pre-registration Data Requirements for Annex II (Part A, Section 4) and Annex III (Part A, Section 5) of Directive 91/414* (1). Petroleum coke is defined as the product formed by subjecting the heavy tar-like residue remaining following oil refining to high temperatures and pressures. It consists of primarily elemental carbon with considerably smaller amounts of hydrocarbons, sulfur and trace amounts of heavy metals. The method will be verified by fortifying freshwater with the test substance and determining the recoveries. Raw data for all work performed at Wildlife International, Ltd. and a copy of the final report will be filed by project number in archives located on the Wildlife International, Ltd. site, or at an alternative location to be specified in the final report.

OBJECTIVE

There are two objectives to this study. One is to verify a Gas Chromatography (GC) or High Performance Liquid Chromatography (HPLC) method for determination of polycyclic aromatic hydrocarbons (PAH) in water accommodated fraction (WAF) solutions of petroleum coke. The second is to employ the method to determine the optimum WAF mixing time to achieve maximum leaching of PAHs from the test substance matrix into freshwater.

EXPERIMENTAL DESIGN

Wildlife International, Ltd. freshwater will be fortified at three different concentrations and analyzed using HPLC or GC methods. Modifications to the methodologies will be made as necessary in order to achieve quantitation of the test substance in the freshwater matrix. Reagent and matrix blanks will be analyzed to evaluate possible analytical interferences that may be present. One or more calibration curves will be prepared and analyzed with each series of matrix fortification samples.

Wildlife International, Ltd.

MATERIALS AND METHODS

Test Substance

The test substance is green coke (CAS Number 64741-79-3) sieved to approximately 2 mm particle size. Information on the characterization of test, control and reference substances is required by Good Laboratory Practice Standards (GLP), 40 CFR Parts 160.31 and 792. The Sponsor is responsible for providing Wildlife International, Ltd. written verification that the test substance has been characterized according to GLPs prior to its use in the study. If written verification of GLP test substance characterization is not provided to Wildlife International, Ltd., it will be noted in the compliance statement of the final report.

The Sponsor is responsible for all information related to test and reference substances and agrees to accept any unused test or reference substance and/or test or reference substance containers remaining at the end of the study.

Reagents and Solvents

All solvents used in the method or procedure will be HPLC grade or equivalent. All reagents will be ACS reagent grade or higher quality. Nanopure water will be used. The solvents and reagents are not expected to contain contaminants capable of interfering with the purpose of this study.

Freshwater

Freshwater to be used for the method verification will be obtained from a well approximately 40-meters deep located on the Wildlife International, Ltd. site. The water will be passed through a sand filter and pumped into a 37,800-L storage tank where the water will be aerated with spray nozzles. Prior to use, the water will be filtered to 0.45 µm in order to remove fine particles. The resulting water is characterized as moderately hard. Typical values for hardness, alkalinity, pH and specific conductance are approximately:

Hardness, mg/L as $CaCO_3$	145
Alkalinity, mg/L as $CaCO_3$	190
pH	8.1
Specific Conductance, µmhos/cm	330

Wildlife International, Ltd.

Hardness, alkalinity, pH and specific conductance will be measured weekly to monitor the consistency of the well water. Means and ranges of the measured parameters for the four-week period preceding the test will be provided in the final report. Analyses will be performed at least once annually to determine the concentrations of selected organic and inorganic constituents of the well water. Results of these analyses will be summarized in the final report. Specifications for acceptable levels of contaminants in well water have not been established. However, there are no known levels of contaminants reasonably expected to be present in the well water that are considered to interfere with the purpose or conduct of the test.

Fortification Stock Solution(s)

Freshwater will be fortified with a stock solution(s) of the test substance. Each stock solution will be assigned a unique identification code which will be recorded on a stock preparation log sheet.

Reference Stock Solution(s), Calibration Standards and Curves

A primary stock solution(s) will be prepared from appropriate reference substances, when available. Calibration standards will be prepared by appropriate dilution of this primary stock solution(s). A minimum of five concentrations of calibration standards will be prepared and analyzed along with each analysis set of verification samples. The calibration standard series will be injected at the beginning and end of the analytical run with, in addition, a minimum of one standard injected following every five samples. One or more calibration curves will be derived from regression analysis of the instrumental responses of the standards.

Method

The analytical methods to be used will be based upon GC (EPA 8270C) or HPLC procedures. The method(s) may be modified to yield a method capable of determining the test substance in freshwater. Samples will be analyzed for the components of petroleum coke listed in Table 1, if possible. The method used will be summarized in the final report.

Verification Analyses - Method Performance

Matrix fortifications (fortified wellwater), prepared at known concentrations of the test substance, will be analyzed to determine recovery and to evaluate method performance. The anticipated verification series will consist of the following analyses:

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SUMMARY OF PROPOSED VERIFICATION ANALYSIS SCHEME

Analysis Type	Concentration	Number of Samples
Reagent Blank	0 (Control)	2
Matrix Blank	0 (Control)	2
Matrix Fortifications	Level 1 -Low[1]	5
	Level 2	5
	Level 3 - High	5
Total Number of Analyses		19

[1] Matrix fortifications will be prepared at three concentrations, the lowest and highest of which will bracket the anticipated treatment range for environmental effects studies.

Matrix fortifications will span the range of concentrations that are anticipated to be used in subsequent environmental effects tests and will be selected in consultation with the Sponsor. Individual fortification samples (identified by project number and a unique sample identification number) will be prepared and analyzed.

If difficulties arise in the validation process (e.g., low recoveries or interferences), the Sponsor will be notified and the need for additional validation and/or method development will be determined through discussions with the Sponsor. Upon completion of the method verification, the Sponsor will review the results and authorize the use of the methodology for analysis of samples, or will authorize further method development trials. Recovery values in the range of 80 to 120% will be used as criteria for method acceptability.

Evaluation of Interferences

Matrix and reagent blanks, if applicable, will be analyzed to assess the presence of potential interferences. Matrix blanks will consist of freshwater without addition of test substance.

Data Analysis

One or more calibration curves will be established for each analytical run. A regression equation of the concentration versus peak area for the calibration standards will be generated. The concentration of the samples will be determined by substituting the respective peak area into the regression equation.

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If the fortification range and calibration curve standard concentrations are significantly higher than method capabilities for quantitation, the limit of quantitation (LOQ) will be defined as the concentration equivalent to the lowest standard accounting for dilutions and other manipulations in the method for matrix blanks. If the fortification range is near the method capability for accurate quantitation of the analyte, the LOQ will be determined from the responses of replicate control samples. The mean (Sb) and standard deviation (σ) of responses will be determined for the replicate control sample and converted to ppm equivalents using a standard curve and method dilution factors. The LOQ will be expressed as the concentration equivalent of Sb + 10 σ. The limit of detection (LOD), defined as the lowest concentration of analyte which can be injected and produce a measurable response above background, will be expressed as the amount equivalent to Sb +3 σ.

Precision and Repeatability

The precision of the method will be reported as the RSD of repeatability at each fortification level and the overall RSD will be reported. In general, the RSD should be $\leq$ 20%.

Determination of Mixing Time for the Preparation of WAF Solutions

Petroleum Coke will be mixed directly with dilution water on a weight:volume basis. A water accommodated fraction (WAF) will be prepared at a single high concentration of 1000 mg coke/L in 13.2L and 4L Pyrex® aspirator bottles with tubulation (Fisher Catalog Numbers 02-972-2 and 02-972F). Solutions will be prepared by mixing the test solutions with a vortex depth of approximately 30% of the test solution height and then allowing settling before sampling. A WAF equilibration test will be performed with analysis of test solutions after approximately 24, 48, 72 and possibly 96 hours of mixing. Sampling intervals may be modified by the Study Director based upon the analytical results. The length of the mixing time required to achieve approximately stable concentration in the WAF will be evaluated and used in aquatic toxicity testing. Analysis of WAF solutions for the analytes listed in Table 1 will be performed using the chromatographic methods.

RECORDS TO BE MAINTAINED

Records to be maintained for data generated by Wildlife International, Ltd. will include:

1. A copy of the signed protocol.

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2. Identification and characterization of the test substance and/or analytical standard, if provided by the Sponsor.
3. Dates of initiation and termination of the test.
4. Storage conditions for test substance, analytical standards, and/or samples.
5. Test substance and/or analytical standard use log.
6. Concentration calculations and records of solution preparation.
7. Instrument operating conditions and chromatograms.
8. Statistical calculations.
9. A copy of the final report.
10. Documentation that the steps in the method were followed.

FINAL REPORT

The report will summarize the findings of the verification, the procedural recoveries obtained, and the methods and instrumentation employed. Upon receipt of these findings, the Sponsor will review the methods and results and evaluate the results for acceptability prior to initiating any fate and/or effects studies.

The final report will include, but not be limited to, the following:

1. Name and address of the facility performing the study.
2. Dates on which the study was initiated and completed.
3. Objectives and procedures stated in the approved protocol, including any changes in the original protocol or deviations from the protocol.
4. The test substance and/or analytical standard identification, including name, chemical abstract number or code number, strength, purity, composition, date of receipt, lot number, storage conditions, physical characteristics, stability and method of preparation of test concentrations, if provided by the Sponsor.
5. A brief summary of the analytical methodology: A description of the experimental measurements, example calculations, sample preparation (sample weights and dilutions), instrumentation employed, reagents and solvents used, class of water used, and any major modifications to the method.
6. A description of all circumstances that may have affected the quality or integrity of the data.

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7. The name of the Study Director, the names of other scientists or professionals, and the names of all supervisory personnel involved in the study.
8. A description of the transformations, calculations, or operations performed on the data.
9. The signed and dated reports of each of the individual scientists or other professionals involved in the study, if applicable.
10. The location where raw data and final report are to be stored.
11. A statement prepared by the Quality Assurance Unit listing the dates that study inspections were made and the dates of any findings reported to the Study Director/Management.

CHANGES TO PROTOCOL

Planned changes to the protocol will be in the form of written amendments signed by the Study Director and approved by the Sponsor's Representative. Amendments will be considered as part of the protocol and will be attached to the final protocol. Any other changes will be in the form of written deviations signed by the Study Director and filed with the raw data. All changes to and deviation from the protocol will be indicated in the final report.

GOOD LABORATORY PRACTICES

This study will be conducted in accordance with Good Laboratory Practice Standards for EPA (40 CFR Part 160 and/or Part 792); and OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17). Each study conducted by Wildlife International, Ltd. is routinely examined by the Wildlife International, Ltd. Quality Assurance Unit for compliance with Good Laboratory Practices, Standard Operating Procedures and the specified protocol. A statement of compliance with Good Laboratory Practices will be prepared for all portions of the study conducted by Wildlife International, Ltd. The Sponsor will be responsible for certification of compliance with Good Laboratory Practices for procedures performed by other laboratories. Raw data for all work performed at Wildlife International, Ltd. and a copy of the final report will be filed by project number in archives located on the Wildlife International, Ltd. site, or at an alternative location to be specified in the final report.

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REFERENCES

1. **European Commission**. 2000. *Residues: Guidance for Generating and Reporting Methods of Analysis in Support of Pre-registration Data Requirements for Annex II (Part A, Section 4) and Annex III (Part A, Section 5) of Directive 91/414*. SANCO/3029/99 rev. 4, 11/07/00

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Table 1.

Analytes of Interest in Petroleum Coke

Acenanhthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(g,h,i)perylene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,e)pyrene
Dibenz(a,h)anthracene
Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
Naphthalene
Perylene
Phenanthrene
Pyrene

Project Number 472C-104

Wildlife International, Ltd.

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Analytical Method Verification for the Determination of Petroleum Coke in Freshwater Using Gas Chromatography (GC) or High Performance Liquid Chromatography (HPLC)

PROTOCOL NO.: 472/033004/MVFW-GC-HPLC/SUB472 **AMENDMENT NO.:** 1

SPONSOR: American Petroleum Institute **PROJECT NUMBER:** 472C-104

EFFECTIVE DATE:

Add to Page 4 – Test Substance

Petroleum Coke pellets (diameter of approximately 2 mm) provided by the Sponsor will be analyzed by GC or HPLC in an attempt to determine concentrations of polyaromatic hydrocarbons (PAH) components in the neat test substance. Results of the analysis will be compared to the analysis of the PAH components in Petroleum Coke provided by the Sponsor.

REASON:

The purpose for analyzing neat petroleum coke is to determine the sensitivity of the selected method for those specific analytes of interest.

Add to Page 5 – Method

Prior to performance of the method verification trial, an evaluation of the HPLC and/or GC method(s) will be performed to determine the background levels of the analytes in water and establish a theoretical limit of quantitation (LOQ) in freshwater. The LOQ will be defined as the product of the low standard and the concentration factor of the matrix blank.

REASON:

At the request of the Sponsor, the steps to be taken to establish the theoretical LOQ were specified in the protocol.

Add and Revise Page 6 – Verification Analysis – Method Performance:

The method verification trial will be performed by fortifying freshwater with known concentrations of analytical standards of the PAH analytes of interest rather than with petroleum coke containing trace quantities of these analytes. Portions of the protocol that refer to fortifying water with petroleum coke are effectively changed to fortifying water with known standards (individual or mixed standards) of the PAH analytes of interest.

Project Number 472C-104

Wildlife International, Ltd.

Fortification levels of each analyte will include the target LOQ and 10X the LOQ to bracket the expected concentrations of each analyte in water to be used for environmental effects testing with petroleum coke. If expected levels of an analyte are above 10X the LOQ, additional fortification levels for that analyte will be added at the appropriate concentration(s).

REASON:

Concentrations of most of the analytes of interest in petroleum coke are < LOQ or are present in trace quantities according to information provided by the Sponsor. Fortifying with known concentrations using analyte standards will verify the efficiency and accuracy of the HPLC and/or GC method for analysis of the analytes in waters to be used for environmental effects testing.

Add to Table 1 – Analytes of Interest

1-methylnapthalene
2-methylnapthalene

REASON:

The analytes were not included in the protocol but were found at relatively high concentrations in the petroleum coke sample provided by the Sponsor.

[redacted]

9/17/04
DATE

9/23/04
DATE

August 6, 2004
DATE

Project Number 472C-104

Wildlife International, Ltd.

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Analytical Method Verification for the Determination of Petroleum Coke in Freshwater Using Gas Chromatography (GC) or High Performance Liquid Chromatography (HPLC)

PROTOCOL NO.: 472/033004/MVFW-GC-HPLC/SUB472 **AMENDMENT NO.:** 2

SPONSOR: American Petroleum Institute **PROJECT NUMBER:** 472C-104

EFFECTIVE DATE: August 20, 2004

Add to Page 2 – Reference Substance Numbers

6705 through 6720, 6765, and 6766.

REASON:

Identification of analytical standards to be used for measurement of the PAH's of interest. The standards will be used for instrument calibration, detection limit determination and matrix fortifications.

Add to Page 2 – Proposed Dates

Experimental Start Date: August 24, 2004
Experimental Termination Date: October 24, 2004

REASON:

Information required to complete the protocol.

[redacted]

9/17/04
DATE

9/23/04
DATE

10/1/04
DATE

Project Number 472C-104

Wildlife International, Ltd.

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Analytical Method Verification for the Determination of Petroleum Coke in Freshwater Using Gas Chromatography (GC) or High Performance Liquid Chromatography (HPLC)

PROTOCOL NO.: 472/033004/MVFW-GC-HPLC/SUB472 AMENDMENT NO.: 3

SPONSOR: American Petroleum Institute PROJECT NUMBER: 472C-104

EFFECTIVE DATE: September 17, 2004

Add to Page 2 – Concentrations to be Verified

10.0, 40.0 and 100 µg a.i./L

REASON:

Definition of test concentrations.

[redacted]

9/17/04
DATE

9/23/04
DATE

10/1/04
DATE

Project Number 472C-104

Wildlife International, Ltd.

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Analytical Method Verification for the Determination of Petroleum Coke in Freshwater Using Gas Chromatography (GC) or High Performance Liquid Chromatography (HPLC)

PROTOCOL NO.: 472/033004/MVFW-GC-HPLC/SUB472 **AMENDMENT NO.:** 4

SPONSOR: American Petroleum Institute **PROJECT NUMBER:** 472C-104

EFFECTIVE DATE: September 28, 2004

REMOVE: The following section from Amendment 1

> Petroleum Coke pellets (diameter of approximately 2 mm) provided by the Sponsor will be analyzed by GC or HPLC in an attempt to determine concentrations of polyaromatic hydrocarbons (PAH) components in the neat test substance. Results of the analysis will be compared to the analysis of the PAH components in Petroleum Coke provided by the Sponsor.

REASON:

Sponsor request since analysis of PAH in neat test substance is not relevant to the analysis of PAH in water. The characterization of Petroleum Coke pellets provided by the Sponsor is adequate to determine the presence of PAH in the test substance.

[redacted]

9/29/04
DATE

9/29/04
DATE

10/1/04
DATE

Project Number 472C-104

Wildlife International, Ltd.

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Analytical Method Verification for the Determination of Petroleum Coke in Freshwater Using Gas Chromatography (GC) or High Performance Liquid Chromatography (HPLC)

PROTOCOL NO.: 472/033004/MVFW-GC-HPLC/SUB472 **AMENDMENT NO.:** 5

SPONSOR: American Petroleum Institute **PROJECT NUMBER:** 472C-104

EFFECTIVE DATE: August 11, 2005

CHANGE: Title of the report

From: Analytical Method Verification for the Determination of Petroleum Coke in Freshwater Using Gas Chromatography (GC) or High Performance Liquid Chromatography (HPLC)

To: Analytical Method Verification for the Determination of Petroleum Coke in Freshwater Using High Performance Liquid Chromatography (HPLC)

REASON:

Request of the Sponsor. Gas Chromatography (GC) was not used for the actual validation.

[redacted]

9/12/05
DATE

9/12/05
DATE

9/2/05
DATE

Appendix 4

Test Article Selection

THE PACE CONSULTANTS INC.
Post Office Box 53473 Houston, Texas 77052 853/351-7800 Fax 853/351-7887
A Member of Jacobs Engineering Group

February 22, 2001

[redacted]
American Petroleum Institute
1220 L Street, NW
Washington, D.C. 20005-4070

Attached is Pace's report covering Task 1 and 2 entitled "U.S. Delayed Coker Petroleum Coke Quality Survey 1998-1999."

We would be pleased to answer any questions concerning this work for API. Please contact me at 832/351-7811 or email [redacted]

For PACE

[redacted]

[redacted]
Attachment

AMENDED

U.S. DELAYED COKER PETROLEUM COKE QUALITY SURVEY 1998-1999

INTRODUCTION

In 1998 the United States Environmental Protection Agency (EPA) challenged chemical producers and importers to provide voluntarily basic toxicity information on their high production volume (HPV) chemicals, defined as those chemicals which are produced in or imported to the U.S. in amounts greater than 1 million pounds per year. The goal of the HPV Challenge Program is to ensure that the American public has access to basic information about the hazards associated with chemicals manufactured and used in the greatest quantities in the United States. It is designed to generate the complete hazard screening data for HPV commercial chemicals.

The American Petroleum Institute (API) serves as administrator of the Petroleum HPV Testing Group, a consortium made up of 72 member companies from API, the National Petrochemical & Refiners Association (NPRA), the Gas Producers Association (GPA) and the Asphalt Institute. These companies represent 92% of the nation's refinery capacity. The Petroleum HPV Testing Group has sponsored 396 substances produced and used by the nation's petroleum industry to meet the EPA's HPV challenge.

Pace was retained by the API HPV Testing Group to assist in identifying potential sources of U.S. petroleum coke samples that could be used in the HPV testing program. As the first step in this process, Pace undertook a review of its quarterly petroleum coke production data to help characterize current U.S. petroleum coke production qualities. Pace has now completed the review of its 1998 and 1999 quarterly petroleum coke production data for all U.S.-based delayed cokers. The results of this review are discussed below.

METHODOLOGY

Pace's petroleum coke production database was used to determine quality characteristics of petroleum coke produced by U.S. refineries. Pace has conducted a survey of U.S. petroleum coker production on a quarterly basis since the second quarter of 1983. Refineries provide the bulk of the data, but some data are also gathered from other market participants. These data are maintained in a database from which the 1998 and 1999 quarterly data were extracted for this study. It was decided that data analysis would concentrate on delayed cokers (excluding needle cokers) since for 1999 our delayed coker data set includes 92+% of all the petroleum coke produced in the United States. Accordingly, fluid and Flexicokers[1] were removed from the data set.

Needle cokers were removed from the delayed coker database because needle cokers represent a special subset of delayed coking production. Needle coke differences include:

[1] Flexicoke is a proprietary coking process developed by Exxon. It involves partially gasifying fluid coke.

1. Needle coke quality is much higher than other delayed coke
2. Needle coke is produced using different feedstock & coking operational procedures because it is a product, not a by-product like other delayed cokes
3. The quantity of needle coke produced is very small
4. Needle coke is handled very carefully due to its high price (typically > $350/metric ton)

SUMMARY AND DATA ANALYSIS

These data were analyzed to determine the ton-weighted average petroleum coke qualities of sulfur (wt%), nickel (ppm), vanadium (ppm), and volatile material (wt%). All data are presented on a dry basis. The results are presented in Table 1 below.

TABLE 1

U.S. DELAYED PETROLEUM COKE QUALITY SUMMARY
TON-WEIGHTED QUARTERLY AVERAGES

Quarter	Sulfur, Wt%		Nickel, ppm		Vanadium, ppm		Vol. Mat., Wt%	
	1998	1999	1998	1999	1998	1999	1998	1999
1Q	4.15	4.11	286	275	758	801	10.9	10.5
2Q	4.22	4.22	277	283	811	821	10.8	11.0
3Q	4.21	4.21	277	282	811	857	10.9	10.9
4Q	4.21	4.22	282	276	854	852	10.7	10.9
Ton-Wt Avg	4.20	4.19	280	279	809	833	10.8	10.8

Ton-weighted average qualities for each quarter were calculated in the following manner:

$$\frac{\sum (\text{quality value})_{\text{delayed coker}} * (\text{quarterly production})_{\text{delayed coker}}}{\text{Total quarterly production}}$$

Where:

quality value = sulfur, vanadium, nickel or volatile content of petroleum coke produced by each delayed coker

quarterly production = petroleum coke produced by that delayed coker

Pace next reviewed the data to determine a ton-weighted frequency distribution for each of the qualities listed. The results of this analysis are presented in Table 2 and in Figures 1 through 4.

TABLE 2

U.S. DELAYED PETROLEUM COKE QUALITY SUMMARY BY PRODUCTION QUARTILE

Cumulative Production	Sulfur, Wt%		Nickel, ppm		Vanadium, ppm		Vol, Wt%	
	1998	1999	1998	1999	1998	1999	1998	1999
min.	0.90	0.50	50	5	45	45	7.0	4.0
25%	3.20	3.10	180	185	400	445	10.0	10.0
50%	4.45	4.60	250	250	650	675	10.7	10.7
75%	5.34	5.30	360	400	1205	1200	12.0	12.0
100%	6.90	6.30	568	568	1900	2000	14.0	14.0

Quality quartiles for each year were calculated in the following manner:

> Annual data were sorted according to each specific quality value (e.g., sulfur, vanadium, nickel, and volatile content) and the cumulative production of petroleum coke by delayed coker was calculated. Quartiles were then calculated for the annual production total, and the quality value at the cumulative total that equaled each quartile was used to determine the quality for that quartile.

TRENDS

Comparing the ton-weighted averages to the 50% production quartile (i.e., the median) reveals the following trends:

- The weighted average nickel and vanadium content of U.S. delayed petroleum coke is higher than the median. This is a direct result of the increasing amount of heavy crudes, particularly Mexican and Venezuelan crudes, processed by U.S. refineries. Because these crudes produce petroleum cokes with nickel and vanadium contents that are significantly above the median, they skew the weighted average away from the median.

- Ton-weighted sulfur content is slightly below the median because some cokers produce petroleum cokes that are well below the median sulfur content (i.e., anode-grade coke which is calcined and primarily used to make anodes for the aluminum smelting industry).

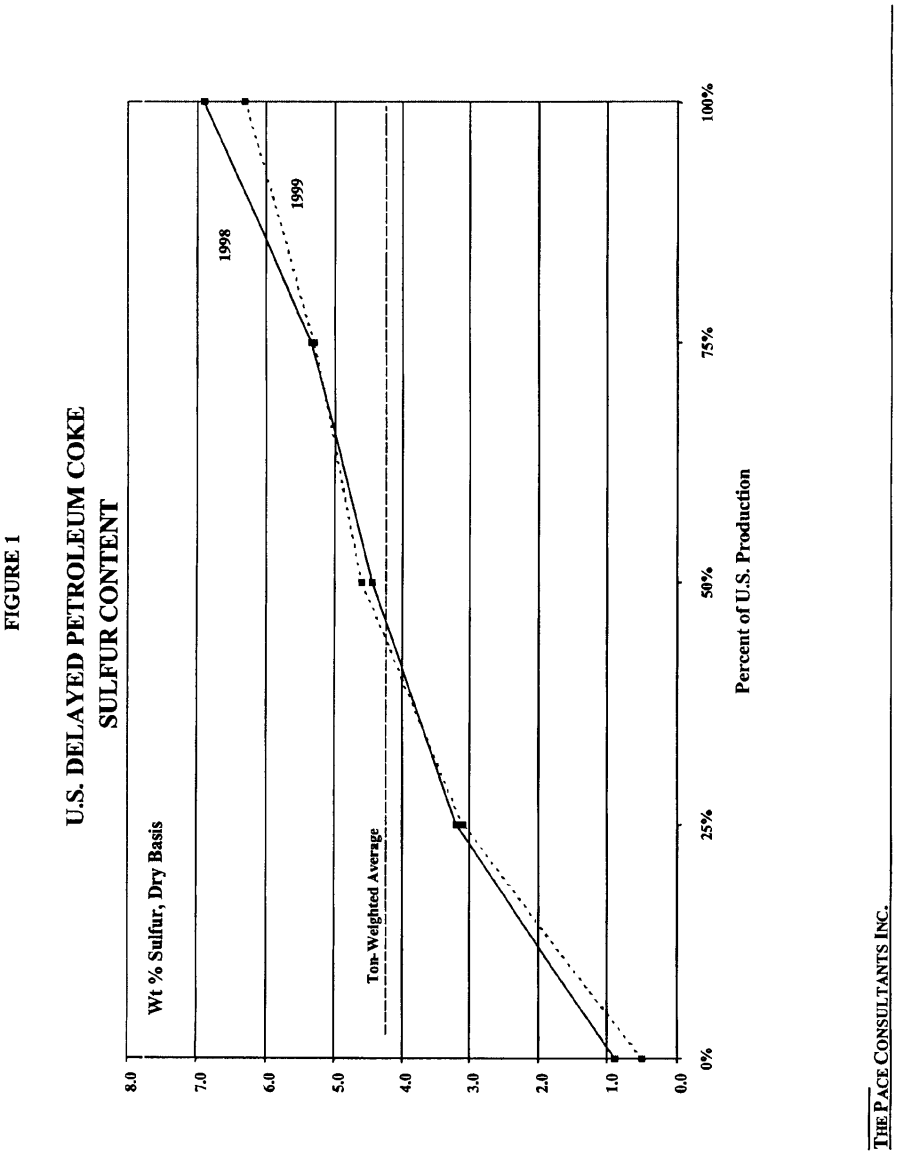
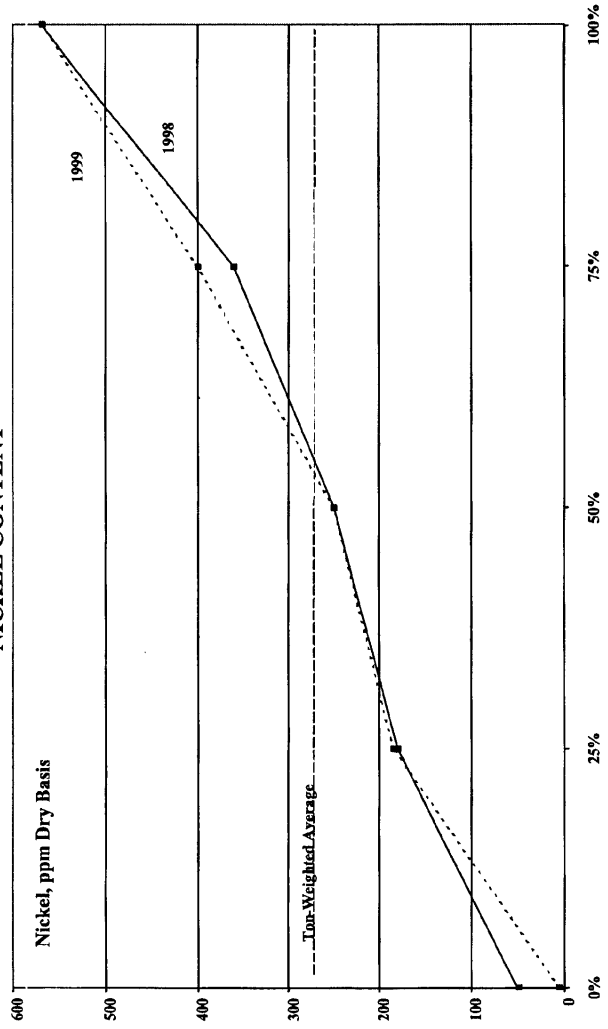


FIGURE 1
U.S. DELAYED PETROLEUM COKE
SULFUR CONTENT
Wt % Sulfur, Dry Basis
1998
1999
Ton-Weighted Average
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0%
25%
50%
75%
100%
Percent of U.S. Production
THE PACE CONSULTANTS INC.

FIGURE 2

U.S. DELAYED PETROLEUM COKE
NICKEL CONTENT



THE PACE CONSULTANTS INC.

FIGURE 3

U.S. DELAYED PETROLEUM COKE
VANADIUM CONTENT

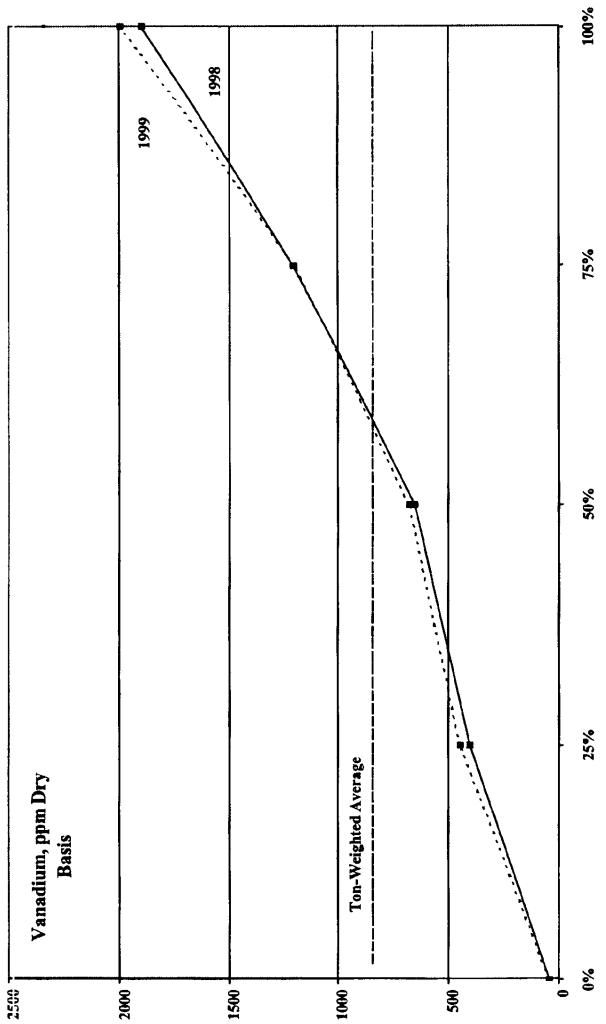


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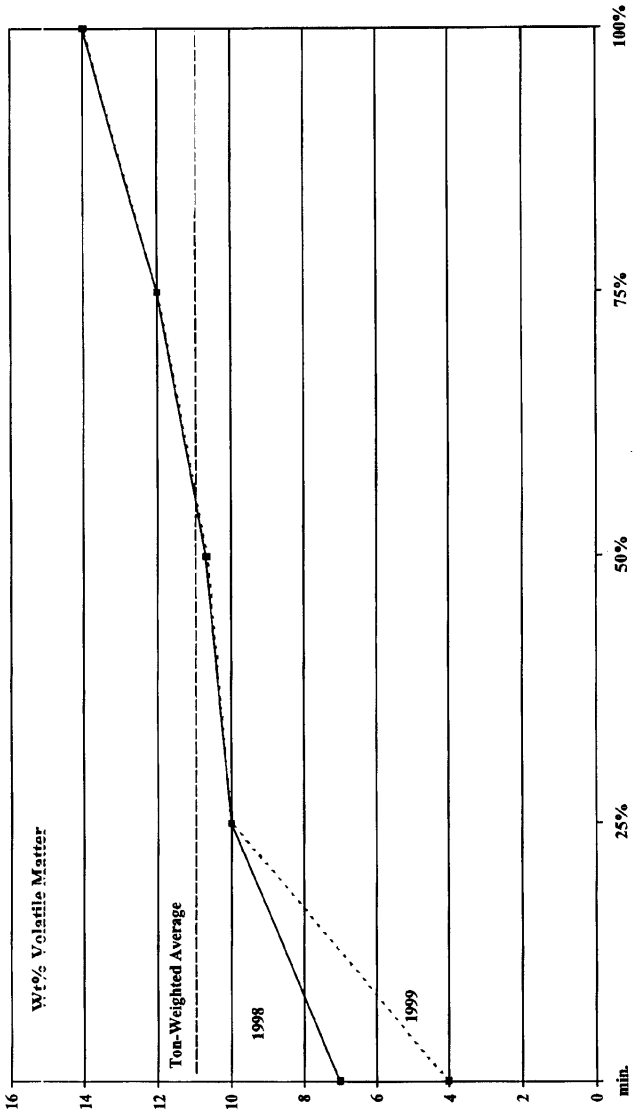
FIGURE 4
U.S. DELAYED PETROLEUM COKE VOLATILE MATTER CONTENT



THE PACE CONSULTANTS INC.

- The sulfur content at the upper and lower ends of the quality spectrum was better in 1999 than in 1998. We believe the lower sulfur content in 1999 was a result of crude production cut-backs by OPEC (Organization of Petroleum Exporting Countries) and other crude oil producers. These producers preferentially reduced the production of their lower quality crude oils in order to minimize the production reductions of their higher quality (i.e. higher priced) crude oils. We see 1999 as an aberration in the general trend of increasing sulfur content in U.S. petroleum cokes.

- We expect the metals content and sulfur content of U.S. petroleum coke will deteriorate beginning in 2001 as new U.S. cokers scheduled to begin operations in the 2000-2002 time frame start up.

- The average volatile matter content is essentially equal to the median.

RECOMMENDATIONS

Pace identified candidate refineries for sampling based on the quality data from the third quarter of 2000, which is the most recent quarter for which data are available. It should be noted that these data may vary slightly from the 1998-1999 averages as increasing amounts of heavy crude are processed. Based on these data, Pace recommends the following candidates for sampling in support of the Petroleum HPV Testing Program:

PETROLEUM HPV TESTING PROGRAM
DELAYED PETROLEUM COKE SAMPLE CANDIDATES

	Candidate A		Candidate B		Candidate C	
	Value	Percentile	Value	Percentile	Value	Percentile
Sulfur, Wt%	6.00	93	5.75	86	5.50	80
Nickel, ppm	500	90	300	58	250	50
Vanadium, ppm	1,500	84	1,200	75	1,000	65
Volatiles, Wt%	10.00	25	12.00	75	13.00	88

PETROLEUM HPV TESTING PROGRAM
DELAYED PETROLEUM COKE SAMPLE CANDIDATES

	Candidate D			Candidate E	
	Value	Percentile		Value	Percentile
Sulfur, Wt%	4.20	43		5.50	80
Nickel, ppm	250	50		350	67
Vanadium, ppm	1,500	84		1,100	70
Volatiles, Wt%	15.00	100		10.00	25

Our analysis indicates that some compromises will have to be made in obtaining a sample for the HPV program since no refinery's petroleum coke is in the upper 75th percentile in all four quality parameters we have evaluated. Additionally, we have spent some time and effort trying to find petroleum cokes which are sampled with automatic sampling equipment that has been bias tested and is operated by an independent laboratory. Unfortunately, we have found that the locations with the best sampling systems have petroleum cokes of generally better quality. Therefore, we do not believe that we will be able to find a "perfect" candidate petroleum coke.

While the sampling at the candidate refineries may not be ideal, the sampling and analysis data have been used for commercial transactions. Substantial quantities of petroleum coke from each of the candidate refineries have been sold in the petroleum coke market. Commercial transactions have relied on the laboratory results for determining quality bonus and penalties and conformance with contract quality specifications. Thus, the samples taken for the HPV study would conform to generally accepted industry sampling practice.

The sampling plan would be to have the sample analyzed for the quality parameters used in this screening analysis (i.e. sulfur, vanadium, nickel, volatile matter) as well as four other commonly tested quality parameters—gross calorific value (Btu/lb), moisture (%), ash (%), and Hardgrove Grindability Index (HGI)–to verify that the sample obtained is similar to the anticipated quality characteristics. This plan would assure that the sample submitted for detailed HPV testing conforms to our quality expectations.

We may not be able to receive authorization from a refinery to use a sample of their petroleum coke for the HPV test. Our present plan would be to approach Refineries B and C regarding obtaining a sample. In the event that these two refineries choose not to participate, then the choice would be either refinery A or E, which have high sulfur and metals but low volatile content or refinery D, which has high vanadium and volatile matter but low sulfur content. (note: each of the five candidate refineries has a different corporate owner).

Pace requests that the HPV Committee confirm Pace's recommended plan to approach refineries B and C regarding obtaining an HPV sample. It is not necessary for the HPV committee to decide now on the preferred refinery to contact in the event that refineries B and C do not wish to participate in the program. However, we would suggest that the committee begin to think about this issue so that decisions can be made expeditiously in the event that refineries B and C choose not participate.

Certificates of Analysis

AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO. H-169N
DESCRIPTION: Benzo(a)pyrene
LOT: 052803MT-AC
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Benzo(a)pyrene (Ames grade)	50-32-8	100	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-001
Rev. 11/02

AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO. H-110N
DESCRIPTION: Anthracene
LOT: A33783
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Anthracene	120-12-7	99.4	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-100N
DESCRIPTION: Benz(a)anthracene
LOT: 19587
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/FID)	Gravimetric Concentration[1]	Analyte Concentration[2]
Benz(a)anthracene	56-55-3	99.5	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-125N
DESCRIPTION: Acenaphthylene
LOT: 011504MS-AC
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/FID)	Gravimetric Concentration[1]	Analyte Concentration[2]
Acenaphthylene	208-96-8	98.5	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-108N
DESCRIPTION: Polynuclear Aromatic Hydrocarbon
LOT: 01915EQ
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Acenaphthene	83-32-9	100	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

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2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-128N
DESCRIPTION: Benzo(b)fluoranthene
LOT: 020402AG-AC
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Benzo(b)fluoranthene	205-99-2	100	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-103N
DESCRIPTION: Benzo(g,h,i)perylene
LOT: 122500MT-AC
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Benzo(g,h,i)perylene	191-24-2	98.3	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-129N
DESCRIPTION: Benzo(k)fluoranthene
LOT: 112603AG-AC
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Benzo(k)fluoranthene	207-08-9	99.7	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-115N
DESCRIPTION: Chrysene
LOT: 13103
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Chrysene	218-01-9	100	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

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1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
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Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-135N
DESCRIPTION: Dibenz(a,h)anthracene
LOT: 13246
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Dibenz(a,h)anthracene	53-70-3	100	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

A comma (,) is used to separate units of one-thousand or greater.
A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-118N
DESCRIPTION: Fluoranthene
LOT: 19762
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Fluoranthene	206-44-0	98.4	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

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A period (.) is used as a decimal place marker.

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480
2. Analyte Concentration = Purity x Gravimetric Concentration
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Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-146N
DESCRIPTION: Fluorene
LOT: 19675
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Fluorene	86-73-7	100	N/A	N/A

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Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-157N
DESCRIPTION: Indeno(1,2,3-cd)pyrene
LOT: 19641
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Indeno(1,2,3-cd)pyrene	193-39-5	100	N/A	N/A

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2. Analyte Concentration = Purity x Gravimetric Concentration
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Certified by: R. Cooper

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-152N
DESCRIPTION: Naphthalene
LOT: 167A-A
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Naphthalene	91-20-3	100	N/A	N/A

Please note: AccuStandard follows the U.S. conventions in reporting numerical values, on both certificates and labels.

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Certified by: R. Cooper

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Rev. 11/02

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-122N
DESCRIPTION: Phenanthrene
LOT: 090903AG-AC-1
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Phenanthrene	85-01-8	99.8	N/A	N/A

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CERTIFICATE OF ANALYSIS

CATALOG NO. H-123N
DESCRIPTION: Pyrene
LOT: 09617LR
SOLVENT: N/A

EXPIRATION: Jun 3, 2007

Maximum uncertainty in the measurement of the purity is ±0.5%.

Component	CAS #	Purity % (GC/MS)	Gravimetric Concentration[1]	Analyte Concentration[2]
Pyrene	129-00-0	98.6	N/A	N/A

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Certified by: R. Cooper

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Rev. 11/02

CIL

CAMBRIDGE ISOTOPE LABORATORIES
50 Frontage Road, Andover, Massachusetts 01810

Certificate of Analysis

Cambridge Isotope Laboratories, Inc. certifies that this material meets or exceeds the specifications stated. Absolute identity as well as chemical and isotopic purities are assured by the use of unambiguous synthetic routes and multiple chemical analyses whenever possible.

Authorized Signature:

Hugh D. Conlon, Ph.D., Quality Control Manager

Product Name: (Isotopic Label, Isotopic Enrichment)

Dibenzo[a,e]pyrene (Unlabeled)

Catalog Number:

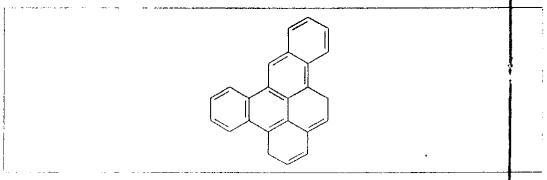
ULM-1226

Unlabeled CAS Number: 192-65-4
Labeled CAS Number: NA

Chemical Purity: 98%+

Molecular Weight: 302.4

Chemical Formula: C24H14



CIL Quality Control Data

Sample compares favorably to known standard for identity and purity. Enrichment meets or exceeds stated specification.

Mass Spectrometry	Pass
GC/FID	Pass
HPLC	Pass
FTIR	Pass

Storage: ***Store at room temperature away from light and moisture.***

Expiration: ***Stable if stored under recommended conditions.***

1-978-749-8000 1-800-322-1174 (USA) 1-800-643-7239 (Canada) 1-978-749-2768 (Fax)

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Cambridge Isotope Laboratories Page 2 of 2

Dibenzo[a,e]pyrene (Unlabeled) ULM-1226

Section 7. Handling and Storage.

General warning:	Possible human carcinogen.
Handling procedures:	Avoid contact with eyes, skin, and clothing.
Storage procedures:	Store at room temperature away from light and moisture. Keep closed.
Hygiene instructions:	Wash thoroughly after handling.
Other:	Use only in a chemical fume hood.

Section 8. Exposure Controls and Personal Protection.

General controls:	Do not breathe vapor. Only experienced personnel should be allowed to handle this material.
Eye/face protection:	Chemical safety goggles.
Skin Protection:	Wear suitable protective clothing.
Respiratory protection:	Cartridge type respirator with organic vapor cartridges recommended.

Section 9. Physical/Chemical Characteristics.

Molecular weight:	302.4	Autoignition temperature:	Unknown.
Appearance:	Pale yellow needles.	Flash point/Method:	Unknown.
Odor:	Unknown.	Melting point:	233°C, 225°C (in vacuo)
Physical state:	Solid.	Boiling point:	Unknown.
pH:	Unknown.	Freezing point:	Unknown.
Vapor pressure:	Unknown.		
Vapor density:	Unknown.		
Solubility in water:	Insoluble.		
Specific gravity/density:	Unknown.		

Section 10. Stability and Reactivity.

Chemical stability:	Stable if stored under recommended conditions.
Conditions to Avoid:	Unknown.
Incompatibility:	Oxidized by sodium dichromate in glacial acetic acid; reacts with Bromine or Acetyl chloride.
Hazardous Decomposition:	Unknown.
Hazardous Polymerization:	Will not occur.

Section 11. Toxicological Information (see Section 3 on first page).

Acute data: May be harmful if swallowed, inhaled, or absorbed through skin. It should be assumed to have toxic effects and, therefore, procedures appropriate for the safe handling of hazardous chemicals should be followed.

Chronic data: IARC 2B The agent is possibly carcinogenic to humans. NTP Group 2 Substances or group of substances which may reasonably be anticipated to be carcinogens.

Section 12. Ecological Information (impact if released into environment).

Data not yet available.

Section 13. Disposal considerations.

Waste materials should be disposed of under conditions which meet Federal, State, and Local environmental control regulations.

Section 14. Transport Information.

Data not available.

Section 15. Regulatory Information.

Data not available.

Section 16. Other Information.

Data not available.

660 Tower Lane • P.O. Box 599 • West Chester, PA 19381-0599
1-800-452-9994 • 1-610-692-3026 • Fax 1-610-692-8729
info@chemservice.com • www.chemservice.com

CERTIFICATE OF ANALYSIS

INVOICE #: CS253100
PO #: 29279

CATALOG #: O-788 CAS #: 91-57-6

DESCRIPTION: 2-Methylnaphthalene

LOT #: 310-43C

PURITY: 98%

EXPIRATION DATE: 09/08

Chem Service, Inc. guarantees the purity of this chemical ± 0.5% deviation prior to the expiration date shown on the label and exclusive of any customer contamination.

Two or more of the following methods of analysis are used to determine purity: Melting point, refractive index, titration, FTIR, IR, TLC, GC/FID, GC/TCD, GC/ECD, GC/MS, HPLC or DSC.

Our standards are suitable for use with all EPA methods.

Certified By:

John Conrad

John Conrad
CSM/TC

ISO 9001
Certificate Number 31610

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1-800-452-9994 • 1-610-692-3026 • Fax 1-610-692-8729
info@chemservice.com • www.chemservice.com

CERTIFICATE OF ANALYSIS

INVOICE #: CS253100
PO #: 29279

CATALOG #: O-787 CAS #: 90-12-0

DESCRIPTION: 1-Methylnaphthalene

LOT #: 325-31A

PURITY: 98%

EXPIRATION DATE: 05/09

Chem Service, Inc. guarantees the purity of this chemical ± 0.5% deviation prior to the expiration date shown on the label and exclusive of any customer contamination.

Two or more of the following methods of analysis are used to determine purity: Melting point, refractive index, titration, FTIR, IR, TLC, GC/FID, GC/TCD, GC/ECD, GC/MS, HPLC or DSC.

Our standards are suitable for use with all EPA methods.

Certified By:

John Conrad
CSM/TC

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ExternalProductDisplay

SIGMA-ALDRICH

Certificate of Analysis

Product Name	Perylene
Product Number	39,447-5
CAS Number	198-55-0
Molecular Formula	$C_{20}H_{12}$
Molecular Weight	252.3

TEST	SPECIFICATION	LOT 20330PO RESULTS
APPEARANCE	ORANGE POWDER OR CRYSTALS AND/OR CHUNKS	ORANGE CRYSTALLINE POWDER
INFRARED SPECTRUM	CONFORMS TO STRUCTURE AND STANDARD AS ILLUSTRATED ON PAGE 968A OF EDITION I, VOLUME 1 OF 'THE ALDRICH LIBRARY OF FT-IR SPECTRA'.	CONFORMS TO STRUCTURE.
HIGH PRESSURE LIQUID CHROMATOGRAPHY	99.50% (MINIMUM)	99.52%
QUALITY CONTROL ACCEPTANCE DATE		DECEMBER 2001

Ronnie J Martin, Supervisor
Quality Control

Chevron Metals Analyses

AUG. 18. 2003 10:47AM [redacted] CRTC THRA [redacted] NO. 2755 P. 2

Analysis Report

Lancaster Laboratories

ANALYTICAL RESULTS

Prepared for:

Chevron Products Company
940 Heneley St. Bldg. 210

Richmond CA 94801
510-242-8191

Prepared by:

Lancaster Laboratories
2425 New Holland Pike
Lancaster, PA 17605-2425

SAMPLE GROUP

The sample group for this submittal is 857532. Samples arrived at the laboratory on Friday, June 27, 2003. The PO# for this group is 99011184 and the release number is BEATTY.

Client Description	Lancaster Labs Number
Pet Coke 2mm Solid Sample	4073301
Pet Coke Micronized Solid Sample	4073302

1 COPY TO Lancaster Laboratories
1 COPY TO Chevron CRTC [redacted]

Questions? Contact your Client Services Representative
Alison M O'Connor at (717) 656-2300.

Respectfully Submitted,

[redacted]

1
A4200
3.00

MEMBER Lancaster Laboratories, Inc.
2425 New Holland Pike
PO Box 12425

AMENDED

AUG. 18. 2003 10:47AM CRTC THRA NO. 2755 P. 3

Analysis Report

Lancaster Laboratories

Lancaster Laboratories Sample No. SW 4073302

Collected:06/26/2003 00:00

Account Number: 10863

Submitted: 06/27/2003 10:40
Reported: 07/09/2003 at 11:42
Discard: 08/09/2003
Pet Coke Micronized Solid Sample
Cost Center# ENG-4066
HPV Petroleum Cake

Chevron Products Company
940 Hensley St. Bldg. 210

Richmond CA 94801

MICPC

CAT No.	Analysis Name	CAS Number	As Received Result		As Received Method Detection Limit	Units	Dilution Factor
07804	PAHs in Soil by GC/MS						
01191	Acenaphthene	83-32-9	N.D.		1,000.	ug/kg	10
01195	Pyrene	129-00-0	8,600.	J	1,000.	ug/kg	10
02751	1-Methylnaphthalene	90-12-0	10,000.		1,000.	ug/kg	10
03761	Naphthalene	91-20-3	11,000.		1,000.	ug/kg	10
03765	Acenaphthylene	208-96-8	N.D.		1,000.	ug/kg	10
03768	Fluorene	86-73-7	1,500.	J	1,000.	ug/kg	10
03775	Phenanthrene	85-01-8	7,800.	J	1,000.	ug/kg	10
03776	Anthracene	120-12-7	3,300.	J	1,000.	ug/kg	10
03779	Fluoranthene	206-44-0	1,400.	J	1,000.	ug/kg	10
03781	Benzo(a)anthracene	56-55-3	7,100.	J	1,000.	ug/kg	10
03782	Chrysene	218-01-9	9,400.	J	1,000.	ug/kg	10
03786	Benzo(b)fluoranthene	205-99-2	3,800.	J	1,000.	ug/kg	10
03787	Benzo(k)fluoranthene	207-08-9	N.D.		1,000.	ug/kg	10
03788	Benzo(a)pyrene	50-32-8	11,000.		1,000.	ug/kg	10
03789	Indeno(1,2,3-cd)pyrene	193-39-5	3,500.	J	1,000.	ug/kg	10
03790	Dibenz(a,h)anthracene	53-70-3	4,100.	J	1,000.	ug/kg	10
03791	Benzo(g,h,i)perylene	191-24-2	8,700.	J	1,000.	ug/kg	10
04694	2-Methylnaphthalene	91-57-6	26,000.		1,000.	ug/kg	10

Due to sample matrix interferences observed during the extraction, the normal reporting limits could not be obtained.

Due to the sample matrix an initial dilution was necessary to perform the analysis. Therefore, the reporting limits for the GC/MS semivolatile compounds were raised.

State of California Lab Certification No. 2116

Laboratory Chronicle

CAT No.	Analysis Name	Method	Trial#	Analysis Date and Time	Analyst	Dilution Factor
07804	PAHs in Soil by GC/MS	SW-846 8270C	1	07/02/2003 18:34	Susan L Scheuering	10

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Lancaster Laboratories, Inc.
2425 New Holland Pike
PO Box 12425

AUG. 18. 2003 10:47AM CRTC THRA NO. 2755 P. 4

Analysis Report

Lancaster Laboratories

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Lancaster Laboratories Sample No. SW 4073302

Collected:06/26/2003 00:00

Account Number: 10863

Submitted: 06/27/2003 10:40
Reported: 07/09/2003 at 11:42
Discard: 08/09/2003
Pet Coke Micronized Solid Sample
Cost Center# ENG-4066
HPV Petroleum Cake

Chevron Products Company
940 Hensley St. Bldg. 210

Richmond CA 94801

MICPC

07805	BNA Soil Extraction	SW-846 3550B	1	06/30/2003 20:00	Sally L Appleyard 1

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2425 New Holland Pike
PO Box 12425
Lancaster PA 17605-2425

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Analysis Report

Lancaster Laboratories

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Lancaster Laboratories Sample No. SW 4073301

Collected:06/26/2003 00:00

Account Number: 10863

Submitted: 06/27/2003 10:40
Reported: 07/09/2003 at 11:42
Discard: 08/09/2003
Pet Coke 2mm Solid Sample
Cost Center# ENG-4066
HPV Petroleum Cake

Chevron Products Company
940 Hensley St. Bldg. 210

Richmond CA 94801

2MMPC

CAT No.	Analysis Name	CAS Number	As Received Result		As Received Method Detection Limit	Units	Dilution Factor
07804	PAHs in Soil by GC/MS						
01191	Acenaphthene	83-32-9	N.D.		330.	ug/kg	10
01195	Pyrene	129-00-0	1,300.	J	330.	ug/kg	10
02751	1-Methylnaphthalene	90-12-0	2,700.	J	330.	ug/kg	10
03761	Naphthalene	91-20-3	3,600.		330.	ug/kg	10
03765	Acenaphthylene	208-96-8	N.D.		330.	ug/kg	10
03768	Fluorene	86-73-7	340.	J	330.	ug/kg	10
03775	Phenanthrene	85-01-8	690.	J	330.	ug/kg	10
03776	Anthracene	120-12-7	N.D.		330.	ug/kg	10
03778	Fluoranthene	206-44-0	N.D.		330.	ug/kg	10
03781	Benzo(a)anthracene	56-55-3	580.	J	330.	ug/kg	10
03782	Chrysene	218-01-9	880.	J	330.	ug/kg	10
03786	Benzo(b)fluoranthene	205-99-2	520.	J	330.	ug/kg	10
03787	Benzo(k)fluoranthene	207-08-9	N.D.		330.	ug/kg	10
03788	Benzo(a)pyrene	50-32-8	1,800.	J	330.	ug/kg	10
03789	Indeno(1,2,3-cd)pyrene	193-39-5	340.	J	330.	ug/kg	10
03790	Dibenz(a,h)anthracene	53-70-3	490.	J	330.	ug/kg	10
03791	Benzo(g,h,i)perylene	191-24-2	1,100.	J	330.	ug/kg	10
04694	2-Methylnaphthalene	91-57-6	11,000.		330.	ug/kg	10

Due to the sample matrix an initial dilution was necessary to perform the analysis. Therefore, the reporting limits for the GC/MS semivolatile compounds were raised.

State of California Lab Certification No. 2116

Laboratory Chronicle

CAT No.	Analysis Name	Method	Trial#	Analysis Date and Time	Analyst	Dilution Factor
07804	PAHs in Soil by GC/MS	SW-846 8270C	1	07/02/2003 15:41	Susan L Schoucring	10
07806	DNA Soil Extraction	SW-846 3550B	1	06/30/2003 20:00	Sally L Appleyard	1

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2425 New Holland Pike
PO Box 12425

AMENDED

Analysis Report

Lancaster Laboratories

Quality Control Summary

Client Name: Chevron Products Company Group Number: 857532
Reported: 07/09/03 at 11:42 AM

Laboratory Compliance Quality Control

Analysis Name	Blank Result	Blank MDL	Report Units	LCS %REC	LCSD %REC	LCS/LCSD Limits	RPD	RPD Max
Batch number: 03181SLA026	Sample number(s): 4073301-4073302							
Acenaphthene	N.D.	33.	ug/kg	91		76-109		
Pyrene	N.D.	33.	ug/kg	89		71-110		
1-Methylnaphthalene	N.D.	33.	ug/kg	87		76-101		
Naphthalene	N.D.	33.	ug/kg	87		73-103		
Acenaphthylene	N.D.	33.	ug/kg	94		73-106		
Fluorene	N.D.	33.	ug/kg	93		66-115		
Phenanthrene	N.D.	33.	ug/kg	88		70-107		
Anthracene	N.D.	33.	ug/kg	86		71-107		
Fluoranthene	N.D.	33.	ug/kg	90		69-107		
Benzo(a)anthracene	N.D.	33.	ug/kg	93		74-107		
Chrysene	N.D.	33.	ug/kg	89		72-109		
Benzo(b)fluoranthene	N.D.	33.	ug/kg	95		71-113		
Benzo(k)fluoranthene	N.D.	33.	ug/kg	97		75-112		
Benzo(a)pyrene	N.D.	33.	ug/kg	94		79-111		
Indeno(1,2,3-cd)pyrene	N.D.	33.	ug/kg	88		74-113		
Dibenz(a,h)anthracene	N.D.	33.	ug/kg	95		81-118		
Benzo(g,h,i)perylene	N.D.	33.	ug/kg	92		74-114		
2-Methylnaphthalene	N.D.	33.	ug/kg	90		70-102		

Sample Matrix Quality Control

Analysis Name	MS %REC	MSD %REC	MS/MSD Limits	RPD	RPD MAX	BKG Conc	DUP Conc	DUP RPD	Dup RPD Max
Batch number: 03181SLA026	Sample number(s): 4073301-4073302								
Acenaphthene	107	93	48-132	14	30				
Pyrene	82	68	28-144	12	30				
1-Methylnaphthalene	75	67*	72-100	5	30				
Naphthalene	77	61	38-132	9	30				
Acenaphthylene	108	91	46-128	18	30				
Fluorene	88	75	39-137	14	30				
Phenanthrene	88	74	29-143	13	30				
Anthracene	101	85	35-138	17	30				
Fluoranthene	81	72	19-145	11	30				
Benzo(a)anthracene	89	75	26-144	14	30				
Chrysene	101	90	23-150	9	30				
Benzo(b)fluoranthene	90	74	32-140	16	30				
Benzo(k)fluoranthene	103	88	36-143	16	30				
Benzo(a)pyrene	90	72	23-154	13	30				
Indeno(1,2,3-cd)pyrene	92	78	13-155	15	30				
Dibenz(a,h)anthracene	110	86	19-163	19	30				
Benzo(g,h,i)perylene	99	83	17-152	13	30				
2-Methylnaphthalene	38	19*	32-133	6	30				

*- 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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Analysis Report

Lancaster Laboratories

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Quality Control Summary

Client Name: Chevron Products Company Group Number: 857532
Reported: 07/09/03 at 11:42 AM

Sample Matrix Quality Control

Analysis Name	MS %REC	MSD %REC	MS/MSD Limits	RPD	RPD MAX	BKG Conc	DUP Conc	DUP RPD	Dup RPD Max

Surrogate Quality Control

Analysis Name: PAHs in Soil by GC/MS
Batch number: 031818LA026

	Nitrobenzene-d5	2-Fluorobiphenyl	Terphenyl-d14
4073301	101	108	92
4073302	101	99	84
Blank	97	85	83
LCS	94	92	93
MS	105	107	88
MSD	90	90	78
Limits:	47-128	55-123	39-128

*- 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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2425 New Holland Pike
PO Box 12425

AMENDED

Lancaster Laboratory PAH Analyses

[redacted]

From: [redacted]
Sent:
To:
Cc:
Subject: Found your results.

micronized

3030999 PETROLEUM COKE 2NM YCJ58009 REGULAR SERVICE
REPORTED 06/13/2003 Marked-up: 06/12/2003 by [redacted] at 50-1118
[redacted]474/0) Prj Id: GLOBETECH

Test code	Test Name/Element/Result	Test Status	Analyst Status date	Test Cost
30258	MICROWAVE DIGST/ICP PLUS	REPORTED	[redacted] 06/13/2003	$200.00

AL	300.200 PPM	AS	<29.61 PPM	B	<29.61 PPM
BA	<29.61 PPM	BE	<14.805 PPM	BI	<29.61 PPM
CA	121.600 PPM	CD	<14.805 PPM	CO	<14.805 PPM
CR	<14.805 PPM	CU	<17.766 PPM	FE	247.000 PPM
K	<44.414 PPM	LI	<14.805 PPM	MG	60.850 PPM
MN	<29.61 PPM	MO	<29.61 PPM	NA	114.600 PPM
NI	351.700 PPM	P	30.300 PPM	PB	<29.61 PPM
S	58060.000 PPM	SB	<74.024 PPM	SE	<29.61 PPM
SI	554.600 PPM	SN	<44.414 PPM	TI	<14.805 PPM
V	1805.000 PPM	ZN	<14.805 PPM		

2mm

3030251 PETROLEUM COKE YCJ58009 REGULAR SERVICE
REPORTED 06/09/2003 Marked-up: 06/09/2003 by [redacted] at 50-1118
[redacted]474/0) Prj Id: [redacted]

Test code	Test Name/Element/Result	Test Status	Analyst Status date	Test Cost
30258	MICROWAVE DIGST/ICP PLUS	REPORTED	TMEAH 06/09/2003	$200.00

AL	321.000 PPM	AS	<19.279 PPM	B	<19.279 PPM
BA	<19.279 PPM	BE	<9.639 PPM	BI	<19.279 PPM
CA	178.000 PPM	CD	<9.639 PPM	CO	<9.639 PPM
CR	<9.639 PPM	CU	<11.567 PPM	FE	310.000 PPM
K	<28.918 PPM	LI	<9.639 PPM	MG	77.370 PPM
MN	<19.279 PPM	MO	<19.279 PPM	NA	133.000 PPM
NI	367.100 PPM	P	<19.279 PPM	PB	<19.279 PPM
S	73920 PPM	SB	<48.197 PPM	SE	<19.279 PPM
SI	743.200 PPM	SN	<28.918 PPM	TI	12.910 PPM
V	1938.000 PPM	ZN	12.010 PPM		

-----Original Message-----
From: [redacted]

1

Aveka, Inc. Milled Particle Size Analysis

AVEKA, INC.
PARTICLE PROCESSING & CUSTOM RESEARCH

Date: May 29, 2003

Make Order #: 5369

Company Name: API

Contact Person: [redacted]

Material: Green Petroleum Coke

Objective: Task 1: Hammermill, Ball-mill and Classify Petroleum Coke to a mean particle size less than 3.6 microns. Task 2: Crush and Classify petroleum coke to a mean particle size of 2 mm.

Equipment: Homoloid JT Hammermill (SN # JT-694) with 0.0093 screen
5 Gallon Ball-mill with 0.25 inch alumina media
Majac A-12 classifier
Horiba LA-910 Laser Light Scattering Particle Sizer
Marcy 4"x 6" Jaw Crusher
Gilson Sonic Sieve

Receipt: Approximately 80 lbs. of material was received 3-19-03 from Federal Express. Confirmation of receipt (EPL Project Identification 1203-001) was returned upon delivery.

Storage: Petroleum coke was stored at room temperature in sealed polyethylene bags when the material was not being processed.

Processing Procedure:

The green petroleum coke showed high moisture content upon inspection. The high moisture content was indicated by condensation on the inside of the received petroleum coke bags. After consulting with Deborah Herron and Jacobs Consultancy, the material was dried according to ASTM D 3302-00 (Standard Test Method for Total Moisture in Coal).

Task 1

All processes were run at room temperature. The dried petroleum coke was then run through a Homoloid JT Hammermill (SN # JT-694) equipped with a 0.0093 screen.

The resulting hammermilled powder was loaded into 5-gallon ball mills loaded with 0.25 inch ceramic (alumina) media. The loading level in the ball mill was 27 lbs. of media with 5.5 lbs. of petroleum coke.

AVEKA, INC.
PARTICLE PROCESSING & CUSTOM RESEARCH

The mills were rotated at 36 rpm for 17.25 hours. The resulting powder had a mean particle size of 9.56 microns (Attch 1) when tested with the Horiba LA-910 in water.

The oversized petroleum coke material was removed using a Majac A-12 Classifier. The Majac was run at 1800 RPM and 8.5 cfm. The resulting particle size of the petroleum coke was a 3.3 micron mean (Attch. 2) when tested with the Horiba LA-910 in water. The Horiba LA-910 test method for the petroleum coke samples is outlined in Attch. 3.

The final yield of product was 10.5 kg of powder.

Task 2

All processes were run at room temperature. An 18" Sweco Screener was set-up with a 7 mesh (2.8 mm) top-screen and a 14 mesh (1.4 mm) bottom-screen. Petroleum coke was fed through the screener and 2-mm material was collected from between the top and bottom screen. Oversized petroleum coke was jaw crushed with a Marcy 4"x 6" Jaw Crusher and rescreened. A Gilson Sonic Sieve particle size analysis (Attch. 4) was run on the screened petroleum coke and the results showed 99.4 % of the material between 1.4 mm – 2.8 mm. Final yield was 3.3 kg of 2 mm Petroleum Coke.

Shipping

All samples were shipped UPS Ground. The following is a summary of the sample disposition.

Sample/Amount	Address	Person
200 grams of 2-3 micron particle size sample	ChevronTexaco Energy Research and Technology Corp. 100 Chevron Way Richmond, CA 94802 Tel: 510-242-7037	Richard Dutta

AVEKA, Inc.

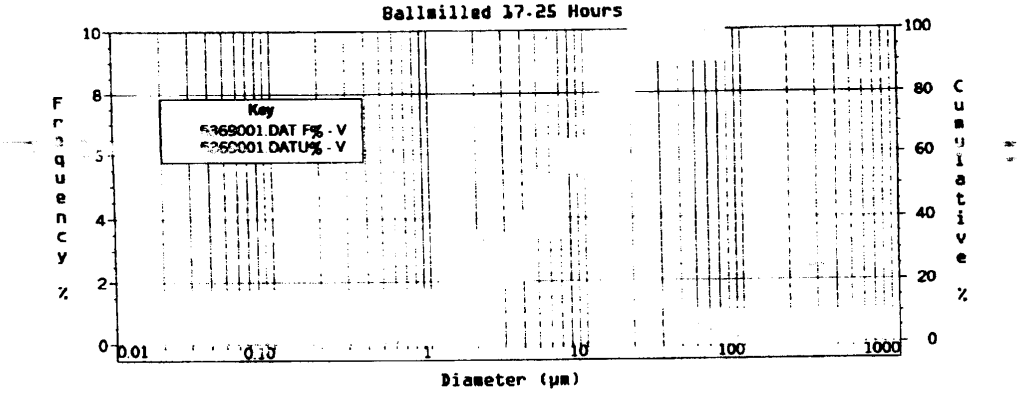
PARTICLE PROCESSING & CUSTOM RESEARCH

200 grams of 2 mm particle sample	ChevronTexaco Energy Research and Technology Corp. 100 Chevron Way Richmond, CA 94802 Tel: 510-242-7037	Richard Dutta
10.5 kg of 2-3 micron particle size sample	EPL Archives, Inc. 45610 Terminal Drive Sterling, Virginia 20166 703/435-8780 ext 201	Sam Busey
Remainder (slightly less than 3 kg) of 2 mm particle size sample)	EPL Archives, Inc. 45610 Terminal Drive Sterling, Virginia 20166 703/435-8780 ext 201	Sam Busey
Leftover petroleum coke material, i.e., that material not used in samples	EPL Archives, Inc. 45610 Terminal Drive Sterling, Virginia 20166 703/435-8780 ext 201	Sam Busey

651-730-1729 ■ 2045 Wooddale Drive, Woodbury, MN 55125 ■ FAX 651-730-1826

Attch 1

HORIBA LA-910
PARTICLE SIZE DISTRIBUTION DATA TABLE Standard 04/23/03
File Name: 5369001.DAT Sample Name: Ballmilled 17.25 Hours ID No: **/04/23-350
Dist. Form: STANDARD R.R. Index: co.rrj□
Dist. Mode: VOLUME Laser: 65.128 % Lamp: 61.185 %
Circulation: 2 Agitation: 7 U.Sonic ** (min)
Material: Petroleum Coke Source: American Petroleum
Lot No: MO5369 Test No: 5369001

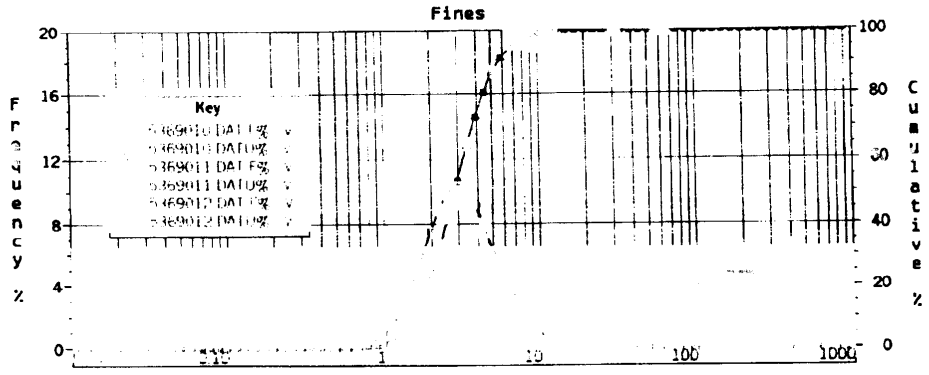


	SIZE(μm)	FREQ%	UNDR%	No.	SIZE(μm)	FREQ%	UNDR%
(1)	0.020	0.0	0.0	(28)	0.766	0.5	0.8	(55)	29.907	1.5	95.4
(2)	0.022	0.0	0.0	(29)	0.877	0.8	1.6	(56)	34.255	1.2	96.6
(3)	0.026	0.0	0.0	(30)	1.005	1.2	2.8	(57)	39.234	0.9	97.5
(4)	0.029	0.0	0.0	(31)	1.151	1.6	4.4	(58)	44.938	0.7	98.2
(5)	0.034	0.0	0.0	(32)	1.318	2.0	6.3	(59)	51.471	0.5	98.8
(6)	0.039	0.0	0.0	(33)	1.510	2.3	8.7	(60)	58.953	0.4	99.2
(7)	0.044	0.0	0.0	(34)	1.729	2.6	11.3	(61)	67.523	0.3	99.5
(8)	0.051	0.0	0.0	(35)	1.981	2.9	14.2	(62)	77.340	0.2	99.7
(9)	0.058	0.0	0.0	(36)	2.269	3.0	17.2	(63)	88.582	0.2	99.9
(10)	0.067	0.0	0.0	(37)	2.599	3.2	20.4	(64)	101.460	0.1	100.0
(11)	0.076	0.0	0.0	(38)	2.976	3.5	23.9	(65)	116.210	0.0	100.0
(12)	0.087	0.0	0.0	(39)	3.409	3.7	27.7	(66)	133.103	0.0	100.0
(13)	0.100	0.0	0.0	(40)	3.905	4.0	31.6	(67)	152.453	0.0	100.0
(14)	0.115	0.0	0.0	(41)	4.472	4.3	35.9	(68)	174.616	0.0	100.0
(15)	0.131	0.0	0.0	(42)	5.122	4.6	40.6	(69)	200.000	0.0	100.0
(16)	0.150	0.0	0.0	(43)	5.867	5.1	45.6	(70)	229.075	0.0	100.0
(17)	0.172	0.0	0.0	(44)	6.720	5.5	51.2	(71)	262.376	0.0	100.0
(18)	0.197	0.0	0.0	(45)	7.697	5.9	57.0	(72)	300.518	0.0	100.0
(19)	0.226	0.0	0.0	(46)	8.816	6.0	63.0	(73)	344.205	0.0	100.0
(20)	0.259	0.0	0.0	(47)	10.097	5.8	68.8	(74)	394.244	0.0	100.0
(21)	0.296	0.0	0.0	(48)	11.565	5.4	74.2	(75)	451.556	0.0	100.0
(22)	0.339	0.0	0.0	(49)	13.246	4.8	79.1	(76)	517.200	0.0	100.0
(23)	0.389	0.0	0.0	(50)	15.172	4.2	83.3	(77)	592.387	0.0	100.0
(24)	0.445	0.0	0.0	(51)	17.377	3.5	86.8	(78)	678.504	0.0	100.0
(25)	0.510	0.0	0.0	(52)	19.904	2.9	89.7	(79)	777.141	0.0	100.0
(26)	0.584	0.1	0.1	(53)	22.797	2.3	92.0	(80)	890.116	0.0	100.0
(27)	0.669	0.2	0.3	(54)	26.111	1.9	93.9	(81)	1019.510	0.0	100.0

Median : 6.533 (μm) Mean: 9.561 (μm) Mode: 8.237 (μm)
Std. Dev.: 10.531 (μm) Span: 10.623
Coef. Var: 110.14% Spec. Area: 15308 (cm2/cm3)

Attch. 2

HORIBA LA-910
PARTICLE SIZE DISTRIBUTION DATA TABLE Standard 05/15/03
File Name: 5369011.DAT Sample Name: Fines ID No: **/04/30-566
Dist. Form: STANDARD R.R. Index: co.rj□
Dist. Mode: VOLUME Laser: 85.118 % Lamp: 86.338 %
Circulation: 3 Agitation: 7 U.Sonic OFF (min)
Material: Petroleum Coke Source: American Petroleum
Lot No: MO5369 Test No: 5369004



No.	SIZE(μm)	FREQ%	UNDR%	No.	SIZE(μm)	FREQ%	UNDR%	No.	SIZE(μm)	FREQ%	UNDR%
(1)	0.020	0.0	0.0	(28)	0.766	0.2	0.2	(55)	29.907	0.0	100.0
(2)	0.022	0.0	0.0	(29)	0.877	0.5	0.7	(56)	34.255	0.0	100.0
(3)	0.026	0.0	0.0	(30)	1.005	1.0	1.7	(57)	39.234	0.0	100.0
(4)	0.029	0.0	0.0	(31)	1.151	1.7	3.5	(58)	44.938	0.0	100.0
(5)	0.034	0.0	0.0	(32)	1.318	2.8	6.3	(59)	51.471	0.0	100.0
(6)	0.039	0.0	0.0	(33)	1.510	4.3	10.6	(60)	58.953	0.0	100.0
(7)	0.044	0.0	0.0	(34)	1.729	5.9	16.5	(61)	67.523	0.0	100.0
(8)	0.051	0.0	0.0	(35)	1.981	7.6	24.1	(62)	77.340	0.0	100.0
(9)	0.058	0.0	0.0	(36)	2.269	9.0	33.0	(63)	88.582	0.0	100.0
(10)	0.067	0.0	0.0	(37)	2.599	10.1	43.1	(64)	101.460	0.0	100.0
(11)	0.076	0.0	0.0	(38)	2.976	10.6	53.7	(65)	116.210	0.0	100.0
(12)	0.087	0.0	0.0	(39)	3.409	10.2	63.8	(66)	133.103	0.0	100.0
(13)	0.100	0.0	0.0	(40)	3.905	9.0	72.9	(67)	152.453	0.0	100.0
(14)	0.115	0.0	0.0	(41)	4.472	7.6	80.4	(68)	174.616	0.0	100.0
(15)	0.131	0.0	0.0	(42)	5.122	6.0	86.5	(69)	200.000	0.0	100.0
(16)	0.150	0.0	0.0	(43)	5.867	4.6	91.1	(70)	229.075	0.0	100.0
(17)	0.172	0.0	0.0	(44)	6.720	3.4	94.5	(71)	262.376	0.0	100.0
(18)	0.197	0.0	0.0	(45)	7.697	2.3	96.8	(72)	300.518	0.0	100.0
(19)	0.226	0.0	0.0	(46)	8.816	1.5	98.3	(73)	344.205	0.0	100.0
(20)	0.259	0.0	0.0	(47)	10.097	0.9	99.1	(74)	394.244	0.0	100.0
(21)	0.296	0.0	0.0	(48)	11.565	0.5	99.6	(75)	451.556	0.0	100.0
(22)	0.339	0.0	0.0	(49)	13.246	0.2	99.9	(76)	517.200	0.0	100.0
(23)	0.389	0.0	0.0	(50)	15.172	0.1	100.0	(77)	592.387	0.0	100.0
(24)	0.445	0.0	0.0	(51)	17.377	0.0	100.0	(78)	678.504	0.0	100.0
(25)	0.510	0.0	0.0	(52)	19.904	0.0	100.0	(79)	777.141	0.0	100.0
(26)	0.584	0.0	0.0	(53)	22.797	0.0	100.0	(80)	890.116	0.0	100.0
(27)	0.669	0.0	0.0	(54)	26.111	0.0	100.0	(81)	1019.510	0.0	100.0

Median : 2.840 (μm) Mean: 3.289 (μm) Mode: 2.781 (μm)
Std. Dev.: 1.844 (μm) Span: 2.780
Coef. Var: 56.08% Spec. Area: 23790 (cm2/cm3)

Attch. 3

TEST METHOD FOR API PETROLEUM COKE

Sample Preparation **May 15, 2003**

Mix 0.15 – 0.2 grams of petroleum coke with 5-6 grams distilled water. Add TX-100 surfactant to aid dispersion. Mix thoroughly until no large concentrations of sample are evident.

LA-910 Preparation

Fill the test chamber to capacity with 140 ml distilled water. Add 3-4 drops of TX-100 surfactant from a 10% concentrate source, resulting in approximately a .1% diluted total. Select the relative refractive index appropriate for this material (1.61-3.02i). Circulate the solvent using a pump speed of 2-3, subtract the background. Add the sample drop by drop until the laser transmission falls into the acceptable range (70 – 95)% transmittance. Activate the sonicator to aid dispersion, cease sonication when sample is completely dispersed.

Sample Test

Measure the sample three times. Save each measurement. Overlay the three measurements on a graph. If they appear stable, the test is complete. If not, investigate. A steady increase in the laser transmission rate indicates more particles are present from pass to pass. That indicates the sample was not completely dispersed yet. A steady decrease in the laser transmission rate indicates the sample is agglomerating, settling, or dissolving.

Report

Using the Display module, graph the three test runs over one another. A stable test will appear as one line, an unstable condition will clearly show all three runs, indicating instability. If stable, select a run (typically the middle run) and print the complete data table along with the graph.

Author: T.J. Roberts
Lab Manager
Aveka, Inc.
(651) 714-4293 ext 208

Attch. 4

5/29/03

MO5369a.xls

Sieve Analysis

Sample ID: American Petroleum Institute 2mm Pet.Coke

US Standard Mesh Size	Mesh Opening (Microns)	Sieve Weight (Grams)	Sieve Weight + Sample (g)	Weight of Sample (g)	% Sample Above Sieve	[illegible]
7	2800	50.951	50.975	0.024	0.31	99.69
8	2360	50.741	52.146	1.405	18.18	81.51
10	2000	48.772	51.024	2.252	29.14	[illegible]
12	1700	47.324	50.173	2.849	36.86	15.51
14	1400	48.450	49.624	1.174	15.19	0.32
catch	0	220.018	220.043	0.025	0.32	0.00
			Totals:	7.729	100.00	

Notes/Comments:

Appendix 5

Personnel Involved in the Study

The following key Wildlife International, Ltd. personnel were involved in the conduct or management of this study:

1. [redacted]
2. [redacted]
3. [redacted]
4. [redacted]

Appendix 6

Report Amendment

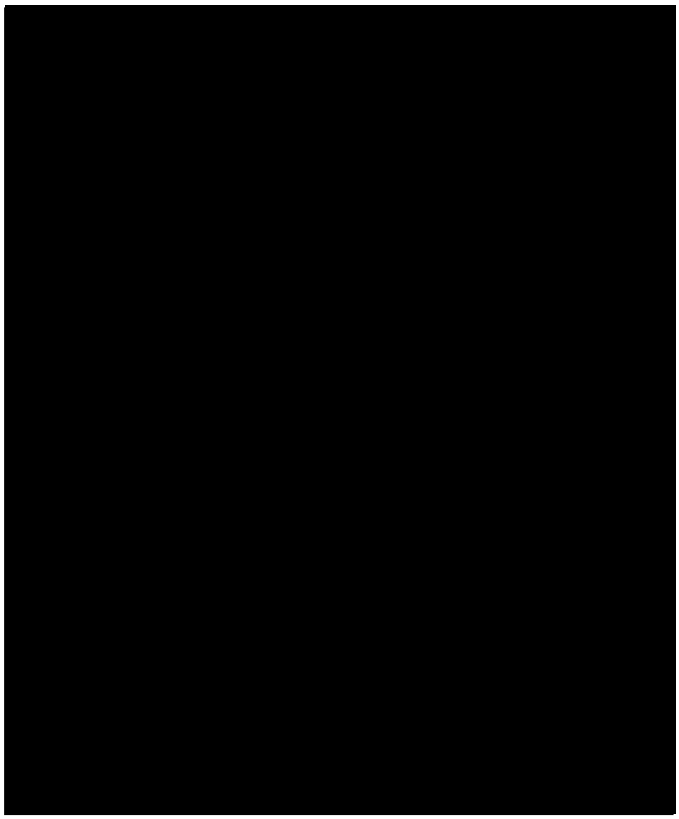
1.	Original Report:	Title Page
	Amended Report:	The amended report date was added. The total number of pages was changed from 150 to 163
	Reason:	To indicate that the report was amended and note change in pagination.
2.	Original Report:	Page 2
	Amended Report:	The amended report date was added and new signatures and dates were added.
	Reason:	To show the amended report date and to provide new signatures and dates for the amended report.
3.	Original Report:	Page 3
	Amended Report:	The audit dates fo r the am ended report were added and a new signature and date were added.
	Reason:	To show the amended report audit dates and to provide a new signature and date for the amended report.
4.	Original Report:	Page 4
	Amended Report:	New signatures and dates were added.
	Reason:	To provide new signatures and dates fothe amended report.
5.	Original Report:	Page 11
	Amended Report:	The Table of Contents was updated to show the addition of the Test Article Selection section in Appendix 4, renumber all appendices from Appendix 4 through the end of the report and added the Report Am endment appendix (Appendix 6).
	Reason:	The Sponsor requested that the Test Article Selection section be added to Appendix 4.
6.	Original Report:	Page 114
	Amended Report:	Test Article Selection was added to Appendix 4.
	Reason:	The Sponsor requested that the Test Article Selection section be added to Appendix 4.
7.	Original Report:	Pages 114-150
	Amended Report:	The Test Article Selection section was added to Appendix 4, therefore all appendices thereafter were renumbered.
	Reason:	The Sponsor requested that the Test Article Selection section be added to Appendix 4.

Appendix 6

(continued)

Report Amendment

AMENDMENT SIGNATURES:



4-10-07
Date

4/10/07
Date

4/26/2007
Date

4/10/2007
Date

AMENDED