

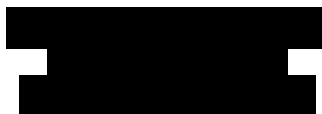
PETROLEUM COKE:
A 14-DAY ACUTE TOXICITY STUDY WITH THE EARTHWORM
IN AN ARTIFICIAL SOIL SUBSTRATE

AMENDED FINAL REPORT

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 472-101

OECD GUIDELINE 207

AUTHORS:



STUDY INITIATION DATE: April 22, 2004

STUDY COMPLETION DATE: June 23, 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

- 2 -

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

SPONSOR: American Petroleum Institute

TITLE: Petroleum Coke: A 14-Day Acute Toxicity Study with the Earthworm in an Artificial Soil Substrate

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 472-101

STUDY COMPLETION: June 23, 2006

AMENDED REPORT DATE: April 10, 2007

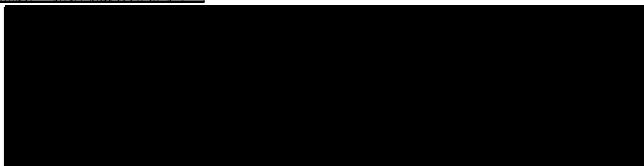
This study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency (40 CFR Part 792, 17 August 1989) (1); OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17, Paris, 1998) (2); and Japan MAFF (11 NohSan, Notification No. 6283, Agricultural Production Bureau, 1 October 1999) (3), 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 and reference substances, and the stability of the substances under conditions of storage at the test site, were not determined in compliance with Good Laboratory Practice Standards.

The concentration, stability and homogeneity of the test substance in the carrier (soil) were not determined analytically.

STUDY DIRECTOR:

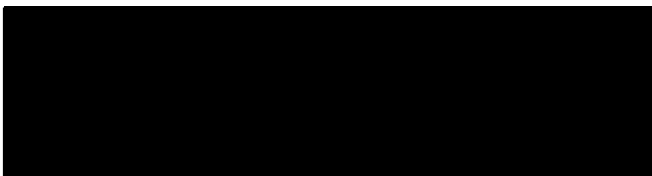


Wildlife International, Ltd.

4/10/07
Date

SPONSOR:

American Petroleum Institute, by:



4/26/2007
Date

AMENDED

- 3 -

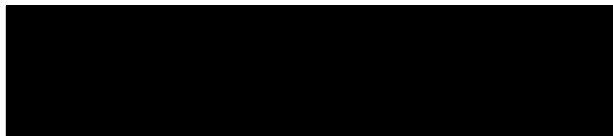
QUALITY ASSURANCE STATEMENT

This study was examined for compliance, with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency (40 CFR Part 792, 17 August 1989) (1); OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17, Paris, 1998) (2); and Japan MAFF (11 NohSan, Notification No. 6283, Agricultural Production Bureau, 1 October 1999) (3). 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 20, 2004	April 20, 2004	April 22, 2004
Test Substance Preparation	February 9, 2005	February 9, 2005	February 10, 2005
Body Weights and Observations	February 23, 2005	February 23, 2005	February 23, 2005
Data and Draft Report	March 8 and 9, 2005	March 9, 2005	March 10, 2005
Final Report	June 22, 2006	June 22, 2006	June 23, 2006
Amended Report	March 30, 2007	March 30, 2007	April 10, 2007

All inspections were study-based unless otherwise noted.



4-10-07
Date

AMENDED

- 4 -

AMENDED REPORT APPROVAL

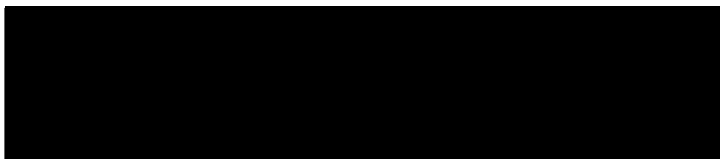
SPONSOR: American Petroleum Institute

TITLE: Petroleum Coke: A 14-Day Acute Toxicity Study with the Earthworm in an Artificial Soil Substrate

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 472-101

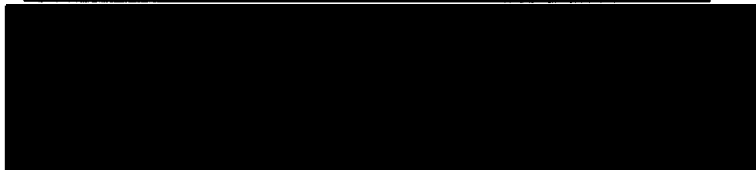
This report was reviewed by the individuals involved in the conduct and management of the study, and was found to be an accurate reflection of the methods used, data collected and results of the study.

STUDY DIRECTOR:



4/10/07
Date

WILDLIFE INTERNATIONAL, LTD. MANAGEMENT:



10 April 07
Date

AMENDED

TABLE OF CONTENTS

Title/Cover Page.....	1
Good Laboratory Practice Compliance Statement	2
Quality Assurance Statement	3
Report Approval	4
Table of Contents	5
Summary	7
Introduction	8
Objective	8
Experimental Design	8
Materials and Methods	9
Test Substance	9
Test Organism.....	9
Artificial Soil and Test Soils Preparation	9
Test Chambers	10
Physical Properties of Test Soils	10
Environmental Conditions.....	11
Observations	11
Body Weights	11
Reference Toxicity Test	11
Conditions for the Validity of the Test.....	12
Data Analyses	12
Results and Discussion	12
Environmental Conditions	12
Physical Properties of Test Soils.....	12
Observations	12
Body Weights.....	13
Reference Toxicity Test	13
Conclusions	13
References	14

TABLE OF CONTENTS (Continued)**TABLES**

Table 1.	Moisture, pH, and Temperature of the Test Soils	15
Table 2.	Cumulative Mortality and Observations of Earthworms Exposed to Petroleum Coke in an Artificial Soil Substrate	16
Table 3.	Average Body Weights of Earthworms Exposed to Petroleum Coke in an Artificial Soil Substrate	17

APPENDICES

Appendix 1.	Exploratory Non-GLP Rangefinding Toxicity Test with Petroleum Coke	18
Appendix 2.	Study Protocol, Amendment and Deviation	20
Appendix 3.	Test Article Selection	37
Appendix 4.	AVEKA, Inc. Particle Processing Report.....	48
Appendix 5.	Laboratory Characterization of 3.3 Micron Particle Size Petroleum Coke	56
Appendix 6.	Artificial Soil and Test Soils Preparation.....	63
Appendix 7.	Personnel Involved in the Study.....	64
Appendix 8.	Report Amendment	66

- 7 -

SUMMARY

SPONSOR: American Petroleum Institute

TITLE: Petroleum Coke: A 14-Day Acute Toxicity Study with the Earthworm in an Artificial Soil Substrate

WILDLIFE INTERNATIONAL, LTD. PROJECT NO.: 472-101

TEST SUBSTANCE: Petroleum Coke

GUIDELINES: OECD Guideline for Testing of Chemicals, 207: *Earthworm, Acute Toxicity Tests*

TEST DATES:	Study Initiation:	April 22, 2004
	Experimental Start (OECD):	February 8, 2005
	Experimental Start (EPA):	February 9, 2005
	Biological Termination:	February 23, 2005
	Experimental Termination:	February 25, 2005

LENGTH OF EXPOSURE: 14 Days

TEST ORGANISM: Earthworm (*Eisenia fetida*)

SOURCE OF TEST ORGANISM: Wildlife International, Ltd. Cultures
Easton, Maryland 21601

AGE OF TEST ORGANISM: Adult (with clitellum)

NOMINAL TEST CONCENTRATIONS: Negative Control
1000 mg petroleum coke / kg dry soil

RESULTS: 14-Day LC50: >1000 mg petroleum coke / kg dry soil
NOEC: 1000 mg petroleum coke / kg dry soil

- 8 -

INTRODUCTION

Wildlife International, Ltd. conducted a 14-day acute toxicity study to determine the effects of petroleum coke on earthworms for the American Petroleum Institute at the Wildlife International, Ltd. toxicology facility in Easton, Maryland. 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 in-life phase of the test was conducted from February 9, 2005 to February 23, 2005. Raw data generated by Wildlife International, Ltd. and a copy of the final report are filed under Project Number 472-101 in archives located on the Wildlife International, Ltd. site.

OBJECTIVE

The objective of this study was to evaluate the acute effects of petroleum coke on earthworms during a 14-day exposure period in an artificial soil substrate.

EXPERIMENTAL DESIGN

Earthworms (*Eisenia fetida*) were exposed to a single limit dose of 1000 mg/kg of petroleum coke in soil. A control group was maintained concurrently in soil without the addition of petroleum coke. This route of administration was selected because it was representative of the natural exposure of earthworms to chemicals. In an exploratory rangefinding toxicity test the LC50 was estimated to be greater than 1000 mg/kg, therefore the definitive test was conducted with a single limit dose. The rangefinding test is summarized in Appendix 1.

Four replicate test chambers were maintained for the treatment group and for the control group, with 10 earthworms in each test chamber. Observations of mortality and clinical signs were conducted once within the first 20 minutes of test initiation, and then on Days 7 and 14. The LC50 was determined by visual examination of the mortality and clinical observation data.

At Wildlife International, Ltd., reference toxicity tests with a reference toxicant, chloroacetamide, are conducted periodically to assess the sensitivity of the test species and test procedures. These studies are conducted under separate protocols, as independent studies. A summary of the results from the most current reference toxicity test is presented in this report.

MATERIALS AND METHODS

The study was conducted according to the procedures in the protocol, “Petroleum Coke: A 14-Day Acute Toxicity Study with the Earthworm in an Artificial Soil Substrate” (Appendix 2). The protocol was based upon procedures outlined in the OECD Guideline for Testing of Chemicals, 207: *Earthworm, Acute Toxicity Tests* (4).

Test Substance

The test substance was green petroleum coke (CAS Number 64741-79-3). The test substance was received from Experimental Pathology Laboratories, Herndon, VA, for API on October 7, 2003. It was assigned Wildlife International, Ltd. identification number 6484 upon receipt and was stored under ambient conditions. The test substance was a black powder identified as 3.3 Micron Mean Petroleum Coke (aka Milled Powder).

The identity, strength, purity, composition (Appendix 5), storage stability, and method of selection, synthesis, fabrication and/or derivation (Appendices 3 and 4) of each batch of the test substance and the maintenance of these records were the responsibility of the Sponsor.

Test Organism

Earthworms (*Eisenia fetida*) for the test were from in-house cultures started with worms obtained from the University of Maryland, Queenstown, Maryland. Approximately 24 hours prior to the test, 100 adult worms (with clitellum) were selected and placed in a container of prepared artificial soil substrate adjusted to a moisture content of approximately 33% by weight, for the acclimation period. On the day of test initiation, the worms were rinsed briefly with deionized water and indiscriminately distributed by pairs into groups of 10 worms each. Each group of worms was weighed then placed on the soil surface in the appropriate test chamber. The worms were not fed during testing.

Artificial Soil and Test Soils Preparation

The artificial soil was prepared in bulk by blending 70% sand, 20% kaolin clay and 10% sphagnum peat (Appendix 6). The pH of the bulk soil prior to hydration was adjusted to 5.5 using calcium carbonate. The bulk artificial soil was stored in a sealed container under ambient conditions until used to prepare the test soils.

- 10 -

Each experimental group consisted of four replicates, each with seven hundred fifty grams of moistened soil individually dosed, mixed and contained in 1-liter glass beakers (Appendix 6). Each test soil replicate was prepared on the day of test initiation by mixing an appropriate amount of test substance with the dry artificial soil needed for each replicate. The negative control replicates were prepared in a similar fashion without the addition of test substance. The eight containers needed to prepare the test and control soils were placed on a rotary mixer for approximately one hour. The mixed soil from each container was transferred to a one-liter glass beaker labeled with study number, treatment group and replicate. Deionized water was added to the artificial soil to achieve a moisture content of approximately 33% by weight and stirred in manually with a stainless steel spoon until evenly mixed. Beakers were covered with plastic wrap to prevent the worms from leaving the test chambers. The test concentration and the LC50 value are reported as milligrams of test substance per kilogram of soil on a dry weight basis (mg/kg).

Test Chambers

The test chambers were one-liter glass beakers covered with plastic wrap that was perforated for air exchange. All test chambers were identified with the project number, test concentration and replicate.

Physical Properties of Test Soils

Soil temperature was measured in one replicate of each treatment and control group at test initiation and termination using a hand-held thermometer. Moisture content and pH measurements were made on composite soil samples collected from each replicate of soil prepared for control and treatment groups at initiation of the experiment. At test termination, samples for moisture content and pH measurements were collected from one replicate each of the control and treatment group. Measurements of pH were made using a Thermo Orion Model 525APlus pH meter. Soil moisture content was determined by measuring the initial weight of the soil sample, then weighing the soil sample after drying for at least 24 hours at approximately 105°C. The percent moisture was calculated using the following formula:

$$\% \text{ Moisture} = [(\text{wet weight} - \text{dry weight}) \div \text{wet weight}] * 100$$

Environmental Conditions

During the test, the worms were maintained in an environmental chamber set to maintain a temperature of approximately $20 \pm 2^{\circ}\text{C}$. Air temperature was measured at least once daily in the environmental chamber. The photoperiod during the test was 24 hours of continuous light per day provided by overhead fluorescent bulbs. The target light intensity during the test was approximately 400 to 800 lux, and was verified on Day 5 of the test.

Observations

At test initiation, the worms were placed on the surface of the soil in each test chamber and were observed once after approximately twenty minutes for burrowing behavior. On Days 7 and 14 of the test, the contents of each test chamber were removed to determine the number of surviving earthworms. All surviving earthworms were observed for behavioral or structural abnormalities. On Day 7, following observations, test soil was returned to the test chambers and the worms were placed on the soil surface in order to observe burrowing behavior. On Day 14, following observations and body weight determinations, surviving earthworms were euthanized by freezing and all carcasses disposed of by incineration.

Body Weights

On Day 0, group weights for all replicate earthworms were collected prior to the earthworms being placed in the test chambers. On Day 14, all surviving worms were removed from each replicate test chamber, rinsed with deionized water and blotted dry on paper towels. Group body weights were measured for each replicate, and average individual body weights were calculated.

Reference Toxicity Test

A reference toxicity test was conducted under a separate protocol to determine the LC50 value for earthworms exposed to the reference toxicant, chloroacetamide, in the soil (5). The test was conducted under conditions similar to those used in this test, and with earthworms from the same source, to monitor the techniques used and sensitivity of the test population. The earthworms were exposed to chloroacetamide in the soil at nominal concentrations of 13, 25 and 50 mg a.i./kg dry soil.

Conditions for the Validity of the Test

The following criteria used to judge the validity of the test was met:

1. mortality in the controls did not exceed 10% at the end of the test.

Data Analyses

The LC50 was determined by visually inspecting the mortality and clinical observation data. Body weights, and change in body weights, were statistically compared by Dunnett's 2-tailed t-test ($\alpha=0.05$) using SAS Version 8 (6). Prior to conducting the t-test, the data were tested for homogeneity of variance and normal distribution.

RESULTS AND DISCUSSION**Environmental Conditions**

Air temperature in the environmental chamber was within the desired range of $20 \pm 2^\circ\text{C}$, remaining at 20°C throughout the test. The earthworms were maintained under continuous lighting at an average intensity of 619 ± 60.2 lux, with a range over the surface of the test chambers of 533 to 680 lux.

Physical Properties of Test Soils

Measurements of soil pH, temperature and moisture content at test initiation and test termination are presented in Table 1. The soil pH was 6.9 in both the treatment and control soil at test initiation and was 8.1 in the treatment and control group at test termination. Soil temperature was 21°C in each test group at test initiation and 20°C at test termination, within the desired range of $20 \pm 2^\circ\text{C}$. Soil moisture content ranged from 32.5 to 32.8% at test initiation and from 31.1 to 31.3% at test termination, indicating that there was little change in soil moisture content during the test.

Observations

The data from weekly observations of the earthworms for mortality and other signs of toxicity are presented in Table 2. There were no mortalities in the control group or the treatment group during the 14-day test. Because mortality was less than 50% in the treatment group the LC50 was judged to be greater than the treatment group concentration. All earthworms in the control group and treatment group were normal in appearance and behavior throughout the test period except for

- 13 -

two worms in one control replicate. One worm was small and one was small and had a reduced reaction to mechanical stimuli on Day 14. Earthworms in both the control and treatment groups exhibited no aversion to the soil during observations of burrowing behavior on Days 0 and 7.

Body Weights

Average individual body weights at test initiation and termination, and the change in body weight from test initiation to test termination, were calculated from the Day 0 and Day 14 replicate measurements (Table 3). Treatment related effects were not observed in the test. A slight loss in body weight from test initiation to test termination was noted in both the control group and treatment group and was not unexpected since the earthworms were not fed during the test. The change in body weight (initial - final) of earthworms in the treatment group was not statistically significant ($p>0.05$) when compared to the control group. The change values were normally distributed. All data analyzed had homogeneous variances.

Reference Toxicity Test

The 14-day LC50 value for earthworms exposed to the reference substance, chloroacetamide, in an artificial soil substrate was approximately 24.5 mg a.i./kg dry soil, with a 95% confidence interval of 13 to 50 mg a.i./kg dry soil (5). These results are consistent with those observed in previous studies, and verify the adequacy and consistency of the methods used in this study.

CONCLUSIONS

The earthworm, *Eisenia fetida*, was exposed for 14 days to petroleum coke in an artificial soil substrate at a single nominal limit concentration of 1000 mg/kg dry soil. The 14-day LC50 estimation was determined to be greater than 1000 mg/kg dry soil, the single concentration tested. The no-observed effect concentration was 1000 mg/kg dry soil.

REFERENCES

- 1 **Title 40 of the Code of Federal Regulations, Part 792** . 1989. *Toxic Substances Control Act (TSCA) Good Laboratory Practice Standards*.
- 2 **OECD**. 1998. *OECD Principles of Good Laboratory Practice*. ENV/MC/CHEM (98) 17. Environmental Directorate, Paris.
- 3 **Ministry of Agriculture, Forestry and Fisheries, Japan (MAFF)**. 1999. Good Laboratory Practice (GLP) for Agricultural Chemicals. 11 NohSan, Notification No. 6283, Agricultural Production Bureau, 1 October 1999.
- 4 **OECD Guideline 207**. 1984. *Guideline for Testing of Chemicals, Earthworm, Acute Toxicity Tests*. Organization for Economic Cooperation and Development.
- 5 **Wildlife International, Ltd.** 2004. *Chloroacetamide: An Acute Toxicity Study with the Earthworm in an Artificial Soil Substrate*. Project No. 100R-110.
- 6 **SAS Institute, Inc.** 1999. SAS/STAT User's Guide, Version 8. Cary, NC, SAS Institute, Inc.

- 15 -

Table 1

Moisture, pH, and Temperature of the Test Soils

Concentration	Test Initiation Moisture Content ¹ (%)	Test Initiation pH ¹	Test Initiation Temperature ² (°C)	Test Termination Moisture Content ² (%)	Test Termination pH ²	Test Termination Temperature ² (°C)
Negative Control	32.8	6.9	21	31.3	8.1	20
1000 mg petroleum coke /kg dry soil	32.5	6.9	21	31.1	8.1	20

¹ Measurements taken from samples collected from each of the replicates and composited prior to test initiation on Day 0.

² Measurements taken from one replicate test chamber at each concentration.

- 16 -

Table 2

Cumulative Mortality and Observations of Earthworms Exposed to Petroleum Coke in an Artificial Soil Substrate

Concentration	Replicate	Day 7		Day 14		Replicate Percent Mortality	Group Percent Mortality
		Number Dead	Effects ¹	Number Dead	Effects ¹		
Negative Control	A	0/10	10 AN	0/10	10 AN	0	0
	B	0/10	10 AN	0/10	10 AN		
	C	0/10	10 AN	0/10	8 AN, 1 S, 1 R & S		
	D	0/10	10 AN	0/10	10 AN		
1000 mg petroleum coke /kg dry soil	A	0/10	10 AN	0/10	10 AN	0	0
	B	0/10	10 AN	0/10	10 AN		
	C	0/10	10 AN	0/10	10 AN		
	D	0/10	10 AN	0/10	10 AN		

¹ Observed Effects: AN = normal in appearance and behavior, S = small, R = reduced reaction to mechanical stimuli.

- 17 -

Table 3

Average Body Weights of Earthworms Exposed to
Petroleum Coke in an Artificial Soil Substrate

Concentration	Replicate	Average Earthworm Body Weights (g)		
		Day 0 ¹	Day 14 ¹	Total Change ²
Negative Control	A	0.39	0.35	-0.04
	B	0.37	0.31	-0.06
	C	0.38	0.28	-0.10
	D	0.42	0.34	-0.08
	Mean $\pm$ Std. Dev.	0.39 $\pm$ 0.022	0.32 $\pm$ 0.032	-0.07 $\pm$ 0.026
1000 mg petroleum coke /kg dry soil	A	0.40	0.30	-0.10
	B	0.39	0.31	-0.08
	C	0.37	0.27	-0.10
	D	0.46	0.36	-0.10
	Mean $\pm$ Std. Dev.	0.41 $\pm$ 0.039	0.31 $\pm$ 0.037	-0.10 $\pm$ 0.010

¹ Test group body weight was not statistically different ($p > 0.05$) when compared to the control group using Dunnett's 2-tailed t-test.

² Test group body weight change was not statistically significant ($p > 0.05$) when compared to the control group using Dunnett's 2-tailed t-test.

Appendix 1

Exploratory Non-GLP Rangefinding Toxicity Test with Petroleum Coke

Introduction

An exploratory non-GLP rangefinding test was conducted from December 8 to 22, 2004 at the Wildlife International, Ltd. invertebrate testing facility. There was a 14-day exposure period with earthworm observations on days 7 and 14.

Methods and Materials

Test soils with nominal petroleum coke concentrations of 10, 100 and 1000 mg/kg dry weight were prepared for the rangefinding test. Each experimental group consisted of two replicates with seven hundred fifty grams of moistened soil individually dosed, mixed and contained in 1-liter glass beakers. Each replicate was prepared by mixing an appropriate amount of test substance with the dry artificial soil on a rotatory mixer for approximately one hour, then adding water and stirring manually with a stainless steel spoon. Test soils were placed in 1-liter glass beakers, which were covered with plastic wrap to prevent the worms from leaving the test chambers. The negative control replicates were prepared in a similar fashion without the addition of test substance.

On the day of test initiation, adult worms (mean weights 420 to 520 mg) were rinsed briefly with deionized water and indiscriminately distributed by pairs into groups of 10 worms each. Each replicate group of worms was weighed then placed on the soil surface in the appropriate test chamber. On Days 7 and 14 of the test, the contents of each test chamber were removed to determine the number of surviving earthworms. All earthworms were observed for behavioral or structural abnormalities and for aversion to the test soil.

The test was maintained in an environmental chamber set to maintain a temperature of approximately $20 \pm 2^{\circ}\text{C}$. The photoperiod during the test was 24 hours of continuous light per day provided by overhead fluorescent bulbs. The target light intensity during the test was approximately 400 to 800 lux. The worms were not fed during testing.

Data Analysis

Statistical analyses were performed using SAS Version 8 statistical software. Dunnett's 2-tailed t-test was used to compare the change in body weight between the control group and the test substance groups. The no-observed-effect-concentration (NOEC) was estimated by visually inspecting the mortality, clinical observation data and a statistical evaluation of body weight.

Results

The results of the rangefinding test are included in the attached tables. Based on the nominal concentrations, the 14-day LC50 value for the rangefinding test was estimated to be greater than 1000 mg/kg, the highest concentration tested. The NOEC was 1000 mg/kg, the highest concentration tested. No aversion to the test soils was noted nor were any behavioral or structural abnormalities noted.

- 19 -

Appendix 1 (Continued)**Exploratory Non-GLP Rangefinding Toxicity Test with Petroleum Coke**

STUDY: Petroleum Coke: A 14-Day Acute Toxicity Study with the Earthworm in an Artificial Soil Substrate
 SPONSOR: American Petroleum Institute
 PROJECT NO.: 472-101

Mortality and Observations

Nominal Concentration ¹ (mg /kg dry soil)	Number Dead and Appearance at 7 and 14 days (Observations ²)		Cumulative Percent Mortality
	Day 7 #dead /appearance	Day 14 #dead /appearance	
Negative Control	0 / 20 AN	0 / 20 AN	0
10	0 / 20 AN	0 / 20 AN	0
100	0 / 20 AN	0 / 20 AN	0
1000	0 / 20 AN	0 / 20 AN	0

¹ Each of two replicates per concentration was mixed individually.
² Observations: AN = appear normal.

Body Weights

Nominal Concentration (mg /kg dry soil)	Mean Body Weights on Days 0 and 14, and Mean Change in Body Weight from Day 0 to Day 14 ¹		
	Day 0 Mean Weight $\pm$ Standard Deviation (grams) ²	Day 14 Mean Weight $\pm$ Standard Deviation (grams) ²	Change in Body Weight Mean $\pm$ Standard Deviation (grams) ²
Negative Control	0.47 $\pm$ 0.028	0.41 $\pm$ 0.021	-0.07 $\pm$ 0.007
10	0.44 $\pm$ 0.028	0.36 $\pm$ 0.021	-0.09 $\pm$ 0.007
100	0.51 $\pm$ 0.021	0.41 $\pm$ 0.000	-0.10 $\pm$ 0.021
1000	0.47 $\pm$ 0.014	0.40 $\pm$ 0.021	-0.08 $\pm$ 0.007

¹ Average individual body weights at test initiation and termination, and the loss in average individual body weight from test initiation to test termination, were calculated from the Day 0 and Day 14 replicate measurements (10 earthworms per replicate, two replicates per concentration).

² Treatment group means were not significantly different from the control mean (Dunnett's 2-tailed t-test, $p > 0.05$).

- 20 -

Appendix 2

Study Protocol, Amendment and Deviation

- 21 -

P R O T O C O L

PETROLEUM COKE: A 14-DAY ACUTE TOXICITY STUDY WITH THE
EARTHWORM IN AN ARTIFICIAL SOIL SUBSTRATE

OECD Guideline 207

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

- 22 -

Wildlife International, Ltd.

- 2 -

PETROLEUM COKE: A 14-DAY ACUTE TOXICITY STUDY WITH THE
EARTHWORM IN AN ARTIFICIAL SOIL SUBSTRATE

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

SPONSOR'S REPRESENTATIVE:

SPONSOR'S TECHNICAL STUDY MONITOR:

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

STUDY DIRECTOR:

Wildlife International, Ltd.

LABORATORY MANAGEMENT:

FOR LABORATORY USE ONLY

Proposed Dates:

Experimental

Start Date:

*

Experimental

Termination Date:

*

Project No.: 472-101

Test Concentrations: *

Test Substance No.: 6484

Reference Substance No. (if applicable): *

PROTOCOL APPROVAL

* to be awarded 4
As 4/22/04

4/22/04

DATE

4/23/04

DATE

01 April, 2004

DATE

PROTOCOL NO.: 472/033004/EWSDTWC/100P-501

- 23 -

Wildlife International, Ltd.

- 3 -

INTRODUCTION

Wildlife International, Ltd. will conduct an acute toxicity test to determine the effects of petroleum coke on the earthworm *Eisenia fetida* for the Sponsor at Wildlife International, Ltd. in Easton, Maryland. 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 study will be performed based on procedures in the Organization for Economic Cooperation and Development (OECD) Guideline 207, *Guideline for Testing of Chemicals, Earthworm, Acute Toxicity Tests* (1). 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

The objective of this study is to determine the acute effects of a test substance on earthworms during a 14-day exposure period in an artificial soil substrate.

EXPERIMENTAL DESIGN

Adult earthworms will be exposed to a geometric series of at least five concentrations of the test substance in artificial soil. A control group, exposed to soil prepared without addition of the test substance, will be maintained concurrently. A soil test is justified because it represents the primary exposure pathway of earthworms to chemicals.

Each treatment and control group will have four replicate test chambers, each containing ten worms. To control bias, worms will be impartially assigned to exposure chambers at test initiation. No other potential sources of bias are expected to affect the results of the study. Observations of mortality and other clinical signs will be made at 7 days and at 14 days when the test is terminated. When possible, the results will be used to determine an LC50 value.

Nominal test concentrations will be selected in consultation with the Sponsor, and will be based upon information such as the results of range-finding toxicity data, known toxicity data, physical/chemical

- 24 -

Wildlife International, Ltd.

- 4 -

properties of the test substance or other relevant information. Concentrations will be expressed as mg/kg (dry weight of soil).

At Wildlife International, Ltd., reference toxicity tests conducted to determine the LC50 for earthworms exposed to the reference toxicant, chloroacetamide, are conducted periodically to assess the sensitivity of the test species and test procedures. These studies are conducted under separate protocols, as independent studies. However, a summary of the results from the most current reference toxicity test will be included in the report for this study.

MATERIALS AND METHODS

Test Substance

The test substance is green coke (CAS Number 64741-79-3) milled to approximately 2-3 micron particle size. Information on the characterization of test, control or reference substances is required by OECD Principles of Good Laboratory Practice. The Sponsor is responsible for providing Wildlife International, Ltd. written verification that the test substance has been characterized according to GLPs. 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 the test substance and agrees to accept any unused test substance and/or test substance containers remaining at the end of the study.

Test System

The organism used in this test will be the earthworm, *Eisenia fetida*. Although not a typical soil species, *E. fetida* occurs naturally in organic-rich soil and its sensitivity to chemicals resembles that of true soil-inhabiting species. It is also very prolific and has a short life cycle which makes it a good laboratory species. Test organisms will be clitellate adults with an average body weight of approximately 300 to 600 mg. The total weight of the ten test organisms in each replicate will not vary by more than 1 gram among all replicates within a test. The organisms will be obtained from in-house cultures or from a reputable supplier. Organisms from in-house cultures will be held in a mixture of moist peat and manure, and fed saturated alfalfa and/or poultry feed. Cultures will be held at a soil temperature of approximately 20-25°C in continuous light. Prior to the experimental start of a study, organisms will be conditioned for

- 25 -

Wildlife International, Ltd.

- 5 -

approximately 24 hours in artificial soil. On the day of test initiation, the earthworms will be allocated to labeled containers in groups of two until each contains 10 worms. The worms will be weighed, then placed in test chambers at test initiation. Earthworms will not be fed during testing.

Test Chambers and Environmental Conditions

Test chambers will be approximately one liter all glass beakers. Each test chamber will be identified by project number, test concentration and replicate. The test chambers will be held at approximately $20 \pm 2^\circ\text{C}$ in continuous light at an intensity of approximately 400-800 lux. Temperature in the environmental chamber will be recorded at least once daily. Light intensity measurements over the surface of the test chambers will be recorded once during the test.

Test Substrate

The artificial soil will be composed of approximately 10% sphagnum peat, 20% kaolin clay and 70% industrial quartz sand. Calcium carbonate will be added as needed to adjust the pH to 6.0 ± 0.5 . The dry constituents of the soil will be mixed in a PK Twinshell or equivalent mixer. Moisture contents will be determined, and adjusted to approximately 33% during the preparation of the test soils.

Neither the well water nor the artificial soil are expected to have contaminants present in quantities known to be capable of interfering with the study. Analyses will be performed at least once annually to determine the concentrations of selected organic and inorganic constituents of water and soil used in this study. Results of the analyses will be stored in the archives located on the Wildlife International Ltd. site.

Preparation of Test Treatments

Concentrations of the test substance in the soil will be prepared on a dry weight basis (e.g., mg test substance/kg dry soil). The treated soils will be prepared immediately before the start of the test by adding an appropriate amount of the test substance to the artificial soil. The treated soil will be thoroughly mixed in a Hobart or equivalent mixer.

Green Coke will be added to the partially moistened soil along with a volume of water to provide the appropriate test concentrations per kilogram dry soil and a soil moisture content of approximately 33%. The mixing time necessary to achieve a homogeneous mixture will be determined in a separate method verification study.

- 26 -

Wildlife International, Ltd.

- 6 -

Seven hundred fifty grams (wet weight) of treated test medium will be placed in each of four replicate chambers. Ten preconditioned (24 h in artificial soil) and rinsed earthworms will be placed on the test medium surface. The chambers will be covered with perforated plastic film to prevent the test medium from drying.

Justification for Route of Exposure

The test substance will be administered to the test organisms in soil. The route of exposure is justified because it is the primary exposure pathway of earthworms to chemicals.

Artificial Soil Sampling

Samples of the experimental soils will be collected on Day 0 from the bulk soil prior to dividing the soil into replicates for chemical analysis to verify/measure test concentrations of the test substance in the artificial soil. All samples will be placed in uniquely identified Nalgene® jars. Samples will be analyzed for the components of petroleum coke listed in Table 1. The soil sampling scheme is summarized below:

ESTIMATED NUMBERS OF VERIFICATION SAMPLES

Experimental Group	Day 0
Control	2
Level 1-Low Concentration	2
Level 2	2
Level 3	2
Level 4	2
Level 5-High Concentration	2
Total = 12 Samples	

The above numbers of samples represent those collected from the test and do not include quality control (QC) samples such as matrix blanks and fortifications prepared and analyzed during the analytical chemistry phase of the study.

- 27 -

Wildlife International, Ltd.

- 7 -

Soil Analyses

Samples of the experimental soils will be stored in a freezer until prepared and/or extracted and analyzed, unless samples will be analyzed immediately following collection. Chemical analyses of soils will be performed by Wildlife International, Ltd. The analytical method used will be based upon chromatographic methodology and/or ICP analysis for metals. The methodology used to analyze the test samples will be documented in the raw data and summarized in the final report.

Observations and Measurements

Biological - The average weight of the live test organisms in each test chamber will be determined at the beginning and end of the test period. Burrowing behavior of the worms will be observed on Day 0 after placement of the worms on the soil surface. Any apparent aversion to the soil will be noted. On Day 7 and at the end of the test on Day 14, the soil will be removed from each test chamber and the worms will be sorted from the test substrate. A mechanical stimulus will be applied to the worms and the reactions will be recorded. Mortality and behavioral or pathological signs will be noted. Dead organisms will be removed, if possible. Earthworms recorded as missing will be considered dead and decomposed and will be treated as such in the data and calculations. On Day 7, the soil will be returned to the test chambers, and the surviving earthworms will be placed on the soil surface and observed for burrowing behavior. After observations on Day 14, the test will be terminated, and the total mass of each batch of surviving earthworms per container will be determined.

Substrate - The pH, temperature, and the moisture content of the artificial soil will be measured at each concentration at the beginning and end of the test.

Disposition of Test Earthworms

After test termination, all surviving earthworms used in the test will be euthanized by freezing or other appropriate methods. The method used will be documented in the raw data. All carcasses then will be incinerated or disposed of using other appropriate methods.

Conditions for the Validity of the Test

The following criteria will be used to judge the validity of the test:

- 28 -

Wildlife International, Ltd.

- 8 -

1. The mortality in the controls should not exceed 10% at the end of the test.

Statistical Calculations

An LC50 value along with 95% confidence limits will be calculated, when possible, using nominal concentrations. One of three methods will be used to calculate the LC50 value. The data will be analyzed, in order of preference, by probit analysis, moving average, or binomial probability (2,3,4,5). The choice of method for calculating the LC50 value will be based upon the mortality pattern observed. Body weight data may be evaluated using Dunnett's t-test to determine any significant differences between treatment group and control group data (6).

RECORDS TO BE MAINTAINED

Records to be maintained for data generated by Wildlife International, Ltd. will include, but not be limited to, the following:

1. A copy of the signed protocol.
2. Identification and characterization of the test substance, if provided by the Sponsor.
3. Dates of experimental start and experimental termination of the test.
4. History of test organism.
5. Preconditioning records.
6. Artificial soil preparation.
7. Test concentrations, calculations, preparation, and administration.
8. Test observations.
9. Average body weight of organisms.
10. Statistical calculations, if any.
11. A copy of the final report.

FINAL REPORT

A report of the results of the study will be prepared by Wildlife International, Ltd. The report will include, but not be limited to, the following:

1. Name and address of the facility performing the study.

- 29 -

Wildlife International, Ltd.

- 9 -

2. Dates on which the test was initiated and completed. It is the responsibility of the Sponsor to provide the final date that data are recorded for chemistry, pathology and/or supporting evaluations that may be generated at other laboratories.
3. A statement of compliance signed by the Study Director addressing any exceptions to Good Laboratory Practice Standards.
4. Objectives and procedures stated in the approved protocol, including any changes in the original protocol.
5. Statistical methods employed for analyzing the data.
6. The test, control and reference substances identified by name, chemical abstracts number or code number, strength, purity, and composition or other appropriate characteristics, if provided by the Sponsor.
7. Stability and, when relevant to the conduct of the study, the solubility of the test, control and reference substances under the conditions of administration, if provided by the Sponsor.
8. A description of the methods used.
9. A description of the test system used. Where applicable, the final report shall include the number of animals used, body weight range, source of supply, species, age, and procedure used for identification.
10. A description of the dosage, dosage regimen, route of administration, and duration.
11. A description of all circumstances that may have affected the quality or integrity of the data.
12. The name of the Study Director, the names of other scientists or professionals, and the names of all supervisory personnel, involved in the study.
13. A description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data, and a statement of the conclusions drawn from the analysis.
14. A copy of the output of statistical programs, if used.
15. The signed and dated reports of each of the individual scientists or other professionals involved in the study, if applicable.
16. The location where all specimens, raw data, and the final report are to be stored.
17. A statement prepared by the Quality Assurance Unit listing the dates that study inspections and audits were made and the dates of any findings reported to the Study Director and Management.
18. If it is necessary to make corrections or additions to a final report after it has been accepted, such changes shall be in the form of amendment by the Study Director. The amendment should clearly

- 30 -

Wildlife International, Ltd.

- 10 -

identify the part of the final report that is being added to or corrected and the reasons for the correction or addition. Amendments shall be signed and dated by the Study Director.

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); OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17); and Japan MAFF (11 NohSan, Notification No. 6283, Agricultural Production Bureau, 1 October 1999). 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 compliance with Good Laboratory Practices for procedures performed by other laboratories (e.g., residue analyses or pathology). 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 may be transferred to an alternative location to be specified in the final report.

- 31 -

Wildlife International, Ltd.

- 11 -

REFERENCES

- 1 **OECD Guideline 207.** 1984. *Guideline for Testing of Chemicals, Earthworm, Acute Toxicity Tests.* Organization for Economic Cooperation and Development.
- 2 **Finney, D.J.** 1971. *Statistical Methods in Biological Assay.* Second edition, Griffin Press, London.
- 3 **Thompson, W.R.** 1947. *Bacteriological Reviews.* Vol II, 2:115-145.
- 4 **Stephan, C.E.** 1977. Methods for Calculating an LC50. *Aquatic Toxicology and Hazard Evaluations.* American Society for Testing and Materials, Publication Number STP 634:65-84.
- 5 **Stephan, C.E.** 1978. U.S. EPA, Environmental Research Laboratory, Duluth, Minnesota. Personal communication.
- 6 **Gulley, D.D.** 1990. *TOXSTAT Release 3.2.* The University of Wyoming.

- 32 -

Wildlife International, Ltd.

- 12 -

Table 1.

Analytes of Interest in Petroleum Coke

PAH	Metals and Sulfur
Acenaphthene	Nickel
Acenaphthylene	Vanadium
Anthracene	Iron
Benzo(a)anthracene	Copper
Benzo(a)pyrene	Selenium
Benzo(b)fluoranthene	Arsenic
Benzo(g,h,i)perylene	Sulfur
Benzo(k)fluoranthene	
Chrysene	
Dibenzo(a,e)pyrene	
Dibenz(a,h)anthracene	
Fluoranthene	
Fluorene	
Indeno(1,2,3-cd)pyrene	
Naphthalene	
Perylene	
Phenanthrene	
Pyrene	

- 33 -

Wildlife International, Ltd.

Project Number 472-101
Page 1 of 3

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE:	Petroleum Coke: A 14-Day Acute Toxicity Study with the Earthworm in an Artificial Soil Substrate	
PROTOCOL NO.:	472/033004/EWSDTWC/100P-501	AMENDMENT NO.: 1
SPONSOR:	American Petroleum Institute	PROJECT NO.: 472-101
EFFECTIVE DATE:	February 3, 2005	

AMENDMENT:

The following information is added to complete page 2 of the Protocol:

Proposed Dates:

OECD Experimental Start Date: February 8, 2005

EPA Experimental Start Date: February 9, 2005

Experimental Termination Date: February 23, 2005

Test Concentrations: 0, 1000 mg/kg dry soil

Reference Substance No.: not applicable

REASON:

This information was not known at the time the Protocol was signed.

AMENDMENT:

Page 3, Experimental Design, replace first sentence.

Old sentence:

Adult earthworms will be exposed to a geometric series of at least five concentrations of the test substance in artificial soil.

New Sentence:

Adult earthworms will be exposed to a single limit dose of 1000 mg/kg dry soil of the test substance in artificial soil.



- 34 -

Wildlife International, Ltd.

Project Number 472-101
Page 2 of 3

REASON:

Based on results of a range-finding test it was decided, in consultation with the Sponsor, that a limit test would be adequate.

AMENDMENT:

Page 4, Test System section, replace sentence.

Old sentence:

Organisms from in-house cultures will be held in a mixture of moist peat and manure, and fed saturated alfalfa and/or poultry feed.

New Sentence:

Organisms from in-house cultures will be held in moist peat and fed saturated alfalfa and/or cow manure.

REASON:

The sentence was amended to accurately describe current culturing practices.

AMENDMENT:

Replace the original Preparation of Test Treatments section (page 5-6) with the following. The new text describes the procedure for mixing each replicate individually rather than mixing a batch for each level in a Hobart mixer.

Preparation of Test Treatments

The concentration of the test substance in the soil will be prepared on a dry weight basis (e.g. mg test substance/kg dry soil). Seven hundred fifty grams (wet weight) of treated test soil will be contained in each of four replicate chambers. The treated soil will be prepared on the day of test initiation by adding an appropriate amount of Green Coke to four containers with the dry artificial soil needed for each replicate. The dry soil for four negative control replicates will be weighed into each of four containers. The eight containers needed to prepare the test and

- 35 -

Wildlife International, Ltd.

Project Number 472-101
Page 3 of 3

control soils will be placed on a rotary mixer for minimum of one hour. The mixed soil from each container will be transferred to a one-liter glass beaker labeled with project number, treatment group and replicate. An appropriate amount of water to provide soil moisture content of approximately 33% will be added and stirred in manually using a stainless steel spoon until evenly mixed.

Ten preconditioned (24 h in artificial soil) and rinsed earthworms will be placed on the test medium surface. The chambers will be covered with perforated plastic film to prevent the test medium from drying.

REASON:

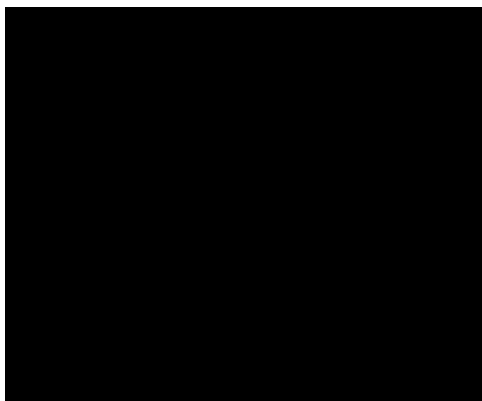
The Sponsor requested that the test soil for each replicate be mixed individually to verify that the proper amount of test substance is added to each replicate since analytical testing will not be done.

AMENDMENT:

Delete Artificial Soil Sampling and Soil Analyses sections (page 6-7) and Table 1 (page 12).

REASON:

These sections are not needed in the Protocol because the test soil will not be analyzed. Since samples of the soil will not be collected for chemical analysis this will be accordingly noted in the Good Laboratory Practice Compliance Statement of the final report.



2/4/05
DATE

2/7/05
DATE

2/3/2005
DATE

- 36 -

Wildlife International, Ltd.

Page 1 of 1

DEVIATION FROM STUDY PROTOCOL

STUDY TITLE: Petroleum Coke: A 14-Day Acute Toxicity Study with the Earthworm in an Artificial Soil Substrate

PROTOCOL NO.: 472/033004/EWSDTWC/100P-501

DEVIATION NO.: 1

SPONSOR: American Petroleum Institute

WIL PROJECT NO.: 472-101

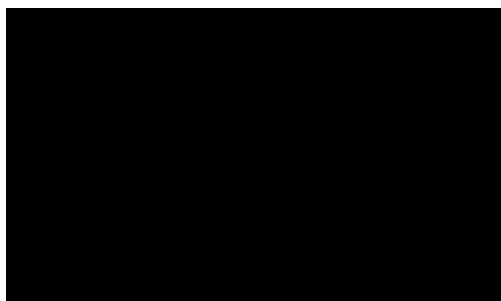
DATE OF DEVIATION: February 8, 2005

DEVIATION:

Analyses to determine the concentrations of selected organic and inorganic constituents of the soil used in this study were not conducted.

REASON:

Some components of the soil, for example the peat moss, were screened for contaminants as components of other media used in other Wildlife International, Ltd. studies, but the earthworm artificial soil mixture is not routinely analyzed. Specifications for acceptable levels of contamination in artificial soil for acute toxicity studies with the earthworm have not been established. Therefore, there was no adverse impact on the study due to this deviation.



4/21/05
DATE

4/21/05
DATE

- 37 -

Appendix 3

Test Article Selection

- 38 -



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

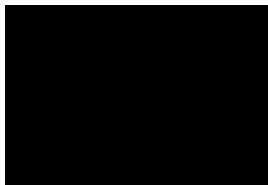


American Petroleum Institute
1220 L Street, NW
Washington, D.C. 20005-4070

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



Attachment

- 39 -

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 Flexicokers¹ 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.

- 40 -

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

- 41 -

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:

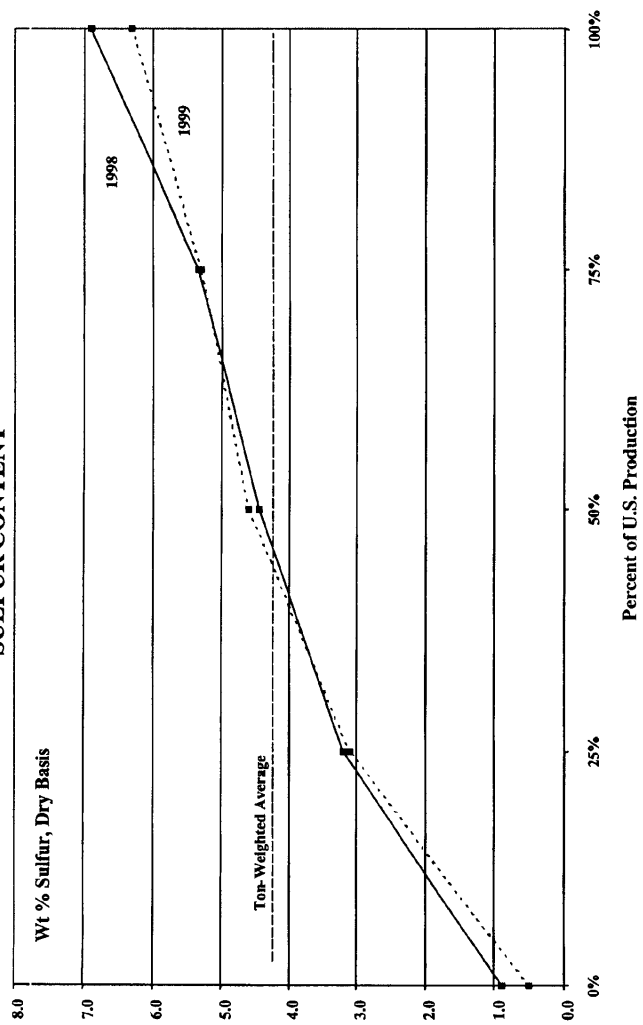
Annual data were sorted according to each specific quality value (e.g., sulfur, vanadium, nickel, and volatile content) and the cumulative production of petroleum coke by delayed coker was calculated. Quartiles were then calculated for the annual production total, and the quality value at the cumulative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

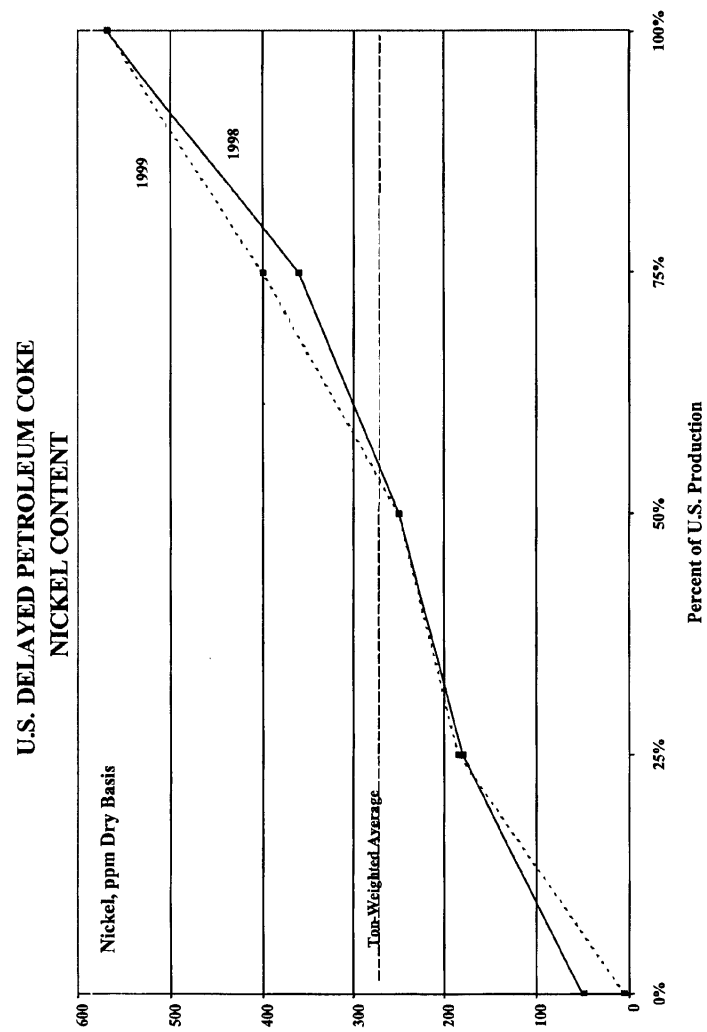
- The weighted average nickel and vanadium content of U.S. delayed petroleum coke is higher than the median. This is a direct result of the increasing amount of heavy crudes, particularly Mexican and Venezuelan crudes, processed by U.S. refineries. Because these crudes produce petroleum cokes with nickel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



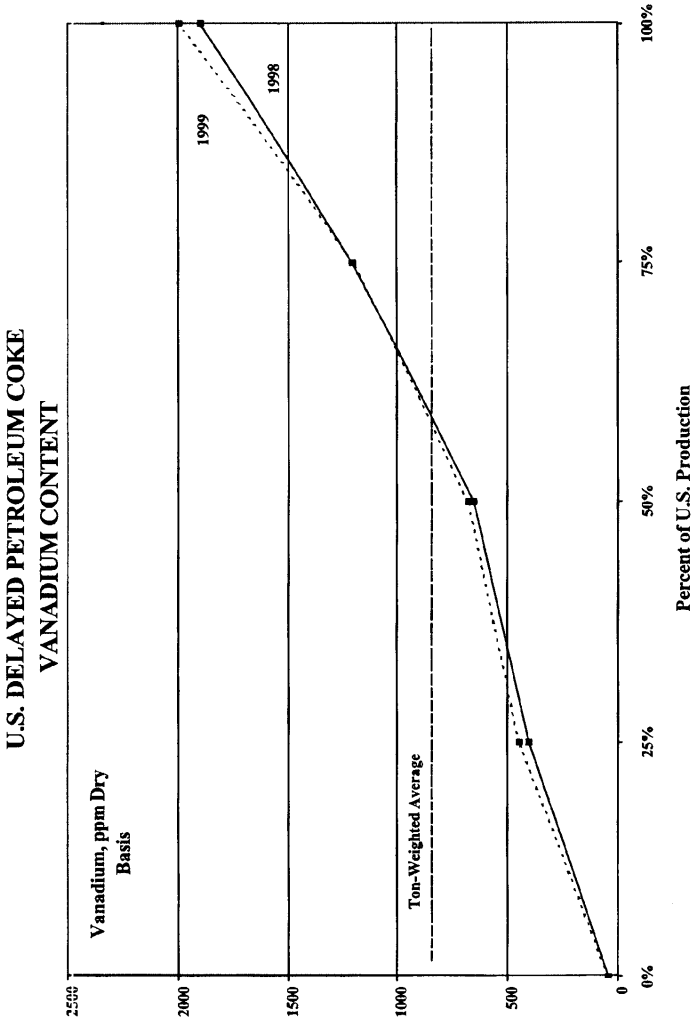
THE PACE CONSULTANTS INC.

FIGURE 2



THE PACE CONSULTANTS INC.

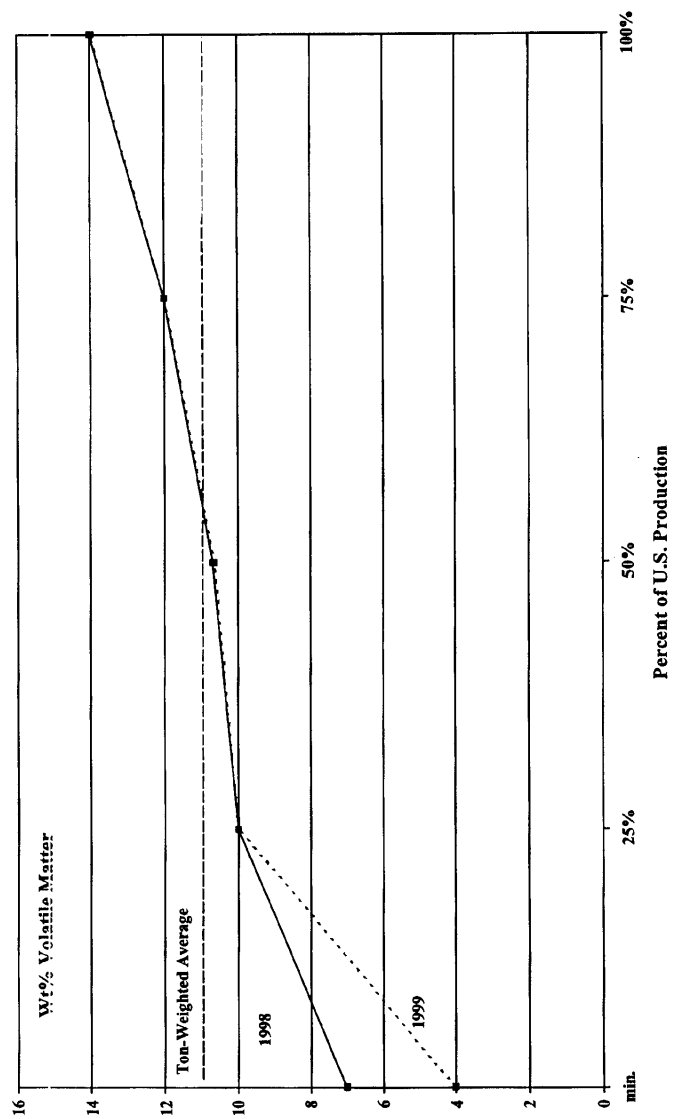
FIGURE 3



THE PACE CONSULTANTS INC.

- 45 -

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



THE PACE CONSULTANTS INC.

AMENDED

- 46 -

- 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

- 47 -

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.

- 48 -

Appendix 4

AVEKA, Inc. Particle Processing Report

- 49 -

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.

651-730-1729

2045 Wooddale Drive, Woodbury, MN 55125

FAX 651-730-1826

AMENDED

- 50 -

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 (Atch 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 (Atch. 2) when tested with the Horiba LA-910 in water. The Horiba LA-910 test method for the petroleum coke samples is outlined in Atch. 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 (Atch. 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.

<u>Sample/Amount</u>	<u>Address</u>	<u>Person</u>
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

- 51 -

AVEKA, Inc.**PARTICLE PROCESSING & CUSTOM RESEARCH**

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

- 52 -

Attach 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.rj□

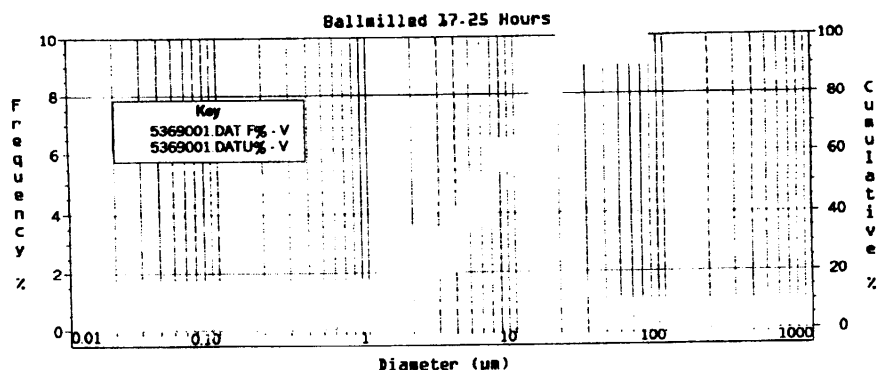
Dist. Mode: VOLUME Laser: 65.128 % Lamp: 61.185 %

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)

- 53 -

Attch. 2

HORIBA LA-910**PARTICLE SIZE DISTRIBUTION DATA TABLE** Standard 05/15/03

File Name: 5369011.DAT Sample Name: Fines ID No: **/04/30-568

Dist. Form: STANDARD R.R. Index: co.rj□

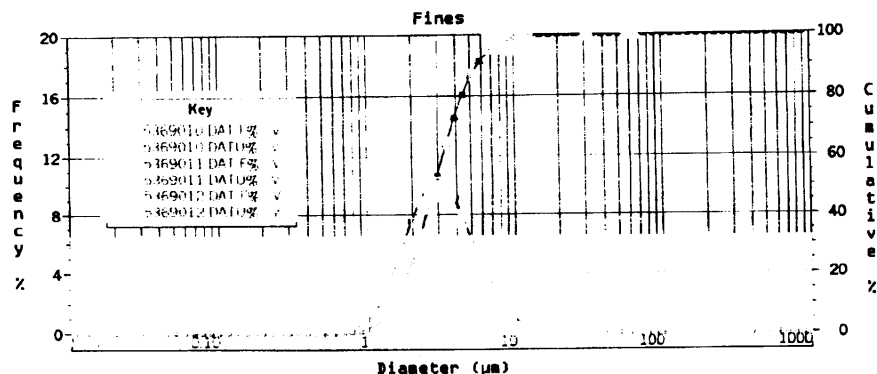
Dist. Mode: VOLUME Laser: 85.118 % Lamp: 86.338 %

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
Avcka, Inc.
(651) 714-4293 ext 208

- 55 -

Attch. 4

5/29/03

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	% Sample Below Sieve
7	2800	50.951	50.975	0.024	0.31	99.69
8	2360	50.741	52.146	1.405	18.18	81.81
10	2000	48.772	51.024	2.252	29.14	70.86
12	1700	47.324	50.173	2.849	36.86	63.14
14	1400	48.450	49.624	1.174	15.19	84.81
catch	0	220.018	220.043	0.025	0.32	99.68
Totals:				7.729	100.00	

Notes/Comments:

MO5388a.xls

AMENDED

- 56 -

Appendix 5

Laboratory Characterization of 3.3 Micron Particle Size Petroleum Coke

- 57 -



ANALYTICAL RESULTS

Prepared for:

Chevron Products Company
940 Hensley 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 [REDACTED]

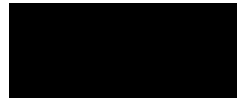
Client Description

Pet Coke 2mm Solid Sample
Pet Coke Micronized Solid Sample

Lancaster Labs Number

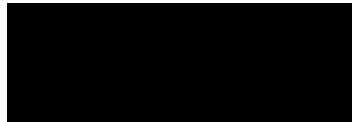
4073301
4073302

1 COPY TO Lancaster Laboratories
1 COPY TO Chevron CRTG



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

Respectfully Submitted,



MEMBER
AMENDED

Lancaster Laboratories, Inc.
2425 New Holland Pike
PO Box 12425
Lancaster, PA 17605-2425

1
A4200
3.66

AMENDED

- 58 -



Analysis Report

Page 1 of 2

Lancaster Laboratories Sample No. SW 4073302

Collected: 06/26/2003 00:00

Account Number: 10863

Submitted: 06/27/2003 10:40

Chevron Products Company

Reported: 07/09/2003 at 11:42

940 Hensley St. Bldg. 210

Discard: 08/09/2003

Pet Coke Micronized Solid Sample

Richmond CA 94801

Cost Center# ENG-4066

HPV Petroleum Cake

MICPC

CAS 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
03778	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 Scheuring	10

MEMBER

Lancaster Laboratories, Inc.
2425 New Holland Pike
PO Box 12425
Richmond CA 94801

AMENDED

- 59 -



Page 2 of 2

Lancaster Laboratories Sample No. SW 4073302

Collected: 06/26/2003 00:00

Account Number: 10863

Submitted: 06/27/2003 10:40

Chevron Products Company

Reported: 07/09/2003 at 11:42

940 Hensley St. Bldg. 210

Discard: 08/09/2003

Pet Coke Micronized Solid Sample

Richmond CA 94801

Cost Center# ENG-4066

HPV Petroleum Cake

MICPC

07806 BRA Soil Extraction

SW-846 35508

1 06/30/2003 20:00 Sally L Appleyard

1

MEMBER
EVENT

Lancaster Laboratories, Inc.
2425 New Holland Pike
PO Box 12425
Lancaster PA 17604-7425

AMENDED

- 60 -



Page 1 of 2

Quality Control Summary

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

Group Number: 857532

Laboratory Compliance Quality Control

<u>Analysis Name</u>	<u>Blank Result</u>	<u>Blank MDL</u>	<u>Report Units</u>	<u>LCS</u> <u>UREC</u>	<u>LCSO</u> <u>UREC</u>	<u>LCS/LCSO</u> <u>Limits</u>	<u>RPD</u>	<u>RPD Max</u>
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

<u>Analysis Name</u>	<u>MS</u> <u>UREC</u>	<u>MSD</u> <u>UREC</u>	<u>MS/MSD</u> <u>Limits</u>	<u>RPD</u>	<u>MXG</u> <u>Conc</u>	<u>DUF</u> <u>Conc</u>	<u>DUF</u> <u>RPD</u>	<u>Dup</u> <u>RPD</u> <u>Max</u>
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	80	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	23-134	13	30			
Indeno(1,2,3-cd)pyrene	92	78	13-135	15	30			
Dibenz(a,h)anthracene	110	88	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.



Lancaster Laboratories, Inc.
2425 New Holland Pike
PO Box 12425
Lawrenceville, GA 30046-2425

AMENDED

- 61 -

Analysis Report



Page 2 of 2

Quality Control Summary

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

Group Number: 857532

Sample Matrix Quality Control

	MS	MSD	MS/MSD	RPD	BKG	DUP	DUP	Dup
<u>Analysis Name</u>	<u>4REC</u>	<u>4REC</u>	<u>Limits</u>	<u>RPD</u>	<u>MAX</u>	<u>Conc</u>	<u>Conc</u>	<u>RPD</u>
								<u>Max</u>

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	87	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.

MEMBER

Lancaster Laboratories, Inc.
2435 New Holland Pike
PO Box 12425
Lancaster, PA 17602-0425

AMENDED

- 62 -

From:
Sent:
To:
Cc:
Subject:

Found your results.

3030999 PETROLEUM COKE 2NM YCJ58009 REGULAR SERVICE *micronized*
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

3030251 PETROLEUM COKE YCJ58009 REGULAR SERVICE *2mm*
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 [REDACTED] 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:

Appendix 6**Artificial Soil and Test Soils Preparation****Artificial Soil Preparation**

The artificial soil was prepared in bulk by mixing the following constituents in a soil mixer for approximately 20 minutes:

quartz sand	31.500 kg
kaolin clay	9.000 kg
sphagnum peat	4.500 kg
calcium carbonate	0.500 kg

Test Soil Preparation

A total of 750 g (wet weight) of test soil with calculated soil moisture content of 33% was prepared for each control group and treatment group replicate. Test substance concentrations were based on the dry weight portion of the soil. The calculated dry weight of 750 kg of prepared soil, with a moisture content of 33%, was 523.4 grams taking into account the estimated 4% moisture content present in the bulk soil stored in ambient conditions. Deionized water, 226.6 mL, was added to each replicate. The nominal contents of bulk soil, water and test substance were as follows:

Nominal Weights and Volumes Used in Preparation of Test Soils			
Concentration	Amount of Bulk Soil (g)	Amount of deionized water (mL)	Amount of Test Substance (g)
Negative Control	523.4	226.6	---
1000 mg petroleum coke /kg dry soil	522.9	226.6	0.5025

Bulk soil was weighed into tared two-liter Nalgene[®] jars. The test substance was weighed onto tared weigh papers and transferred to the Nalgene[®] jars. The weigh papers were rinsed with bulk soil and all rinsates returned to the jar. Negative control jars received no test substance. All jars were capped and placed on a rotary mixer for approximately one hour. The contents of the jars were transferred to one-liter glass beakers labeled with study number, treatment group and replicate. A graduated cylinder and a two-mL glass pipette were used to measure the deionized water. A portion of the water was used to rinse the Nalgene[®] jars, and added to each replicate along with the remainder of the water. A stainless steel spoon was used to manually stir the soil for each replicate until evenly mixed. Test chambers were covered with plastic wrap and held in an incubator under the environmental conditions of the test until test initiation.

- 64 -

Appendix 7

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]

- 65 -

Appendix 8

Report Amendment

- | | | |
|----|--|--|
| 1. | Original Report:
Amended Report:

Reason: | Title Page
The amended report date was added. The total number of pages was changed from 53 to 66.
To indicate that the report was amended and note change in pagination. |
| 2. | Original Report:
Amended Report:

Reason: | Page 2
The amended report date was added and new signatures and dates were added.
To show the amended report date and to provide new signatures and dates for the amended report. |
| 3. | Original Report:
Amended Report:

Reason: | Page 3
The audit dates for the amended report were added and a new signature and date were added.
To show the amended report audit dates and to provide a new signature and date for the amended report. |
| 4. | Original Report:
Amended Report:
Reason: | Page 4
New signatures and dates were added.
To provide new signatures and dates for the amended report. |
| 5. | Original Report:
Amended Report:

Reason: | Page 6
The Table of Contents was updated to show the addition of Appendix 3, to renumber all appendices from Appendix 3 through the end of the report and to add the Report Amendment appendix (Appendix 8).
The Sponsor requested that Appendix 3 be added to the final report. |
| 6. | Original Report:
Amended Report:

Reason: | Page 9
Appendix 3 was referenced in the Test Substance Section of the report and the appendix references were renumbered due to addition of Appendix 3.
The Sponsor requested that Appendix 3 be added to the final report. |

- 66 -

Appendix 8
(continued)

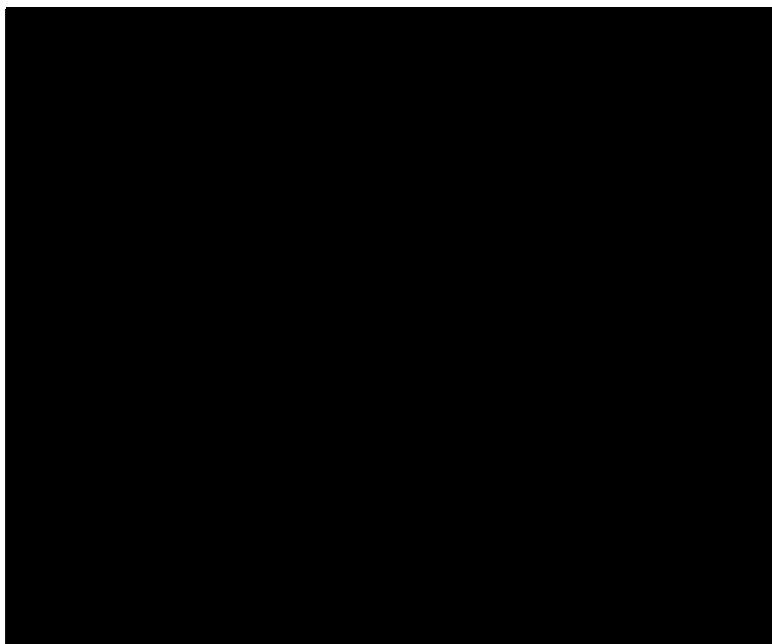
Report Amendment

- | | | |
|----|--|---|
| 7. | Original Report:
Amended Report:

Reason: | Page 10
The appendix reference was renumbered due to addition of Appendix 3.
The Sponsor requested that Appendix 3 be added to the final report. |
| 8. | Original Report:
Amended Report:
Reason: | Page 37
Appendix 3 was added to the report.
The Sponsor requested that Appendix 3 be added to the final report. |
| 9. | Original Report:
Amended Report:

Reason: | Pages 37-53
Appendix 3 was added to the report, therefore all pages thereafter were renumbered.
The Sponsor requested that Appendix 3 be added to the final report. |

AMENDMENT SIGNATURES:



4/10/07
Date

10 April 07
Date

4-10-07
Date

AMENDED