FINAL REPORT

Study Title:	Analytical Validation and Stability Study of Distillates (Petroleum), Light Catalytic Cracked in Mineral Oil Formulations
Study Number:	WIL-402027
Study Director:	Eric S. Bodle, PhD
Data Requirements:	Not Applicable
Study Initiation Date:	18 March 2011
Study Completion Date:	17 October 2011
Performing Analytical Laboratory:	WIL Research Laboratories, LLC 1407 George Road Ashland, OH 44805-8946
Sponsor:	American Petroleum Institute 1220 L Street, NW Washington, DC 20005

COMPLIANCE STATEMENT

This study, designated WIL-402027, was conducted in compliance with the United States EPA GLP Standards (40 CFR Part 792), 18 September 1989; the OECD Principles of GLP [C(97) 186/Final], 26 November 1997; the WIL Research SOPs; and the protocol and protocol amendments as approved by the Sponsor. A Report of Analysis was provided by the Sponsor (presented in Appendix B); the characterization analyses were conducted according to unknown standards. A sufficient amount of analytical sample stability was inadvertently not established in this study. The lack of sufficient analytical stability had no impact on the quality or integrity of the study as all samples analyzed during the analysis in question met all protocol acceptance criteria.

Eric S. Bodle, PhD
Assistant Director, Analytical Chemistry
Study Director

17 Oct 2011
Date

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1. SUMMARY

A high performance liquid chromatography method using ultraviolet absorbance detection at a wavelength of 254 nm for the determination of distillates (petroleum), light catalytic cracked (CAS# 64741-59-9) concentration in mineral oil formulations containing test substance ranging in concentration from 50.0 to 500 mg/mL was validated in this study. In addition, test substance homogeneity and, following 8 and 15 days of room temperature storage, resuspension homogeneity and stability were assessed in formulations prepared at target concentrations of 50 and 500 mg CAS# 64741-59-9/mL.

The CAS# 64741-59-9 assay procedure was validated in this study with 3 validation sessions. Quantitation was performed using calibration standards ranging in test substance concentration from 25.0 to 200 µg/mL. The mean back-calculated standard concentrations had inter-session variability ranging from 0.77% to 5.8% relative standard deviation (RSD) and percent relative error (%RE) ranging from -4.8% to 1.8%, which met the protocol-specified acceptance criteria for calibration standards, *i.e.*, RSD ≤10% and %RE within ± 10% (except at the lowest level where RSD ≤15% and %RE within ± 15% were acceptable). Assay precision and accuracy were verified by the analysis of quality control (QC) samples prepared at 50.0, 250, and 500 mg CAS# 64741-59-9/mL. The mean calculated QC concentrations had inter-session variability (precision) ranging from 3.9% to 4.7% RSD and %RE (accuracy) ranging from 1.5% to 3.9%. The results met the WIL Research SOP acceptance criteria for precision and accuracy, *i.e.*, RSD ≤15% and %RE within ± 15%.

The results of the test substance homogeneity assessment in formulations prepared at target concentrations of 50 and 500 mg CAS# 64741-59-9/mL met the protocol-specified acceptance criteria, *i.e.*, the RSD for the mean concentration was ≤10% at a concentration within the acceptable limits (85% to 115% of target). Assessment of test substance resuspension homogeneity and stability in formulations prepared at target concentrations of 50 and 500 mg CAS# 64741-59-9/mL and following up to 15 days of room

temperature storage met the protocol-specified acceptance criteria for resuspension homogeneity, *i.e.*, the RSD for the mean concentration was ≤10%, and stability, *i.e.*, the post-storage concentration was not <90% of the pre-storage value.

2. INTRODUCTION

This report provides a detailed description and validation of a high performance liquid chromatography (HPLC) method using ultraviolet (UV) absorbance detection at a wavelength of 254 nm for the determination of distillates (petroleum), light catalytic cracked (CAS# 64741-59-9) concentration in mineral oil formulations containing test substance ranging in concentration from 50.0 to 500 mg/mL. Assay specificity/selectivity, calibration reproducibility, precision, accuracy, and ruggedness were assessed. In addition, formulations prepared at target concentrations of 50 and 500 mg CAS# 64741-59-9/mL were analyzed to assess test substance homogeneity and, following up to 15 days of room temperature storage, resuspension homogeneity and stability.

The study protocol and deviations from the protocol are presented in Appendix A.

A list of abbreviations potentially used in this report is presented in Section 9. (Abbreviations).

2.1. KEY STUDY DATES

Date(s)	Event(s)
16 May 2011	Experimental start/starting date (first date of analysis)
8 June 2011	Experimental termination/completion date (last date of analysis)

2.2. WIL RESEARCH KEY STUDY PERSONNEL

Jonathan G. Bernauer, BA	Chemist II, Analytical Chemistry
Eric S. Bodle, PhD	Assistant Director, Analytical Chemistry
Dennis J. Hoobler, BS	Chemist III, Analytical Chemistry
Nicole L. Unsworth	Chemist II, Analytical Chemistry
Gregory A. Hawks, AS	Data Coordinator, Reporting & Technical Support Services
Melissa A. Hull, BS	Group Manager, Reporting & Technical Support Services

3. EXPERIMENTAL PROCEDURES - MATERIALS AND METHODS

3.1. TEST SUBSTANCE AND VEHICLE

3.1.1. TEST SUBSTANCE IDENTIFICATION

The test substance, distillates (petroleum), light catalytic cracked (CAS# 64741-59-9), was received from EPL Archives, Sterling, VA on 10 November 2010 as follows:

Identification	Physical Description
Gas oil; distillates (petroleum), LCC (CAS# 64741-59-9, Site #26, Sample #18) [WIL log no. ARS-8471A]	Brown, viscous liquid

A Report of Analysis for the test substance was provided by the Sponsor and is presented in Appendix B. The test substance was stored at room temperature, protected from light and was considered stable under this condition. A reserve sample of the test substance was collected and stored in the WIL Research Archives.

3.1.2. VEHICLE IDENTIFICATION

The vehicle used in preparation of the test substance formulations was mineral oil, prepared using:

- Mineral oil (Batch no. ZH1000, exp. date: 3 March 2012, received from Spectrum Chemicals Laboratory, New Brunswick, NJ)

3.2. FORMULATION PREPARATION

Formulations were prepared at the test substance concentrations indicated in the following table:

Group No.	Dose Concentration (mg/mL)
Low	50
High	500

The appropriate amount of the test substance for each formulation was weighed in a tared weighing vessel and quantitatively transferred to a tared glass container containing a stir bar. Approximately 80% of the vehicle was added to each container and mixed with a magnetic stirrer until uniform (approximately 15 minutes). The formulations were then brought to the calibration target with vehicle. The formulations were mixed until uniform using a magnetic stirrer. The test substance formulations were stirred continuously throughout the preparation and sampling procedures.

3.3. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Instrument: Agilent 1100 liquid chromatograph equipped with a variable wavelength detector, autosampler, and Dionex Chromeleon® software, or equivalent system

Column: Phenomenex Jupiter® C4, 150 × 4.6 mm, 5-µm particle-size, 30 nm

Mobile Phase: A: Deionized (DI) water
B: Methanol (MeOH)

Gradient:

Time (minutes)	A (%)	B (%)
0.0	50.0	50.0
2.0	50.0	50.0
7.0	10.0	90.0
9.0	10.0	90.0
9.1	50.0	50.0
11.5 or 11.8	50.0	50.0

Flow Rate :	1.50 mL/minute
Column Temperature:	30°C
Detector:	UV absorbance at 254 nm
Injection Volume:	10 µL
Retention Time:	Approximately 5.5 to 9.0 minutes for CAS# 64741-59-9 peak group
Run Time:	11.5 minutes or 11.8 minutes

3.4. PREPARATION OF CALIBRATION STOCK SOLUTION

The calibration standard stock solution was prepared at a concentration of 0.500 mg CAS# 64741-59-9/mL by transferring 28 µL of CAS# 64741-59-9 (WIL log no. ARS-8471A, density of 0.92851 mg/mL) to a 50-mL volumetric flask. Approximately 40 mL of ethyl acetate (EtOAc) was added, and the preparation was mixed as needed to achieve dissolution of the test substance. Additional EtOAc was added as needed to obtain the desired concentration, and the solution was stirred to mix.

3.5. PREPARATION OF CALIBRATION STANDARDS

Aliquots of the calibration stock solution were combined in amber autosampler vials with appropriate volumes of EtOAc to obtain calibration standards ranging in concentration from 25.0 to 200 µg CAS# 64741-59-9/mL. Triplicate calibration standards were prepared at each concentration for the validation sessions; at least single calibration standards were prepared at each concentration thereafter.

3.6. PREPARATION OF QUALITY CONTROL STOCK SOLUTIONS

The quality control (QC) stock solution was prepared at a concentration of 100 mg CAS# 64741-59-9/mL by transferring 2.70 mL of CAS# 64741-59-9 (WIL log no. 8471A, density of 0.92851 mg/mL) into a 25-mL volumetric flask. Approximately 20 mL of EtOAc was added and the preparation was mixed as needed to achieve dissolution of the test substance. Additional EtOAc was added as needed to obtain the desired concentration, and the solution was stirred to mix.

3.7. PREPARATION OF QUALITY CONTROL SAMPLES

As detailed in the following table, QC samples were prepared to simulate the processing of formulations at concentrations of 50.0, 250, and 500 mg CAS# 64741-59-9/mL (nominal QC concentrations) by combining aliquots of the QC stock solution, vehicle (mineral oil), and EtOAc in polypropylene tubes. The processed samples were mixed with vortex action. Portions of the processed samples were further diluted with EtOAc in amber autosampler vials or polypropylene tubes. QC samples were prepared in triplicate at each concentration; a single blank was prepared.

QC Level	QC Concentration (mg/mL)	Vehicle Volume (mL)	QC Stock Concentration (mg/mL)	QC Stock Volume (mL)	EtOAc Volume (mL)	Secondary Dilution	Final Concentration (µg/mL)
BQC	0	1.00	NA	NA	39.00	10-fold	0
QC1	50.0	1.00	100	0.500	38.50	10-fold	125
QC2	250	1.00	100	2.50	36.50	50-fold	125
QC3	500	1.00	100	5.00	34.00	100-fold	125

NA = Not applicable

3.8. FORMULATION SAMPLE PROCESSING

Quadruplicate formulation samples were collected using a syringe and dosing cannula and placed in polypropylene tubes. Two samples from each quadruplicate set were processed for analysis, and the remaining 2 samples (back-up samples) were stored at room temperature and, if not processed for analysis, were discarded after the Study Director's acceptance of results. As detailed in the following table, formulation samples were processed by adding EtOAc and mixing with vortex action. Portions of the processed samples were further diluted with EtOAc in amber autosampler vials or polypropylene tubes. The vials were capped, and the diluted samples mixed with vortex action.

Group	Theoretical Test Substance Concentration (mg/mL)	Sample Volume (mL)	EtOAc Volume (mL)	Secondary Dilution	Theoretical Final Concentration (µg/mL)
Low	50	1.0	39.00	10-fold	125
High	500	1.0	39.00	100-fold	125

3.9. CONCENTRATION QUANTITATION

Single injections were made of each calibration standard and processed QC and formulation sample. A calibration curve was constructed for each set of analyses. The CAS# 64741-59-9 peak group areas (y) and the theoretical concentrations (x) of the calibration standards were fit with least-squares regression analysis to the linear function:

$$y = ax + b$$

Concentrations were calculated from the results of the regression analysis using Dionex Chromeleon® software. The concentration data were transferred to a Microsoft Excel® spreadsheet, where appropriate summary statistics, *i.e.*, mean, standard deviation (SD), relative standard deviation (RSD), percent relative error (%RE), and concentration as a percent of target concentration, were calculated and presented in tabular form. The concentrations of the formulation and QC samples were calculated by applying any necessary factors to correct for dilution and/or unit conversion.

3.10. WIL RESEARCH COMPUTER SYSTEMS

3.10.1. REPORTING AND ANCILLARY SYSTEMS

Program/System	Description
Archive Management System (AMS)	In-house developed application for storage, maintenance, and retrieval of information for archived materials (*e.g.*, lab books, study data, wet tissues, slides, *etc.*)
InSight® Publisher	Electronic publishing system (output is Adobe Acrobat, PDF)
Master Schedule	Maintains the master schedule for the company.
Microsoft® Office 2002 and 2007; GraphPad Prism® 2008	Used in conjunction with the publishing software to generate study reports.

4. RESULTS AND DISCUSSION

Under the described chromatographic conditions, the retention time of the test substance peak group was approximately 5.5 to 9.0 minutes. Figure 1, Figure 2, Figure 3, and Figure 4 are typical chromatograms of a calibration standard, a processed QC sample, a processed formulation sample, and a processed vehicle blank sample, respectively. The total analysis time required for each run was 11.5 minutes or 11.8 minutes.

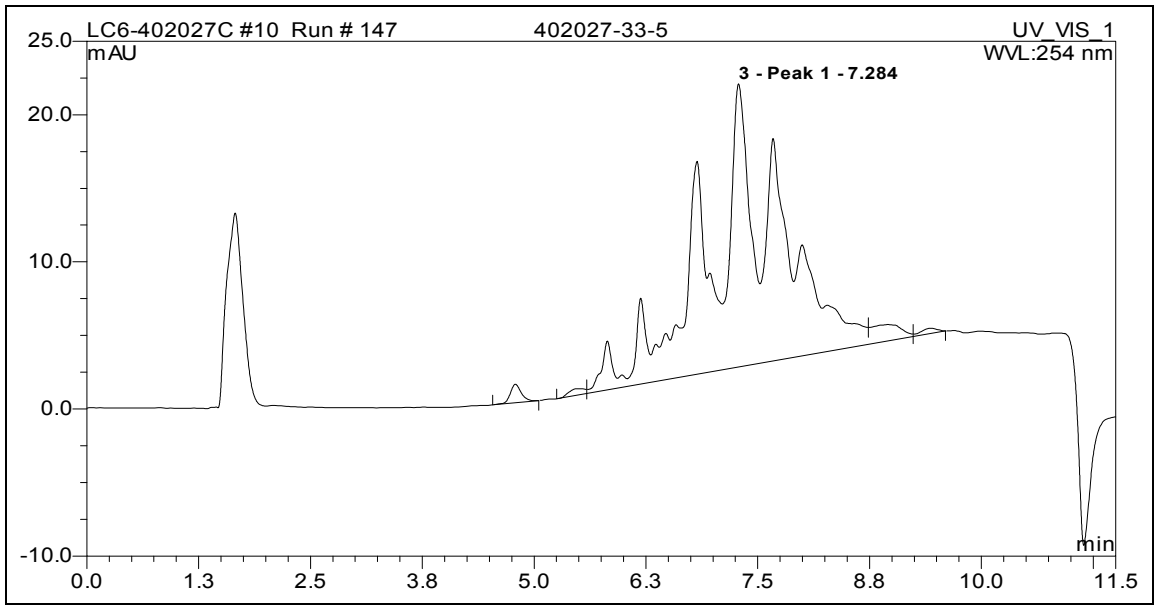


Figure 1: Representative Chromatogram of a 75.0 µg CAS# 64741-59-9/mL Calibration Standard

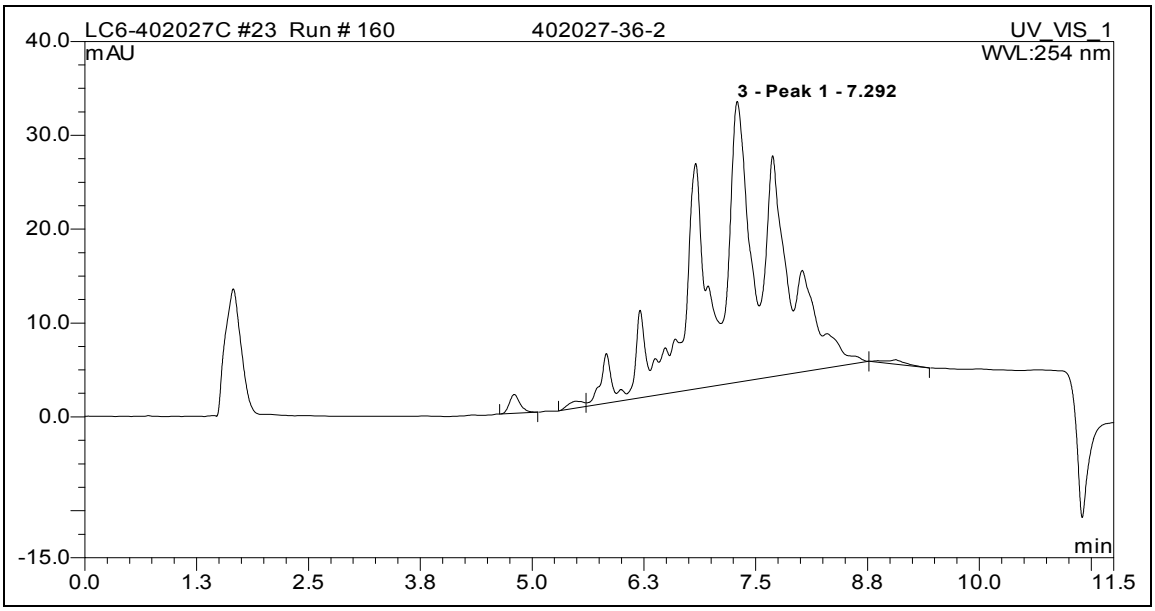


Figure 2: Representative Chromatogram of a Processed 50.0 mg CAS# 64741-59-9/mL Quality Control Sample

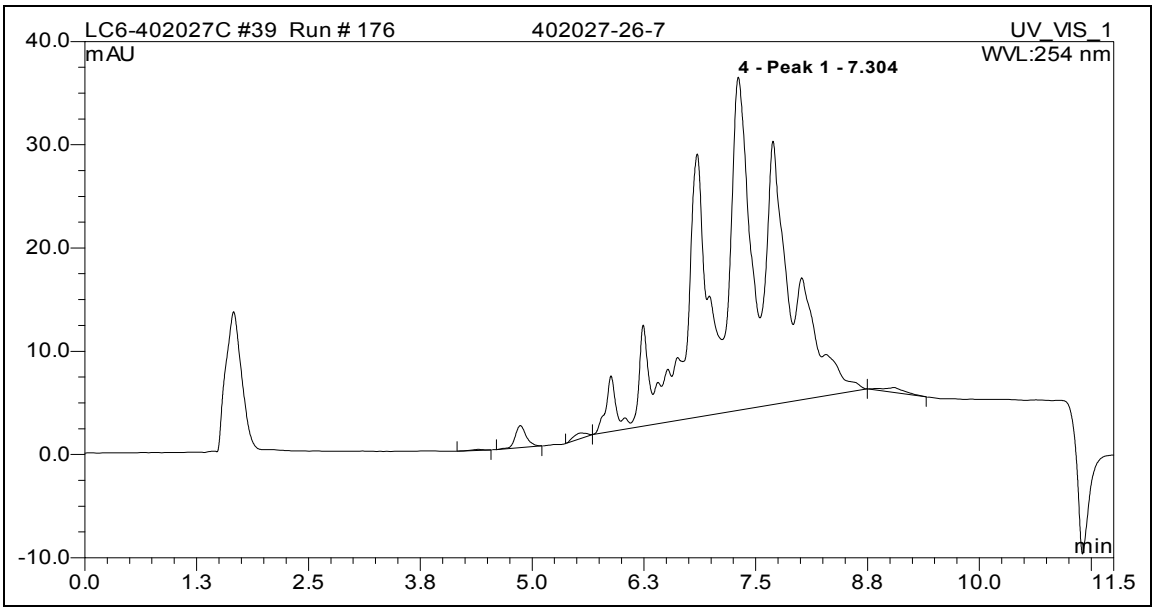


Figure 3: Representative Chromatogram of a Processed 500 mg CAS# 64741-59-9/mL Formulation Sample

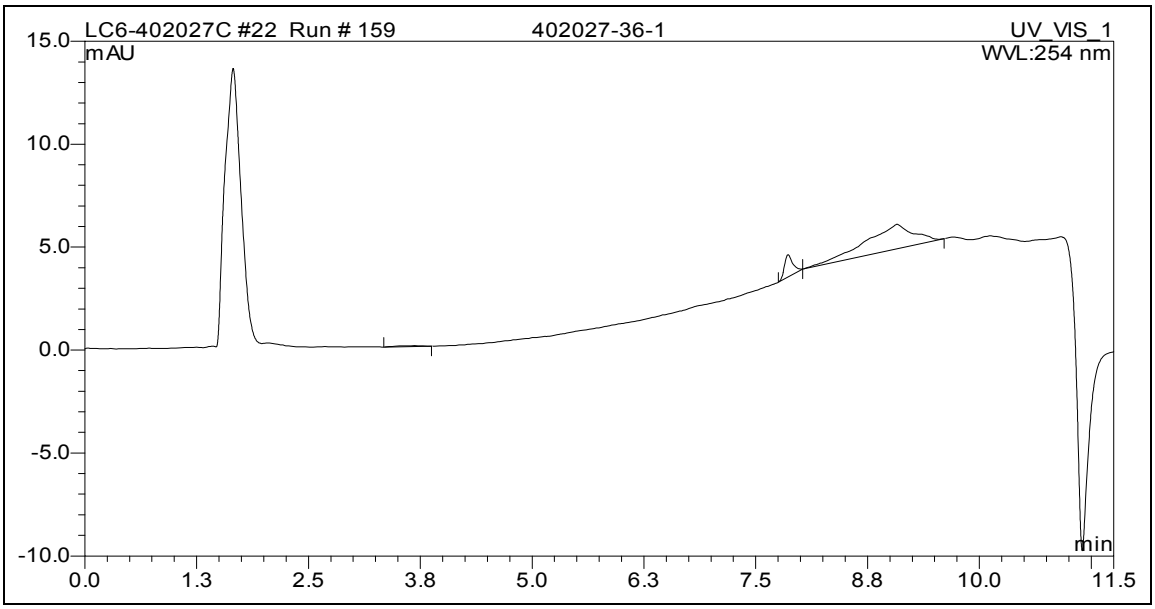


Figure 4: Chromatogram of a Processed Vehicle Blank Sample

4.1. SPECIFICITY/SELECTIVITY

As shown in Figure 4 (and in contrast to the chromatograms shown in Figure 1, Figure 2, and Figure 3), assay specificity/selectivity was confirmed when HPLC/UV analysis of processed vehicle samples revealed that there were no significant peaks (with signal-to-noise ratio [S/N] >10) at or near the retention time for the test substance peaks (approximately 5.5 to 9.0 minutes).

4.2. ASSAY VALIDATION: CALIBRATION REPRODUCIBILITY

During each of 3 validation sessions, triplicate calibration standards at 5 concentrations were prepared and analyzed as described previously. Single injections were made of each calibration standard. The resulting CAS# 64741-59-9 peak group area versus theoretical CAS# 64741-59-9 concentration data were fit to the linear function using least-squares regression analysis. The results of the regression analyses were used to back-calculate the corresponding concentrations from the peak group area data. As per the protocol, the reproducibility of the calibration curve data was considered valid when 1) the inter-session variability, expressed as RSD, of the back-calculated concentrations

at each calibration level was ≤10% RSD, except at the lowest calibration level where ≤15% was acceptable; and 2) the mean back-calculated concentrations at each calibration level were within ± 10% of the theoretical values (%RE within ± 10%), except at the lowest calibration level where %RE within ± 15% was acceptable.

The back-calculated concentrations and the associated intra- and inter-session statistics for the CAS# 64741-59-9 assay calibration standards are summarized in Table 1. The inter-session variability (RSD) of the back-calculated concentrations ranged from 0.77% to 5.8% RSD. The inter-session mean concentrations had %RE values ranging from -4.8% to 1.8%. Based on the stated criteria, the reproducibility of the calibration data was acceptable.

4.3. ASSAY VALIDATION: PRECISION AND ACCURACY

During each of 3 validation sessions, triplicate QC samples at 3 concentrations were prepared and analyzed as described previously. Single injections were made of each processed QC sample. The results of the regression analyses were used to calculate the corresponding concentrations from the QC peak group area data. The variability (RSD) of the calculated QC concentration data was used as a measure of assay precision, and the difference between the theoretical and calculated mean QC concentrations (%RE) was used as a measure of assay accuracy. According to the protocol, the precision of the method was considered acceptable when the inter-session RSD of the calculated concentrations at each QC level was ≤15%, and the accuracy of the method was considered acceptable when the inter-session calculated mean concentration at each QC level had a %RE value within ± 15%.

The calculated concentrations and the associated intra- and inter-session statistics for the CAS# 64741-59-9 assay QC samples are summarized in Table 2. The inter-session variability (RSD) of the calculated concentrations of each QC sample (precision) ranged from 3.9% to 4.7% RSD. The inter-session mean concentrations of the QC samples had

%RE values (accuracy) ranging from 1.5% to 3.9%. Based on the stated criteria, the precision and accuracy of the CAS# 64741-59-9 assay were acceptable.

4.4. ASSAY RUGGEDNESS

Assay ruggedness, as required by WIL Research SOP, was successfully demonstrated for this method because at least 2 of the 3 validation sessions were performed by different analysts.

4.5. ASSAY ACCEPTABILITY

In addition to the experimental samples, each analytical session consisted of (but was not limited to) calibration standards at 5 concentrations and triplicate QC samples prepared at each of 3 concentrations. In this study, the formulations were prepared at target concentrations of 50 and 500 mg CAS# 64741-59-9/mL, and the QC samples were prepared at nominal concentrations of 50.0, 250, and 500 mg CAS# 64741-59-9/mL. For an analytical session to be considered valid, at least two-thirds of the calculated QC concentrations with at least 1 sample at each concentration had to be 85% to 115% of the nominal QC concentration. All reported results were from analytical sessions that met the acceptance criteria.

4.6. TEST SUBSTANCE HOMOGENEITY AND RESUSPENSION HOMOGENEITY ASSESSMENT OF FORMULATIONS

Duplicate samples from the top, middle, and bottom strata of the formulations prepared on 24 May 2011 at target test substance concentrations of 50 and 500 mg/mL were analyzed to assess test substance homogeneity. The formulations that remained after sampling were divided into aliquots as would be used for daily dispensation. Representative aliquots were stored at room temperature for 8 and 15 days, at which time the test substance was resuspended by stirring. Duplicate samples were collected from the top and bottom strata of the aliquots and analyzed to assess 8- and 15-day resuspension homogeneity. The results of the homogeneity and resuspension

homogeneity analyses are presented in Table 3, Table 4, and Table 5, respectively, with the overall statistics summarized as follows:

Homogeneity Assessment of the 24 May 2011 Formulations

	Group Low (50 mg/mL)	Group High (500 mg/mL)
Mean Concentration (mg/mL)	50.6	517
SD	1.3	8.6
RSD (%)	2.6	1.7
Mean Concentration % of Target	101	103

8-Day Room Temperature Resuspension Homogeneity Assessment of the 24 May 2011 Formulations

	Group Low (50 mg/mL)	Group High (500 mg/mL)
Mean Concentration (mg/mL)	56.4	556
SD	1.3	6.4
RSD (%)	2.3	1.1
Mean Concentration % of Target	113	111

15-Day Room Temperature Resuspension Homogeneity Assessment of the 24 May 2011 Formulations

	Group Low (50 mg/mL)	Group High (500 mg/mL)
Mean Concentration (mg/mL)	49.8	500
SD	0.50	11
RSD (%)	1.0	2.2
Mean Concentration % of Target	99.6	99.9

The homogeneity assessment of the 24 May 2011 formulations met the protocol-specified requirement, *i.e.*, the RSD for the mean concentration was ≤10% at a concentration within the acceptable limits (within 85% to 115% of target concentration). The resuspension homogeneity assessments of the 24 May 2011 formulations met the protocol-specified requirement, *i.e.*, the RSD for the mean concentration was ≤10%.

4.7. TEST SUBSTANCE STABILITY IN FORMULATIONS

The formulations prepared and analyzed on 24 May 2011 were stored at room temperature for 8 and 15 days before being re-analyzed to assess test substance stability. The results of the stability analysis are presented in Table 4 and Table 5. The mean concentrations and percent of time-zero are summarized in the following table.

		Mean Concentration, mg/mL (% of Time-Zero)	
Storage Condition	Storage Duration	Group Low (50 mg/mL)	Group High (500 mg/mL)
Room Temperature	8 Days	56.4 (111)	556 (108)
	15 Days	49.8 (98.4)	500 (96.7)

The post-storage test substance concentrations ranged from 96.7% to 111% of the pre-storage values, which met the protocol-specified requirement for stability, *i.e.*, the mean post-storage concentration was not <90% of the pre-storage value.

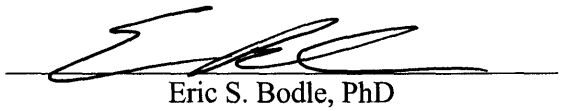
5. CONCLUSIONS

An HPLC/UV method for the determination of CAS# 64741-59-9 concentration in mineral oil formulations containing test substance ranging in concentration from 50.0 to 500 mg/mL was validated in this study. Method specificity/selectivity, ruggedness, calibration reproducibility, precision, and accuracy, were assessed and validated, satisfying protocol criteria.

Formulations prepared at target test substance concentrations of 50 and 500 mg CAS# 64741-59-9/mL met the WIL Research SOP requirement for homogeneity and, after 8 and 15 days of room temperature storage, resuspension homogeneity and stability.

6. REPORT REVIEW AND APPROVAL

Report Prepared and Approved by:

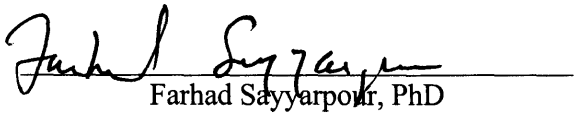


17 Oct 2011

Eric S. Bodle, PhD
Assistant Director, Analytical Chemistry
Study Director

Date

Report Reviewed by:



17 Oct 2011

Farhad Sayyarpour, PhD
Director, Bioanalytical Chemistry

Date

7. QUALITY ASSURANCE STATEMENT

7.1. PHASES INSPECTED

Date(s) of Inspection(s)	Phase Inspected	Dates(s) Findings Reported to Study Director	Date(s) Findings Reported to Management	Auditor(s)
01-Jun-2011	Test Article Analysis	01-Jun-2011	27-Jul-2011	C.Winkler
16-Aug-2011	Study Records (A-1)	16-Aug-2011	26-Sep-2011	C.Heifner
16-Aug-2011	Study Records (Rx-1)	16-Aug-2011	26-Sep-2011	C.Heifner
06-Sep-2011	Analytical Chemistry Report	06-Sep-2011	17-Oct-2011	C.Heifner
09-Sep-2011	Audited Analytical Chemistry Report	09-Sep-2011	17-Oct-2011	C.Heifner
17-Oct-2011	Final Report	17-Oct-2011	17-Oct-2011	E.Crookshank

This study was inspected in accordance with the United States EPA GLP Regulations (40 CFR Part 792), the OECD Principles of GLP, the WIL Research SOPs, and the protocol and protocol amendments as approved by the Sponsor. The data located in Appendix B (Report of Analysis) were the responsibility of the Sponsor. Review of the protocol and protocol amendments (if applicable) as well as a yearly internal facility inspection are conducted by the WIL Research Quality Assurance Department. A status report is submitted to management monthly.

This report accurately reflects the data generated during the study. The methods and procedures used in the study were those specified in the protocol, its amendments, and the WIL Research SOPs.

7.2. APPROVAL

This study was inspected according to the criteria discussed in Section 7.1.

Report Audited by:

Elizabeth B. Crookshank, BS
Group Supervisor, Quality Assurance

17 Oct 2011
Date

Carrie A. Heifner, BA
Compliance Specialist

17 Oct 2011
Date

Report Released by:

Heather L. Johnson, BS, RQAP-GLP
Manager, Quality Assurance

17 OCT 2011
Date

8. DATA RETENTION

The raw data, the retention sample(s) if applicable, pertinent electronic storage media, and the original final report are retained in the WIL Research Archives in compliance with regulatory requirements.

9. ABBREVIATIONS

The following abbreviations may apply to this report:

µ	-	micro
µL	-	microliter
ACN	-	acetonitrile
btm	-	bottom
cm	-	centimeter
DI	-	deionized
DMSO	-	dimethylsulfoxide
EPA	-	Environmental Protection Agency
g	-	gram
GLP	-	Good Laboratory Practices
HPLC	-	high performance liquid chromatography
hr	-	hour(s)
IS	-	internal standard
kg	-	kilogram
L	-	liter
LLOQ	-	lower limit of quantitation
MC	-	methylcellulose
MeOH	-	methanol
mg	-	milligram
mL	-	milliliter
mm	-	millimeter
msec	-	milliseconds
MS	-	mass spectrometry
NA	-	not applicable
ND	-	not detected
ng	-	nanogram
nm	-	nanometer
OECD	-	Organisation for Economic Cooperation and Development
ppm	-	parts per million
QC	-	quality control
%RE	-	percent relative error
RSD	-	relative standard deviation
SD	-	standard deviation
SOP	-	standard operating procedure
UV	-	ultraviolet
v	-	volume
w	-	weight
WIL Research	-	WIL Research Laboratories, LLC

TABLES 1 - 5

Table 1. Back-Calculated Concentrations of the Validation Calibration Standards

Theoretical Concentration (µg/mL)	**25.0**	**75.0**	**100**	**150**	**200**
Set 1 (16 May 2011)	26.0	72.7	102	151	198
	24.0	76.7	100	148	200
	24.4	75.0	101	149	202
Mean	24.8	74.8	101	149	200
SD	1.0	2.0	0.78	1.6	2.5
%RSD	4.2	2.7	0.78	1.1	1.2
%RE	-0.8	-0.31	0.98	-0.48	0.079
Set 2 (17 - 18 May 2011) *Ruggedness*	24.7	71.3	103	150	198
	24.3	71.4	102	151	199
	24.5	77.8	102	152	199
Mean	24.5	73.5	102	151	199
SD	0.19	3.7	0.80	0.92	0.55
%RSD	0.76	5.0	0.78	0.61	0.27
%RE	-2.1	-2.0	2.4	0.59	-0.61
Set 3 (17 - 18 May 2011)	22.2	77.4	102	150	199
	22.2	77.0	103	149	198
	21.9	77.9	102	151	199
Mean	22.1	77.4	102	150	198
SD	0.15	0.46	0.51	0.59	0.68
%RSD	0.67	0.59	0.50	0.39	0.34
%RE	-12	3.2	2.0	0.048	-0.80
Interset Statistics					
n	9	9	9	9	9
Mean	23.8	75.2	102	150	199
SD	1.4	2.7	0.87	1.2	1.5
%RSD	5.8	3.6	0.86	0.79	0.77
%RE	-4.8	0.30	1.8	0.053	-0.44

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Table 2. Calculated Concentrations of the Validation Quality Control Samples

Theoretical Concentration (ppm)	**50.0**	**250**	**500**
Set 1 (16 May 2011)	49.9	252	481
	49.5	251	487
	48.8	249	488
Mean	49.4	251	486
SD	0.56	1.6	3.9
%RSD	1.1	0.63	0.81
%RE	-1.2	0.28	-2.9
Set 2 (17 - 18 May 2011) *Ruggedness*	54.3	272	523
	54.8	274	538
	55.3	273	546
Mean	54.8	273	536
SD	0.48	0.84	11
%RSD	0.87	0.31	2.1
%RE	9.6	9.2	7.1
Set 3 (17 - 18 May 2011)	50.7	257	502
	51.5	254	501
	50.8	256	503
Mean	51.0	256	502
SD	0.42	1.5	1.4
%RSD	0.83	0.59	0.27
%RE	2.0	2.3	0.41
Interset Statistics			
n	9	9	9
Mean	51.7	260	508
SD	2.4	10	23
%RSD	4.7	3.9	4.5
%RE	3.5	3.9	1.5

results.xls II
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Table 3. Homogeneity Assessment of the 24 May 2011 Formulations

(Analyzed 24May 2011)

Group/Strata	Dose Conc (mg/mL)	Ref # (402027-)	Run # (402027-)	Analyzed Conc (mg/mL)	Percent of Target (%)	Mean Conc (mg/mL)	SD	RSD (%)	Mean Conc % of Target (%)
Low/Top	50	30 - 1	170	48.4	96.7	50.6	1.3	2.6	101
		30 - 2	171	51.5	103				
Low/Mid	50	30 - 3	172	51.9	104				
		30 - 4	173	50.0	100				
Low/Btm	50	30 - 5	174	50.5	101				
		30 - 6	175	51.3	103				
High/Top	500	30 - 7	176	509	102	517	8.6	1.7	103
		30 - 8	177	530	106				
High/Mid	500	30 - 9	178	508	102				
		30 - 10	179	523	105				
High/Btm	500	30 - 11	180	516	103				
		30 - 12	181	514	103				

Table 4. 8-Day Room Temperature Storage Resuspension Homogeneity and Stability Assessment of the 24 May 2011 Formulations
(Analyzed 1 June 2011)

Group/ Strata	Dose Conc. (mg/mL)	Ref # (402027 -)	Run #	Analyzed Conc. (mg/mL)	Percent of Target (%)	Mean Conc. (mg/mL)	SD	RSD (%)	Mean Conc % of Target (%)	Mean Conc % of Time Zero (%)
Low/Top	50	52 - 1	214	58.3	117	56.4	1.3	2.3	113	111
		52 - 2	215	55.8	112					
Low/Btm		52 - 3	216	55.7	111					
		52 - 4	217	55.7	111					
High/Top	500	52 - 5	218	558	112	556	6.4	1.1	111	108
		52 - 6	219	564	113					
High/Btm		52 - 7	220	551	110					
		52 - 8	221	551	110					

Time Zero Conc. (mg/mL)	
Low Group	50.6
High Group	517

results.xls 2RHS
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Table 5. 15-Day Room Temperature Storage Resuspension Homogeneity and Stability Assessment of the 24 May 2011 Formulations
(Analyzed 8 June 2011)

Group/ Strata	Dose Conc. (mg/mL)	Ref # (402027 -)	Run #	Analyzed Conc. (mg/mL)	Percent of Target (%)	Mean Conc. (mg/mL)	SD	RSD (%)	Mean Conc % of Target (%)	Mean Conc % of Time Zero (%)
Low/Top	50	67 - 1	254	50.3	101	49.8	0.50	1.0	99.6	98.4
		67 - 2	255	49.7	99.3					
Low/Btm		67 - 3	256	49.2	98.4					
		67 - 4	257	50.1	100					
High/Top	500	52 - 5	258	493	98.5	500	11	2.2	99.9	96.7
		52 - 6	259	516	103					
High/Btm		52 - 7	260	495	99.0					
		52 - 8	261	495	99.0					

Time Zero Conc. (mg/mL)	
Low Group	50.6
High Group	517

results.xls 3RHS
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APPENDIX A

Study Protocol and Deviations

DEVIATIONS FROM THE PROTOCOL

This study was conducted in accordance with the protocol and protocol amendments, except for the following.

- **The Sponsor** requested that the test substance be protected from light. The WIL Research Formulations Dept. was notified of this change via email from the Study Director on 23 November 2010. The retention sample collected on 15 November 2010 was stored in a clear glass vial, not light protected. The WIL Research Archives Dept. was contacted and informed that the material should be protected from light. The sample was foil-wrapped for light protection by WIL Research Archive personnel on 20 December 2010.

  Reason for Deviation: Retention sample was originally stored in a manner that was not light protected.

These deviations did not negatively impact the quality or integrity of the data nor the outcome of the study.

Study Number: WIL-402027

PROTOCOL AMENDMENT 1

Sponsor: American Petroleum Institute

Title of Study:

Analytical Validation and Stability Study of Distillates (Petroleum) Light Catalytic Cracked in Mineral Oil Formulations

Protocol Modifications:

1) Reference to a gas chromatograph (GC) method with flame ionization or mass spectrometric detection is mentioned throughout the protocol. However, the method developed and validated for use on this study was a high performance liquid chromatography (HPLC) method with ultraviolet (UV) absorbance detection.

Reasons for Protocol Modification:

1) Based on the characteristics of the compound, an HPLC method was more feasible for the evaluation of formulations concentration as stated in the Study Director Notification dated 13 May 2011.

Approval:

Sponsor's approval was obtained via email on 3 October 2011.
Date

WIL Research Laboratories, LLC

Eric S. Bodle, PhD
Study Director
30 Oct 2011
Date

for
Farhad Sayyarpour, PhD
Director, Bioanalytical Chemistry
30 Oct 2011
Date

American Petroleum Institute

Russell White
Sponsor Representative
4 Oct. 2011
Date

Departmental/Study Director Notification

Study Director: Eric Bodle

Study No.: 402027

Please note the following:

The protocol states a GC study will be developed and validated for the determination of distillates (petroleum) light catalytically cracked in mineral oil formulations. However, an HPLC/UV method will be developed and validated for the analysis of this compound in mineral oil formulations.

Reason for change:

Based on the characteristics of the compound, an HPLC method was more feasible for the evaluation of formulations concentration.

Sponsor approval on: NA

■ **Protocol amendment forthcoming**

☐ **Clarification of procedure, no protocol amendment required**

Study Director: _____ **Date:** 13 May 2011

Original: QAU
Copies as per protocol distribution

A-186-9

PROTOCOL

ANALYTICAL VALIDATION AND STABILTIY STUDY OF DISTILLATES (PETROLEUM), LIGHT CATALYTIC CRACKED IN MINERAL OIL FORMULATIONS

Submitted To:

American Petroleum Institute
1220 L Street, NW
Washington, DC 20005

WIL Research Laboratories, LLC
1407 George Road
Ashland, OH 44805-8946

1 OBJECTIVE:

To develop and validate a method for the determination of distillates (petroleum), light catalytic cracked concentration in mineral oil formulations using gas chromatography (GC) with flame ionization or mass spectrometric detection. Mineral oil formulations prepared at test substance concentrations of 50 and 500 mg/mL will be assessed for test substance homogeneity and, following 8 and 15 days of room temperature storage, resuspension homogeneity and stability.

This study will be conducted in compliance with the U.S. EPA/TSCA, 40 CFR Part 792, and the OECD, [C(97)186/Final], Good Laboratory Practice Standards. The study will also be conducted in accordance with the protocol and WIL Research Standard Operating Procedures.

2 PERSONNEL INVOLVED IN THE STUDY:

2.1 Sponsor Representative:

Russell White
American Petroleum Institute
1220 L Street, NW
Washington, DC 20005
Tel: (202) 682-8344
Email: whiter@api.org

2.2 WIL Study Director:

Eric. S. Bodle, PhD
Assistant Director, Analytical Chemistry
Phone: (419) 289-8700
Fax: (419) 289-3650
E-mail: ebodle@wilresearch.com

2.3 WIL Departmental Responsibilities:

Amanda M. Stanton, BA
Group Supervisor/Training Coordinator, Analytical Chemistry
Emergency Contact
Tel: (419) 289-8700
Fax: (419) 289-3650
E-mail: astanton@wilresearch.com

Mark D. Nemec, BS, DABT
President and Chief Operating Officer

Michael J. Schlosser, PhD, DABT
Vice President, Analytical, Metabolism,
and *In Vitro* Toxicology Services

Heather L. Johnson, BS, RQAP-GLP
Manager, Quality Assurance

Robert A. Wally, BS
Operations Manager, Reporting and
Regulatory Technical Services

3 STUDY SCHEDULE:

Proposed Experimental Starting Date:	March 2011
Proposed Experimental Completion Date:	April 2011
Proposed Audited Report Date:	Typically 6 weeks after the completion of validation activities.

4 TEST SUBSTANCE INFORMATION:

4.1 Test Substance:

4.1.1 Identification:

Distillates (petroleum), light catalytic cracked

4.1.2 CAS#:

64741-59-9

4.1.3 CAS definition:

A complex combination of hydrocarbons produced by the distillation of products from a catalytic cracking process. It consists of hydrocarbons having carbon numbers predominantly in the range of C9 through C25 and boiling in the range of approximately 150°C to 400°C (302°F to 752°F). It contains a relatively large proportion of bicyclic aromatic hydrocarbons.

4.1.4 Lot Number:

26:18

4.1.5 Expiration/Retest Date:

To be provided by the sponsor. Retest in 5 years

4.1.6 Purity:

100%

4.1.7 Storage Conditions:

Room temperature

4.1.8 Stability:

The test substance is considered to be stable under the storage conditions provided by the Sponsor.

4.1.9 Physical Description:

To be documented by WIL Research Laboratories, LLC.

4.1.10 Reserve Samples:

Reserve samples of the test substance will be taken in accordance with WIL Standard Operating Procedures and stored in the Archives at WIL Research Laboratories, LLC indefinitely, unless otherwise specified.

4.1.11 Personnel Safety Data:

It is the responsibility of the Sponsor to notify the testing facility of any special handling requirements for the test substance. A Material Safety Data Sheet (MSDS) should accompany the test substance upon arrival at the laboratory.

4.1.12 Test Substance Disposition:

With the exception of the reserve sample for each batch of test substance, all neat test substance remaining at study completion will be returned to the Sponsor. Alternatively, the test substance can be retained for subsequent studies.

5 TEST SYSTEM:

- Mineral Oil with and without test substance

6 EXPERIMENTAL DESIGN:

6.1 Overview of the Study:

Distillates (petroleum), light catalytic cracked is the test substance for this study and will be referred to as the analyte. The method to be validated is for the determination of the analyte concentration in mineral oil formulations. This study will provide the necessary data that demonstrates the analytical method as valid.

6.2 Method Details

6.2.1 Instrument

A GC equipped with a mass spectrometer and/or flame ionization detector, an autosampler, and MS workstation software, or equivalent system. Possible systems include:

- Varian 3800 GC System
- Varian 2200 Ion-Trap mass spectrometer

6.2.2 Carrier:

Mineral Oil, USP (Spectrum Chemicals and Laboratory Products)

6.2.3 Method:

The method validation activities include two phases: (1) method evaluation and development, and (2) formal method validation.

Method evaluation of sponsor-supplied methodology usually includes (but is not limited to) the following activities: (1) the analysis of standards prepared in an appropriate solvent to establish chromatography, including retention times, resolution, sensitivity, and to check proportionality of response; (2) the analysis of the analyte prepared in the matrix to confirm the presence or absence of interferences, to evaluate potential stability limitations, and to evaluate response proportionality. Sponsor supplied methodology and other literature will be used as a starting point for method evaluation/development. Method development/evaluation will not be audited by the WIL Quality Assurance Unit.

6.3 Study Details and Criteria:

6.3.1 Specificity:

The specificity of the method will be determined by analyzing representative blank samples. The retention time window(s) corresponding to the analyte and internal standard (if applicable) will be examined for interferences and, if needed, appropriate efforts to minimize interfering peaks will be taken such as: adjustment or change of chromatographic parameters to maximize resolution of interference and analyte peaks; use of a more analyte-specific wavelength; and change in sample preparation procedure to minimize the presence of the interference in the sample to be analyzed. The success of these efforts will be determined when the method validation either passes or fails the accuracy and precision acceptance criteria for calibration and quality control samples.

6.3.2 Calibration Reproducibility:

A minimum of 3 validation sessions will be performed to validate the method for the determination of the analyte concentration in formulations. For each validation session, at least triplicate calibration standards at a minimum of 5 different analyte concentrations will be prepared and analyzed. The concentration of the calibration standards and the regression model used for the regression analysis will be specified in the written method to be validated. The results of the regression analysis will be used to back-calculate the calibration standard concentrations. The inter-session back-calculated concentration data at each calibration level must be precise (relative standard deviation [RSD] less than or equal to 10%, except at the lowest concentration level where it should not exceed 15%) and accurate (percent relative error [%RE] within ± 10% except at the lowest concentration level where it should not exceed ± 15%).

6.3.3 Accuracy and Precision:

Quality control samples will be prepared at a minimum of 3 concentrations in blank matrix – one near the lowest, one near the middle and one near the highest formulation concentration expected for future studies. The concentration of the QC samples will be specified in the written method to be validated. At least 3 replicate quality control samples at each concentration level will be analyzed with the calibration standards during each validation session. The inter-session accuracy and precision will be established based on the analyzed concentrations of the quality control samples. The inter-session analyzed concentration data

at each QC level must be precise (RSD less than or equal to 15%, except at the lowest concentration level where 20% is acceptable), and accurate (RE is within ± 15%, except at the lowest concentration level where ±20% is acceptable).

6.3.4 Stability of Calibration Standards and Quality Control Samples:

The room temperature and/or autosampler temperature stability of calibration standards and processed quality control samples will be evaluated after a minimum of 24 hours of storage.

At least duplicate samples at the highest and lowest concentration levels evaluated will be analyzed post-storage, and the results will be compared to pre-storage values, results from analysis of freshly prepared samples, or the theoretical pre-storage values.

The analyte will be considered stable if the post-storage value is not less than 90% of the pre-storage (or pre-storage equivalent) value. If a >10% reduction occurs under the intended storage conditions, alternate storage conditions and/or durations may be evaluated as necessary to identify conditions that allow for stability during sample storage and processing.

6.3.5 Homogeneity, Resuspension Homogeneity, and Stability of Mineral Oil Formulations:

Test substance homogeneity, resuspension homogeneity, and stability in mineral oil formulations prepared at test substance concentrations of 50 and 500 mg/mL will be assessed immediately after preparation and after at least 8 and 15 days of room temperature storage. The formulations will be prepared according to instructions reviewed and authorized by the Study Director. The carrier and dose formulation preparations will be stirred during sample collection.

For the homogeneity assessment, samples (in at least duplicate) will be collected from the top, middle, and bottom strata of the formulations on the day of preparation and analyzed to assess test substance homogeneity in the formulations. Additional samples may be collected on the day of preparation from the middle stratum and stored appropriately for the assessment of stability. Following sample collection the formulations will be divided into aliquots representative of those used for daily dispensation and stored at room temperature for at least 8 and 15 days. After the intended storage, aliquots of the formulations will be resuspended by stirring for a minimum of 30 minutes and duplicate samples from the top and bottom strata of the

formulations will be collected and analyzed to assess resuspension homogeneity.

In order for the formulations to be considered homogeneous, the RSD for the mean concentration of the analyzed samples must be less than or equal to 10% at a concentration within the acceptable limits (90% to 110% of the target concentration). In order for the formulations to be considered homogeneous after resuspension, the RSD for the mean concentration of the analyzed samples must be less than or equal to 10%. In order for the test substance to be considered stable in the formulation, the post-storage assay concentration cannot be less than 90% of the pre-storage concentration.

7 QUALITY ASSURANCE:

The study will be audited by the WIL Quality Assurance Unit while in progress to assure compliance with GLP regulations, adherence to the protocol and to WIL SOP. The raw data and draft report will be audited by the WIL Quality Assurance Unit prior to submission to the Sponsor to assure that the final report accurately describes the conduct and the findings of the study.

This study will be included on the WIL master list of regulated studies.

8 RECORDS TO BE MAINTAINED:

All original raw data records, as defined by WIL SOPs and the applicable GLPs, will be stored in the Archives at WIL Research Laboratories, LLC. Records to be retained will include, but are not limited to the following:

- Protocol and protocol amendments
- A list of WIL study personnel involved in the conduct of the study
- The original chromatograms, spectra and other instrument generated data
- Calculations of concentration levels and appropriate test parameters

9 WORK PRODUCT:

The Sponsor will have title to all documentation records, raw data, and other work product generated during the performance of the study. All work product, including raw paper data and magnetically encoded records, will be retained at no charge for a period of six months following issuance of the final report in the Archives at WIL Research Laboratories, LLC. Thereafter, WIL Research Laboratories, LLC will charge a monthly archiving fee for retention of all work product. All work product will be stored in compliance with regulatory requirements.

Any work product, including documents, and samples, that are required by this protocol, its amendments, or other written instructions of the Sponsor, to be shipped by WIL Research Laboratories, LLC to another location will be appropriately packaged and labeled as defined by WIL's SOPs and delivered to a common carrier for shipment. WIL Research Laboratories, LLC will not be responsible for shipment following delivery to the common carrier.

10 REPORTS:

The final report will contain a summary, test substance data, methods and procedures, and an interpretation and discussion of the study results. The report will contain all information necessary to conform with current EPA and OECD specifications.

The contents of the report will be as follows:

- The study will be summarized in a formal report.
- Details of all experimental procedures and methods of calculation will be described.
- Sample preparation, chromatographic or other test conditions, calibration reproducibility, accuracy and precision will be detailed.
- Copies of chromatograms obtained in the analysis will be entered as appropriate.
- Any protocol or GLP deviations that may occur during the study will be detailed.
- A compliance statement and a Quality Assurance Unit statement will be included.

WIL Research Laboratories, LLC will provide one (1) electronic copy of an Audited Draft Report, submitted 6-8 weeks upon completion of the study prior to issuance of the final report. One (1) revision will be permitted as part of the cost of the study, from which the Sponsor's reasonable revisions and suggestions will be incorporated into the Final Report as appropriate. Additional changes or revisions may be made at extra cost. It is expected that the Sponsor will review the draft report and provide comments to WIL within a two (2) month time frame following submission. WIL will submit the Final Report within one (1) month following receipt of comments. If the Sponsor's comments/authorization to finalize the report have not been received at WIL Research Laboratories, LLC within one year following submission of the draft report, WIL Research Laboratories, LLC may elect to finalize the report following appropriate written notification to the Sponsor. Two (2) electronic copies of the Final Report (PDF) will be provided; requests for additional copies of the Final Report may result in additional charges.

11 PROTOCOL MODIFICATION:

Modification of the protocol may be accomplished during the course of this study. However, no changes will be made in the study design without the verbal or written permission of the Sponsor. In the event that the Sponsor verbally requests or approves a change in the protocol, such changes will be made by appropriate

documentation in the form of a protocol amendment. All alterations of the protocol and reasons for the modification(s) will be signed by the Study Director and the Sponsor Representative.

12 PROTOCOL APPROVAL:

Sponsor approval received via e-mail on 15 Mar 2011.
Date

American Petroleum Institute

Russell D. White — 21 March 2011
Russell White — Date
Sponsor Representative

WIL Research Laboratories, LLC

[signature] — 18 Mar 2011
Eric S. Bodle, PhD — Date
Study Director

[signature] — 18 March 2011
for Michael J. Schlosser, PhD, DABT — Date
Vice President,
Analytical, Metabolism, and *In Vitro* Toxicology Services

APPENDIX B

Report of Analysis

Intertek

Report of Analysis

Sample ID:2009-DRPK-000651-041
Drawn By: Client
Sample Designated As: Crude Oil
Representing: Site#26 Sx.#18 (As Received)

Date Taken:16-January-2009
Date Submitted:16-January-2009
Date Tested:04-March-2009

Method	Test	Result	Units
ASTM D4052	Density of Liquids by Digital Density Meter		
	Relative Density @ 60/60°F	0.9390	
	API Gravity @ 60°F	19.2	°API
ASTM D2887	Boiling Range Distribution of Petroleum Fractions by GC (Simulated Distillation)		
	Boiling Point Distribution	See Attached Report	
ASTM D1319	Hydrocarbon Types (Aromatics, Olefins, Saturates) by FIA		
	Comment	Couldn't Run (Water)	
ASTM D5186	Determination of Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels by Supercritical Fluid Chromatography		
	Monoaromatics by SFC	35.3	Wt %
	Polynuclear Aromatics by SFC	45.7	Wt %
	Total Aromatics	80.9	Wt %

Sample ID:2009-DRPK-000651-042
Drawn By: Client
Sample Designated As: Crude Oil
Representing: Site#26 Sx.#21 (As Received)

Date Taken:16-January-2009
Date Submitted:16-January-2009
Date Tested:26-January-2009

Method	Test	Result	Units
ASTM D4052	Density of Liquids by Digital Density Meter		
	Relative Density @ 60/60°F	0.8057	
	API Gravity @ 60°F	44.1	°API
ASTM D2887	Boiling Range Distribution of Petroleum Fractions by GC (Simulated Distillation)		
	Boiling Point Distribution	See Attached Report	
ASTM D1319	Hydrocarbon Types (Aromatics, Olefins, Saturates) by FIA		
	Aromatics	14.7	Vol %
	Olefins	2.3	Vol %
	Saturates	83.0	Vol %
ASTM D5186	Determination of Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels by Supercritical Fluid Chromatography		
	Monoaromatics by SFC	19.9	Wt %
	Polynuclear Aromatics by SFC	1.1	Wt %
	Total Aromatics	21.0	Wt %

Simdist-2000
ITS Caleb Brett - Houston

SAMPLE:	09-0651-41 (Site #26 Sx. #18)	Injection Date:	0090117095350-0600
		Report Date:	1/18/09 8:08
FILE:	C:\CP32 Instruments\D2887 & D3710\Data\2009\JAN-09\09-0651-41.0001.CDF		
PROCEDURE:	C:\CP32 Instruments\D2887 & D3710\PROCEDURES\122308-D2887.prc		
EXCEL FILE:	C:\CP32 Instruments\D2887 & D3710\Reports\2009\JAN-09\09-0651-41_0001_CDF.xls		

Boiling Point Distribution Report
ASTM D2887 Simulated Distillation

%Off	BP °F	BP °C	%Off	BP °F	BP °C	%Off	BP °F	BP °C
IBP	254.7	123.7	40%	473.4	245.2	80%	607.4	319.7
1%	288.2	142.3	41%	478.6	248.1	81%	613.0	322.8
2%	314.5	156.9	42%	481.0	249.4	82%	615.8	324.3
3%	330.8	166.0	43%	483.4	250.8	83%	620.0	326.7
4%	340.8	171.6	44%	484.6	251.4	84%	625.7	329.9
5%	351.3	177.4	45%	485.7	252.1	85%	632.8	333.8
6%	364.6	184.8	46%	488.0	253.4	86%	639.9	337.7
7%	369.8	187.7	47%	489.0	253.9	87%	645.2	340.7
8%	376.0	191.1	48%	489.7	254.3	88%	649.2	342.9
9%	379.2	192.9	49%	490.6	254.8	89%	653.5	345.3
10%	389.4	198.5	50%	493.8	256.6	90%	660.9	349.4
11%	391.2	199.6	51%	495.6	257.6	91%	669.4	354.1
12%	395.9	202.2	52%	499.2	259.5	92%	676.5	358.1
13%	398.6	203.7	53%	506.6	263.7	93%	682.7	361.5
14%	400.1	204.5	54%	510.9	266.1	94%	691.9	366.6
15%	401.9	205.5	55%	514.5	268.1	95%	700.1	371.1
16%	405.2	207.3	56%	516.3	269.1	96%	711.0	377.2
17%	407.7	208.7	57%	519.9	271.0	97%	722.1	383.4
18%	408.7	209.3	58%	522.3	272.4	98%	739.4	393.0
19%	410.4	210.2	59%	524.5	273.6	99%	768.2	409.0
20%	413.3	211.8	60%	527.5	275.3	FBP	800.2	426.8
21%	415.2	212.9	61%	530.1	276.7			
22%	417.9	214.4	62%	533.1	278.4			
23%	421.9	216.6	63%	537.3	280.7			
24%	428.0	220.0	64%	540.7	282.6			
25%	431.7	222.0	65%	543.5	284.2			
26%	435.5	224.2	66%	547.3	286.3			
27%	438.9	226.0	67%	551.0	288.4			
28%	441.9	227.7	68%	554.9	290.5			
29%	446.4	230.2	69%	559.0	292.8			
30%	447.6	230.9	70%	566.0	296.7			
31%	448.3	231.3	71%	568.8	298.2			
32%	448.9	231.6	72%	571.9	300.0			
33%	449.5	231.9	73%	574.6	301.5			
34%	451.2	232.9	74%	578.8	303.8			
35%	452.8	233.8	75%	582.7	305.9			
36%	453.7	234.3	76%	585.7	307.6			
37%	456.4	235.8	77%	592.1	311.2			
38%	462.2	239.0	78%	597.5	314.2			
39%	467.0	241.7	79%	602.2	316.8			

Start Elution Time (mins):	0.151	**Sample Wt:**	0 g
End Elution Time (mins):	23.761	**Solvent Wt:**	0 g
		Material Balance:	100.0 Wt%
Blank File:	C:\CP32 Instruments\D2887 & D3710\Data\2009\JAN-09\CS2-BLANK.0009.CDF		
Calib File:	D:\CP32 Instruments\D2887 & D3710\DATA\RTMIX-060905.0006.CDF		
Resp Factor:	1.000E+00		