FINAL REPORT

Study Title:	Analytical Validation and Stability Study of Catalytically Cracked Slurry Oil in Acetone Formulations
Study Number:	WIL-402029
Study Director:	[redacted]
Study Initiation Date:	18 March 2011
Study Completion Date:	18 November 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-402029, 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 Certificate of Analysis was not provided by the Sponsor.

[redacted]

Research Chemist, Analytical Chemistry
Study Director

18 Nov 2011
Date

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1. SUMMARY

A gas chromatography method using flame ionization detection for the determination of catalytically cracked slurry oil concentration in acetone formulations (high purity solvent, 99.8+% pure) and test substance ranging in concentration from 1.00 to 100 mg/mL was validated in this study. Also in this study, the assay was cross-validated in acetone formulations (minimum 99.0+% pure) and test substance ranging in concentration from 1.00 to 100 mg/mL. In addition, test substance stability in calibration standards and processed QC samples stored at room temperature for 2 days was assessed.

Also in this study, test substance homogeneity and, following 11 and 18 days of room temperature storage, resuspension homogeneity and stability were assessed in formulations prepared at target concentrations of 1 and 100 mg catalytically cracked slurry oil/mL.

The catalytically cracked slurry oil assay procedure was validated in this study with 3 validation sessions and subsequently cross-validated with a single validation session. Quantitation was performed using calibration standards ranging from 500 to 1000 µg catalytically cracked slurry oil/mL. The inter- or intra-session variability (relative standard deviation [RSD]) and percent relative error (%RE) of the mean back-calculated standard concentrations of the calibration standards prepared for the validation and cross validation, respectively, are summarized in the following table.

Validation	RSD Range of Values (%)	%RE Range of Values (%)
Full (3 sessions)	0.82 to 2.3	-0.58 to 0.64
Cross (1 session)	0.39 to 1.1	-3.0 to 0.66

The results met the protocol-specified acceptance criteria for calibration standards, *i.e.*, RSD ≤10% and %RE within ± 10% (except at the lowest calibration level where RSD ≤15% and %RE within ± 15% were acceptable).

Assay precision and accuracy were verified by the analysis of QC samples. The inter- or intra-session variability (precision) and %RE (accuracy) of the mean calculated QC concentrations of the samples prepared for the validation and cross-validation, respectively, are summarized in the following table.

Validation	QC Range (mg/mL)	RSD Range of Values (%)	%RE Range of Values (%)
Full (3 sessions)	1.00 to 100	2.0 to 2.6	-1.6 to -0.14
Cross (1 session)	1.00 to 100	0.41 to 3.0	-0.72 to -0.080

The results met the protocol-specified acceptance criteria for precision and accuracy, *i.e.*, RSD ≤15% and %RE within ± 15% (except at the lowest calibration level where RSD ≤20% and %RE within ± 20% were acceptable).

The results of the test substance homogeneity assessment in formulations prepared at target concentrations of 1 and 100 mg catalytically cracked slurry oil/mL met the protocol-specified acceptance criteria, *i.e.*, the RSD for the mean concentration was ≤10% at a concentration within the acceptable limits (90% to 110% of target). Assessment of test substance resuspension homogeneity and stability in formulations prepared at target concentrations of 1 and 100 mg catalytically cracked slurry oil/mL and following 11 and 18 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 gas chromatography (GC) method using flame ionization detection (FID) for the determination of catalytically cracked slurry oil concentration in acetone formulations and test substance ranging in concentration from 1.00 to 100 mg/mL. Assay specificity/selectivity, calibration reproducibility, precision, accuracy, ruggedness, and test substance stability in calibration

standards and processed QC samples stored at room temperature for 2 days were assessed. In addition, formulations prepared at target concentrations of 1 and 100 mg catalytically cracked slurry oil/mL were analyzed to assess test substance homogeneity and, following 11 and 18 days of room temperature storage, resuspension homogeneity and stability.

The study protocol and deviations 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)
12 April 2011	First date of analysis (Experimental start/starting date)
23 June 2011	Last date of analysis (Experimental termination/completion date)

2.2. WIL RESEARCH KEY STUDY PERSONNEL

[redacted]	Chemist III, Analytical Chemistry
	Chemist II, Analytical Chemistry
	Group Supervisor/Associate Research Chemist, Analytical Chemistry
[redacted]	Publishing Specialist, Reporting & Technical Support Services
[redacted]	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, clarified oils (petroleum), catalytic cracked, CAS no. 64741-62-4, also known as catalytically cracked slurry oil, was received from EPL Archives, Sterling, VA on behalf of American Petroleum Institute on 10 November 2010 as follows:

Identification	Quantity Received	Physical Description
Catalytically Cracked Slurry Oil CAS no. 64741-62-4, Site 12, Sample 2 WIL log no.8473A	4 Bottles	Dark Brown, Very Viscous Liquid

The test substance was stored at room temperature and protected from light and was considered stable under this condition. A reserve sample of the test substance (approximately 0.834 g) was collected on 15 November 2010 and stored in the Archives of WIL Research.

3.1.2. VEHICLE IDENTIFICATION

The vehicle used in preparation of the test substance formulations was acetone:

- Acetone, min. 99.0+% (received from Spectrum Chemical Mfg. Corp., New Brunswick, NJ)
- Each lot used was documented in the raw data.

3.2. FORMULATION PREPARATION

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

Group Number	Test Substance	Concentration (mg/mL)
Low	Catalytically cracked slurry oil	1
High	Catalytically cracked slurry oil	100

The appropriate amount of the test substance for each formulation was weighed in a calibrated glass container. A stir bar was added and the appropriate amount of vehicle was added to each container and mixed with a magnetic stirrer until uniform. The test substance formulations were stirred continuously throughout the preparation and sampling procedures.

3.3. GAS CHROMATOGRAPHY

Instrument:	Agilent 6890 gas chromatograph (GC) equipped with a flame ionization detector, an Agilent 7673 autosampler, and Dionex Chromeleon® data system, or equivalent
Column:	Zebron ZB-1HT Inferno column, 15 m × 0.32 mm ID, 0.25-µm film-thickness
Temperature Program:	120°C, hold for 1 minute, ramp at 40°C/minute to 400°C, hold for 2 minutes
Carrier Gas:	Helium
Carrier Gas Flow Rate:	1.5 mL/minute
Injector Temperature:	300°C
Injection Volume:	1 µL split (5:1)
Detector:	Flame ionization detector at 400°C
Retention Time:	Approximately 3.75 to 7.0 minutes for light catalytically cracked slurry oil peak group
Run Time:	10 minutes
Wash Vial:	Hexane

3.4. PREPARATION OF CALIBRATION STOCK SOLUTION

A calibration standard stock solution was prepared at a concentration of 1.00 mg catalytic cracked slurry oil/mL as follows. Approximately 25 mg of catalytic cracked slurry oil (WIL log no. 8473A, no correction for purity) was accurately weighed in a tared glass funnel and transferred to a 25-mL volumetric flask with rinses of ethyl acetate (EtOAc). EtOAc used for preparation of stock solutions and dilutions were HPLC grade >99%. The contents were mixed as needed to achieve dissolution of the test substance. Additional EtOAc was added to yield the desired concentration, and the solution was stirred to mix.

3.5. PREPARATION OF CALIBRATION STANDARDS

Calibration standards were prepared at 500, 600, 750, 850, and 1000 µg catalytic cracked slurry oil/mL by thoroughly mixing the appropriate volumes of calibration stock solution

and EtOAc in amber autosampler vials. Calibration standards were prepared in triplicate at each concentration level for the validation sessions; at least single calibration standards were prepared at each concentration thereafter.

3.6. PREPARATION OF THE QUALITY CONTROL STOCK SOLUTION

A quality control (QC) stock solution was prepared at a concentration of 20.0 mg catalytic cracked slurry oil/mL as follows. Approximately 0.2 g of catalytic cracked slurry oil (WIL log no. 8473A, no correction for purity) was accurately weighed in a tared glass funnel and transferred to a 10-mL volumetric flask with rinses of EtOAc. The contents were mixed as needed to achieve dissolution of the test substance. Additional EtOAc was added to yield the desired concentration, and the solution was stirred to mix.

3.7. PREPARATION AND PROCESSING OF QUALITY CONTROL SAMPLES

As detailed in the following table, QC samples were prepared to simulate the processing of formulation samples at concentrations of 1.00, 10.0, and 100 mg catalytic cracked slurry oil/mL (nominal QC concentrations) by combining aliquots of the QC stock solution, vehicle (acetone), and EtOAc in polypropylene tubes or amber autosampler vials. The QC samples were capped and mixed with vortex action. The processed samples were further diluted as necessary with EtOAc in amber autosampler vials. The samples were capped and thoroughly mixed with vortex action. Triplicate QC samples at each concentration were prepared; a single blank sample was prepared.

QC Level	Nominal QC Concentration (mg/mL)	Vehicle Volume (mL)	QC Stock Volume (mL)	EtOAc Volume (mL)	Secondary Dilution	Theoretical Final Concentration (µg/mL)
Blank	0	0.500	0	0.300	NA	0
QC1	1.00	0.500	0.0250	0.275	NA	625
QC2	10.0	0.500	0.250	7.25	NA	625
QC3	100	0.500	2.50	7.00	6.67-fold	750

NA = Not Applicable

3.8. FORMULATION SAMPLE PROCESSING

Quadruplicate formulation samples were collected using a 1-mL Class A volumetric pipette 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 discarded upon Study Director's acceptance of the formulations. 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. 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	1	1.0	0.600	NA	625
High	100	1.0	9.000	12.5-fold	800

NA = Not applicable

3.9. CALIBRATION AND QUANTITATION

Single injections were made of each calibration standard, processed QC and formulation samples. A calibration curve was constructed for each set of analyses. The catalytic cracked slurry oil peak group area (y) and the theoretical concentrations (x) of the calibration standards were fit with least-squares regression analysis to the quadratic function:

$$y = ax^2 + b\,x + c$$

Concentrations were back-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, were calculated and presented in tabular form. The concentrations

of the formulation and QC samples were calculated by applying any necessary multiplication factors to correct for dilution and/or unit conversions.

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 information retrieval 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 3.75 to 7.0 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 10 minutes.

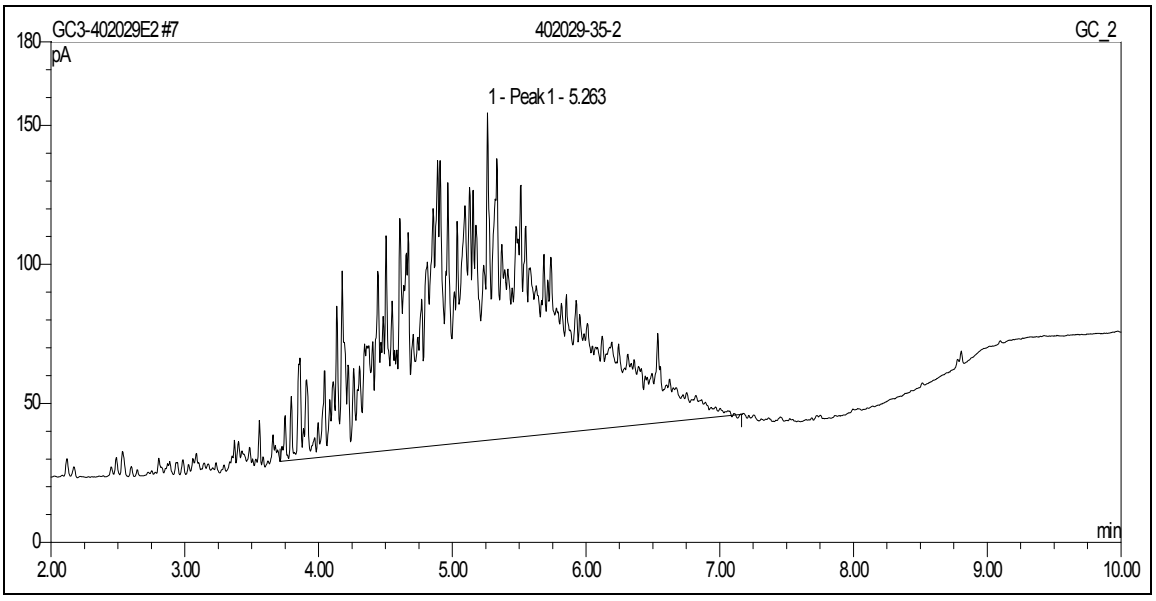

Figure 1: Representative Chromatogram of a 500 µg Catalytically Cracked Slurry Oil/mL Calibration Standard

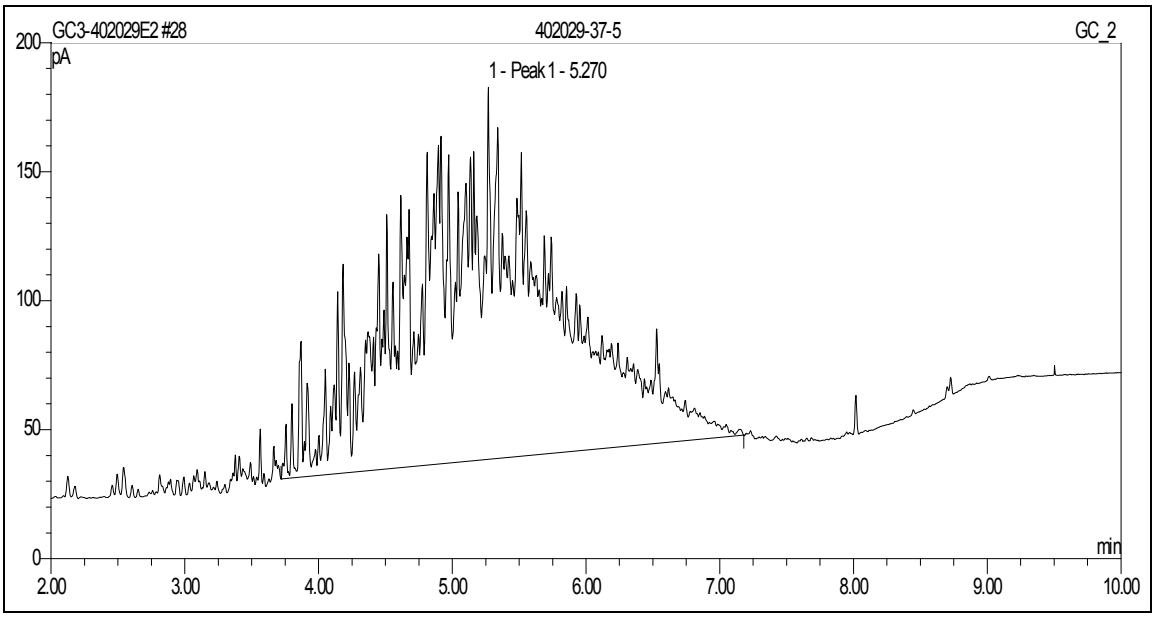

Figure 2: Representative Chromatogram of a Processed 10.0 mg Catalytically Cracked Slurry Oil/mL Quality Control Sample

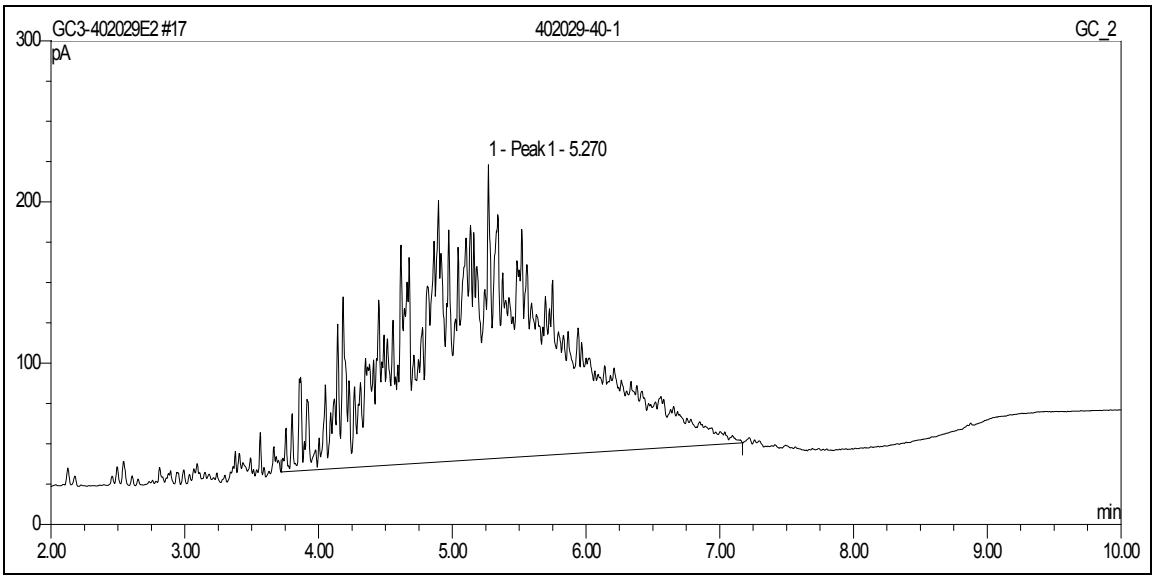

Figure 3: Representative Chromatogram of a Processed 100 mg Catalytically Cracked Slurry Oil/mL Formulation Sample

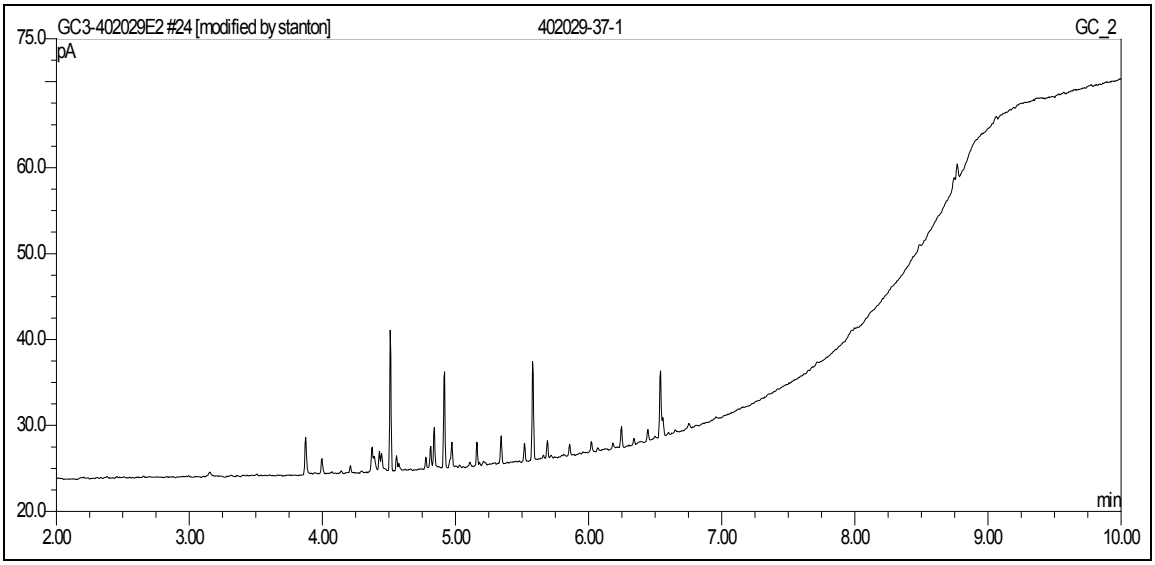

Figure 4: Chromatogram of a Processed Vehicle Blank Sample

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 GC/FID 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 group (approximately 3.75 to 7.0 minutes).

4.2. ASSAY VALIDATION: CALIBRATION REPRODUCIBILITY

During each of the 3 method validation sessions and the subsequent single cross-validation session, triplicate calibration standards at 5 concentrations were prepared and analyzed as described previously. Single injections were made of each calibration standard. The resulting catalytically cracked slurry oil peak areas versus theoretical catalytically cracked slurry oil concentration data were fit to the quadratic function using least-squares regression analysis. The results of the regression analyses were used to back-calculate the corresponding concentrations from the peak area data. As per protocol-specified acceptance criteria, 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%, 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 (percent relative error [%RE] within ± 10%), except at the lowest calibration level where %RE within ± 15% was acceptable. Intra-session statistics were used to evaluate the single cross-validation session.

The back-calculated concentrations and the associated inter- and/or intra-session statistics for the catalytically cracked slurry oil assay validation and cross-validation calibration standards are summarized in Table 1 and Table 2, respectively, with the inter- or intra-session variability (RSD) of the back-calculated concentrations and the %RE of the inter- or intra-session mean concentrations summarized as follows.

Validation	RSD Range of Values (%)	%RE Range of Values (%)
Full (3 sessions)	0.82 to 2.3	-0.58 to 0.64
Cross (1 session)	0.39 to 1.1	-3.0 to 0.66

Based on the stated criteria, the reproducibility of the catalytically cracked slurry oil calibration data was acceptable.

4.3. ASSAY VALIDATION: PRECISION AND ACCURACY

During each of the 3 method validation sessions and the subsequent single cross-validation session, 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 area data. The variability (RSD) of the calculated QC concentration data was used as a measure of assay precision, and the difference between theoretical and the calculated mean QC concentrations (%RE) was used as a measure of assay accuracy. According to protocol-specified acceptability criteria, the precision of the method was considered acceptable when the inter-session RSD of the calculated concentrations at each QC level was ≤15% except at the lowest QC level where ≤20% was acceptable, 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% except at the lowest QC level where ≤20% was acceptable. Intra-session statistics were used to evaluate the single cross-validation session.

The calculated concentrations and the associated inter- and/or intra-session statistics for the catalytically cracked slurry oil assay validation and validation extension QC samples are summarized in Table 3 and Table 4, respectively, with the inter- or intra-session variability (RSD) of the calculated concentrations of each QC sample (precision), and the %RE values (accuracy) of the inter- or intra-session mean concentrations of the QC samples summarized as follows.

Validation	QC Range (mg/mL)	RSD Range of Values (%)	%RE Range of Values (%)
Full (3 sessions)	1.00 to 100	2.0 to 2.6	-1.6 to -0.14
Cross (1 session)	1.00 to 100	0.41 to 3.0	-0.72 to -0.080

Based on the previously stated criteria, the precision and accuracy of the catalytically cracked slurry oil assay was 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 1 and 100 mg catalytically cracked slurry oil/mL, and the QC samples were prepared at nominal concentrations of 1.00, 10.0, and 100 mg catalytically cracked slurry oil/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 STABILITY IN CALIBRATION STANDARDS

Calibration standards prepared at 500 and 1000 µg/mL and analyzed on 21 June 2011 were stored at room temperature for 2 days before being re-analyzed to assess test substance stability. The mean post-storage concentrations were 99.7% and 100% of the pre-storage values (Table 5), which met the protocol-specified acceptance requirement for stability *i.e.*, the mean post-storage concentration was not <90% of the pre-storage value.

4.7. TEST SUBSTANCE STABILITY IN PROCESSED SAMPLES

QC samples prepared at nominal test article concentrations of 1.00 and 100 mg/mL were analyzed on 21 June 2011. The processed samples were stored at room temperature for 2 days before being re-analyzed to assess test substance stability. The mean post-storage concentrations were 94.4% and 95.7% of the pre-storage values (Table 5), which met the previously stated protocol-specified acceptance requirement for stability.

4.8. TEST SUBSTANCE HOMOGENEITY AND RESUSPENSION HOMOGENEITY ASSESSMENT OF FORMULATIONS

Duplicate samples from the top, middle, and bottom strata of the formulations prepared on 14 April 2011 at target test substance concentrations of 1 and 100 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 11 and 18 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 11- and 18-day resuspension homogeneity. The results of the homogeneity and resuspension homogeneity analyses are presented in Table 6, Table 7, and Table 8, respectively, with the overall statistics summarized as follows:

Homogeneity Assessment of the 14 April 2011 Formulations

	Low Group (1 mg/mL)	High Group (100 mg/mL)
Mean Concentration (mg/mL)	1.00	95.9
SD	0.018	9.9
RSD (%)	1.8	10
Mean Concentration % of Target	100	95.9

11-Day Room Temperature Resuspension Homogeneity Assessment of the 14 April 2011 Formulations

	Low Group (1 mg/mL)	High Group (100 mg/mL)
Mean Concentration (mg/mL)	1.03	99.8
SD	0.018	0.82
RSD (%)	1.7	0.82
Mean Concentration % of Target	103	99.8

18-Day Room Temperature Resuspension Homogeneity Assessment of the 14 April 2011 Formulations

	Low Group (1 mg/mL)	High Group (100 mg/mL)
Mean Concentration (mg/mL)	0.991	95.4
SD	0.014	1.8
RSD (%)	1.4	1.9
Mean Concentration % of Target	99.1	95.4

The homogeneity assessment of the 14 April 2011 formulations met the protocol-specified acceptance 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 14 April 2011 formulations met the protocol-specified acceptance requirement, *i.e.*, the RSD for the mean concentration was ≤10%.

4.9. TEST SUBSTANCE STABILITY IN FORMULATIONS

The formulations prepared and analyzed on 14 April 2011 were stored at room temperature for 11 and 18 days before being re-analyzed to assess test substance stability. The results of the stability analyses are presented in Table 7 and Table 8. The mean concentrations and percent of time-zero are summarized in the following table.

Storage Condition	Storage Duration	Mean Concentration, mg/mL (% of Time-Zero)	
		Low Group (1 mg/mL)	High Group (100 mg/mL)
Room Temperature	11 Days	1.03 (103)	99.8 (104)
	18 Days	0.991 (98.7)	95.4 (99.4)

The post-storage test substance concentrations ranged from 98.7% to 104% of the pre-storage values, which met the previously stated protocol-specified acceptance requirement for stability.

5. CONCLUSIONS

A GC/FID method for the determination of catalytically cracked slurry oil concentration in acetone formulations and test substance ranging in concentration from 1.00 to 100 mg/mL was validated in this study. Method specificity/selectivity, ruggedness, calibration reproducibility, precision, accuracy, and test substance stability in calibration standards and processed QC samples stored at room temperature for 2 days were assessed and validated, satisfying protocol-specified acceptance criteria.

Formulations prepared at target test substance concentrations of 1 and 100 mg catalytically cracked slurry oil/mL met the protocol-specified acceptance requirement for homogeneity and, after 11 and 18 days of room temperature storage, resuspension homogeneity and stability.

6. REPORT REVIEW AND APPROVAL

Report Approved by:

[redacted]

18 Nov 2011
Date

Research Chemist, Analytical Chemistry
Study Director

Report Prepared by:

[redacted] Chemist,
Analytical Chemistry

Report Reviewed by:

[redacted]

18 Nov 2011
Date

Assistant Director, Analytical Chemistry

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)
25-Apr-2011	Test Article Analysis	25-Apr-2011	27-May-2011	C.Winkler
16-Jun-2011, 17-Jun-2011	Study Record (A-1)	17-Jun-2011	27-Jul-2011	M.Stauffer
28-Jun-2011	Analytical Chemistry Report	28-Jun-2011	27-Jul-2011	M.Stauffer
30-Jun-2011	Study Records (A-1, supplement)	30-Jun-2011	27-Jul-2011	M.Stauffer
05-Jul-2011	Audited Analytical Chemistry Report	05-Jul-2011	26-Aug-2011	M.Stauffer
17-Nov-2011	Final Report	17-Nov-2011	18-Nov-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 [C(97) 186/Final], the WIL Research SOPs, and the protocol and protocol amendments as approved by the Sponsor. Quality Assurance findings, derived from the inspections during the conduct of the study and from the inspections of the raw data and draft report, are documented and have been reported to the Study Director. 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:

[redacted]
Group Supervisor, Quality Assurance

18 NOV 2011
Date

[redacted]
Senior Compliance Specialist

18 Nov. 2011
Date

Report Released by:

[redacted]
Manager, Quality Assurance

18/Nov/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
conc.	-	concentration
DI	-	deionized
DMSO	-	dimethylsulfoxide
EPA	-	Environmental Protection Agency
ESI+	-	positive electrospray ionization
g	-	gram
GLP	-	Good Laboratory Practices
GMP	-	Good Manufacturing Practices
HPLC	-	high performance liquid chromatography
hr	-	hour(s)
IS	-	internal standard
kg	-	kilogram
L	-	liter
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 - 8

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

Concentration (µg/mL)	**500**	**600**	**750**	**850**	**1000**
Set 1	509	607	753	857	1002
(12Apr2011)	494	603	756	836	1003
Ruggedness	489	603	738	850	999
Mean	498	605	749	848	1001
SD	10	2.5	9.9	11	2.1
%RSD	2.0	0.41	1.3	1.3	0.21
%RE	-0.49	0.76	-0.14	-0.25	0.11
Set 2	500	605	746	853	1023
(13Apr2011)	503	592	745	859	1025
	501	599	744	861	946
Mean	501	599	745	858	998
SD	1.4	6.0	0.95	4.0	45
%RSD	0.27	1.0	0.13	0.47	4.5
%RE	0.29	-0.24	-0.68	0.89	-0.22
Set 3	492	610	749	847	994
(14-15Apr2011)	494	615	739	851	1011
	503	601	741	858	996
Mean	496	608	743	852	1000
SD	5.9	7.0	4.9	5.3	9.6
%RSD	1.2	1.2	0.66	0.62	0.96
%RE	-0.76	1.4	-0.93	0.24	0.041
Interset Statistics					
n	9	9	9	9	9
Mean	498	604	746	852	1000
SD	6.4	6.4	6.1	7.6	23
%RSD	1.3	1.1	0.82	0.89	2.3
%RE	-0.32	0.64	-0.58	0.29	-0.021

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Table 2. Back-Calculated Concentrations of the Cross-Validation Calibration Standards

Concentration (µg/mL)	**500**	**600**	**750**	**875**	**1000**
Cross-Validation (14Apr2011)	501	599	748	845	991
	502	606	745	850	1011
	492	607	751	852	1000
Intraset Statistics					
n	3	3	3	3	3
Mean	498	604	748	849	1001
SD	5.6	4.1	2.9	3.4	9.8
%RSD	1.1	0.67	0.39	0.40	0.98
%RE	-0.41	0.66	-0.22	-3.0	0.071

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Table 3. Calculated Concentrations of the Validation Quality Control Samples
Vehicle - Acetone (OmniSolv high purity solvent, 99.8+% pure)

Concentration (mg/mL)	**1.00**	**10.0**	**100**
Set 1	1.03	10.1	101
(12Apr2011)	1.03	10.1	100
Ruggedness	1.04	10.1	100
Mean	1.03	10.1	100
SD	0.0057	0.030	0.64
%RSD	0.56	0.30	0.63
%RE	3.0	0.87	0.46
Set 2	1.00	10.0	101
(13Apr2011)	0.995	9.81	101
	0.989	9.49	102
Mean	0.994	9.77	101
SD	0.0057	0.27	0.21
%RSD	0.57	2.8	0.21
%RE	-0.56	-2.3	1.5
Set 3	0.970	9.57	96.6
(15Apr2011)	0.975	9.79	96.7
	0.970	9.59	98.7
Mean	0.971	9.65	97.4
SD	0.0030	0.12	1.2
%RSD	0.31	1.3	1.2
%RE	-2.9	-3.5	-2.6
Interset Statistics			
n	9	9	9
Mean	0.999	9.84	99.8
SD	0.026	0.25	2.0
%RSD	2.6	2.5	2.0
%RE	-0.14	-1.6	-0.23

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Table 4. Calculated Concentrations of the Cross-Validation Quality Control Samples
Vehicle - Acetone (Min. 99.0+%)

Concentration (mg/mL)	1.00	10.0	100
Cross-Validation (14Apr2011)	0.999	9.71	97.0
	0.991	10.1	99.9
	0.992	14.5*	103
Intraset Statistics			
n	3	2	3
Mean	0.994	9.93	99.9
SD	0.0041	0.30	2.9
%RSD	0.41	3.0	2.9
%RE	-0.60	-0.72	-0.080

*Sample is an outlier and will not be used in statistics

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Table 5. 2-Day Room Temperature Stability Analysis of the
20 June 2011 Calibration Standards And Processed Quality Control Samples

Date Analyzed	Theo. Conc (µg/mL)	Ref # (402029 -)	Run #	Conc (µg/mL)	Percent of Time Zero (%)
Calibration Standards					
21Jun2011	500	68 - 1	262	502	N/A
23Jun2011		68 - 2	287	501	100
21Jun2011	1000	68 - 13	266	1003	N/A
23Jun2011		68 - 13	289	1000	99.7

Date Analyzed	Theo. Conc (mg/mL)	Ref # (402029 -)	Run #	Conc (mg/mL)	Percent of Time Zero (%)	Overall Percent of Time Zero (%)
QC Samples						
21Jun2011	1.00	70 - 2	269	1.02	N/A	94.4
23Jun2011		70 - 2	293	0.970	94.7	
21Jun2011	1.00	70 - 4	271	1.04	N/A	
23Jun2011		70 - 4	295	0.977	94.1	
21Jun2011	100	71 - 1	275	101	N/A	95.7
23Jun2011		71 - 1	299	95.8	94.6	
21Jun2011	100	71 - 2	276	100	N/A	
23Jun2011		71 - 2	300	96.7	96.5	
21Jun2011	100	71 - 3	277	100	N/A	
23Jun2011		71 - 3	301	96.3	95.9	

N/A = Not applicable

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Table 6. Homogeneity Assessment of the 14 April 2011 Formulations
(Analyzed 14 April 2011)

Group	Strata	Dose Conc (mg/mL)	Ref # (402029-)	Run #	Analyzed Conc (mg/mL)	Percent of Target (%)	Mean Conc (mg/mL)	SD	RSD (%)	Mean Conc % of Target (%)
Low	Top	1	18 - 1	113	1.02	102	1.00	0.018	1.8	100
			18 - 2	114	1.01	101				
	Mid	1	18 - 3	115	0.971	97.1				
			18 - 4	116	1.00	100				
	Btm	1	18 - 5	117	1.01	101				
			18 - 6	118	1.01	101				
High	Top	100	19 - 1	119	101	101	95.9	9.9	10	95.9
			19 - 2	120	98.6	98.6				
	Mid	100	19 - 3	121	102	102				
			19 - 4	122	75.8	75.8				
	Btm	100	19 - 5	123	100	100				
			19 - 6	124	98.9	98.9				

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Table 7. 11-Day Room Temperature Resuspension Homogeneity and Stability Assessment of the 14 April 2011 Formulations
(Analyzed 25-27 April 2011)

Group/Strata	Conc. (mg/mL)	Ref # (402029 -)	Run #	Analyzed Conc. (mg/mL)	Percent of Target (%)	Mean Conc. (mg/mL)	SD	RSD (%)	Mean Conc % of Target (%)	Percent of Time Zero (%)
Low/Top	1.00	39 - 1	291	1.05	105	1.03	0.018	1.7	103	103
		39 - 2	292	1.05	105					
Low/Btm		39 - 3	293	1.01	101					
		39 - 4	294	1.02	102					
High/Top	100	40 - 1	295	100	100	99.8	0.82	0.82	99.8	104
		40 - 2	296	99.6	99.6					
High/Btm		40 - 3	297	98.7	98.7					
		40 - 4	300	100	100					

Group	Mean Time-Zero Conc. (mg/mL)
low	1.00
high	95.9

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Table 8. 18-Day Room Temperature Resuspension Homogeneity and Stability Assessment of the 14 April 2011 Formulations
(Analyzed 2 May 2011)

Group/Strata	Conc. (mg/mL)	Ref # (402029 -)	Run # (402023-)	Analyzed Conc. (mg/mL)	Percent of Target (%)	Mean Conc. (mg/mL)	SD	RSD (%)	Mean Conc % of Target (%)	Percent of Time Zero (%)
Low/Top	1.00	56 - 1	232	0.985	98.5	0.991	0.014	1.4	99.1	98.7
		56 - 2	233	0.979	97.9					
Low/Btm		56 - 3	234	1.01	101					
		56 - 4	235	0.990	99.0					
High/Top	100	57 - 1	236	96.0	96.0	95.4	1.8	1.9	95.4	99.4
		57 - 2	237	93.4	93.4					
High/Btm		57 - 3	238	94.4	94.4					
		57 - 4	239	97.5	97.5					

Group	Mean Time-Zero Conc. (mg/mL)
low	1.00
high	95.9

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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.

- **Protocol Section 4.1.7** states that the test substance is to be stored at an ambient temperature, protected from light. However, the retention sample collected on 15 November 2010 was stored in a clear glass vial, therefore not protected from light, until it was wrapped in foil on 20 December 2010.

  Reason for Deviation: Inadvertent technician error. The formulations department was notified of the light protection requirement via email on 23 November 2010.

- **Protocol Section 6.2.2** states that the vehicle to be used is OmniSolv acetone. However, a protocol amendment was written on 12 April 2011 changing the vehicle from OmniSolv acetone to Spectrum acetone. The first and second validation sets were prepared on 12 April 2011 and 13 April 2011 using OmniSolv acetone. On 14 April 2011, the third validation set was prepared using OmniSolv acetone as well as a cross-validation set using Spectrum acetone.

  Reason for Deviation: Technician error.

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

Study Number: WIL-402029

PROTOCOL AMENDMENT 1

Sponsor: American Petroleum Institute

Title of Study:

Analytical Validation and Stability Study of Catalitically Cracked Slurry Oil in Acetone Formulations

Protocol Modifications:

1) **Title:**

   Analytical Validation and Stability Study of Catalytically Cracked Slurry Oil in Acetone Formulations

2) **2.2 WIL Study Director:**

   E-mail: [redacted]

3) **6.2.2 Carrier:**

   Acetone, Min. 99.0+% (2-propanone, CAS# 67-64-1, Spectrum Chemical Mfg. Corp., product code AC115)

Reasons for Protocol Modification:

1) Correction of spelling.

2) Correction of Study Director e-mail address.

3) Modification of source of vehicle (acetone).

Approval:

Sponsor's approval was obtained via e-mail on 4/13/11 ①
Date

WIL Research Laboratories, LLC

[redacted] 12 APR 2011
Study Director Date

[redacted] 12 Apr 2011
Assistant Director, Analytical Chemistry Date

American Petroleum Institute

[redacted] 12-April-2011
Sponsor Representative Date

① This section was filled in per MEH2 4/13/11

PROTOCOL

ANALYTICAL VALIDATION AND STABILTIY STUDY OF CATALITICALLY CRACKED SLURRY OIL IN ACETONE 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 catalytically cracked slurry oil concentration in acetone formulations using gas chromatography (GC) with flame ionization or mass spectrometric detection. Acetone formulations prepared at test substance concentrations of 1.00 and 100 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:

[redacted]
American Petroleum Institute
1220 L Street, NW
Washington, DC 20005
Tel: (202) 682-8344
Email: [redacted]

2.2 WIL Study Director:

[redacted]
Research Chemist, Analytical Chemistry
Tel: (419) 289-8700
Fax: (419) 289-3650
E-mail: [redacted]

2.3 WIL Departmental Responsibilities:

[redacted]
Associate Research Chemist, Analytical Chemistry
Emergency Contact
Tel: (419) 289-8700
Fax: (419) 289-3650
E-mail: [redacted]

[redacted]
President and Chief Operating Officer

[redacted]
Vice President, Analytical, Metabolism, and *In Vitro* Toxicology Services

[redacted]
Assistant Director, Analytical Chemistry

[redacted]
Manager, Quality Assurance

[redacted]
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:

Clarified oils (petroleum), catalytic cracked, commonly referred to as catalytically cracked slurry oil

4.1.2 CAS#:

64741-62-4

4.1.3 CAS Definition:

A complex combination of hydrocarbons produced as the residual fraction from distillation of the products from a catalytic cracking process. It consists of hydrocarbons having carbon numbers predominantly greater than C20 and boiling above approximately 350°C (662°F). This stream is likely to contain 5 wt % or more of 4- to 6-membered condensed ring aromatic hydrocarbons.

4.1.4 Lot Number:

Site 12: Sample 2

4.1.5 Expiration/Retest Date:

Retest in 5 years.

4.1.6 Purity:

100%

4.1.7 Storage Conditions:

Ambient temperature, protected from light.

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:

- Acetone with and without test substance

6 EXPERIMENTAL DESIGN

6.1 Overview of the Study:

Catalytically cracked slurry oil 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 acetone 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:

Acetone (OmniSolv high purity solvent, 99.8+% pure)

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 each carrier 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:

The room temperature (or autosampler temperature if a cooled autosampler proves appropriate and necessary for adequate analyte stability) stability of processed samples will be evaluated over a minimum of 24 hours.

If a significant degradation (>10% reduction in the mean analyte concentration or response from the time zero samples) occurs under the tested conditions, then special precautions should be taken.

6.3.5 Homogeneity, Resuspension Homogeneity, and Stability of Acetone Formulations:

Test substance homogeneity, resuspension homogeneity, and stability in acetone formulations prepared at anticipated test substance concentrations of 1 and 100 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 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 email on 17 MAR 11.
Date

American Petroleum Institute

[redacted] 22-March-2011
Sponsor Representative Date

WIL Research Laboratories, LLC

[redacted] 18 MAR 11
Study Director Date

[redacted] 18 Mar 11
Assistant Director, Analytical Chemistry Date