QUALITY ASSURANCE PROJECT PLAN

Pressurized Hydrocarbon Liquids Sampling and Analysis Study (DJCD-PHLSAS-RFP-0002)

Prepared for:
Noble Energy, Inc. (“Noble”)
1625 Broadway Street, Suite 2200
Denver, CO 80202

Prepared by:
SPL
8820 Interchange Drive
Houston, TX 77054

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Section A – Project Management

A.1 QAPP for Noble Energy, Inc. – Pressurized Hydrocarbon Liquids Sampling and Analysis Study (DJCD-PHLAS-RFP-0002)

Prepared by:

____ Date: _________________________
Diana Bowling, Quality Director
Southern Petroleum Laboratories, Inc.

Approved by:

____ Date: _________________________
Susan Gomez, EHSR Manager
Noble Energy, Inc.

____ Date: _________________________
Joe Landes, Executive V.P. of Technical Services
Southern Petroleum Laboratories, Inc.

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Documents Referenced in the Quality Assurance Project Plan

This Quality Assurance Project Plan ("QAPP") is intended as a complimentary document to be used in conjunction with the *Pressurized HC Liquids Sampling and Analysis Study Work Plan* (the "Work Plan"). Extensive references to those documents are made throughout this QAPP. It is necessary to have the referenced versions of the Work Plan and Uncertainty Analysis Processes readily available when reviewing the QAPP, as many of the tables and references are not repeated throughout the various documents. Additional documents referenced in this QAPP are listed in *QAPP Table 1* below; SPL SOPs are not included in the QAPP and are available by request. The reference methodology (ASTM, GPA documents, etc.) listed in *QAPP Table 1* must be purchased independently by the reader due to licensing and copyright agreements.

Table 1. List of Documents Referenced in the QAPP

ID/Version	Brief Description
Work Plan	PHLSA Study Work Plan
GPA 2198	Selection, Preparation, Validation, Care, and Storage of Natural Gas and Natural Gas Liquids Reference Standard Blends
GPA 2177	Analysis of Natural Gas Liquid Mixtures Containing Nitrogen and Carbon Dioxide by Gas Chromatography
GPA 2186	Method for the Extended Analysis of Hydrocarbon Liquid Mixtures Containing Nitrogen and Carbon Dioxide by Temperature Programmed Gas Chromatography
GPA 2261	Analysis for Natural Gas and Similar Gaseous Mixtures by Gas Chromatography
GPA 2286	Method of Extended Analysis for Natural Gas and Similar Gaseous Mixtures by Temperature Programmed Gas Chromatography
GPA 2103	Method for the Analysis of Natural Gas Condensate Mixtures Containing Nitrogen and Carbon Dioxide by Gas Chromatography
GPA 2174	Obtaining Liquid Hydrocarbon Samples for Analysis by Gas Chromatography
GPA 2166	Obtaining Natural Gas Samples for Analysis by Gas Chromatography
API MPMS 8.1	Standard Practice for Manual Sampling of Petroleum and Petroleum Products (Liquid Sampling – Thief Method)
GPA 2145	Table of Physical Properties for Hydrocarbon and Other Compounds of Interest to the Natural Gas Industry
GPA TP 17	Table for Physical Properties of Hydrocarbons for Extended Analysis of Natural Gases
Yaws, 2005	Yaw's Handbook of Physical Properties, published 2005
ASTM D2887	Boiling Range Distribution of Petroleum Fractions by Gas Chromatography
FLD-L8.1	(SPL SOP) Liquid Sampling
FLD-G14.1	(SPL SOP)Gas Sampling
HY-M10	(SPL SOP) GPA 2261 – C6 Gas Analysis
HY-M9	(SPL SOP) GPA 2286 – Extended Gas Analysis
HY-M6	(SPL SOP) GPA 2177 – C6 Liquid Analysis
HY-M8	(SPL SOP) GPA 2186 – Extended Liquid Analysis
HY-M15	(SPL SOP) GPA 2103 – Condensate Analysis
HY-M33	(SPL SOP) Physical Shrinkage Determination
HY-M32	(SPL SOP) ASTM D2001M / D86M – Distillations for Condensate Analysis
HY-M23	(SPL SOP) ASTM D5001 – Digital Densitometer
HY-13	(SPL SOP) Molecular Weight by Cryette
HY/SM-P15	(SPL SOP) Sample Receipt and Login
QA-P41	(SPL SOP) Validation of New Calibration Standards

A.3 Distribution and Contact List

This section lists all individuals from each organization who require copies of this Quality Assurance Project Plan (“QAPP”) and any subsequent revisions, including all persons responsible for implementation.

Table 2. QAPP Distribution and Contact List

Name	Company	Title	Email	Phone
Joe Landes	SPL	VP Hydrocarbon Services	jlandes@spl-inc.com	713-660-0901 x1242
Jonathan Ochterbeck	SPL	QA Coordinator/Analyst	jochterbeck@spl-inc.com	713-660-0901 x1189
Joel Grice	SPL	GM, Houston/Pleasanton	jgrice@spl-inc.com	713-660-0901 x2207
Meridith Spurlock	SPL	Safety Director	mspurlock@spl-inc.com	713-660-0901 x1255
Diana Bowling	SPL	Quality Director	dbowling@spl-inc.com	713-660-0901 x1181
Royce Miller	SPL	VP Technical Services	rmiller@spl-inc.com	713-660-0901 x1241
Chris Staley	SPL	Lab Manager, Houston/Pleasanton	cstaley@spl-inc.com	713-660-0901 x1188
Tom Benz	SPL	Houston Asst. Lab Manager	tbenz@spl-inc.com	713-660-0901 x1182
Ricky Rayon	SPL	Blending Manager	rrayon@spl-inc.com	713-660-0901 x1171
Bill Theriot	SPL	Technical Director - Allocations	btheriot@spl-inc.com	281-485-8705 x115
Ricardo Aguiar	Movilab	General	ricardo.aguiar@Movilab.com	552-129-3434
Bill Obermann	Noble Energy	EHSR Manager	bill.obermann@nblenergy.com	M: 303-947-1446 D: 720-587-2204
Susan	Noble	EHSR Manager	Susan.gomez@nblenergy.com	303-228-4089

Gomez	Energy			
Alon Mandel	Noble Energy	Env. Engineer	Alon.Mandel@nblenergy.com	M: 303-717-0369 D: 303-228-4053
Scott Patefield	EPA	TAP Member	Patefield.Scott@epa.gov	303-312-6248
Alicia Frazier	CDPHE	TAP Member	Alicia.Frazier@state.co.us	M: 720-253-3382 D: 303-692-3179
Mike Pearson	APT Laboratories	TAP Member	MPearson@airtest.net	303-420-5949
Thomas McGrath	IES	Engineer	tmcgrath.mea@gmail.com	714-315-4040
Alex Casetta	Eagle Automation	Instrumentation Lead	Alex.Casetta@nblenergy.com	970-201-3940
Dave Picard		TAP Member		

Table 3. Results and Reporting Distribution List

Name	Company	Title
Alon Mandel	Noble	Environmental Engineer
Bill Obermann	Noble Energy	EHSR Manager
Susan Gomez	Noble Energy	EHSR Manager
Scott Patefield	EPA	TAP Member
Alicia Frazier	CDPHE	TAP Member
Thomas McGrath	IES	Engineer

A.4 Project/Task Organization

This section identifies the roles and responsibilities for each of the key individuals participating in the project. A summary of this section may be referenced in *QAPP Table 4.*

Joe Landes (SPL), Project Lead – The Project Lead will coordinate and oversee all aspects of the PHLSA study; specific responsibilities will include:

- Final review and approval of the QAPP in terms of program specific requirements.
- Oversee initial consultation and audit for measurement equipment.
- Perform data entry in BRE PSM/EOS software.
- Reviewing reports and ensuring plans are implemented according to schedule.
- Making final project decisions with the authority to commit the necessary resources to conduct the project.

- Reporting to Noble regarding status of PHLSA study and benchmark results and preparing interim and final reports.

Chris Staley (SPL), Assistant Project Lead and Houston /Pleasanton Laboratory Manager:

- Coordinating receipt of samples at the laboratory.
- Selecting the analytical team members.
- Distribute applicable sections of the QAPP and subsequent revisions to members of the analytical team.
- Coordinating the analysis of the sample and laboratory validation data.
- Report any laboratory problems affecting the project data to the Project Lead.
- Will serve as backup for Project Lead for reporting status to Noble, preparing reports, and making final project decisions as needed.

Joel Grice (SPL), Environmental Regulatory Advisor:

- Will remain independent of the groups responsible for data generation.
- Provide technical assistance to the Project Lead regarding environmental regulatory issues.
- Oversee and coordinate field and laboratory activities
- Will serve as backup for Project Lead for review and approval of QAPP.

Meridith Spurlock (SPL), Advisor for Environmental Health and Safety:

- Provide technical assistance to the Project Lead regarding EHS and air quality issues.
- Perform data entry and PSM/EOS sensitivity study with E&P Tank software.

Diana Bowling (SPL), Advisor for Quality Assurance and Quality Control:

- Will remain independent of the groups responsible for data generation.
- Provide technical assistance to the Project Lead regarding QAQC.
- Developing, maintaining, and distributing the QAPP to all personnel as detailed in *QAPP Table 2*.
- Perform laboratory QA audits of the project to verify conformance to QAPP. At this time, no audits are planned; however, Noble may request an audit at any time during the project. If an audit is conducted, results will be reported to the Project Lead who will then submit to Noble.

Royce Miller (SPL), Consulting, Sampling, and Measurement:

- Perform initial audit and consultation for measurement equipment needed for study.

- Select the field technician team members.
- Ensure sampling and measurement procedures are performed per the QAPP.
- Will serve as backup for Project lead for consultation and audit of measurement equipment.
- Calibration of meters and gauges.

Tom Benz (SPL), ASTM Technical Advisor:

- Provide technical assistance to Project Lead regarding ASTM procedures.
- Oversee ASTM analytical procedures and verify they are performed per the QAPP.
- Review and approve data before reporting.
- Perform depentanization of initial samples to obtain hexanes plus “lot” for CRMs.

Jonathan Ochterbeck (SPL), ASTM Analyst and QA Coordinator:

- Assist with analytical data review prior to reporting results.
- Assist with PSM modeling.

Richard Rayon (SPL), Blending Manager:

- Generate Certified Reference Materials (CRMs) in accordance with stated composition for PHLSA study.
- Validate CRMs against alternate vendor’s calibration standards to ensure composition.

Bill Theriot (SPL), Technical Director Allocations:

- Develop PSM model for all PSM/EOS users to implement.
- Develop SOP for set up and data entry into PSM/EOS.
- Perform data entry in VMG software.
- Perform mass balance calculations.
- Serve as backup for Project Lead for data entry in BRE PSM/EOS software.

Ricardo Aguiar (Movilab), Consulting, and Measurement:

- Perform sensitivity studies.
- Perform uncertainty analysis.

Bill Oberman (Noble), EHSR Manager:

- Project oversight.
- Document review and approval.

Susan Gomez (Noble), EHSR Manager:

- Project oversight.
- Document review and approval.

Alon Mandel (Noble), Environmental Engineer:

- Responsible for reviewing QAPP, work plans, and reports.
- Coordination between SPL sampling team, instrumentation, site operations, and instrumentation data analysis.
- Perform calculations in HYSYS and Promax based on SPL's modeling instructions.

Alex Casetta (Eagle Automation), Instrumentation Lead:

- Responsible for installing, calibrating, and operating instrumentations (e.g. process temperature, pressure, and flow rate measurements.
- Data download and management.

Ed Riggs (Noble), Site Operator and System Foreman:

- Well Site Operations.

Scott Patefield, TAP Member:

- Project oversight.
- Document review and approval.

Alicia Frazier, TAP Member:

- Project oversight.
- Document review and approval.

Mike Pearson, TAP Member:

- Project oversight.
- Document review and approval.

Thomas McGrath (IES), Technical Oversight:

- Technical Consultant
- Project Oversight

Table 4. Responsibilities of Key Personnel
(corresponds to "Personnel" Table in Section 5 of Work Plan)

Responsibilities	Leader	Key Personnel	Associated Study Tasks
Project Management	J. Landes	C. Staley J. Ochterbeck	(ALL)
Process measurement skid	R. Miller	J. Landes	*(Measurement), Work Plan Sections 2c and 2g*

design, process measurement uncertainty			
Field sampling	R. Miller	B. Burns A. Hartman	*(Sampling), Work Plan Sections 2a, 2f, 2g*
Analytical Testing	C. Staley	T. Benz J. Ochterbeck	*(Analysis), Work Plan Sections 2b, 2d, 2e, 2f*
CRM Blending	R. Rayon		*(CRM), Work Plan Section 2a*
QAPP, QA/QC	D. Bowling		*(QC), Work Plan Section 2e*
PSM/EOS Modeling, Mass Balance	B. Theriot	J. Landes M. Spurlock	*(Mass Balance), Work Plan Section 2g*
Uncertainty Analysis, Sensitivity Studies	R. Aguiar	R. Aguiar	*(Uncertainty), Work Plan Sections 2a, 2b, 2c, 2d, 2f* *(Data Validation), Work Plan Section 2e* *(Uncertainty & Sensitivity), 2g* *(Data Analyses), 2h*

A.5 Problem Definition/Background

A Consent Decree ("CD") between the United States, the State of Colorado, and Noble Energy, Inc. ("Noble"), Civil Action No. 1:15-cv-00841-RBJ,was entered by the U.S. District Court for the District of Colorado as final judgment on June 2, 2015. This CD addressed volatile organic compound ("VOC") emissions from condensate storage tanks. As part of the settlement with the EPA, Noble Energy has been tasked with conducting a Supplemental Environmental Project ("SEP") "*to improve the reliability of hydrocarbon liquids sampling and analysis procedures, which can be used to estimate emissions and properly size condensate storage tanks' vapor control systems. By identifying techniques that eliminate potential sources of error, the study should facilitate more accurate and reliable sampling results that provide better emission estimates for properly engineering and sizing vapor control systems. This SEP is likely to lead to future emission reductions*."

The purpose of the pressurized hydrocarbon (HC) liquid sampling and analysis (PHLSA) study is described in paragraph 37 of the Consent Decree:

"The purpose of the study is to isolate individual variables of the sampling and analytical methods typically used to obtain information regarding the flash potential and makeup of pressurized hydrocarbon liquids and to identify protocols for determining how these samples can be reliably obtained, handled, and analyzed to produce accurate analytical results for practical application in modeling flashing losses."

Based on this purpose,

- The primary objective of the study is to develop guidelines for the sampling and analysis of pressurized liquid hydrocarbons; and
- The secondary objective of the study is to collect data to support the use of process simulation models to estimate flash gas generation when pressurized hydrocarbon liquids are dumped to atmospheric storage tanks.

Key measurements to achieve these objectives include:

- Pressurized hydrocarbon liquids composition and sample collection performance checks, and
- Flash gas-to-oil ratio (FGOR) by mass balance measurements (i.e., measurement of storage tank inlet and outlet hydrocarbon liquid and gas flows), process simulation software, and laboratory procedures.

A.6 Summary of Project/Task Description

QAPP Table 5 is identical to the “Tasks” table in Section 2 of the Work Plan, which provides an overall summary of the project tasks. This table also references various other tables in the Work Plan detailing sample collection and analyses, associated process measurements, and uncertainty analyses.

Table 5. PHLSA Study Summary(corresponds to “Tasks” Table in Work Plan Section 2)

Work Plan Task	Description
Initial Sampling and Development of Certified Reference Material (CRM) (*Work Plan Section 2a*)	The objectives of this task are to develop a Certified Reference Material, and to evaluate the collection of simultaneous (i.e., multiple sample locations/probes) and sequential (i.e., from a single sample location/probe) pressurized HC liquid samples (i.e., do simultaneous and/or sequential sampling impact the pressurized HC liquids composition). The CRM will be used to evaluate analytical method uncertainty, to validate operational performance checks, to conduct the sample handling perturbation study, and as the CRM for calibrations throughout the PHLSA Study.
Analytical Method Performance and Uncertainty (*Work Plan Section 2b*)	The objective of this task is to define the accuracy, precision, and overall uncertainty of four different methods used for pressurized condensate analyses.

Work Plan Task	Description
Process Measurement Uncertainty Analysis (*Work Plan Section 2c*)	The objective of this task is to minimize process measurement uncertainty so that the data used for mass balance and flashing emission models will be of optimum quality.
Sample Handling Perturbation Study (*Work Plan Section 2d*)	The objective of this task is to collect data to develop guidelines for proper sample handling and to estimate the additional uncertainty produced from improper sample handling.
Operational Performance Checks (*Work Plan Section 2e*)	The objective of this task is to evaluate operational performance checks for sampling and analysis of pressurized condensates.
Sampling Perturbation Study (*Work Plan Section 2f*)	The objective of this task is to collect data to develop guidelines for proper sample collection and to estimate the additional uncertainty introduced from improper sampling techniques.
Three Separator Pressure Range and Seasonal PHLSA Studies (*Work Plan Section 2g*)	The objectives of this task are to 1.) Assess seasonal effects on pressurized condensate sampling and analysis, and modeling of flashing losses (Winter and Summer testing), and 2.) Assess the effect of changes in separator operating pressure on pressurized condensate sampling and analysis, and modeling of flashing losses (Three separator pressure ranges).
Data Analyses (*Work Plan Section 2h*)	The objective of this task is to isolate individual variables of the sampling, sample handling, and analysis process, as well as PSM/EOS calculations, to estimate the uncertainties of each variable and the sensitivity to the mass balance when modeling flashing losses.

A.7 Quality Objectives and Criteria for Measurement Data

A.7.1 Data Quality Indicators (DQIs) and Definitions- Data Quality Indicators (DQIs) to be implemented throughout the PHSLA study include precision, accuracy, representativeness, comparability, and completeness. The definition for each DQI is listed as referenced in *EPA's Guidance for Quality Assurance Project Plans (EPA QA/G-5).*

A.7.1.1 Precision is the measurement of agreement among repeated measurements of the same property under identical or substantially similar conditions.

A.7.1.2 Accuracy is a measure of the overall agreement of a measurement to a known value.

A.7.1.3 Representativeness is a qualitative term that expresses “the degree to which date accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition” (ANSI/ASQC 1995).

A.7.1.4 Comparability is a qualitative term that expresses the measure of confidence that one data set can be compared to another and combined.

A.7.1.5 Completeness is a measure of the amount of valid data needed to be obtained from a measurement system.

A.7.2 Additional Details for Data Quality Indicators (DQIs)

A.7.2.1 Precision – GPA and ASTM methodology implement repeatability criteria to evaluate the precision of two results obtained under identical operating conditions.

A.7.2.2 Accuracy - Depending on the method referenced, either reproducibility criteria or the performance evaluation acceptance criteria is intended to evaluate the accuracy of the results.

A.7.2.3 Representativeness - The Sampling Perturbation Study (*Work Plan Section 2f*) will evaluate various protocols for obtaining pressurized hydrocarbon liquid samples to determine the procedure for obtaining the most representative samples. Once identified, these procedures will be implemented for the Three Pressure Range and Seasonal studies (*Work Plan Section 2g*) to ensure that all samples are obtained in the same manner. To ensure that a representative sample is obtained once the protocols have been established, sampling will be performed in accordance with the specified sampling protocols and sampled in triplicate with additional samples obtained to provide backup samples, if needed, for analysis.

A.7.2.4 Comparability - The use of standard test methods and/or technically accepted methods will assure that data may be comparable to other sources of data. The Sampling Perturbation study (*Work Plan Section 2f*) and the Sample Handling Perturbation study (*Work Plan Section 2d*) will evaluate various protocols for the sampling and laboratory handling procedures. Once the best procedures are identified, these will be implemented for the remainder of the PHLSA study to ensure that all samples are obtained and handled in the same manner.

A.7.2.5 Completeness- The Work Plan specifies the minimum number of samples and analytical measurements needed for satisfying the requirements of the Consent Decree and for the statistical uncertainty analyses. 100% of the samples indicated in the Work Plan must be obtained and analyzed. The use of backup samples may be implemented to meet the completeness requirements if necessary. To calculate the uncertainty associated with each measurement, replicate measurements will be performed.

A.7.3 Operational Performance Checks

A.7.3.1 Operational Performance Checks (*Work Plan Section 2e*) will be established throughout the study to provide a means of comparison to determine acceptance criteria for accepting field samples at the laboratory for testing. These checks will include the Initial Pressure Test (Pressure Verification) and High Pressure Densitometer Bubble Point Verification. Details for the criteria implemented will be provided with the final report submitted for the PHLSA study; additional checks may be developed as determined necessary.

A.7.4 Correlation of Data Quality Indicators (DQIs) and Data Quality Objectives (DQOs)

A.7.4.1 The DQIs are key factors that have been identified to determine the validity of the overall results of the PHLSA study. The DQOs are the acceptance criteria applied to individual analytical results as they are obtained, and are used to measure the performance of the test method as it correlates to those analytical results. It should be noted that not all DQIs and/or DQOs are applicable to the results of all test methods; the *QAPP Tables 6* and *7* below show the correlation of each DQO as it pertains to the key DQIs identified for the PHLSA study. The tables are segregated by GC test methods and ASTM test methods. Additional details for the DQOs are provided in *QAPP Section B.5*.

Table 6. Data Quality Indicators (DQIs) for GC Test Methods

DQI		Precision	Accuracy	Accuracy	Representative	Comparability	Comparability	Completeness
DQO		**Repeatability**	**Reproducibility**	**Performance Evaluation Criteria**	**Recovery**	**Fidelity Plots**	**Control Charts**	**Completeness**
Data Needed from GC Analysis and Supporting Documentation that must be Available	**Blank Checks**	NA	NA	NA	Un-normalized results for each component from blank analysis; analyzed as an unknown sample.	NA	NA	100% of planned QC Checks required; Check should be performed at the beginning of each day samples are analyzed.
	Cal Std and/or CRM	Results from two consecutive analyses from the same instrument under identical operating conditions.	**(1)** Results from analysis; analyzed as an unknown sample (non-calibration run). **(2)** NIST traceable cert. for cal. material stating concentrations for each component.	**(1)** Results from analysis; analyzed as an unknown sample (non-calibration run). **(2)** NIST traceable cert. for cal. material stating concentrations for each component, must include uncertainty.	**(1)** Results from analysis; analyzed as an unknown sample (non-calibration run). **(2)** NIST traceable cert. for cal. material stating concentrations for each component.	**(1)** Results from analysis; analyzed as an unknown sample (non-calibration run). **(2)** NIST traceable cert. for cal. material stating concentrations for each component.	Area counts for each component from analysis.	100% of planned QC Checks required; Checks performed at least once per batch of samples analyzed. Duplicate analyses may be required (as applicable) to meet the precision DQO.
	Samples	NA	NA	NA	NA	NA	NA	100% of planned analyses of samples indicated as non-backup sample in the Work Plan. Backup samples may be used to meet completion requirements.
Criteria		See each GC Test Method DQO Table.	See each GC Test Method DQO Table.	See each GC Test Method DQO Table.	See each GC Test Method DQO Table.	See each GC Test Method DQO Table. Refer to GPA 2198 for additional details.	Western Electric Control Chart Rule Violations as stated in GPA 2198.	

Table 7. Data Quality Indicators (DQIs) for ASTM Test Methods

DQI		**Precision**	**Accuracy**	**Accuracy**	**Accuracy**	**Completeness**
DQO		**Repeatability**	**Reproducibility**	**Calibration Check**	**Equipment Verification**	**Completeness**
Data Needed from ASTM Analysis and Supporting Documentation that must be Available	**Cal Std and/or CRM**	Results from two consecutive analyses from the same instrument under identical operating conditions.	**(1)** Results from analysis; analyzed as an unknown sample **(2)** NIST traceable cert. (or cert. of purity) for check material used, stating relevant properties for each component.	**(1)** Results from analysis; analyzed as an unknown sample **(2)** NIST traceable cert. (or cert. of purity) for check material used, stating relevant properties for each component.	**(1)** Results from verification procedure; **(2)** NIST traceable cert. of piston volume (if applicable).	100% of planned QC Checks required. Requirements for frequency of checks are provided for each test method in the ASTM Test Method DQO Table. Duplicate analyses may be required (as applicable) to meet the precision DQO.
	Samples	Results from two consecutive analyses from the same instrument under identical operating conditions.	NA	NA	NA	100% of planned analyses of samples indicated as non-backup sample in the Work Plan. Backup samples may be used to meet completion requirements. Requirements for frequency of checks are provided for each test method in the ASTM Test Method DQO Table. Duplicate analyses may be required (as applicable) to meet the precision DQO.
Criteria		See ASTM Test Method DQO Table.	See ASTM Test Method DQO Table.	See ASTM Test Method DQO Table.	See ASTM Test Method DQO Table.	

A.8 Special Training Requirements/Certifications

Special training or certifications (in addition to the required initial qualification and any periodic refresher training) for field sampling, analytical services, blending services, PSM/EOS modeling, and/or uncertainty analyses have not been identified as necessary for this project. The supervising manager for each phase will oversee that all technicians involved in the PHLSA study will be up to date in regard to the routine training required for the services being performed. The training information will be maintained in personnel training files at the location where the services are performed, and will be available for review upon request.

A.9 Documents and Records

The QAPP, and any subsequent revisions, will be distributed by the QAQC Advisor as detailed in *QAPP Table 2* to each of the key personnel indicated. Additional copies may be available for distribution within workgroups upon request. Any revisions to the QAPP will be dated and numbered sequentially.

The sampling and measurement records generated for this project will include field data sheets (sample tags), chains of custody (COCs), and verification/calibration, proving, and maintenance records for equipment. Refer to *Work Plan Figure5* for details regarding the minimum information required for the field data sheets. Each field data sheet will be recorded with no more than one sample cylinder per data sheet, and will be used to record all pertinent information regarding the sample collection process (cylinder number, date, and time of sampling). Field measurement data will be electronically captured in Noble's SCADA system and the information will be provided to SPL to populate the remaining fields in the field data sheets. SPL will review the field data sheets prior to shipping samples to the lab, and any discrepancies identified will be reported to Noble to determine if action is required.

SPL's laboratory login department will review the field data sheets upon receipt. Records retained for the analytical services will include shipping documents, COCs, and field data sheets (sample tags) sent from the field services group. Additional records will consist of calibration and/or maintenance information for equipment, reference standard certificates, original results and observations (electronic and/or hard copy as applicable for testing performed), and any additional login information generated. As samples are received in the laboratory they will be logged in and each sample container will be assigned a unique internal identifier. All similar samples received at the same time will be logged under the same work order, with each individual sample being assigned as a fraction number. Samples that are from the same sampling location, but designated as backups will have the same fraction number with letters assigned to indicate the container as a backup. All samples for each work order will be included in a single analytical report, with individual sample fraction results identified in the report. A summary of the calibration and QC data, along with raw analytical data will be included with the final report. The laboratory manager will review all data and associated documentation prior to generating reports and submitting to the Project Lead. Any discrepancies identified will be reported to Noble to determine if action is required.

The blending services group will provide records for receipt and verification of feedstock prior to blending, calibration and/or maintenance information for equipment, and all original data sheets generated during the blending process. Any discrepancies noted during the blending process will be reported to the Project Lead who will discuss with Noble to determine if additional action is required.

The Project Lead will review all field data sheets, analytical reports, and blending data to identify any suspect data to flag and discuss with Noble if necessary. Once approved by the Project Lead, the all data will be sent to Noble and Movilab to begin statistical uncertainty analysis.

SPL will develop the PSM/EOS model and reports, along with required input parameters and procedures for the mass balance and flashing emissions calculations. All PSM/EOS reports along with input parameters used for calculations will be reviewed by SPL to identify any suspect data to flag and discuss with Noble if necessary. Once approved by the Project Lead, the all data will be sent to Noble and Movilab to begin statistical uncertainty analysis.

All source data along with data analysis records will be provided by Movilab from the statistical uncertainty analysis.SPL and Movilab will review to identify any suspect data to flag and discuss with Noble to determine if action is required.

Upon completion of individual perturbation studies, results from each work group will be sent to the Project Lead to review and compile a midterm report to submit to the distribution group identified in *QAPP Table 2.* As the final phases of the overall PHLSA study are completed, the same convention will be followed by submitting all data to the Project Lead to compile the final PHLSA report.

Refer to *Work Plan Section 3* for details of the final PHLSA report deliverable.

A.9.1 Record Retention and Archival

All data and records for this project will be maintained according to SPL's standard policy regarding data retention; therefore, records will be retained for seven years from the date of submitting the final PHLSA report to Noble, unless requested to do otherwise. Key personnel representing each work group identified in *QAPP Table 3* will be responsible for ensuring that all necessary records are submitted to the Project Lead. All hard copy records mentioned in *QAPP Section A.9* will be included in the data archival. All data will be retained and retrievable via storage/archival accommodations and/or electronic means throughout the duration of the archival period.

Section B – Data Generation and Acquisition

B.1 Sampling Process Design – refer to *QAPP Section A.6* and/or *Work Plan Section 2* for discussion of the project tasks.

B.2 Sampling Methods

The following sampling methods will be implemented for this study. Brief descriptions for each of the sampling procedures implemented for this study are detailed below; refer to the referenced documents for additional details if necessary. As the hydrocarbon samples will be transported, the sample containers must meet the specifications of and be labeled and packaged according to the Hazardous Materials Regulations of the Department of Transportation. All procedures must begin with clean, leak free sample containers appropriate for the type of sampling to be performed. SPL will provide the cylinders for all sampling events and will be responsible for ensuring that all cylinders are properly cleaned and inspected prior to use for sampling.

Table 8. List of Sampling Methods

Method Reference	Brief Description
GPA 2174	Liquid Sampling – Piston Cylinder Method
GPA 2174	Liquid Sampling – Displacement Method
GPA 2166	Gas Sampling – Purge, Fill, and Empty Method
GPA 2166	Gas Sampling – Evacuated Container Method
GPA 2166	Gas Sampling – Portable GC Method
API MPMS 8.1	Liquid Sampling – Thief Method

B.2.1 <u>GPA 2174</u> – A hydrocarbon fluid sample will be transferred under pressure from a source into a sample container, using a sample probe designed to secure product from the center one-third of the flowing stream to obtain a representative sample. For floating piston cylinders, it is recommended to use an "inert" charge gas that is not present in the sample, so that a leak in the cylinder itself can be easily detected during the analysis. The use of a natural gas as a back-pressure fluid is not recommended because a leak across the piston may result in compromising the sample with components that were not present at the source.

B.2.1.1 Piston Cylinder Method – This method requires the displacement end of the piston cylinder to be filled with an inert gas to a pressure at least 100psi above the sampling pressure. The product end of the piston cylinder is connected to the sampling source and the sample material is allowed to purge into the cylinder as the pressure is verified not to drop below the original sample pressure (preventing flashing and altering the sample composition). The inert gas is allowed to vent from the displacement end to reduce sample volume to 80% while still verifying that the pressure does not drop below the original sample pressure. This type of sampling method does not require outage or reducing the pressure for transportation.

B.2.1.2 Displacement Method – This method uses single cavity (constant volume) cylinders that have been steam cleaned and water filled with a displacement liquid such as water prior to being placed into service for sampling. The sample cylinder is connected to the sampling source as per GPA 2174 requirements with the cylinder positioned vertically (outage tube upward) over a graduated cylinder. Sample material is allowed to purge into cylinder, while allowing the displacement liquid to drain into the graduated cylinder without allowing the pressure to drop below sampling pressure. This is performed until 80% of the cylinder volume has been displaced into the graduated cylinder. The bottom valve is then opened again to allow the remaining displacement liquid to drain from the sample cylinder.

B.2.2 GPA 2166 – The sampling methods listed for these procedures require that the sampling components are clean and free of contaminants. These methods are designed for sampling natural gas from systems that are at or above the hydrocarbon dew point temperature. Ambient cooling of the sample system can cause heavy hydrocarbons to condense out of the vapor phase. The presence of any condensation in the sample inlet system will cause the sample to be non-representative. Condensation must be avoided in the entire sample system from sample probe to sample cylinder outlet or chromatogram inlet.

Pressure drops due to pressure regulators or flow control devices cause reduction in temperature due to the Joule-Thompson effect which may cause condensation in the sampling system. Therefore, it may be necessary to heat regulators that are used to reduce the sample pressure. Where applicable, flow control devices such as flow control plugs or valves should be located at the outlet of the sample container. These flow-controlling devices can cause cooling due to the Joule-Thompson effect. To thermally isolate this cooling effect from the sample container, the flow control devices should be placed at the outlet end of an extension tube (“pigtail”), which is attached to the outlet of the sample container.

Entrained or free-flowing hydrocarbon liquids are generally collected and accounted for in a condensate recovery system, and are not desired in the vapor phase portion of the accounting system. Other contaminants such as water, oil, etc. are not desirable in the sample container. Since free flowing liquids tend to flow along the bottom of the pipe or, or along the walls of the pipe, it is necessary to have a sample probe extending into the pipe.

B.2.2.1 Purge, Fill, and Empty Method – refer to *QAPP Table 9* the number of cycles required for this procedure. This method is applicable when the temperature of the sample equipment downstream of the sample is maintained above the hydrocarbon dew point throughout the entire sampling process and the source pressure is at least 15psig. A "pigtail" extension tube with the valve on the discharge end must be connected to the outlet valve of the sample container. This arrangement places the temperature drop associated with the Joule-Thomson effect at the end of the extension tube and eliminates the possibility of heavy hydrocarbon condensation in the sample container outlet valve. The sample container is held in a vertical position and the entire apparatus is purged and filled from the sampling point with the sample material at line pressure. The sampling point valve is closed, and then the entire apparatus is depressurized to nearly ambient pressure to complete one fill and empty cycle. The entire fill and empty process must be repeated according to *QAPP Table 9* below. After the appropriate number of purge cycles, the apparatus is filled to line pressure, the valves are closed, and the pigtail extension is removed for sample transportation.

Table 9. Number of Purge Cycles for PF&E Method

Pressure Range (psig)	Purge Cycles
15 – 30	13
30 – 60	8
60 – 90	6
90 – 150	5
150 – 500	4
500+	3

B.2.2.2 Evacuated Container – This method is applicable when source pressure is at least 15psig. It should be noted that most valve styles are not vacuum-tight, and is recommended that the cylinder be evacuated immediately prior to sample procurement. The sample container must be evacuated to a pressure of 1mm Hg or less and installed in a vertical position over the sampling point valve. The sample line should be purged to displace any air and then the sample container valve opened to allow the container pressure to increase to the desired sampling pressure. The sample container is removed and checked for leaks prior to transporting.

B.2.2.3 Portable GC – It may be necessary to use a regulator to reduce the line pressure to the required pressure for the portable or online GC. To counteract the effects of the Joule-Thompson effect it may be necessary to heat trace the regulator and sample tubing.

B.2.3 API 8.1 – Thief Method – This method is designed such that a core type sampling thief, when lowered slowly, is capable of trapping a vertical column of liquid at any selected level below the surface. The closing of the lower end of the tube to trap the sample at the desired level may be achieved by various closure mechanisms. If samples are required at more than one location (refer to *QAPP Table 10*), the sample must be measured out with a graduated cylinder and poured into the sample container. The amount of sample measured will depend on the size of the thief and the tests to be performed.

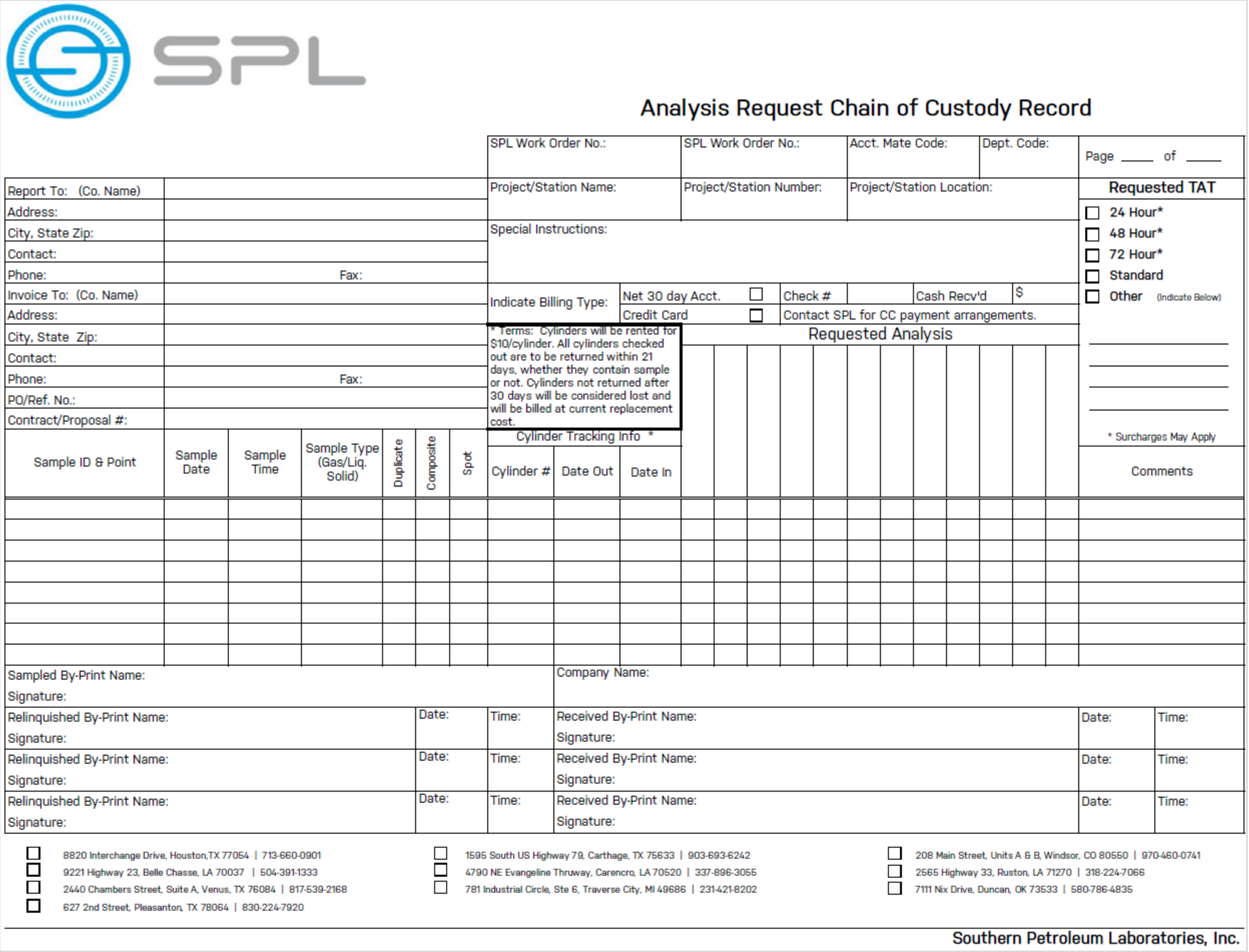
Table 10. Spot Sample Minimum Requirements

Liquid Level	Number of Samples		
	Upper	Middle	Lower
Liquid Level ≤ 3m		X	
3m < Liquid Level ≤ 6m	X		X
6m < Liquid Level	X	X	X

B.3 Sample Handling, Custody, and Preparation

B.3.1 Field Data Sheets (Sample Tags) - The example Field Data Sheet in *Work Plan Figure 5 (Section 2g)* have been designed specifically for the needs of this PHLSA study, and should contain the minimum required information requested on the form.

B.3.2 Chain of Custody (COC)- The COC is intended as a means of physically tracking the sample up to the point where it is submitted to the laboratory for analysis. It serves as a legally defensible document stating the specific date and time that a sample was relinquished to the sample custodian of the laboratory. Additionally, pertinent information for testing, reporting, and invoicing is included on this document. It is SPL's policy that a COC be completed for every sample (or "batch" of samples) relinquished to the laboratory. The example Chain of Custody (COC) *QAPP Figure 1 – SPL Chain of Custody (COC)* is available for use for the PHLSA study.



SPL

Analysis Request Chain of Custody Record

SPL Work Order No.:
SPL Work Order No.:
Acct. Mate Code:
Dept. Code:
Page ____ of ____

Report To: (Co. Name)
Address:
City, State Zip:
Contact:
Phone: Fax:
Invoice To: (Co. Name)
Address:
City, State Zip:
Contact:
Phone: Fax:
PO/Ref. No.:
Contract/Proposal #:

Project/Station Name:
Project/Station Number:
Project/Station Location:
Special Instructions:

Indicate Billing Type: Net 30 day Acct. ☐ Check # Cash Recv'd $
Credit Card ☐ Contact SPL for CC payment arrangements.

* Terms: Cylinders will be rented for $10/cylinder. All cylinders checked out are to be returned within 21 days, whether they contain sample or not. Cylinders not returned after 30 days will be considered lost and will be billed at current replacement cost.

Requested TAT

☐ 24 Hour*
☐ 48 Hour*
☐ 72 Hour*
☐ Standard
☐ Other (Indicate Below)

* Surcharges May Apply

							Cylinder Tracking Info *			Requested Analysis												
Sample ID & Point	Sample Date	Sample Time	Sample Type (Gas/Liq. Solid)	Duplicate	Composite	Spot	Cylinder #	Date Out	Date In													Comments

Sampled By-Print Name:
Signature:
Company Name:

Relinquished By-Print Name: Signature:	Date:	Time:	Received By-Print Name: Signature:	Date:	Time:
Relinquished By-Print Name: Signature:	Date:	Time:	Received By-Print Name: Signature:	Date:	Time:
Relinquished By-Print Name: Signature:	Date:	Time:	Received By-Print Name: Signature:	Date:	Time:

☐ 8820 Interchange Drive, Houston,TX 77054 | 713-660-0901
☐ 9221 Highway 23, Belle Chasse, LA 70037 | 504-391-1333
☐ 2440 Chambers Street, Suite A, Venus, TX 76084 | 817-539-2168
☐ 627 2nd Street, Pleasanton, TX 78064 | 830-224-7920

☐ 1595 South US Highway 79, Carthage, TX 75633 | 903-693-6242
☐ 4790 NE Evangeline Thruway, Carencro, LA 70520 | 337-896-3055
☐ 781 Industrial Circle, Ste 6, Traverse City, MI 49686 | 231-421-8202

☐ 208 Main Street, Units A & B, Windsor, CO 80550 | 970-460-0741
☐ 2565 Highway 33, Ruston, LA 71270 | 318-224-7066
☐ 7111 Nix Drive, Duncan, OK 73533 | 580-786-4835

Southern Petroleum Laboratories, Inc.

Figure 1 - SPL Chain of Custody (COC).

B.3.3 Sample Preparation for Gas Samples – There is no prescribed preservation technique or holding time for samples collected for this analysis. Samples shall be heated prior to testing. Samples collected at temperatures below 120°F shall be equilibrated in the laboratory at 20-50°F above the source temperature of the field sampling. For small sample containers of approximately 300cc's, an equilibration time of approximately 2 hours using a heating blanket or oven is recommended. The time the sample is placed under the heating blanket or in the oven must be documented and retained with the sample records. Due to safety reasons, samples collected above 120°F will not be heated 20°F above collection temperature.

B.3.4 Sample Preparation for Liquid Samples - There is no prescribed preservation technique or holding time for samples collected for this type of analysis. The sample handling perturbation study will investigate various sample handling procedures as part of the baseline study. The normal, routine procedure for sample handling is described below; however, the deviations explored as part of the perturbation study will be documented to determine the best handling practice to implement for the remainder of the PHLSA project.

All samples should be collected, transported and stored prior to analysis in containers and using sampling methodologies that minimize any chance of sample loss or leakage. Normally samples should be stored at ambient temperature prior to analysis. Samples should not be chilled as this may lead to condensation (however, this will be investigated in more detail as part of the Sample Handling Perturbation study). Samples shall not be stored at elevated temperatures due to safety concerns, as liquid samples have a tremendous expansion capability. Immediately prior to analysis, samples shall have a back pressure of a minimum of 200psi over the pressure indicated on the sample tag to ensure that flashing does not occur.

B.4 Analytical Methods

The following analytical methods will be implemented for this study. Due to the nature of testing to be performed, a single/main method reference may be listed in conjunction with a suite of other procedures to obtain physical properties and/or direct measurements needed for calculations and unit conversions required to complete the main method reference. Methods may have an "M" added to the end of the method reference to indicate a modification not listed in the reference method, with the modification detailed in the SPL SOP for that procedure. Brief descriptions for each of the analytical procedures implemented for this study are detailed below; refer to the reference document(s) for additional details. *QAPP Table 11* lists the analytical reference methods used for this study (including methods where a suite of testing is required, but are listed as the Main Method Reference on the COA report).

Table 11. List of Analytical Methods

Main Method References	Associated Testing References	Brief Description of Procedure
Bubble Point Verification	ASTM D4052	Bubble Point Verification
Initial Pressure Test	Proprietary	Initial Pressure Test
GPA 2186 (Extended Liquid Analysis)	GPA 2177	TCD C_6+ Liquid Analysis
	GPA 2186	FID Extended Liquid Analysis
GPA 2286 (Extended Gas Analysis)	GPA 2261	TCD C_6+ Gas Analysis
	GPA 2286	FID Extended Gas Analysis
GPA 2103 (Condensate Analysis)	GPA 2103	TCD C_1-C_6Condensate Analysis
	Proprietary	Physical Shrinkage Determination
	ASTM D2001	Depentanization (C_6+ residue)
	ASTM D86	Naphtha Cut (C_6 – C_{10} cut, C_{11}+ residue)
	Proprietary	Molecular Weight Determination
	ASTM D4052	Digital Densitometer
	ASTM D2887	High Temperature Simulated Distillation
ASTM D323	ASTM D323	Reid Vapor Pressure
ASTM D1945-1946 (Proprietary Refinery Gas Analysis)	ASTM D1945	FID Analysis for C_2–C_6 and Olefin Components
	ASTM D1946	TCD Analysis for C_1–C_2 and Inert Components

B.4.1 HP Densitometer Bubble Point Verification- The high-pressure density measurements by densitometer procedure will not only give a means of QC for calculated density, but also provide a bubble point determination as a QC measure for the calculated bubble point. A sample will be introduced into the high-pressure densitometer and the density will be measured. The pressure will be slowly released in increments with the density measured at each increment of pressure. The resulting plot of measured density vs. measured pressure will have a slope change at the bubble point of the fluid (refer to *Work Plan Figure 4, Section 2e*). The CRM analysis calculated density vs. measured density and CRM analysis calculated bubble point vs. measured bubble point form the baseline for Sampling & Analysis QC. All subsequent analytical results will be validated on this basis to measure the combined uncertainty.

B.4.2 Initial Pressure Test (Pressure Verification) -The Initial Pressure Test is used to estimate the bubble point of a high-pressure liquid sample. It will be performed in the field at the time of sampling prior to outage being taken, and upon receipt of the sample in the lab after re-pressurization and mixing. In the lab, it may also be used to validate the samples prior to analysis to ensure that the sample has not been compromised in transit. If the lab pressure differs significantly from the tag pressure, the sample will be rejected without being analyzed and an alternate or backup sample will be verified and analyzed instead. The lab may also analyze the bubble point by high pressure densitometer and then compare the densitometer result to the field estimate as an additional QC parameter to validate samples prior to analysis.

For single cavity cylinders (constant volume), if a sample tag indicates that 250psi sample has been obtained, back pressure will be applied until the digital pressure gauge attached to the cylinder reads 500psi. This can be achieved by using a sprague pump to pump water into the bottom of a cylinder held in a vertical position. Once the desired pressure has been reached, the Sprague pump should be shut off, and then the valve should be opened to allow a few drops of water to fall out of the cylinder. The resulting pressure (for example now at 259psi) would be the bubble point.

B.4.3 GPA 2186 (Extended Liquid Analysis) - This method covers the compositional analysis of natural gas liquid streams where precise physical property data of the Hexanes and heavier (C_7+) fraction are required by combining the analysis described in GPA 2177 with an extended analysis of the Hexanes and heavier components using capillary gas chromatography according to GPA 2186. This method is applicable for de-methanized mixes through light gasoline fractions that may contain components such as nitrogen, carbon dioxide and/or hydrocarbon complexes (C_1 –C_{14}) that fall within the ranges shown in *QAPP Table 12*, from GPA 2186.

Components to be determined in the hydrocarbon liquid mixture are physically separated by gas chromatography and compared to calibration data previously obtained under identical operating conditions on a mixture of known composition. Two separate fixed volumes of sample in the liquid phase are isolated in suitable sample inlet valves.

Each volume is injected into its corresponding chromatographic system, effectively splitting the analysis into two sections. These two chromatographic systems may be in the form of two different instruments, such as a TCD instrument with a packed column and an FID instrument with a capillary column.

The front end TCD instrument is essentially a GPA 2177 analysis which will analyze from Nitrogen through N-Pentane, with the Hexanes plus (C_6+) components grouped as a single composite peak at the beginning of the chromatogram. This is accomplished by using a precut column, which is back-flushed when N-Pentane has eluted into the main analytical column. The advantages of this method are that better precision is achieved in measuring the Hexanes plus portion of the sample and the analysis time is reduced compared to the single column approach.

The same sample will be injected on to the extended instrument with an FID. Once the raw area counts for the hexanes plus (C_6+) components have been obtained, they are normalized so that each component of hexanes plus from the FID can be allocated to the total hexanes plus determined from the TCD analysis. The FID type detector is effectively a mass sensitive device. With the exception of benzene and toluene, the response (area percent) is proportional to component mass (weight percent). The response factors correct the benzene and toluene peaks so that the determination of weight percent is simply a ratio of component peak area to total peak area multiplied by the hexanes plus weight percent determined in the TCD portion of the analysis.

The molecular weight and density is calculated for the hexanes plus from the FID portion of the analysis, and then used in the calculation of weight percent for the TCD portion, since the molecular weight of the Hexanes plus (C_6+) fraction is an unknown in the TCD analysis. All other compounds from the TCD analysis will use the molecular weights and densities from GPA Standard 2145. The weight percent of the TCD is calculated, and the Hexanes plus in the FID portion is allocated to the weight percent of the Hexanes plus in the TCD portion.

B.4.4 GPA 2186M (Extended Liquid Analysis - modified) - This method is analytically similar to the GPA 2186 described above, however FID portion of the analysis is not normalized before adding to the TCD portion of the analysis. Any difference from 100% will be added to the C10+ component. Also, the molecular weight and density of the C_{10}+ will be physically determined and used in subsequent calculations. The chromatographic data from the GPA 2186 analysis will be used in this modification.

B.4.5 GPA 2103M-C10 (Extended Condensate Analysis) - Due to the nature of this test method, several analytical methods are necessary to directly measure the physical properties to perform the calculations needed to complete the overall analysis. The additional analyses required to calculate the heptanes and heavier (C_7+)fraction include a physical shrinkage determination (modified from API 20.1), de-pentanization (ASTM D2001), an atmospheric distillation (modified ASTM D86), a molecular weight determination(by cryoscopic method), a density by digital density meter (ASTM D4052), and an extended liquid analysis (GPA 2186).

B.4.5.1 GPA 2103 TCD Analysis - This analysis is intended for mixtures containing greater than 20mol% hexanes and heavier (C_6+), or 5 mol% of heptanes and heavier (C_7+) fractions. The 2103 TCD analysis is similar to the 2177 chromatographic analysis, except that the back-flushed heptanes plus is directed to vent out of the chromatographic system instead of onto the column; therefore, this fraction is ignored in the chromatographic analysis. The physical properties of the heptanes and heavier are determined volumetrically by direct measurements.

Table 12. Scope for GPA 2103, GPA 2177, and GPA 2186

Component	Applicable Concentration Ranges		
	GPA 2103 (Mol %)	GPA 2177 (Wt %)	GPA 2186 (Wt %)
Nitrogen	0.01 – 5.0	0.005 – 5.0	0.005 – 5.000
Carbon Dioxide	0.01 – 5.0	0.005 – 5.0	0.005 – 5.000
Methane	0.01 – 40.0	0.001 – 5.0	0.001 – 5.000
Ethane	0.01 – 15.0	0.001 – 95.0	0.001 – 95.000
Propane	0.01 – 15.0	0.001 – 100.0	0.001 – 100.000
Iso-Butane	0.01 – 15.0	0.001 – 100.0	0.001 – 100.000
N-Butane (2,2-Dimethylpropane)	0.01 – 15.0	0.001 – 100.0	0.001 – 100.000
Iso-Pentane	0.01 – 15.0	0.001 – 15.0	0.001 – 50.000
N-Pentane	0.01 – 15.0	0.001 – 15.0	0.001 – 50.000
Hexanes	0.01 – 50.0	0.001 – 15.0	0.001 – 30.000
Heptanes+	5.0 – 80.0	0.001 – 5.0	0.001 – 30.000

B.4.5.2 Physical Shrinkage Determination – This method covers the determination of the physical volume shrinkage of pressurized crudes and condensates from meter conditions to stock tank conditions at atmospheric pressure and ambient temperature. After the TCD portion of the analysis is complete, the sample is physically flashed to atmospheric pressure. Approximately 200cc of liquid sample will be displaced by water into a piston with known volume from the pressurized sample cylinder at ambient temperature and a pressure slightly above sampling conditions. The sample will be slowly flashed from the top of the piston and the remaining liquid portion is collected and measured in a graduated buret. The amount of gas flashed will be measured and a physical shrink factor calculated for the sample material.

B.4.5.3 ASTM D2001 Depentanization - This test method covers the removal of Pentanes and lighter hydrocarbons from gasolines, naphthas, and similar petroleum distillates to prepare samples suitable for further analysis. This test method determines the volume percent of bottoms remaining after depentanization. An aliquot of sample is distilled into an overhead (C_5 and lighter) fraction and a bottoms hexanes and heavier (C_6+) fraction or “cut”. The volume of bottoms is measured and the volume present, based on the charge, is calculated as per ASTM D2001 methodology.

B.4.5.4 ASTM D86M Modified Atmospheric Distillation – This distillation process is performed in the D86 manual distillation apparatus but the procedure is modified to distill only the hexanes and heaver (C_6+) bottom cut from the ASTM D2001 depentanization process described above. Instead of measuring the entire boiling point range of the sample material, the cut off temperature corresponds to a predetermined decane component(C_{10}) temperature in order to produce a bottom cut of components.

B.4.5.5 ASTM D4052 Density by Digital Density Meter – This test method covers the determination of the density, API gravity, and specific gravity of petroleum distillates and viscous oils that can be handled as liquids with vapor pressures below 14.5psi and viscosities below approximately 15,000cSt at the temperature of the test. A small volume of sample liquid is introduced into an oscillating sample tube and the change in oscillating frequency cause by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample. This procedure is performed on the original liquid volume from the shrinkage determination, the hexanes and heaver (C_6+) cut from the depentanization, and the undecanes and heavier (C_{11}+) cut from the additional D86 modified distillation.

B.4.5.6 Molecular Weight by Cryette - This test method covers the calibration and operation of a cryette for the determining the molecular weight of an unknown sample by freeze point depression of a non-aqueous solution of the compound. The instrument is calibrated with a solvent blank and a known calibration standard on each day, prior to analysis of samples and throughout each batch of samples to verify that the calibration has not shifted.

B.4.6 GPA 2103M-C_{30}+ (Modified Extended Condensate Analysis) –the GPA 2103 C_{10}+ analysis listed above may implement an additional modification to extend values out to triacontane (C_{30}+) carbon group values. In these instances, the ASTM D2887M procedure listed below would be performed in addition to those listed above.

B.4.6.1 ASTM D2887M (High Temperature Simulated Distillation) – This is a chromatographic method utilizing an inlet and a capillary column, both of which are subject to a temperature program. An FID is used with a data acquisition system operating in the slice mode. A retention time calibration mixture is used to develop a retention time versus a boiling point curve. A solution of the reference oil is used to determine the response factor. Solvent injections are made and the resulting signal is subtracted from both the response factor standard and the sample chromatogram. Finally, the sample solution is injected and, with the use of the response factor, the amount of sample recovered is calculated. After converting the retention times of the sample slices to temperature, the boiling point distribution can be calculated up to the recovered amount.

Since the FID is essentially a mass sensitive device, the area percent is equivalent to the weight percent of each carbon grouping (sample sliced by carbon number) from the D2887 analysis. This procedure is modified to allocate the weight percent for each carbon number grouping to the total undecanes and heavier (C11+) determined from the GPA 2186 extended liquid analysis.

B.4.7 ASTM D323 (Reid Vapor Pressure) – The procedure implemented for this test method covers the determination of vapor pressure of non-oxygenated gasoline and other volatile petroleum products with a vapor pressure of less than 26psi. The pre-chilled liquid chamber of the vapor pressure apparatus is filled with a sample that is also pre-chilled, and is then connected to the vapor chamber that has been heated to 100°F in a bath. The assembled apparatus is immersed in a bath at 100°F until a constant pressure is observed. The reading, suitably corrected by comparison of the RVP gauge with a pressure measuring device, is reported as the Reid Vapor Pressure.

B.4.8 ASTM D1945-1946 (Proprietary Refinery Gas Analysis) - This test method covers the determination of the chemical composition of reformed gases and similar gaseous mixtures by injecting a gaseous sample into the instrument through separate injection points for analysis by three separate chromatographic systems housed within the same instrument. When the sample is purged through the sample inlet, small aliquots of sample are retained in each valve and the sample is injected simultaneously from each valve and is routed to all columns simultaneously. The first chromatographic system has a separate carrier gas and is used exclusively for the detection of helium and hydrogen via an adsorption column and a TCD type detector. The second system is used to detect carbon monoxide, oxygen, nitrogen, methane, carbon dioxide, ethane, and ethylene components via a partition column and TCD type detector. The third system will analyze the olefin (alkene) components and C_2-C_6 components, and requires an alumina PLOT column and FID type of detector. The composition of the components determined by the TCD detectors are calculated by comparison of the response of each component in the sample to the corresponding value of that component in the reference or calibration standard. The weight percent of the components determined from the FID-type detector will be obtained after bridging to TCD components and subsequent normalization. Calculations and reports for this analysis will use the physical properties from GPA Standard 2145 for all components detected.

B.5 Quality Control –Analytical Procedures and Requirements for Data Quality Objectives (DQOs)

B.5.1 Data Quality Objectives (DQOs) - Provided below are brief descriptions of the laboratory quality control procedures implemented for the PHLSA study, information regarding applicability and/or limitations of each DQO, and an explanation for interpreting the results. Additional details and information regarding the quality control procedures for the laboratory may be referenced in the method SOP's and are available upon request. The DQO Tables in *QAPP Section B.5*showthe QC procedures applied to each of the analytical methods and lists the calculations and specific criteria used for each DQO.A summary of the results of the quality control performed for each phase of testing will be provided with the analytical results in the final PHLSA report; additional details and supporting documentation of the quality control performed will be available a part of the QC report submitted with the final PHLSA report.

B.5.2 Explanation of "Pass/Fail/Review" Evaluations Used for DQOs

B.5.2.1 Each data quality objective applied will be listed with either a "Pass/Fail" or a "Pass/Review" type of evaluation in the QC Reports to provide a means of differentiating and interpreting the various DQOs; additional details are provided for the "Pass/Fail/Review" evaluation applied to each DQO below. Overall, if method specified criteria is published and available, a "Pass/Fail" evaluator will be listed on the QC Report. The "Pass/Review" evaluator will be used in various other instances such as when the criteria from one test method is substituted for another test method, in cases where no published or method specified criteria is available, or where the method criteria provides guidance but does not list exact specifications or requirements.

B.5.2.2 The “Fail” or “Review” evaluation shown on the QC Report also provides a means of gauging the severity of any suspect data that is flagged on a QC Report. A “Fail” shows deviation from definitive data quality objectives specified by the reference method and will carry more weight for the suspect data to be investigated, whereas a “Review” may be interpreted as a warning to monitor data for trends before a major problem arises.

B.5.2.3 Additionally, it should be noted that for the purposes of simplification, the “Pass/Fail” evaluations listed on the QC summaries provided with the final results may be listed as a “% Pass” on the QC summary reports to provide a more accurate description in cases where the DQO is applied to multiple components (i.e., for a 10-component calibration blend, if 8 components pass the DQO but 2 components fail the DQO, an “80% Pass” evaluator will be listed rather than a “Fail”, which would imply 100% failure for all components).

B.5.3 Repeatability DQO

B.5.3.1 Description of DQO - Repeatability criteria provides a means to determine if an instrument can produce a repeatable response by comparing the results of two consecutive analyses. GPA defines it as “the expected precision within a laboratory using the same equipment and same analyst, and represents the difference in analyzed values between two sequential runs.” The two analyses must be obtained under identical operating conditions with identical test material.

B.5.3.2 Applicability and Limitations - Repeatability may be applied to the analyses of a calibration standard and/or sample material, provided enough sample material is available to perform the duplicate analysis. For some test methods, the nature of the testing prevents the same sample material from being analyzed consecutively, as the original sample composition may be permanently altered as part of the normal testing process.

B.5.3.3 “Pass/Fail/Review” QC Evaluation – The Repeatability DQO is gauged with a “Pass/Fail” type of evaluation for the test methods that have published criteria available. It should be noted that the GPA 2103 test method does not have published criteria available and GPA 2177 criteria is implemented for comparison purposes throughout the PHLSA study. As the composition of materials analyzed for GPA 2103 are outside the scope of GPA 2177, a “Pass/Review” type of evaluator would the most appropriate for gauging the repeatability DQOs for the GPA 2103 analysis. However; the LIMS used to generate the QC reports does not differentiate between the test methods that the formulas are used for, and will automatically generate the QC reports using a “Pass/Fail” evaluator for the repeatability DQO of the GPA 2103 analysis.

B.5.4 Reproducibility DQO

B.5.4.1 Description of DQO - Overall, reproducibility is used to determine if an instrument can produce reliable and accurate results by comparing the analysis of calibration standard to the results listed on the certificate for that standard; however, GPA and ASTM methodology have slight differences in how the criteria is implemented. Reproducibility is defined by GPA as the difference between two analyzed values by different laboratories using different equipment and different analysts, but identical operating conditions. For GPA test methods, reproducibility does not represent the difference between an analyzed value and the certified value listed on a blend; however, in cases where the uncertainty of the blend is not known, reproducibility will be implemented in lieu of the performance evaluation criteria (see below). ASTM methodology implements reproducibility to compare the results of a calibration standard or certified reference material to the values listed on the certificate of the standard or reference material.

B.5.4.2 Applicability and Limitations - As implemented for the purposes of the PHLSA study, reproducibility criteria are only applied to the analyses of calibration standards and/or certified reference materials; it is not applicable to the analyses of sample materials.

B.5.4.3 “Pass/Fail/Review” QC Evaluation - Reproducibility is gauged with a Pass/Fail type of indicator for the test methods that have published criteria available. It should be noted that the GPA 2103 test method does not have published criteria available and GPA 2177 criteria is implemented for comparison purposes throughout the PHLSA study. As the composition of materials analyzed for GPA 2103 are outside the scope of GPA 2177, a “Pass/Review” type of evaluator would be the most appropriate for gauging the reproducibility DQOs for the GPA 2103 analysis. However; the LIMS used to generate the QC reports does not differentiate between the test methods that the formulas are used for, and will automatically generate the QC reports using a “Pass/Fail” evaluator for the reproducibility DQO of the GPA 2103 analysis.

B.5.5 Performance Evaluation Acceptance Criteria DQO

B.5.5.1 Description of DQO - The performance evaluation criteria is the true means by which GPA test methods will determine if an instrument can produce reliable and accurate results. GPA defines this as “the ability of an instrument to match the certified values from a gravimetric blend”. The uncertainty of the blend must be known, as both the reproducibility criteria and the uncertainty of the blend are used to calculate the performance evaluation criteria.

B.5.5.2 Applicability and Limitations - ASTM methods do not implement this type of DQO. The performance evaluation DQO is only applied to the GC analyses of calibration standards and/or certified reference materials; it is not applicable to the analyses of sample materials.

B.5.5.3 “Pass/Fail/Review” QC Evaluation - Performance evaluation criteria is gauged with a “Pass/Fail” type of evaluator for the test methods that have published criteria available. It should be noted that the GPA 2103 test method does not have published criteria available and GPA 2177 criteria is implemented for comparison purposes throughout the PHLSA study. As the composition of materials analyzed for GPA 2103 are outside the scope of GPA 2177, a “Pass/Review” type of evaluator would be the most appropriate for gauging the reproducibility DQOs for the GPA 2103 analysis. However; the LIMS used to generate the QC reports does not differentiate between the test methods that the formulas are used for, and will automatically generate the QC reports using a “Pass/Fail” evaluator for the performance evaluation DQO of the GPA 2103 analysis.

B.5.6 Recovery DQO (Calibration Standards for GC Test Methods)

B.5.6.1 Description of DQO- For analysis of the calibration standard, the recovery criterium is implemented to provide a means of monitoring the calibration of the instrument as well as verifying that a repeatable sample volume is introduced into the GC. The recovery is considered to be the un-normalized sum of all components when the standard is analyzed as an unknown sample (i.e., a non-calibration run such that the un-normalized sum will not be equal to the normalized sum). The un-normalized sum is compared against the calibration amount for the test method analyzed.

B.5.6.2 Applicability and Limitations - The GPA 2261 and GPA 2177 test methods calibrate to 100% of the sample composition. The GPA 2103 analysis does not calibrate for the heptanes plus (C_7+) components, therefore the calibration amount will vary based on the composition of each blend.

B.5.6.3 “Pass/Fail/Review” QC Evaluation - For the analysis of a calibration standard, the recovery DQO criteria are gauged with a “Pass/Review” evaluation of the QC data.

B.5.7 Recovery DQO (Instrument Blanks for GC Test Methods)

B.5.7.1 An instrument blank is performed to verify that the analytical system is free from residual sample material and/or contaminants that would interfere with the results of an analysis. The blank check consists of the analysis of a component free matrix such as the carrier gas of the instrument. In this case, the recovery is defined to be the un-normalized result of each component.

B.5.7.2 This DQO is only applicable for the hexanes plus (C_6+) and heptanes plus (C_7+)GC test methods implemented on thermal conductivity detectors (TCDs). The extended analysis test methods implemented on flame ionization detectors (FIDs) do not require calibration and the analyst must visually gauge the raw instrument response to determine if the analytical system is free of residual sample material.

B.5.7.3 “Pass/Fail/Review” QC Evaluation - For the instrument blank checks, the recovery DQO criteria are gauged with a “Pass/Review” evaluation of the QC data.

B.5.8 Recovery DQO (Analysis of Unknown Sample for GC Test Methods)

B.5.8.1 For analysis of an unknown sample, the recovery may be used as a rough means of comparing how similar the compositions are between the calibration standard and the sample analyzed. In this case, the recovery is referring to the un-normalized sum of all components detected from the analysis of the unknown sample relative to the calibration amount.

B.5.8.2 The composition of the calibration standard must be selected such that it resembles the composition of the samples expected to be analyzed and a sample recovery that differs greatly from that of the calibration standard may be an indication that the composition of the calibration standard is not representative of the composition of the sample that was analyzed. This similarity comparison is only applicable for the hexanes plus (C_6+) and heptanes plus (C_7+) GC test methods implemented on thermal conductivity detectors (TCDs). For purposes of the PHLSA study, the composition of the calibration standards may be intentionally selected with varying compositions as a predetermined investigation and is expected due to the nature of this project.

B.5.8.3 “Pass/Fail/Review” QC Evaluation - For the analysis of a calibration standard and the instrument blank checks, the recovery DQO criteria are gauged with a “Pass/Review” evaluation of the QC data. The recovery comparison implemented with the analysis of an unknown sample is a simple ballpark observation to assist with troubleshooting if needed, and no definitive “Pass/Fail/Review” type of evaluator is required.

B.5.9 Fidelity Plot DQOs

B.5.9.1 Description of DQO - Fidelity plots are log-log plots of the molecular weight vs. the response factor of the instrument for each component in the reference blend. The plot provides a means for comparison of the actual vs. expected response relative to the concentration of each component stated on the certificate. The molecular weight increases incrementally for the normal alkane components, and a linear response of the TCD type detector should provide a proportionate response for each of those components (regardless of the actual concentration of the components). The Pearson Correlation Coefficient (R^2 Value) is determined for the normal alkane series, with a 1.0 representing a linear response of the instrument. A non-linear fidelity plot may indicate a potential problem with the GC or with the calibration standard. For an instrument that has been previously verified to be in control, the plot provides a means for determining if the standard is suitable for service. Prior to placing the standard in service, the composition of the standard must be verified to match the values stated on the certificate for the blend. Once placed in service, the fidelity plots monitor the condition of the standard to verify that it has not become compromised over time. For example, if the standard is not stored or handled properly and the lighter components are allowed to flash out of the blend, the composition of the standard is permanently altered and the standard may no longer be used for instrument calibrations. GPA 2198 should be referenced if needed to provide additional details and information regarding fidelity plots.

B.5.9.2 Applicability and Limitations-Fidelity Plots are applicable to calibration standards analyzed by the C_6+ and C_7+ GC test methods implemented on thermal conductivity detectors (TCDs). As the fidelity plots are dependent on a stable and linear instrument response, all components must be plotted from the same detector. This DQO is not applicable to test methods that implement multiple columns and detectors to analyze the complete sample such as the portable GCs and the ASTM D1945-1946 methods. Additionally, the components plotted must have a linear correlation between the molecular weight; therefore, the plot is not applicable to components with identical molecular weights such as isomers.

B.5.9.3 “Pass/Fail/Review” QC Evaluation - There are no published criteria available from GPA for the fidelity plot DQOs; therefore, the criteria implemented is gauged with a “Pass/Review” type of evaluator.

B.5.10 Control Charts DQO

B.5.10.1 Description of DQO - Control charts are a statistical tool used to provide a means for distinguishing a pattern of normal, random variations of the instrument response. The charts are generated to monitor both the instrument as well as the condition of the calibration standard over a period of time. Individual charts must be maintained for each component in each reference standard, for each detector the standards are consistently analyzed on. A maximum of 20 data points of history will be plotted on the control charts for each component, along with the average of the most recent 20 data points, and ±1 standard deviations, ±2 standard deviations, and ±3 standard deviations. As new data from current analyses are added, the older data in the chart is replaced by the FIFO (first in, first out) method. Trends are identified and flagged using the *Western Electric Control Chart Rule Violations* listed in GPA 2198, as stated below.

- 4 of 5 consecutive points above or below the ± 1 Standard Deviation Limits (± 1σ).
- Any point outside of the Control Limits (± 3σ).
- 2 of 3 points outside the Warning Limits (± 2σ).
- 7 consecutive points, all above or all below the average
- 8 consecutive points within ± 1 Standard Deviation Limits (± 1σ).
- 5 consecutive points moving in the same vertical direction, either increasing or decreasing

B.5.10.2 Applicability and Limitations - Control charting is applicable to all components for nearly all GC test methods used throughout the PHLSA study, provided that the operating conditions, instrument response, and the composition of the calibration standard must all remain stable throughout the duration of the data plotted. If operating conditions change and/or outlier data is included, the control chart will not be useful in flagging data trends or to diagnose potential problems. If a new standard is placed in service that differs in composition from the previous standard, or if major maintenance is performed that results in a change of instrument response, new charts must be started for all components for that instrument. Additionally, control charts must be generated with consistent data inputs over the period of time that the data is plotted. Charts that are generated with infrequent or inconsistent data inputs will not provide a true indication of the normal variation of instrument response, and will not be useful in flagging data trends (i.e., if large periods of time elapse without data input, or if large amounts of data are added within a small amount of time).

B.5.10.3 "Pass/Fail/Review" QC Evaluation – Control charts provide a means to focus on data trends that have the potential to indicate minor problems with an instrument or calibration standard, before the problem develops into a more severe problem. Flagged data does not necessarily indicate that the analysis is out of control and/or that corrective actions are necessary. GPA 2198 specifies the control chart rule violations that should be used to monitor and flag trends and/or suspect data; however, there are no requirements or guidelines for gauging the severity of the flagged data. Therefore, the criteria implemented for the control chart rule violations are gauged with a "Pass/Review" type of evaluator. An analyst that is familiar with the day to day operation and maintenance of the instrument, how long a particular standard has been in service, and the normal deviation of the instrument response may use their discretion in the interpretation of any flagged data.

B.5.11 Equipment Verification DQO (Physical Shrinkage Determination)

B.5.11.1 Description and Applicability of DQO- Equipment verifications are performed to verify that the equipment used for analyses have not been damaged and are suitable for continued service. The physical shrinkage determination uses a water draw procedure to compare the volume within the piston to the NIST traceable volume stated on the corresponding certificate for that piston.

B.5.11.2 Applicability and Limitations - This DQO is only applicable to the physical shrinkage determination.

B.5.11.3 "Pass/Fail/Review" QC Evaluation - The physical shrinkage determination does not have published criteria available and this DQO is gauged with a "Pass/Review" type of evaluator.

B.5.12 Equipment Verification DQO (Reid Vapor Pressure)

B.5.12.1 Description and Applicability of DQO- Equipment verifications are performed to verify that the equipment used for analyses have not been damaged and are suitable for continued service. The ASTM D323 RVP test method specifies a procedure to verify the accuracy of the RVP gauges used for the analytical testing process. After the sample analysis is complete, the RVP gauge pressure is compared against a manometer with an equal amount of pressure applied.

B.5.12.2 Applicability and Limitations - This DQO is only applicable to the Reid Vapor Pressure analysis.

B.5.12.3 "Pass/Fail/Review" QC Evaluation - The ASTM D323 Reid Vapor Pressure (RVP) method published criteria is implemented for the verification; however, a "Pass/Review" type of evaluator is used to gauge this DQO criterium since the method does not specify the frequency of the verifications and/or provide guidelines regarding when to remove a gauge from service. This verification is performed with each sample analyzed and is implemented as a means to monitor the accuracy of each gauge in service. Experience has shown that a particular sample result may result in a "Review" QC evaluation of the

RVP gauge used for one analysis, but may result in a "Pass" once the gauge is properly cleaned and used for the next sample analysis. Gauges that frequently result in a "Review" may be removed from service at the discretion of the laboratory supervisor. Additionally, it should be noted that regardless of the resulting "Pass/Review" evaluation, all final results are reported from the manometer reading and not the RVP gauge reading.

B.5.13 Calibration Check DQO

B.5.13.1 Description of DQO–The calibration check DQO is performed to verify the calibration of a balance. On each day that the balance is used, NIST traceable weights will be used to verify the calibration across the working range of the balance; and the weights are selected such that verifications are performed at approximately 1%, 50%, and 100% of the balance capacity. The balance result for each weight is then compared against the corresponding NIST value stated on the certificate for that weight. This DQO is a separate requirement from the annual verifications/calibrations that are provided by an external vendor for the NIST traceability of the balance.

B.5.13.2 Applicability and Limitations - This DQO is only applicable for use to verify the calibration of a balance.

B.5.13.3 "Pass/Fail/Review" QC Evaluation - The balance acceptance limits published for ANSI/ASTM class I weights are implemented as the balance calibration DOQs criteria; therefore, a "Pass/Fail" type of evaluator is used.

B.5.14 Data Quality Objectives (DQOs) for Each Analysis

The DQO tables below provide additional details for each DQO as it pertains to each test method and, if applicable, each component in that test method. These tables also include the formulas used to determine the criteria along with brief definitions and notes that are helpful to understand how the calculations are applied. For ease of reference, the DQO Tables are grouped similarly to the DQI Tables in *QAPP Section A.7*, and are also segregated by GC and ASTM testing. However, to provide the detail needed, each GC test method has an individual table listing the DQOs for each GC test method. All of the ASTM test methods are grouped in a single table.

Table 13. Applicable concentration ranges for GPA 2261 (C6 Gas Analysis) and GPA 2177 (C6 Liquid Analysis) Precision Criteria

Component	2261 Range (Mol %)	2177 Range (Mol %) (*also implemented for GPA 2103)
Nitrogen	0.02 - 15	*0.01 – 5.0
Methane	50 - 100	*0.01 – 5.0
Carbon Dioxide	0.2 – 15	*0.01 – 5.0
Ethane	0.2 - 15	*0.01 – 95.0
Propane	0.2 - 15	*0.01 – 100.0
Iso-Butane	0.2 - 8	*0.01 – 100.0
N-Butane	0.2 - 8	*0.01 – 100.0
Iso-Pentane	0.2 – 8	*0.01 – 15.0
N-Pentane	0.2 - 4	*0.01 – 15.0
Hexanes Plus	0.2 - 2	0.01 – 15.0

Table 14. Data Quality Objectives (DQOs) for GPA 2103 C7+ Condensate Test Method[a]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)[b]	Performance Evaluation Criteria (PE)[c]	Method Repeatability (r)[b]	Fidelity Plot	Control Charts
Blank Check	**Result** ≤ 0.01 LV %	-	-	-	-	-	-
Nitrogen	-	**Sum** ± 5% of Cal Amt	$R = 0.8*\mathbf{X}^{(2/7)}$	$PE = X \pm (U^2 + R^2)^{(1/2)}$	$r = 0.1233*X^{(1/3)}$	N Alkane Series $R^2 > 0.990$	Area counts plotted for each component with average, ± 1, ±2, and ± 3 standard deviations.
Methane							
Carbon Dioxide							
Ethane							
Propane							
Iso-Butane							
N-Butane							
Iso-Pentane							
N-Pentane							
DQO Notes	**Result** is the un-normalized concentration of each component detected from analysis of a component free matrix, such as the carrier gas.	**Sum** is the sum of all un-normalized concentrations of a standard. **Cal Amt** refers to the total concentration that the method is calibrated for. GPA 2103 does not calibrate for the C6+ components.	**X** is component concentration listed on the certificate of the blend.	**U** is the uncertainty of blend component. **R** is Reprod for component. **X** is component concentration listed on the certificate of the blend.	**X** is the component concentration of run 1 out of 2 consecutive runs.	**R^2** is the Pearson Correlation Coefficient indicating a measure of the linear correlation between the two variables (in this case, molecular weight vs. instrument response for each component).	Trending of each component is monitored as per GPA 2198.

a. All DQOs apply to all components, QC checks must be performed at least once per batch of samples analyzed.

b. No GPA published criteria available for GPA 2103, therefore GPA 2177-13 Reproducibility (R) and Repeatability (r) criteria are applied for comparison purposes. GPA 2177 Reproducibility and Repeatability criteria may be applied in any unit of concentration, but is listed here in LV%.

c. If the Uncertainty (U) of the calibration blend is known, the Performance Evaluation (PE) criteria will be used in lieu of Reproducibility (R).

Table 15. Data Quality Objectives (DQOs) for GPA 2177 C6+ Liquids Test Method[d]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)[e]	Performance Evaluation Criteria (PE)[f]	Method Repeatability (r)[e]	Fidelity Plot	Control Charts
Blank Check	**Result** ≤ 0.01 LV %	-	-	-	-	-	-
Nitrogen	-	**Sum** ± 5% of Cal Amt	R = 0.8*$\mathbf{X}^{(2/7)}$	PE = $X \pm (U^2 + R^2)^{(1/2)}$	r = 0.1233*$X^{(1/3)}$	N Alkane Series $R^2 > 0.990$	Area counts plotted for each component with average, ± 1, ±2, and ± 3 standard deviations.
Methane							
Carbon Dioxide							
Ethane							
Propane							
Iso-Butane							
N-Butane							
Iso-Pentane							
N-Pentane							
Hexanes Plus							
DQO Notes	**Result** is the un-normalized concentration of each component detected from analysis of a component free matrix, such as the carrier gas.	**Sum** is the sum of all un-normalized concentrations of a standard. **Cal Amt** refers to the total concentration that the method is calibrated for. GPA 2177 is calibrated for 100% of sample components present.	**X** is component concentration listed on the certificate of the blend.	**U** is the uncertainty of blend component. **R** is Reprod for component. **X** is component concentration listed on the certificate of the blend.	**X** is the component concentration of run 1 out of 2 consecutive runs.	**R²** is the Pearson Correlation Coefficient indicating a measure of the linear correlation between the two variables (in this case, molecular weight vs. instrument response for each component).	Trending of each component is monitored as per GPA 2198.

d. All DQOs apply to all components, QC checks must be performed at least once per batch of samples analyzed.

e. GPA 2177-13 Reproducibility (R) and Repeatability (r) criteria applied; may be used in any unit of concentration, but is listed here in LV%.

f. If the Uncertainty (U) of the calibration blend is known, the Performance Evaluation (PE) criteria will be used in lieu of Reproducibility (R).

Table 16. Data Quality Objectives (DQOs) for GPA 2186 Extended Liquids Test Method[g]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)	Performance Evaluation Criteria (PE)	Method Repeatability (r)	Fidelity Plot	Control Charts
Blank Check	Baseline visually gauged	-	-	-	-	-	-
2,2-Dimethylbutane	-	-	-	-	-	-	Area counts plotted for each component with average, ± 1, ±2, and ± 3 standard deviations.
2,3-Dimethylbutane							
2-Methylpentane							
3-Methylpentane							
N-Hexane							
Benzene							
N-Heptane							
Toluene							
N-Octane							
Ethylbenzene							
M, P-Xylene							
O-Xylene							
N-Nonane							
N-Decane							
DQO Notes	Instrument is not calibrated; therefore, baseline is visually gauged to verify that the instrument does not contain residual sample material from previous analyses.	-	-	-	-	-	Trending of each component is monitored as per GPA 2198.

g. No GPA published criteria available for this test method, QC checks must be performed at least once per batch of samples analyzed.

Table 17. Data Quality Objectives (DQOs) for GPA 2103 Extended Condensate (C6-C10 Cuts) Test Method[h]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)	Performance Evaluation Criteria (PE)	Method Repeatability (r)	Fidelity Plot	Control Charts
Blank Check	Baseline visually gauged	-	-	-	-	-	-
2,2-Dimethylbutane	-	-	-	-	-	-	Area counts plotted for each component with average, ± 1, ±2, and ± 3 standard deviations.
2,3-Dimethylbutane							
2-Methylpentane							
3-Methylpentane							
N-Hexane							
Benzene							
N-Heptane							
Toluene							
N-Octane							
Ethylbenzene							
M, P-Xylene							
O-Xylene							
N-Nonane							
N-Decane							
DQO Notes	Instrument is not calibrated; therefore, baseline is visually gauged to verify that the instrument does not contain residual sample material from previous analyses.	-	-	-	-	-	Trending of each component is monitored as per GPA 2198.

h. No GPA published criteria available for this test method, QC checks must be performed at least once per batch of samples analyzed.

Table 18. Data Quality Objectives (DQOs) for GPA 2261 C6+ Gas (Benchtop GC) Test Method[i]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)[j]	Performance Evaluation Criteria (PE)[k]	Method Repeatability (r)[j]	Fidelity Plot	Control Charts
Blank Check	Result ≤ 0.01 Mol%	-	-	-	-	-	-
Nitrogen	-	Sum ± 5% of Cal Amt	$R = 0.158*X^{(1/2)}$	$PE = X \pm (U^2 + R^2)^{(1/2)}$	$r = 0.039*X^{(1/4)}$	N Alkane Series $R^2 > 0.990$	Area counts plotted for each component with average, ± 1, ±2, and ± 3 standard deviations.
Methane			$R = 91000*X^{(-3)}$		$r = 0.0079*X^{(1/3)}$		
Carbon Dioxide			$R = 0.12*X^{(1/3)}$		$r = 0.0042*X^{(1/3)}$		
Ethane			$R = 0.0315*X^{(1/3)}$		$r = 0.0124*X^{(1/3)}$		
Propane			$R = 0.026*X^{(1/2)}$		$r = 0.0084*X^{(1/8)}$		
Iso-Butane			$R = 0.018*X^{(1/2)}$		$r = 0.01*X^{(1/5)}$		
N-Butane			$R = 0.033*X^{(1/2)}$		$r = 0.0117*X^{(2/5)}$		
Iso-Pentane			$R = 0.025*X^{(1/4)}$		$r = 0.009*X^{(1/4)}$		
N-Pentane			$R = 0.026*X^{(1/3)}$		$r = 0.01*X^{(1/5)}$		
Hexanes Plus			$R = 0.051*X^{(1/2)}$		$r = 0.0135*X^{(1/4)}$		
DQO Notes	**Result** is the un-normalized concentration of each component detected from analysis of a component free matrix, such as the carrier gas.	**Sum** is the sum of all un-normalized concentrations of a standard. **Cal Amt** refers to the total concentration that the method is calibrated for. GPA 2261 is calibrated for 100% of sample components present.	**X** is component concentration listed on the certificate of the blend.	**U** is the uncertainty of blend component. **R** is Reprod for component. **X** is component concentration listed on the certificate of the blend.	**X** is the component concentration of run 1 out of 2 consecutive runs.	**R^2** is the Pearson Correlation Coefficient indicating a measure of the linear correlation between the two variables (in this case, molecular weight vs. instrument response for each component).	Trending of each component is monitored as per GPA 2198.

i. All DQOs apply to all components, unless specific formula is present for that component. QC checks must be performed at least once per batch of samples analyzed.

j. GPA 2261-13 Reproducibility (R) and Repeatability (r) criteria applied, must be implemented in Mol%.

k. If the Uncertainty (U) of the calibration blend is known, the Performance Evaluation (PE) criteria will be used in lieu of Reproducibility (R).

Table 19. Data Quality Objectives (DQOs) for GPA 2261 C6+ Gas (Portable GC) Test Method[l]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)[m]	Performance Evaluation Criteria (PE)[n]	Method Repeatability (r)[m]	Fidelity Plot	Control Charts
Blank Check	Result ≤ 0.01 Mol%	-	-	-	-	-	-
Nitrogen	-	-	$R = 0.158*X^{(1/2)}$	$PE = X \pm (U^2 + R^2)^{(1/2)}$	$r = 0.039*X^{(1/4)}$	-	-
Methane			$R = 91000*X^{(-3)}$		$r = 0.0079*X^{(1/3)}$		
Carbon Dioxide			$R = 0.12*X^{(1/3)}$		$r = 0.0042*X^{(1/3)}$		
Ethane			$R = 0.0315*X^{(1/3)}$		$r = 0.0124*X^{(1/3)}$		
Propane			$R = 0.026*X^{(1/2)}$		$r = 0.0084*X^{(1/8)}$		
Iso-Butane			$R = 0.018*X^{(1/2)}$		$r = 0.01*X^{(1/5)}$		
N-Butane			$R = 0.033*X^{(1/2)}$		$r = 0.0117*X^{(2/5)}$		
Iso-Pentane			$R = 0.025*X^{(1/4)}$		$r = 0.009*X^{(1/4)}$		
N-Pentane			$R = 0.026*X^{(1/3)}$		$r = 0.01*X^{(1/5)}$		
Hexanes Plus			$R = 0.051*X^{(1/2)}$		$r = 0.0135*X^{(1/4)}$		
DQO Notes	**Result** is the un-normalized concentration of each component detected from analysis of a component free matrix, such as the carrier gas.	Port. GCs use multiple columns/detectors to calculate groups of components and recovery criteria is not applicable.	**X** is component concentration listed on the certificate of the blend.	**U** is the uncertainty of blend component. **R** is Reprod for component. **X** is component concentration listed on the certificate of the blend.	**X** is the component concentration of run 1 out of 2 consecutive runs.	Port. GCs use multiple columns/detectors to calculate groups of components. Fidelity plots must have all data available from same detector, and are not applicable.	Control charts must have consistent use and data to plot a stable response over time. Port. GCs used intermittently throughout study.

l. All DQOs apply to all components, unless specific formula is present for that component. QC checks must be performed at least once per batch of samples analyzed.

m. GPA 2261-13 Reproducibility (R) and Repeatability (r) criteria applied, must be implemented in Mol%.

n. If the Uncertainty (U) of the calibration blend is known, the Performance Evaluation (PE) criteria will be used in lieu of Reproducibility (R).

Table 20. Data Quality Objectives (DQOs) for GPA 2286 Extended Gas Test Method[o]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)	Performance Evaluation Criteria (PE)	Method Repeatability (r)	Fidelity Plot	Control Charts
Blank Check	Baseline visually gauged	-	-	-	-	-	-
2,2-Dimethylbutane	-	-	-	-	-	-	Area counts plotted for each component with average, ± 1, ±2, and ± 3 standard deviations.
2,3-Dimethylbutane							
2-Methylpentane							
3-Methylpentane							
N-Hexane							
Benzene							
N-Heptane							
Toluene							
N-Octane							
Ethylbenzene							
M, P-Xylene							
O-Xylene							
N-Nonane							
N-Decane							
DQO Notes	Instrument is not calibrated; therefore, baseline is visually gauged to verify that the instrument does not contain residual sample material from previous analyses.	-	-	-	-	-	Trending of each component is monitored as per GPA 2198.

o. No GPA published criteria available for this test method, QC checks must be performed at least once per batch of samples analyzed.

Table 21. Data Quality Objectives (DQOs) for ASTM D1945-1946 Refinery Gas Test Method (TCD Channels)[p]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)[q]	Performance Evaluation Criteria (PE)	Method Repeatability (r)[q]	Fidelity Plot	Control Charts
Helium	-	-	Reprod calculated for each component based on concentration, see below.	-	Repeat calculated for each component based on concentration, see below.	-	Area counts plotted for each component with average, ± 1, ±2, and ± 3 standard deviations.
Hydrogen							
Carbon Dioxide							
Ethylene							
Ethane							
Argon/Oxygen							
Nitrogen							
Methane							
Carbon Dioxide							
Mo l% (0 ≤ X ≤ 1)			R = 0.1		r = 0.05		
Mol % (1 ≤ X ≤ 5)			R = 0.2		r = 0.1		
Mol % (5 ≤ X ≤ 25)			R = 0.5		r = 0.3		
Mol %(25 ≤ X ≤ 100)			R = 1.0		r = 0.5		
DQO Notes	**Result** is the un-normalized concentration of each component detected from analysis of a component free matrix, such as the carrier gas.	This instrument configuration uses multiple columns/detectors to calculate groups of components and recovery criteria is not applicable.	**X** is component concentration listed on the certificate of the blend.	-	**X** is the component concentration of run 1 out of 2.	This instrument uses multiple columns/detectors to calculate groups of components. Fidelity plots must have all data available from same detector, and are not applicable.	Trending of each component is monitored as per GPA 2198.

p. All DQOs apply to all components; formula used is dependent on the concentration detected for each component. QC checks must be performed at least once per batch of samples analyzed.

q. ASTM D1946-90 (2015) Reproducibility (R) and Repeatability (r) criteria applied, must be implemented in Mol%.

Table 22. Data Quality Objectives (DQOs) for ASTM D1945-1946 Refinery Gas Test Method (FID Channel)[r]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)[s]	Performance Evaluation Criteria (PE)	Method Repeatability (r)[s]	Fidelity Plot	Control Charts
Propane	-	-	Reprod calculated for each component based on concentration, see below.	-	Repeat calculated for each component based on concentration, see below.	-	Area counts plotted for each component with average, ± 1, ±2, and ± 3 standard deviations.
Propylene							
Iso-Butane							
N-Butane							
Propadiene							
Trans-2-Butene							
1-Butene							
Iso-Butylene							
Cis-2-Butene							
Iso-Pentane							
N-Pentane							
1,3-Butadiene							
Hexanes Plus							
Mol %(0 ≤ X ≤ 0.09)			R = 0.02		r = 0.01		
Mol %(0.1 ≤ X ≤ 0.9)			R = 0.07		r = 0.04		
Mol%(1.0 ≤ X ≤ 4.9)			R = 0.10		r = 0.07		
Mol %(5.0 ≤ X ≤ 10)			R = 0.12		r = 0.08		
Mol %(10 ≤ X)			R = 0.15		r = 0.10		
DQO Notes	**Result** is the un-normalized concentration of each component detected from analysis of a component free matrix, such as the carrier gas.	This instrument configuration uses multiple columns/detectors to calculate groups of components and recovery criteria is not applicable.	**X** is component concentration listed on the certificate of the blend.	-	**X** is the component concentration of run 1 out of 2.	This instrument uses multiple columns/detectors, fidelity plots must have all data available from same detector, and are not applicable.	Trending of each component is monitored as per GPA 2198.

r. All DQOs apply to all components, unless specific formula is present for the concentration detected for that component. QC checks must be performed at least once per batch of samples analyzed

s. ASTM D1945-14 Reproducibility (R) and Repeatability (r) criteria applied, must be implemented in Mol%.

Table 23. Data Quality Objectives (DQOs) for ASTM D2887 High Temperature Simulated Distillation Test Method[t]

	Blank Component Recovery	Sum of Component Recoveries	Method Reproducibility (R)[u]	Performance Evaluation Criteria (PE)	Method Repeatability (r)	Fidelity Plot	Control Charts
% Off - IBP	-	-	R = 0.066*X	-	-	-	-
% Off – 5%			R = 0.015*(X + 100)				
% Off – 10 - 20%			R = 0.015*(X + 100)				
% Off – 30%			R = 0.015*(X + 100)				
% Off – 40%			R = 4.3				
% Off – 50 - 90%			R = 4.3				
% Off – 95%			R = 5				
% Off – FBP			R = 11.8				
DQO Notes	-	-	**X** = average of two results (in °C). Reproducibility criteria used for both Diesel Fuel Crosscheck and Lab Control Sample (LCS).	-	-	-	-

t. ASTM D2887-15 Reproducibility (R) criteria applied. QC checks must be performed at the beginning of each batch of samples, and after every 10th sample analyzed.

u. Reproducibility Criteria used for both Diesel Fuel Crosscheck and Lab Control Sample (LCS).

Table 24. Data Quality Objectives (DQOs) for ASTM Test Methods

Test Method	Check Performed	Check Performed with:	Frequency of Check Performed:	Equipment Verification	Calibration Check	Reproducibility (R)	Repeatability (r)
Physical Shrink Determination[v]	Piston Volume Verification by Water Draw Procedure	Water (House Source)	Daily, prior to analysis of samples	± 1mL of NIST	-	-	-
ASTM D4052 Density by Digital Densitometer[w]	Calibration Check	DI Water (House Source)	Daily, prior to analysis of samples	-	-	R = 0.00412*X	-
ASTM D4052 Density by Digital Densitometer[w]	Calibration Check	Toluene (Reagent Grade)	Daily, prior to analysis of samples	-	-	R = 0.00412*X	-
ASTM D2001 Depentanization Distillation (C6+ Cuts)[x]	Balance Verification (@ 500g, 1000g, 1500g)	Weight Set (NIST Traceable)	Daily, prior to analysis of samples	-	1% of NIST	-	-
Molecular Weight by Cryette[x]	Balance Verification (@ 2g, 100g, 100g)	Weight Set (NIST Traceable)	Daily, prior to analysis of samples	-	1% of NIST	-	-
Molecular Weight by Cryette[y]	Calibration Check	N-Nonane (Reagent grade)	Daily, prior to analysis of samples and after every 10th sample analyzed	-	-	R = ± 2amu	-
Molecular Weight by Cryette[z]	Duplicate Analysis	Sample Material	Every 10th sample analyzed	-	-	-	r = ± 2amu
ASTM D323 Reid Vapor Pressure[aa]	RVP Gauge Accuracy Check	Compressed Air (Air Compressor in Lab)	Each sample analyzed	1% of scale range of RVP Gauge	-	-	-

v. Water draw procedure compares the volume of water displaced from the piston against the volume of the piston, as stated on the NIST traceable certificate.

w. X is sample mean of two values (certified value and result). Result of analysis of check material must be within Reproducibility (R), relative to certified value.

x. Balance result for each weight must be within 1% of NIST value stated on certificate for weight.

y. Amu is atomic mass units. Result of analysis of check material must be within Reproducibility (R), relative to the certified value of material.

z. Amu is atomic mass units. Result from duplicate analysis must be within Repeatability (r), relative to original result.

aa. Results of RVP gauge reading and manometer reading are compared to monitor accuracy of RVP gauge. This test is performed with each sample analyzed, at the same pressure as the results indicated on the RVP gauge. All results are corrected and reported from manometer.

B.6 Instrument / Equipment Testing, Inspection, & Maintenance Requirements

B.6.1 All laboratory instrumentation and equipment used for process measurements will be verified for calibration as per the procedures detailed in *QAPP Section B.7.* All equipment that requires periodic certifications and/or calibrations will have records available showing that the equipment has been either certified by an external vendor or verified in-house with NIST traceable equipment. This is required for equipment such as thermometers, pressure gauges, balances, weights, flowmeters, and deadweight testers. A detailed report of all equipment used throughout the study, along with dates and records of certification, will be provided as part of the final QC Report submitted.

B.6.2 GC instruments used in the laboratory for analysis will verified for calibration as per the schedule detailed in *QAPP Section B.7*; and as a general rule, the GC instrumentation will not be recalibrated unless necessary. If the GC is found to be out of calibration, it will be investigated to determine if a corrective action is necessary or if it simply needs to be recalibrated. If the findings of the investigations warrant maintenance or repairs, the maintenance will be documented in dedicated maintenance logbooks, and include symptoms/cause for maintenance, repairs or modifications made, the date the maintenance was performed, and the initials of the individual performing the maintenance.

B.7 Instrument Calibration& Frequency

B.7.1 <u>GC Laboratory</u> **-** Instruments will have the calibration procedures (as listed in *QAPP Section B.5)* for each analysis performed according to the frequency stated in the GC test method DQO tables (*QAPP Tables 14 -23*).

B.7.2 <u>ASTM Laboratory</u>

B.7.2.1 Primary calibration equipment thermometers, weights, balances, pressure gauges, and dead weight testers will be calibrated annually by an external vendor to a NIST traceable source, unless the certificate provided by the vendor states otherwise for the recertification period. Secondary working thermometers will be verified against the primary calibration thermometer annually. Balances will be verified daily, prior to use with the NIST traceable weights. Pressure gauges will be verified annually with NIST traceable dead weight testers. Documentation for outside calibrations will be maintained by the individual responsible for coordinating the calibration services. Documentation of daily verifications will be maintained in dedicated logbooks and/or with worksheets specific for each set of analytical data.

B.7.2.2 The DQOs required for the ASTM test methods will be performed according to the frequency stated in the ASTM Test Method DQO Table (*QAPP Table 24*).

B.7.3 Blending Equipment – weights and balances will be calibrated annually by an external vendor to a NIST traceable source. Balances will be verified daily, prior to use, with the NIST traceable weights.

B.7.4 Field Equipment

B.7.4.1 Pressure gauges implemented for measurements made in the field will be verified in SPL's corporate office prior to each sampling event, using a NIST traceable dead weight tester and/or pressure gauge.

B.7.4.2 Process measurement instrumentation will be maintained by Noble. Documentation and maintenance records will be provided to SPL and Movilab for all applicable instrumentation used for the PHLSA study.

Table 25. Bernhardt PC-J31Well Production Facility Instrumentation List and Specifications

Parameter	Datalogger ID	Location	Instrument	Range	Accuracy	Calibration Method	Data Collection Frequency[B]	Units
Ambient pressure (P_{amb})	PIT 4	Automation Stand	Pressure transducer	0-1.5 psig (0-24 oz)	+/- 2% of measured value	3 Point linear	1 second	PSIG
Ambient temperature ($T_{ambient}$)	RTD 7	Vicinity of tank, upwind	RTD	-25 – 175ºF	+/- 2 ºF		1 second	ºF
Separator Oil Temperature ($T_{sep\ oil}$)	RTD 1	Separator oil layer	RTD	0 – 200ºF	+/- 2 ºF		1 second	ºF
Separator Gas Temperature ($T_{sep\ gas}$)	RTD 2	Separator gas headspace	RTD	0 – 200ºF	+/- 2 ºF		1 second	ºF
Separator Pressure (P_{sep})	PIT 1	Separator headspace	Pressure transducer	0-500 psi	+/- 2 psi	3 Point linear	1 second	PSIG
Separator produced gas Velocity ($Q_{sales\ gas}$)	ABB Velocity	Separator gas leg	TBD	TBD (scfm)	+/- 2% of measured value?		1 second	Standard MCF per day
Separator produced gas flowrate ($Q_{sales\ gas}$)	ABB Flow	Separator gas leg	TBD	TBD (scfm)	+/- 2% of measured value?		1 second	Standard MCF per day
Separator produced gas Temperature ($Q_{sales\ gas}$)	ABB Temp	Separator gas leg	TBD	TBD (scfm)	+/- 2% of measured value?		1 second	Fahrenheit
Separator oil flowrate to tank (Q_{oil})	CM Flow	Separator oil leg upstream of dump valve	Coriolis meter	TBD, Coriolis meters Standard?	Coriolis meters “prove” spec? +/- 2% of measured value?		1 second	Barrels Per Day
Separator oil to tank density (ρ_{oil})	CM Density	Separator oil leg upstream of dump valve	Coriolis meter	TBD	+/- 2 ºF		1 second	SGU
Separator oil to tank temperature ($T_{CM\ oil}$)	CM RTD	Separator oil leg upstream of dump valve	Coriolis meter	0 – 200ºF	+/- 2 ºF		1 second	Fahrenheit
Coriolis meter drive gain	CM DG	Separator oil leg upstream of dump valve	Coriolis meter	TBD	TBD		1 second	Percent

Parameter	Datalogger ID	Location	Instrument	Range	Accuracy	Calibration Method	Data Collection Frequency[B]	Units
Coriolis meter left output	CM Left	Separator oil leg upstream of dump valve	Coriolis meter	TBD	TBD		1 second	Volts
Coriolis meter right output	CM Right	Separator oil leg upstream of dump valve	Coriolis meter	TBD	TBD		1 second	Volts
Coriolis meter damping	CM Damp	Separator oil leg upstream of dump valve	Coriolis meter	TBD	TBD		1 second	Seconds
Separator water flowrate to tank (Q_{water})	CM W Flow	Separator water leg upstream of dump valve	Coriolis meter	TBD, Coriolis meters Standard?	Coriolis meters "prove" spec? +/- 2% of measured value?		1 second	Barrels Per Day
Coriolis water meter drive gain	CM W DG	Separator water leg upstream of dump valve	Coriolis meter	TBD	TBD		1 second	Percent
Oil dump valve on/off position & dump time/ duration (τ_{dump}, I_{dump})	O Dump Po	Oil dump valve	Valve position indicator	0 or 1	NA	NA	1 second	0,1,2,3
Separator-to-oil tank pipe gas/liquids temperature ($T_{dump\ flow}$)	RTD 4	Separator-to-oil tank pipe, just prior to entering the tank on the horizontal section	Thermocouple	25 – 175ºF	+/- 2ºF		1 second	ºF
Separator-to-oil tank pipe gas/liquids pressure ($P_{dump\ flow}$)	PIT 5	Where the sep-to-oil tank pipeline comes to the surface, base of upcomer	Pressure transducer	0 – 15 psig	+/- 2% of measured value	3 Point linear	1 second	PSIG
Oil tank headspace gas temperature ($T_{tank\ gas}$)	RTD 3	In tank, at top of tank, centerline	RTD	-25 – 175ºF	+/- 2ºF		1 second	ºF
Oil tank gas/liquids temperature ($T_{tank\ 1}$)	RTD 15	In tank, cernterline,152" above tank bottom	RTD	-30 – 150ºF	+/- 2ºF		1 second	°F
Oil tank gas/liquids temperature ($T_{tank\ 2}$)	RTD 14	In tank, centerline, 132" above tank bottom	RTD	-30 – 150ºF	+/- 2ºF		1 second	°F
Oil tank gas/liquids temperature ($T_{tank\ 3}$)	RTD 13	In tank, centerline, 112" above tank bottom	RTD	-30 – 150ºF	+/- 2ºF		1 second	°F
Oil tank gas/ liquids	RTD 12	In tank, centerline, 92"	RTD	-30 – 150ºF	+/- 2ºF		1 second	°F

Parameter	Datalogger ID	Location	Instrument	Range	Accuracy	Calibration Method	Data Collection Frequency[B]	Units
temperature ($T_{tank\ 4}$)		above tank bottom						
Oil tank gas/liquids temperature ($T_{tank\ 5}$)	RTD 11	In tank, centerline, 72" above tank bottom	RTD	-30 – 150ºF	+/- 2ºF		1 second	°F
Oil tank gas/liquid temperature ($T_{tank\ 6}$)	RTD 10	In tank, centerline, 52" above tank bottom	RTD	-30 – 150ºF	+/- 2ºF		1 second	°F
Oil tank gas/liquids temperature ($T_{tank\ 7}$)	RTD 9	In tank, centerline, 32" above tank bottom	RTD	-30 – 150ºF	+/- 2ºF		1 second	°F
Oil tank gas/liquids temperature ($T_{tank\ 8}$)	RTD 8	In tank, centerline, 12" above tank bottom[A]	RTD	-30 – 150ºF	+/- 2ºF		1 second	°F
Oil tank liquid level ($H_{tank\ liquids}$)	LL1	Oil tank liquid surface	Tank level sensor	0 – 180 Inches	0.25 inch		1 second	Inches
Oil tank headspace gas pressure ($P_{tank\ gas}$)	PIT 2	Bulk tank headspace pressure (gauge pressure)	Pressure transducer	0-1.5 psig (0-24 oz)	+/- 2% of measured value	3 Point linear	1 second	PSIG
Oil tank VOC burner line gas temperature ($T_{VOC\ pipe}$)	RTD 6	In tank VOC burner line downcomer upstream of flowmeter(s)	RTD	-30 – 150ºF	+/- 2ºF		1 second	ºF
Oil tank VOC burner line gas pressure($P_{VOC\ pipe}$)	PIT 3	In tank VOC burner line downcomer upstream of flowmeter(s) (gauge P)	Pressure transducer	0-1.5 psig (0-24 oz)	+/- 2% of measured value	3 Point linear	1 second	PSIG
Oil tank VOC burner line gas velocity	Fox1 Velocity	In tank VOC burner line downcomer upstream of knockout[C]	Thermal flowmeter	TBD	TBD		1 second	NMPH
Oil tank VOC burner line gas flowrate($Q_{tank\ gas\ 1}$)	Fox1 Flow	In tank VOC burner line downcomer upstream of knockout[C]	Thermal flowmeter	TBD	TBD		1 second	Standard MCF per day
Oil tank VOC burner line gas temp	Fox1 Temp	In tank VOC burner line downcomer upstream of knockout[C]	Thermal flowmeter	TBD	TBD		1 second	Fahrenheit
Oil tank VOC burner line gas flowrate($Q_{tank\ gas\ 2}$)	Vane Anemometer	In tank VOC burner line downcomer upstream of knockout[C]	Vane anemometer	TBD	TBD		1 second	Meters per Second??
Oil tank VOC burner	Fox2 Velocity	In tank VOC burner line	Thermal	TBD	TBD		1 second	NMPH

Parameter	Datalogger ID	Location	Instrument	Range	Accuracy	Calibration Method	Data Collection Frequency[B]	Units
line gas Velocity		downcomer upstream of knockout[C]	flowmeter					
Oil tank VOC burner line gas Flowrate($Q_{tank\ gas}$)	Fox2 Flow	In tank VOC burner line downcomer upstream of knockout[C]	Thermal flowmeter	TBD	TBD		1 second	Standard MCF per day
Oil tank VOC burner line gas Temp	Fox2 Temp	In tank VOC burner line downcomer upstream of knockout[C]	Thermal flowmeter	TBD	TBD		1 second	Fahrenheit
Separator Dump Temp, Just prior to coriolis meter.	RTD 5	Separator Dump leg, Just prior to coriolis meter.	RTD	0 – 200ºF	+/- 2ºF		1 second	ºF
Separator Dump Pressure, Just prior to coriolis meter.	PIT 6	Separator Dump leg, Just prior to coriolis meter.	Pressure transducer	0-500 psig	+/- 2% of measured value	3 Point linear	1 second	PSIG
Oil tank PRV vent gas flowrate($Q_{PRV\ gas}$)	Fox3 Velocity	In tank PRV vent line upstream of the PRV[C]	Thermal flowmeter	TBD	+/- 2% of measured value		1 second	NMPH
Oil tank PRV vent gas flowrate($Q_{PRV\ gas}$)	Fox3 Flow	In tank PRV vent line upstream of the PRV[C]	Thermal flowmeter	TBD	+/- 2% of measured value		1 second	Standard MCF per day
Oil tank PRV vent gas flowrate($Q_{PRV\ gas}$)	Fox3 Temp	In tank PRV vent line upstream of the PRV[C]	Thermal flowmeter	TBD	+/- 2% of measured value		1 second	Fahrenheit
Auxiliary	AUX 1	Extra datalog space for additional variable					1 Second	
Auxiliary	AUX 2	Extra datalog space for additional variable					1 Second	
Auxiliary	AUX 3	Extra datalog space for additional variable					1 Second	
Auxiliary	AUX 4	Extra datalog space for additional variable					1 Second	
Auxiliary	AUX 5	Extra datalog space for additional variable					1 Second	

B.8 Inspection and Acceptance Requirements for Supplies and Consumables

B.8.1 Standard Receipt and Validation Procedures

B.8.1.1 Analytical Calibration Standards– As new calibration standards are received by the laboratory, they are logged into a dedicated standard receipt log and assigned a unique internal identification number (however, in some cases the lot or QC number of the material may be used as the internal identification number). Information such as concentration, manufacture date, and NIST traceability are recorded in the log book. Calibration standards must be validated before placing into service to verify that the concentrations listed on the certificate accompanying the standard are correct, and that the standard is suitable for the intended use. This is accomplished by analyzing the new standard as an unknown in duplicate on an instrument that has been previously verified to be in calibration. Applicable QC criteria for the material being validated and the method being performed will be used to determine if the material is representative of what is listed on the certificate. Records of the validation procedure is maintained with the vendor certificate and any other documentation accompanying the material such as the MSD.

B.8.1.2 Laboratory Reagents and Materials - As new reagents and pure materials/solvents are received by the laboratory, they are logged into a dedicated standard receipt log and assigned a unique internal identification number. Information regarding concentration, manufacture date, NIST traceability, etc. are recorded in the log book. Documentation accompanying the material such as the MSD sheet is maintained along with the certificate provided by the material manufacturer.

B.8.1.3 Blending Feedstock – All raw materials received are logged in and assigned a unique internal identification number for future reference. Vendor or manufacturer records regarding the purity of the material must accompany the material, or the material may not be accepted. The material will be validated by GC analysis from an instrument that is calibrated to detect the component of interest and verified for purity (unless analytical capability for a given component is not available). Documentation of the validation procedure is maintained with the certificate and any other documentation accompanying the material such as the MSD.

B.9 Data Acquisition Requirements for Non-Direct Measurements

GPA Standard 2145 lists the constants and physical properties for components up through N-Decane. GPA Technical Publication 17 (TP-17) lists the physical properties and constants for each component of interest through Tetradecane. When components greater than Tetradecane are identified (such as with the ASTM D2887 analysis), Yaws Handbook of Physical Properties will be used to reference the properties and constants of the normal alkane for the corresponding carbon number grouping identified in the analysis.

B.10 Data Management

Data for this project will be produced in multiple locations. Key personnel representing each work group identified in *QAPP Table 4* will be responsible for ensuring that all necessary records are submitted to the Project Lead, who will review and approve results before sending to the panel.

The sampling and measurement records generated for this project will include field data sheets (sample tags), chains of custody (COCs), and calibration/maintenance/service records for equipment. The VP of Technical Services overseeing the sampling and measurement procedures will review the field data sheets prior to shipping samples to the lab, and any problems identified will be reported to the Project Lead. Field data sheets (sample tags) and COCs will accompany the samples to the laboratory.

The laboratory login department will review field data sheets and COCs upon receipt, and log the samples in according to the testing required. Final analytical results generated from the laboratory will have a minimum of two levels of review before being submitted to the Project Lead, along with the field data sheets and COCs. The Project Lead will review all field data sheets and analytical reports to identify any suspect data to flag and discuss with the panel if necessary. Once approved by the Project Lead, the all data will be sent to Movilab to begin statistical uncertainty analysis.

The blending services group will generate certificates for each CRM blend that will accompany each CRM. Each certificate will include information regarding composition of each component in the CRM, details of the hexanes plus (C6+) lot composition, verification records and data, and date of completion.

Upon completion of individual perturbation studies, results from each work group will be sent to the Project Lead to review and compile a midterm report to submit to the distribution group identified in *QAPP Table 2*. As the final phases of the overall PHLSA study are completed, the same convention will be followed by submitting all data to the Project Lead to compile the final PHLSA report.

All data and records for this project will be maintained according to SPL's policy regarding data retention; therefore records will be retained for seven years from the date of submitting the final PHLSA report to Noble, unless requested to do otherwise. All hard copy records listed in *QAPP Section A.9* will be included in the data archival.

Electronic records (from data acquisition software, PSM/EOS data, etc.) will be able to be retrieved electronically for review for the duration of the archival period, unless provisions are made to retain hard copies of data in sufficient enough detail to reconstruct the original results if necessary.

Section C – Assessment and Oversight

C.1 Assessment and Response Actions

The laboratory is subject to an annual internal performance audit. Additional audits have not been scheduled for this project specifically, however Noble may request to schedule an audit to be performed throughout the duration of this project.

C.2 Reports to Management

Due to the nature of this project, it is anticipated that the Project Lead will be in frequent contact with the Noble TAP giving informal status updates throughout the study. Upon completion of the perturbations and methods assessment, a formal midterm report will be presented to the panel by the Project Lead per the anticipated timeline as shown in the schedule (refer to *Work Plan Section 4*). This report will include a summary of QA/QC activities, measurements, calculations, and list and discuss deviations and exceedances. A semiannual status report is expected to be completed in January 2016 with the final report to be completed in March 2017.

Section D – Data Validation and Usability

D.1 Data Review, Verification, and Validation

Data will be analyzed by an independent third party (Movilab) to ensure the validity of the results and estimate the uncertainties of measured and calculated values. The results and all intermediate steps to reach the results will be documented in a data analysis report which the third party will supply along with any relevant calculations, Excel worksheets, etc.

D.2 Data Verification and Validation Methods

D.2.1 Data Validation and Verification

D.2.1.1 Storage tank input and output process measurements, and mass balance calculations - To perform an accurate mass balance of a storage tank, it is imperative that all inputs, outputs, and storage in the tank be measured for both quantity and quality. The uncertainty will be estimated for each measurement based on best available information. This information may come from the manufacturer, industry research, or engineering judgment of the company performing the data analyses. The mass balance will be completed on a component basis from the quantity and quality determinations. While the details associated with making these measurements are yet to be determined, the basic mass balance and related process measurement calculations are well understood.

D.2.1.2 Comparison of various pressurized HC liquids S&A procedures and modifications - Sampling and analysis methodologies will be separately compared and evaluated. Two sampling methods (constant volume and constant pressure) will be evaluated, and four analysis methods will be evaluated. A certified reference standard, with low uncertainty, will be utilized as a basis for comparison. First, four analysis methods will be tested for accuracy and repeatability. Based on the test results the "best" methodology will be identified. This method may be a modified method based on the expertise of the testing company and the results. Second, with customer agreement, the selected analysis methodology along with the GPA 2103M C_{10}+ methodology will be utilized to evaluate several sampling methodologies. The two analysis methodologies and sampling methodologies will be utilized over the remainder of the testing.

D.2.1.3 Sensitivity of measured values to changes in S&A procedures and/or changes to separator and storage tank operation - For example, impacts of S&A modifications on pressurized HC liquids results, and impacts of instrumented well production facility process parameters on flash gas-to-oil ratio (FGOR) (i.e., experimentation and measurements sensitivity analysis). To accurately determine uncertainties and sensitivities associated with each component of the S&A procedures, data must be collected for each component. The testing as described in Section B above will allow us to independently analyze each process component and assign uncertainty and sensitivity values to each component based on the methodology implemented.

D.2.1.4 Sensitivity of process simulation software program outputs, such as FGOR, to variations in required input values (i.e., calculations and simulations sensitivity analysis) - Each software package to be tested will be analyzed over a range of values to determine the sensitivities of output values to input data. A test matrix will be developed by the independent third party and agreed upon by all parties involved. This test matrix will be based on real world data that has been collected to this point. Comparisons between software packages will be made on both calculated values and input data sensitivities.

D.2.1.5 Uncertainty of primary study parameters - including, but not limited to:

- Measured FGORs -Uncertainties of the measured FGORs will be estimated based on the measurement equipment and techniques used to obtain the information. System balance information will also be utilized to verify the measured FGORs. All comparisons will be made with respect to the uncertainty associated with each measured value.

- Bubble point pressures estimated from process simulation software and a pressurized HC liquids analysis- Bubble point pressures will be determined experimentally from the certified reference blends. These values will be compared to calculated values to estimate the uncertainty associated with each software package.
- FGORs estimated from process simulation software and a pressurized HC liquids analysis - Information gathered from the S&A testing will be combined with data from the FGOR testing and the software simulation testing to determine an overall uncertainty associated with the final tested methodology.

Maximum potential flash gas generation rates during separator dumps estimated from process simulation software and a pressurized HC liquids analysis. For example, pressurized HC liquid samples are often collected at a separator pressure less than the maximum, and a tank control system designer would like to understand the uncertainty in process simulation software estimates of flash gas generation rate at the maximum separator operating pressure and tank temperature.

Based on all of the information gathered, it will be possible to estimate (with an associated uncertainty) the maximum potential flash gas generation rates. By combining all uncertainties (in accordance with the ISO Guide to Uncertainty in Measurement), the overall uncertainty of this value will also be determined. Only once the data has been analyzed will the sensitivities of each component be understood. It appears there will be enough data collected to determine the uncertainty of the processes from experimental data. If there is not, the third party may run Monte Carlo simulations to estimate the overall uncertainty. Utilizing real data to determine uncertainties is always the preferred method for determining the overall uncertainty of a process, and Monte Carlo is meant to be utilized when that data is not present.

D.2.2 Reconciliation with Data Quality Objectives

The sampling plan allows enough data to be collected to determine the uncertainty of the processes from experimental data. If extenuating circumstances prevent enough data from being collected, Movilab may run Monte Carlo simulations to estimate the overall uncertainty. Utilizing real data to determine uncertainties is always the preferred method for determining the overall uncertainty of a process, and Monte Carlo is meant to be utilized when that data is not present. Once the statistical method selection is determined, this information will be submitted to the Project Lead and the Quality Director to update the QAPP. Uncertainty reports will then be submitted to the Project Lead for review.

Upon receipt of the uncertainty results, the Project Lead will compile a midterm report containing applicable information regarding sample collection, laboratory handling and analysis results, and the uncertainty results. Midterm reports will then be submitted and/or presented to the Noble TAP for review.

D.3 Reconciliation with User Requirements

D.3.1 Data Validation and Usability - Data will be accepted providing the following criteria are met:

D.3.1.1 Sample collection procedures are documented on the chain of custody (COC) forms, and actual sample collection procedures match the collection procedures identified in *QAPP Section B.2*.

D.3.1.2 Field data sheets are complete. Refer to *Work Plan Figure5* for details regarding the minimum information required for each sample obtained.

D.3.1.3 Field data and laboratory data were validated to correspond to each other to verify that the samples were not compromised in transit (this is the Initial pressure verification and bubble point verification).

D.3.1.4 Actual sample handling procedures in the laboratory correspond to the handling procedures identified in *Work Plan Tables 5a* and *5b*. Any measurement or analytical deviations from the QAPP shall be documented in detail in either the sampling information sheet or with the analytical report. The Project Lead will review all documented deviations and flag items to discuss with the project team and/or Noble panel to determine whether to include the data for statistical evaluation or to reject and resample.

Appendix 1 – Summary of Updates to QAPP Revision 2

Version 1 Reference	Description of Update
QAPP	Reformatted entire document to reflect PHCLSA Report Format Guidelines. All updates to QAPP sections as described below reference the original section/table ID from the version 1 document.
Cover Page	Date Submitted changed for QAPP revisions to reflect target date of final report.
Section A.1	Approval from Alon Mandel changed to Susan Gomez.
Section A.4	Susan Gomez responsibilities added
Section A.7	Additional clarifications made for Data Quality Indicators (DQIs) and corresponding tables
Section B.4	Updated to include descriptions for the additional analyses performed throughout PHLSA study (not included in original scope).
Section B.5	Updated to better correlate analytical QC procedures and DQOs. Updates were made to each DQO and included a description of the DQO, the applicability and limitations of the DQO, and an explanation of how each DQO is evaluated and/or represented in the QC documentation provided with the final report.
Section B.7.2	Consolidated instrument calibration requirements for ASTM. Much of the detail provided in original document is included in DQO Tables and Section B.5.
Section E.1	Placeholder for the Brief Description of Uncertainty Analysis was removed, as this will be provided in final PHLSA report.
Table 3 – QAPP Distribution and Contact List	Susan Gomez contact information added.
Table 4 – Results and Reporting Distribution List	Removed Alon Mandel, added Susan Gomez.
Table 6 – Applicable Concentration Ranges for GPA 2177 and GPA 2261 Precision	Moved from Section A.7.2 to Section B.5 to better correlate with Data Quality Objectives (DQOs) and quality control procedures. Now table 13.
Tables 7–12 – DQOs for Methods	Moved from Section A.7.2 to Section B.5 to maintain correlation with Data Quality Objectives (DQOs) and quality control procedures. Reformatted and provided additional clarifications/information. Now tables 14-24 (includes DQOs for additional analyses not included in original scope).
Table 13 - Bernhardt Instrumentation List and Specifications	Moved from Section A.7.2 to Section B.7 to maintain correlation with Data Quality Objectives (DQOs) and quality control procedures. Reformatted and provided additional clarifications/information. Now table 25.

Table 14 – List of Sampling Methods	Now table 8.
Table 15 – Number of Purge Cycles for PF&E Method	Now table 9.
Table 16 – Spot Sample Minimum Requirements	Now table 10.
Table 17 – List of Analytical Methods	Updated to include additional analyses performed throughout PHLSA study (not included in original scope). Now table 11.
Tables 18-21 - Scopes for Methods	Merged tables for GPA 2103, GPA 2177, and GPA 2186 into a single table Now table 12.
New table added	Data Quality Indicators for GC Test Methods (table 6).
New table added	Data Quality Indicators for ASTM Test Methods (table 7).