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New Technology Research & Development Program
Grant Contract 582-5-70807-M028

Task 1 Report
Approved Test Plan

The preparation of this report is based on work funded in part
by the State of Texas through a Grant from the
Texas Commission on Environmental Quality.

26914 cc: G. [illegible] excluding conf. info

ORYXE

ORYXE ENERGY INTERNATIONAL, INC.

6 Thomas Avenue
Irvine, California 92618 USA
Telephone: (949) 452-9272
Facsimile: (949) 452-9274

RECEIVED
JUL 18 2006
EXECUTIVE DIRECTOR

July 17, 2006

VIA FEDERAL EXPRESS

Mr. Glenn Shankle
Executive Director
Texas Commission on Environmental Quality
12100 Park 35 Circle
Austin, Texas 78753

Re: Request for Approval to Conduct Texas Low Emission Diesel Alternative Fuel Formulation Testing for Biodiesel

Dear Mr. Shankle:

Under the Texas Commission on Environmental Quality's (TCEQ's) New Technology Research and Development grant contract number 582-5-70807-M028, ORYXE Energy International, Inc. (ORYXE Energy) intends to begin official testing of our additive, OR-B20, pursuant to Section 114.312(f) and 114.315(c) of Title 30 of the Texas Administrative Code (Alternative Diesel Fuel Formulations). Please note this is the same protocol and procedures approved by David Schanbacher of the TCEQ via correspondence dated June 2, 2006. The same protocol is being resubmitted for approval with the only changes made in the testing location, West Virginia University, and the test engine, 1992 DDC Series 60. The purpose of this testing is to qualify ORYXE Energy's additive under the alternative diesel fuel formulation provisions of the Texas Low Emission Diesel (TxLED) rule for biodiesel, a blend that will be comprised of 20%, by volume, of biodiesel, and 80%, by volume, of conventional no. 2 diesel fuel, otherwise commonly referred to as "B20." ORYXE Energy understands by qualifying a B20 blend, all blends at or below this level (e.g., B15, B10, B5, B2, etc.) will automatically qualify as well without any further testing. Please indicate to us whether or not this is correct.

Attached to this document, you will find four (4) Exhibits. Exhibit A is the detailed testing protocol developed by ORYXE Energy. The specific protocol as proposed conforms with Section 114.315(c)(4)(C)(ii)(IV) of Title 30 of the Texas Administrative Code, alternative 4. We request your approval of the protocol as official testing is planned to begin at West Virginia University (WVU) shortly after approval has been received. Notification of the start date will be provided to the TCEQ 7 days prior to beginning the official test.

Exhibit A also discusses the reference fuel, the candidate fuel, and the pure biodiesel ("B100") selected for this test. The reference fuel has been analyzed for its required fuel properties by Caleb Brett in Signal Hill, California and is attached. The B20 candidate fuel (untreated with OR-B20) has been analyzed for its required fuel properties in triplicate by Caleb Brett in Signal Hill, California and is attached. Finally, the B100 has been analyzed for its required fuel properties by Caleb Brett in Signal Hill, California and is attached, along with a specification sheet provided by the B100 supplier (West Central). Please know that the reference fuel and the 80%, by volume, part of the B20 candidate fuel/blend are the same fuel.

Exhibit B indicates the detailed instructions on how to blend/mix the OR-B20 additive into the B20 candidate fuel, the chemical composition of OR-B20, and the treat rate of OR-B20. Attached to Exhibit C are the analytical test procedures used to identify the presence and concentration of each component used in the OR-B20 additive. Finally, attached to Exhibit D are the quality assurance and quality control procedures that will be used by WVU during this testing campaign.

Please know that all of the contents of Exhibit B and Exhibit C can be found within a separate brown envelope and marked "Confidential/Proprietary: inform applicant and seek AG opinion before releasing."

After you and your staff have had the opportunity to review the enclosed information, please contact me to discuss the details of the protocol or to answer any specific questions you might have regarding this topic.

All of the information contained in this letter constitutes secret processes of manufacturing or production and trade secrets. Please maintain the confidentiality of all of the information contained herein to the fullest extent of the law.

Best Regards,

Nick Economides
Executive Vice President,
Commercial Products and Technology Management

Enclosures (8):
- Exhibit A – Proposed Test Protocol
- Exhibit B – Chemical Composition of OR-B20
- Exhibit C – Analytical Procedures
- Exhibit D – UCR QA/QC Procedures
- Reference Fuel Analysis
- Candidate Fuel Analysis
- B100 Fuel Analysis
- B100 Specification Sheet

EXHIBIT A

PROPOSED TEST PROTOCOL

Abstract

The purpose of this project is to demonstrate that a technically verified fuel additive product from ORYXE Energy International, Inc. (referred to herein as OR-B20) can be used to produce Texas Low Emission Diesel (TxLED) biodiesel—a "B20" blend of 20%/80% of biodiesel/conventional no. 2 diesel, by volume—under the Texas alternative diesel fuel formulation provisions specified in Title 30 of the Texas Administrative Code, Part 1, Chapter 114, Subchapter H, Division 2, Rule §114.312(f) and §114.315(c).

Testing will include verifying and quantifying the nitrogen oxides (NO_X) reduction benefits of OR-B20 by carrying out a test protocol approved by the Texas Commission on Environmental Quality (TCEQ). The total hydrocarbon (THC), non-methane hydrocarbon (NMHC), and particulate matter (PM) emission impacts of the OR-B20-treated B20 candidate fuel will also be evaluated and compared to those of the untreated reference fuel.

The ultimate goal of the project is to treat standard B20 (or lower blends) with an additive (OR-B20) so that they qualify as TxLED and help the State of Texas maximize both on-road and off-road mobile NO_X reductions.

Success of this project will demonstrate the applicability of ORYXE Energy International, Inc.'s OR-B20 product to achieve cost effective TxLED emission reduction compliance in B20 or lower blends with various downstream implementation options (the terminal and/or refinery) to the fuel provider.

Test Plan

The overall test plan will adhere to the appropriate test procedures listed in the requirements specified in Title 30 of the Texas Administrative Code (TAC), Part 1, Chapter 114, Subchapter H, Division 2, Rule §114.315.

The testing program ORYXE Energy International, Inc. (ORYXE Energy) proposes to carry out includes the following five aspects:

(1) Testing Lab.
(2) Testing Engine.
(3) Testing Fuels.
(4) Testing Protocol.
(5) Data Analysis.

Each of these five aspects is discussed in further detail below.

(1) Testing Lab

ORYXE Energy proposes to use the National Center for Alternative Fuels, Engines and Emissions at West Virginia University (WVU) in Morgantown, West Virginia. WVU is a U.S. Environmental Protection Agency (EPA) and California Air Resources Board (CARB) recognized and industry respected emissions testing research laboratory that has conducted hundreds of research projects over the last 13 years. These projects were or are currently sponsored by fuel suppliers (BP, ARCO, Chevron, and others), engine manufacturers (Cummins, Caterpillar, Detroit Diesel, and others), vehicle manufacturers (Ford, GM, and others), Federal Government Agencies (Department of Energy, Department of Transportation, EPA, and others), and State Government Agencies (CARB, Arizona Department of Environmental Quality, New York State Department of Environmental Conservation, and others). These projects have addressed issues associated with the full spectrum of fuels, engines, and vehicle performance. Many projects have specifically focused on fuels and fuels effects.

Given its experience and capability, WVU can and will meet the Quality Assurance/Quality Control (QA/QC) requirements that the TCEQ needs in order to assure that the test results achieved are accurate and repeatable within a 95% confidence level. As proof, WVU's QA/QC procedures that will be used during this testing campaign are attached hereto as Exhibit D. WVU has previously been approved by the TCEQ and the California Air Resources Board (CARB) as a qualified and capable facility to carry out a test program similar to the one being proposed in this application.

(2) Testing Engine

The engine ORYXE Energy proposes to use at WVU is a Detroit Diesel Corporation Series 60 (DDC-60), 1992 model year, which has been used in past TCEQ-approved programs by ORYXE Energy. The displacement of this engine is 12.7 liters and the horsepower rating is 360 at 1,810 revolutions per minute (RPMs). The DDC-60 diesel engine is the engine specified in TAC §114.315(c)(4)(A) for carrying out the test program being proposed in this application.

(3) Testing Fuels

ORYXE Energy proposes to test two fuels at WVU (as marked) using the DDC-60, but lists here three fuels that have been analyzed. The three fuels that have been analyzed are the reference fuel, the B20 candidate fuel, and the pure or "B100" biodiesel. Each of these fuels is discussed in further detail, below.

Reference Fuel (to be tested)

ORYXE Energy proposes to use and test a low total aromatics reference fuel. The attached fuel property analysis was carried out on the reference fuel by Caleb Brett in Signal Hill, California and is attached. The reference fuel properties satisfy the characteristics identified in TAC §114.315(c)(3)(A)-(I).

B20 Candidate Fuel (to be tested)

ORYXE Energy proposes to use and test a low total aromatics candidate fuel blend comprising of 20%, by volume, of soy-based biodiesel (or "B100") from West Central Co-Op in Ralston, Iowa, and 80%, by volume, of the reference fuel described herein above (this total blend will be hereinafter referred to as "B20"). Soybeans were chosen for this test because they have the least positive effect on emissions reductions.

A fuel property analysis (untreated with OR-B20) was carried out on the B20 candidate fuel in triplicate by Caleb Brett in Signal Hill, California and is attached. This was done in accordance with TAC §114.315(c)(2)(A). The B20 candidate fuel conforms to the diesel fuel standards listed in the American Society for Testing and Materials (ASTM) D-975 method, except for lubricity, and therefore satisfies TAC §114.315(c)(1)(C).

B100 (will not be tested)

Although ORYXE Energy will not test the B100, the attached fuel property analysis (untreated with OR-B20) was carried out by Caleb Brett in Signal Hill, California and is attached. This analysis was carried out only to confirm that the B100 met ASTM D-6751 and not for any other purpose. In addition, the specification sheet/Certificate of Analysis provided by the supplier (West Central) is attached to further confirm the B100 specifications.

(4) Testing Protocol

The testing protocol ORYXE Energy proposes to run at WVU using the DDC-60 and the reference and candidate fuels described above is one that will conform to TAC §114.315(c)(4)(C)(ii)(IV). The testing method sequence selected will be the protocol identified as "Alternative 4." More specifically, Alternative 4 refers to a testing sequence consisting of seven (7) Federal Test Procedures (FTPs) (as defined in 40 Code of Federal Regulations, Part 86, Subpart N) totaling twenty one (21) "hot" starts in the following sequence: RR CCC RR (where "R" represents a reference fuel FTP and "C" represents a **OR-B20-treated** candidate fuel FTP). Details of our proposed testing protocol are discussed and described below.

Overview

Proposed Testing Protocol
Day 1 – R R
Days 2-4 – C [Conditioning the engine (not to exceed 72 total hours)]
Day 5 – C C
Day 6 – C R R

Key

R	Reference fuel meeting specifications in TAC §114.315(c)(3)(A-I) (1 FTP = 3 "hot" starts)
C	Candidate fuel meeting specifications in TAC §114.315(c)(1)(C) (1 FTP = 3 "hot" starts) and treated with OR-B20

Specific Proposed Testing Protocol Discussion

On Day 1, ORYXE Energy proposes to carry out six FTP "hot" start tests using the reference fuel. The engine mapping procedures and an engine conditioning transient cycle will be conducted after every fuel change and/or at the beginning of each testing day. The reference cycle generated from the reference fuel for the first test (on Day 1) will be used for all subsequent tests in accordance with TAC §114.315(c)(4)(C)(iv).

On Day 2, ORYXE Energy proposes to initiate the conditioning of the DDC-60 engine using the candidate fuel for no more than 72 total hours (3 days) of engine operation. This conditioning period will be run in accordance with and will represent normal engine operation.

On Day 5, ORYXE Energy proposes to carry out six FTP "hot" start tests using the candidate fuel.

On Day 6, ORYXE Energy proposes to carry out three FTP "hot" start tests using the candidate fuel.

On Day 7, the last day of the protocol, ORYXE Energy proposes to carry out six FTP "hot" start tests using the reference fuel.

(5) Data Analysis

The average of only the six (6) "hot" start emission values from the reference fuel run on Day 1 will be used to calculate the average emission value of the reference fuel for the entire testing program. Although the average of the six "hot" start emission values from the reference fuel run on Day 7 will be carried out to determine engine drift and/or carry-over effect (if any), these values will not be used to calculate the average and final emission values of the reference fuel.

The average of only the nine (9) "hot" start emission values from the candidate fuel run on Day 5 and Day 6 (this includes the six "hots" on Day 5 and the three "hots" on Day 6) will be used to calculate the average emission value of the candidate fuel for the entire testing program. Although the conditioning of the DDC-60 engine will most likely be run with the candidate fuel on Day 2, Day 3 and Day 4 in accordance with TAC §114.315(c)(4)(C)(ii)(IV), these values will not be used to calculate the average and final emission values of the candidate fuel.

The average emission values of the nine (9) specified candidate fuel "hot" starts will be compared to the average emission values of the six (6) specified reference fuel "hot" starts in accordance with TAC §114.315(c)(5).

Finally, the test data set generated from this testing program will be reviewed by WVU personnel for outliers using ASTM E-178, "Recommended Practice for Dealing with Outlying Observations."

EXHIBIT D

WVU QA/QC PROCEDURES

WVU's quality assurance and quality control procedures that will be used during this testing campaign are attached hereto.

* * * *

Texas Commission on Environmental Quality - TxLED Fuel Evaluation

Prepared by: Gregory J. Thompson, Ph.D.
23 January 2006

West Virginia University
Center for Alternative Fuels, Engines and Emissions
Mechanical and Aerospace Engineering
West Virginia University
PO Box 6106
Morgantown, WV 26506-6106
304.293.3111
Gregory.Thompson@mail.wvu.edu

Reviewed and approved by:

Gregory J. Thompson, Principal Investigator	Date
Nigel Clark, CAFEE Director	Date
David McKain, CAFEE Quality Assurance Officer	Date
Byron Rapp, CAFEE Test Engineer	Date

Distribution List:
Gregory Thompson
Nigel Clark
David McKain
Byron Rapp

Table of Contents

A PROJECT MANAGEMENT

A.1 Project Organization

Project management and execution responsibilities are divided among CAFEE staff as described below.

A.1.1 Project Management

Project management will be the responsibility of Dr. Gregory Thompson. His responsibility will be:

- Oversight of the Test Engineer and Quality Assurance Officer in the performance of their duties.
- Serve as the liaison between CAFEE and the program sponsors.
- Treat the candidate fuel with the applicant's additive.
- Oversight of preparation and delivery of the final report.

A.1.2 Test Execution

Engineering Scientist Byron Rapp will be responsible for all required pre-test procedures including calibrations, checks, and inspections. He will also supervise testing and perform the initial verification of test data and supply that data for quality review.

A.1.3 Report Generation

Dr. Thompson will be responsible for final data reduction and generation of the final report. He will provide the final data to David McKain for final quality assurance review.

A.1.4 Quality Review

Mr. McKain will be responsible for performing the final quality review of the data and data reduction processes.

A.2 Problem Definition

A candidate Texas market diesel fuel is being evaluated against a reference diesel fuel. The aim is to show equivalency of the candidate fuel to the reference fuel per the Texas Administrative Code (TAC) §114.315. Alternative 4 will be used as the test sequence for the work as described in TAC §114.315.c.3.ii.IV.

A.3 Project Description

Emissions will be evaluated from a 1992 Detroit Diesel Corporation, Series 60 engine for the reference fuel and candidate fuel. The engine will be exercised of the heavy-duty engine dynamometer test procedure using multiple hot start tests.

A.4 Quality Objectives and Criteria

The quality objective of this program is to collect data in a manner adhering to the procedures and requirements set forth by 40 CFR Part 86, Subpart N. These requirements define the procedures for collecting and reporting emissions data from engines over the FTP. In addition to ensuring the quality of individual emissions tests, the repeatability of these test is also measured and only considered acceptable if the coefficient of variance for carbon dioxide (CO_2), oxides of nitrogen (NO_X), fuel consumption, and work are below 1%; the coefficient of variance for carbon monoxide (CO) and particulate matter (PM) are below 3%; and the coefficient of variance for total hydrocarbons (HC) and non-methane hydrocarbons (NMHC) are below 10%. Although CO does not need to be reported, it will be collected as part of the test program.

A.5 Special Training and Certification

No special training or certification is required for this test program.

A.6 Documentation and Records

A draft report summarizing the emissions will be supplied to the program sponsors after completion of the testing. The report will detail the comparison of the candidate fuel to the reference fuel. A final report will be issued after the quality assurance review and the project approved by CAFEE personnel. The sponsors may see results if they are present at the time of testing, but CAFEE will not approve the use of results other than those that have been subjected to quality auditing and presented in the final report.

B DATA GENERATION AND ACQUISITION

B.1 Experimental Design

The CAFEE engine dynamometer employs a full-scale dilution tunnel and is capable of measuring emissions from heavy-duty engines up to 550 hp. Hot start emissions from the test engine will be measured using the reference and candidate fuels, the results from the candidate fuel will be compared to the reference fuel using the criteria listed in TAC §114.315.c.5.

B.1.1 Pre Test Tasks

The engine will be installed on the engine dynamometer and prepared for emissions testing. The engine oil, oil filters, and fuel filters will be changed at the start of the test program. Commercially available filters and oil will be used. The emission analyzers and engine dynamometer will be calibrated during this time. Any other required check or calibrations by 40 CFR Part 86, Subpart N will be performed at this time.

B.1.2 Test Tasks

The test plan approved by the TCEQ will be executed. Any deviation from the approved test plan or from the TAC §114.315 requirements will be immediately reported to the sponsor and to the TCEQ via email.

B.2 Sampling Methods

Engine emissions will be sampled using a full dilution tunnel and emissions sampling system conforming to the directives of 40 CFR Part 86, Subpart N. Both regulated and non-regulated

(NMHC) emissions will be examined from the engine with the emissions measurement procedures adhering to 40 CFR Part 86, Subpart N.

B.3 Sample Handling and Custody

All particulate filters, oil samples, and fuel samples will be retained for one year after testing is complete. Reference and candidate fuel samples will be collected from the fuel storage barrels and sent to Intertek Caleb Brett in California for analysis per the requirements set forth in TAC §114.315.

B.4 Analytical Methods

Analyzers and equipment utilized for regulated emissions measurement (CO, NO_X, HC, PM) and engine dynamometer will meet the requirements of 40 CFR Part 86, Subpart N. The method used for NMHC measurements will conform to the requirements of SAE J1151 using a gas chromatograph (GC) or using an online HC analyzer with a non-methane cutter.

B.5 Quality Control

Quality control in sampling and measurement is ensured by following the standard operating procedures which were developed in adherence to CFR 40, Part 86, Subpart N. Copies of all instrument calibrations will be permanently retained for future inspection and review.

B.5.1 Dilution Tunnel

The WVU laboratory employs a dilution tunnel in their engine emissions laboratory where the engine exhaust is mixed with ambient air conditioned to maintain a temperature of 77°F ± 5°F and a relative humidity of 50% ± 5%. The temperature and humidity are recorded at a frequency of 5Hz. Mass flow through the tunnel is controlled by a set of critical flow venturis that can be used in parallel, depending on the flow required to adequately dilute the engine exhaust. These critical flow venturis were initially calibrated using primary flow device from the US Environmental Protection Agency. To ensure that the calculated flow through the dilution tunnel is correct, propane is injected into the dilution tunnel using a Horiba Model 201B propane injection kit which is equipped with a calibrated orifice, precision thermocouple and pressure gage. The propane injection kit is capable of accurately metering the mass flow of propane into the tunnel. While the propane is being injected, a calibrated hydrocarbon analyzer is used to measure the concentration of propane in the dilution tunnel. The mass of propane injected by the propane injection kit is compared to the mass of propane recovered by the hydrocarbon analyzer to ensure the integrity of the dilution tunnel. A minimum of three propane injection tests, each lasting a minimum of 5 minutes, are performed and considered satisfactory when the difference between injected and recovered propane less then and with ±2.0% and three repeat tests are 0.5%, on a mass basis.

B.5.2 Gaseous Emissions

The WVU engine emissions laboratory employs the following analyzers to measure gaseous emissions concentrations from their dilution tunnel:

- Oxides of Nitrogen (NOx): Rosemount Model 955 Heated Chemiluminescent Analyzer and EcoPhysics Model 822 Heated Chemiluminescent Analyzer
- Total Hydrocarbons (THC): Rosemount Model 402 HFID Analyzer

- Non-Methane Hydrocarbons (NMHC): Varian 3600 GC or California Analytical Model 600-HFID Analyzer with Non-methane cutter
- Carbon Monoxide (CO): Horiba Model AIA-210LE Non-Dispersive Infrared Detector
- Carbon Dioxide (CO_2): Horiba Model AIA-210 Non-Dispersive Infrared Detector

Each analyzer is calibrated using a commercially available gas standard and maintained as per the Code of Federal Regulations, Volume 40, Part 86. This calibration procedure involves passing gas at varying percentages of the concentration of the selected range (0% to 100% in 10% increments) through the analyzer and recording its electronic response. In the case of NOx, THC and NMHC, the sample is routed to the analyzer by flooding the emissions sample probe which provides the additional check of the integrity of the sampling system. Up to a third order least squares fit of the analyzer response versus concentration curve is then determined and utilized to translate analyzer response to sample concentration. To verify the concentration of the commercial gas standard, as indicated by the supplier, WVU verifies that the indicated bottle concentration is within 1.5% when compared to an NIST standard reference (NIST-SRM).

WVU records each analyzers response to 0% (zero) and 100% (span) gas samples prior to and following each emissions test. If the analyzer response at the zero or span is not within ±0.5%, the analyzer is re-zeroed and re-spanned until both the zero and span responses are within tolerance. If the comparison between pre and post test zero or span exceeds ±2.0 the test is considered invalid.

Dilute exhaust samples are drawn from the dilution tunnel using heated sample probes and lines. The temperature of these devices are monitored and recorded throughout each tests and maintained in the ranges set forth by the Code of Federal Regulations using PID temperature controllers. Any tests where the temperature strays outside the tolerances of the CFR are considered invalid.

While continuous emissions are used to calculate the final emissions result, measurements from a bag sample taken over the duration of the test (integrated bag) are used to check the accuracy of the continuous emissions. While no set deviation limits exist, the test engineer follows good engineering practice and, upon observing excessive deviation between continuous and bag emissions measurement, action is taken to determine and correct the source causing the difference.

B.5.3 Particulate Matter

Particulate matter emissions are determined by passing a proportional sample from the dilution tunnel through two 70mm filters in series as per the CFR. The temperature at the filter face is monitored and recorded throughout the test and maintained below 125°F. Any test where the filter face temperature exceeds 125°F is considered invalid. The mass of particulate on the filters is determined gravimetrically by weighing the filters before and after the test. Prior to either pre-test weighing, use, and post-test weighing, the filters are stored in an environmentally controlled environment for no less than 1 hours with the temperature and humidity maintained at 70°F±5°F and 50%±5% respectively. The weighing device, a Mettler-Toledo Model UMX2, is calibrated according to the manufacturer's procedures and verified using NIST calibration weight prior to each use. Reference filters are also used prior to and within four hours of each weighing session.

B.5.4 Fuel Consumption

Three independent means of determining engine fuel consumption are employed by the laboratory. The primary (reported) means of calculating fuel consumption is accomplished using an inline metering device installed in the fuel supply system and the fuel specific gravity. A second measurement of fuel consumption is taken using the measured carbon (corrected for background) in the dilute exhaust, consisting of CO_2, CO, and THC, and the carbon-to-hydrogen ratio of the fuel as per the Code of Federal Regulations. The integrated fuel flows over a test from both systems are compared to ensure that both devices are working properly. The third method is determined gravimetrically by recording the fuel mass before and after the test. The comparisons between the measurements are examined. Firstly, if the difference between the measurements exceeds 5%, an investigation into the cause for the difference is initiated and, if the problem is found to be in the primary fuel consumption measurement system and the test measurement cannot be corrected for the error, the test is considered invalid. Secondly, any test-to-test variance of the ratio of the fuel consumption determined using a carbon balance (CFR) and that measured by the in-line fuel meter that exceeds 5% for a set of runs using the same test procedure and engine configuration must be investigated, the reason for the excessive variation noted and, if necessary, additional tests run to satisfy the demands of the test protocol.

B.5.5 Dynamometer and Engine Work

Engine torque is calculated from the torque measured from a side-arm torque cell mounted on the engine dynamometer and the torque arising from the engine having to accelerate the mass of the dynamometer rotating elements. The side-arm torque cell is calibrated by hanging weights from the side-arms mounted on either side of the dynamometer while recording the electronic response of the cell. A third order least squares fit is made to the applied torque vs. measured response data to establish a calibration curve. Engine (and dynamometer) speed is measured using a counter which measures the frequency at which the teeth on a toothed wheel attached to the dynamometer shaft pass through a magnetic field. This frequency is converted to a voltage which is passed to the data acquisition computer where a fixed calibration coefficient is used to convert the voltage to a rotational speed. Speed calibration is accomplished by attaching a function generator to the input of the frequency to voltage converter and adjusting the frequency to voltage converter to such that the indicated speed is correct.

The work performed by the engine is calculated using the speed and torque recorded during each engine test and compared to that of other tests performed using the same test procedure and engine configuration.

B.5.6 Data Review, Oversight and Record Keeping

An independent Quality Assurance Quality Control (QA/QC) engineer will oversee the data collection and data processing. The engineer will not participate in the data collection or data reduction phases of the work. The QA/QC engineer will review and identify discrepancies in the data or data collection process, in writing, to the Project Director. Any changes or modifications in the dataset will be identified in the final report.

WVU will maintain a test log that identifies all tests conducted, all engine mapping procedures, all physical modifications or operational tests of the engine, all re-calibrations or other changes to the test instruments, and any interruption between tests and the reason for the interruption. In addition, WVU will maintain chain of custody logs for the fuel, fuel samples retained at WVU, TPM filters, and NMHC bag samples. WVU will conduct data reduction and report average results for the regulated and non regulated measurements for the reference and candidate fuels.

B.6 Instrument/Equipment Testing, Inspection and Maintenance

All dynamometer and emissions measurement equipment will be tested and inspected as per laboratory standard operating procedures. The procedures listed in section E must be preformed prior to initiating the test sequence and a copy of the completed record for each procedure must be made and retained in the project record.

B.7 Instrument/Equipment Calibration and Frequency

The dynamometer test bed will be calibrated as per standard operating procedures while the primary and secondary analyzers, dilution tunnel and associated instrumentation will be calibrated as per 40 CFR Part 86. The instrumentation used for the NMHC by the GC method will be calibrated as outlined in SAE J1151.

B.8 Inspection/Acceptance of Supplies and Consumables

The fuel, analytical gases, filters, gas sample bags, fuel sample containers, and oil sample containers will be purchased and remain in the custody of WVU personnel. Existing suppliers of these consumables will be used for the test program; no deviations of normal laboratory operating procedures are planned. Any deviation from normal practices will be noted.

B.9 Non-Direct Measurements

All measurements will be direct.

B.10 Data Management

Electronic data collected during this testing will be transferred to the data reduction machine for post-test processing. The data will be reduced on the reduction computer using the latest revision of the reduction programs and short reports will be generated for QA review. Upon completion of the test program and delivery of the final report, a copy of the electronic data and of the final report will be archived using a removable electronic storage device (CD/DVD).

C ASSESSMENT AND OVERSIGHT

C.1 Assessments and Response Actions

The first level of data review will take place at the level of the Test Engineer. Using data from previous tests on this engine and good engineering practice, the test engineer will review engine and dynamometer operating parameters as well as the recorded output data from selected temperature, pressure, and analyzer responses throughout the test. The Test Engineer will observe the response of the analyzers and sampling system flow rates and temperatures. Post-test, on-site data review is the responsibility of the Test Engineer.

C.1.1 Identification of Outliers

Outliers will be identified in a two-tier approach. First, if a test is invalid due to deviation from the specified test procedure or an identifiable equipment failure and it cannot be corrected using a redundant measurement then it will be marked as invalid and the data will be removed from the dataset. The dataset will be annotated for the known physical reason(s) for excluding the run(s) from the rest of the data. Second, if there are no known physical reasons why the run(s) should be excluded from the dataset, the statistical significance of the disputed run(s) will be examined. The methodology described in Schiff and D' Agostino (1996) will be employed for the dataset with a 1% significance level. It is noted that the second approach is the same methodology employed in ASTM E178-02 (2002), Section 6.

C.2 Reports to Management

The data reported to the QA Officer for review will include both the test program package containing the records of all calibrations and checks preformed prior to initiating testing, the analyzer calibration record, the Test Day History, and Test History sheets for each test.

D DATA VALIDATION AND USABILITY

D.1 Data Review, Verification, and Validation

The QA Officer will review both the continuous data and integrated results from the data reduction program to verify that the data was acquired and recorded correctly. The QA Officer will also verify the operation of the electronic reduction program. Validation of the final reported data will be contingent upon the continuous and integrated data meeting the requirements of 40 CFR Part 86, Subpart N with test-to-test coefficient of variance below those given above.

D.2 Verification and Validation Methods

The short reports and continuous data generated by the laboratory reduction program will be compared with records of prior data for this engine. Additionally, independent measurements will be compared to the reported test methods. These measurements include low and high range CO, gravimetric and fuel flow comparison to dilution tunnel reported fuel flow, public broadcast of the engine control unit (ECU) data, a second independent NOx sample system, and the comparison of the PM to the Rupprecht and Patashnick Co., Inc. 1105 TEOM.

D.3 Reconciliation with User Requirements

CAFEE will supply the reduced data in the draft and final report and will provide a basic interpretation of that data. Coefficient of variance of reported data requirements are as specified in A.4. Any deviation from the test program, any outlier determinations, or any data discarded will be noted in the report

E FORMS AND RECORDS

E.1 Analytical Equipment History

West Virginia University

Engine and Emissions Laboratories Gas anlyzers and Bottle History

Engine Model No.: ____________

Engine Serial No.: ____________

Engineer: ____________ Date: ____________

Project: ____________

Dynamometer

Model Number: ____________

Serial Number: ____________

Comment: ____________

Gas Analyzers

Analyzer	Model No.	Serial No.	Comment
THC			
lo CO			
hi CO			
CO_2			
NOx			
Zero Air			
Nox -2			

Gas Bottles

Gas Comp.	Conc.	Named Conc.	Pressure (psig)	Part No	Cyl No
Propane					
lo CO					
hi CO					
CO_2					
NOx					
Zero Air					
N_2					
FID					

Changes

Date	Description

E.2 Test Day History

West Virginia University

Engine and Emissions Laboratories Test Day History

Engine Model No.: ____________________

Engine Serial No.: ____________________

Engineer: ____________________ Date: __________

Project:

Time	Event/Test Name	Event/Test Description

E.3 Fuel System Barrel History

West Virginia University

Engine and Emissions Laboratories Fuel Barrel Tracking

BRL

XX

Fuel Description: ____________________

Refinery/Manuf. Barrel ID: ____________________

WVU ID: ____________________

Arrival Date: ____________________ Time: ____________________

Engineer: ____________________

Project:

Comments: ____________________

Modification(s) to Fuel

Date	Time	Engineer	Description

E.4 Fuel System History

West Virginia University

Engine and Emissions Laboratories Engine Fuel Tracking

Engine Model No.: ____________________

Engine Serial No.: ____________________

Project:

Fuel Filter Change Information

Date of Fuel Filter Change: ________ Fuel Type: ________ Fuel Name: ________

Filter Manufacturer(s): ____________________

Filter Number(s): ____________________

Fuel Change/Sample

Date	Time	Engineer	Description

E.5 Oil History

West Virginia University

Engine and Emissions Laboratories Engine Oil Consumption

Engine Model No.: ____________________

Engine Serial No.: ____________________

Project: ____________________

Oil Change Information

Date of Oil/Filter Change: ________ Oil Type: ________ Oil Brand: ________

Filter Manufacturer(s): ____________________

Filter Number(s): ____________________

Oil Check/Addition/Sample

Date	Time	Engineer	Description

E.6 Engine Setup and Mapping

West Virginia University

Engine and Emissions Laboratories Engine Intake and Exhaust Setup

Project: ______________________ Engineer: ______________

Date: ______________ Time: ________ Fuel: ______________

Engine Model No.: ______________________________________

Engine Serial No.: ______________________________________

Parameter	Unit	Laboratory (ECU) Value
Engine Speed	rpm	
Engine Torque	ft-lb	
Intake Air Temperature	deg F	
Intake Flow	scfm	
Intake Depression	in H_2O	
Manifold Air Pressure	psig	
Manifold Air Temperature	deg F	
Intercooler Water Inlet Temperature	deg F	
Intercooler Diff Pressure	in H_2O	
Exh Back Press	in H_2O	
Exhaust (Ch 80)	deg F	
Exhaust (Ch 86)	deg F	
Coolant Temperature.	deg F	
Oil Temperature	deg F	
Oil Press	psig	
Fuel Temperature	deg F	
Barometric Pressure	in Hg	
Relative Humidity	%	
Other, Specify		
Other, Specify		
Other, Specify		
Other, Specify		

Comments:

Engine Mapping History

Date	Time	Fuel	Description/Comment

E.7 Field Custody Log

The field custody log is electronic in nature and is accessible to the laboratory. Once testing is complete, a print copy of the field custody log will be generated and included with the other test records.

E.8 Media Tracking Sheet

West Virginia University

Engine and Emissions Laboratories Test Information

Project: ______________________ Test Date: ____________

Test Desc.: ______________________

Test Fuel: ______________________

Engineer: ______________________

Test No.: ____________ Start Type: ☐ Cold ☐ Hot ☐ Warm ☐ Background

	THC	NMHC	Lo CO	Hi CO	CO_2	NOx	NOx²
Pre Test Zero:	☐	☐	☐	☐	☐	☐	☐
Pre Test Span:	☐	☐	☐	☐	☐	☐	☐
Post Test Zero:	☐	☐	☐	☐	☐	☐	☐
Post Test Span:	☐	☐	☐	☐	☐	☐	☐

(Check box(s) if adjustments were made.)

	375 ± 20 °F		235 ± 20 °F	235 ± 20 °F	
Probe	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Line	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Filter	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Pump	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐

(Check boxes if the pre and post test temperatures were within range.)

Pre Test Flow:							
Post Test Flow:							

TPM

TPM Bar Code

Pre: ____________

Post: ____________

Holder: ____________

Test Comment: ____________

NMHC Bags — Dilute ______ Background ______

Fuel Weight — Pre ______ Post ______

Test No.: ____________ Start Type: ☐ Cold ☐ Hot ☐ Warm ☐ Background

	THC	NMHC	Lo CO	Hi CO	CO_2	NOx	NOx²
Pre Test Zero:	☐	☐	☐	☐	☐	☐	☐
Pre Test Span:	☐	☐	☐	☐	☐	☐	☐
Post Test Zero:	☐	☐	☐	☐	☐	☐	☐
Post Test Span:	☐	☐	☐	☐	☐	☐	☐

(Check box(s) if adjustments were made.)

	375 ± 20 °F		235 ± 20 °F	235 ± 20 °F	
Probe	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Line	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Filter	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Pump	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐

(Check boxes if the pre and post test temperatures were within range.)

Pre Test Flow:							
Post Test Flow:							

TPM

TPM Bar Code

Pre: ____________

Post: ____________

Holder: ____________

Test Comment: ____________

NMHC Bags — Dilute ______ Background ______

Fuel Weight — Pre ______ Post ______

Test No.: ______________________ Start Type: ☐ Cold ☐ Hot ☐ Warm ☐ Background

	THC	NMHC	Lo CO	Hi CO	CO_2	NOx	NOx[2]
Pre Test Zero:	☐	☐	☐	☐	☐	☐	☐
Pre Test Span:	☐	☐	☐	☐	☐	☐	☐
Post Test Zero:	☐	☐	☐	☐	☐	☐	☐
Post Test Span:	☐	☐	☐	☐	☐	☐	☐

(Check box(s) if adjustments were made.)

	375 ± 20 °F		235 ± 20 °F	235 ± 20 °F	
Probe	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Line	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Filter	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Pump	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐

(Check boxes if the pre and post test temperatures were within range.)

Pre Test Flow:							
Post Test Flow:							

TPM

TPM Bar Code

Pre: ______________________

Post: ______________________

Holder: ______________________

Test Comment:

NMHC Bags (Date / Background)

Fuel Weight (Pre / Post)

Test No.: ______________________ Start Type: ☐ Cold ☐ Hot ☐ Warm ☐ Background

	THC	NMHC	Lo CO	Hi CO	CO_2	NOx	NOx[2]
Pre Test Zero:	☐	☐	☐	☐	☐	☐	☐
Pre Test Span:	☐	☐	☐	☐	☐	☐	☐
Post Test Zero:	☐	☐	☐	☐	☐	☐	☐
Post Test Span:	☐	☐	☐	☐	☐	☐	☐

(Check box(s) if adjustments were made.)

	375 ± 20 °F		235 ± 20 °F	235 ± 20 °F	
Probe	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Line	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Filter	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐
Pump	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐	☐ / ☐

(Check boxes if the pre and post test temperatures were within range.)

Pre Test Flow:							
Post Test Flow:							

TPM

TPM Bar Code

Pre: ______________________

Post: ______________________

Holder: ______________________

Test Comment:

NMHC Bags (Date / Background)

Fuel Weight (Pre / Post)

Overall Comment:

Data Valid?

Test Engineer: ______________________ Date: __________ Yes ☐ No ☐

QA/QC Engineer: Date: Yes ☐ No ☐

Intertek Caleb Brett

REPORT OF ANALYSIS

Vessel	: SUBMITTED SAMPLE AND ANALYSIS
Port/Terminal	: ORYXE ENERGY
Customer Reference	: ---
Our Reference	: 260-0004211
Date Sample Taken	: ---
Date Submitted	: 05/01/06
Date Tested	: 05/01-02/06
Sample Designated As	: DIESEL FUEL
Drawn By	: AS SUBMITTED
Representing	: OFFICIAL TCEQ BIODIESEL REFERENCE FUEL
Lab Reference	: 06-27620

TEST	METHOD	RESULT	UNITS
Gravity, API	D 287	36.7	---
Viscosity @ 40°C	D 445	3.075	cSt
Flash Point (P.M.C.C.)	D 93A	182	°F
Cloud Point	D 2500	-17	°C
Water & Sediment	D 2709	<0.005	Vol%
Ash Content	D 482	<0.001	Wt%
Total Aromatics by SFC	D 5186	9.1	Wt%
Total Aromatics by SFC	D 5186	9.7	Vol%
PNA by SFC	D 5186	0.6	Wt%
Carbon Residue, Ramsbottom (10% Btms)	D 524	0.08	Wt%
Copper Corrosion (3hrs @ 50°C)	D 130	1a	---
Cetane Number	D 613	49.0	---
Distillation			
-Initial Boiling Point	D 86	392	°F
-5% Recovered	D 86	431	°F
-10% Recovered	D 86	447	°F
-20% Recovered	D 86	466	°F
-30% Recovered	D 86	482	°F
-40% Recovered	D 86	498	°F
-50% Recovered	D 86	511	°F
-60% Recovered	D 86	522	°F
-70% Recovered	D 86	535	°F
-80% Recovered	D 86	548	°F
-90% Recovered	D 86	572	°F
-95% Recovered	D 86	601	°F
-End Point	D 86	620	°F
-%Recovered	D 86	97.5	Vol%
-%Residue	D 86	1.5	Vol%
-%Loss	D 86	1.0	Vol%
Sulfur Content	D 5453	12	ppm
Nitrogen Content	D 4629	6	ppm

INTERTEK Caleb Brett

Los Angeles Laboratory
1941 Freeman Ave, Suite A Signal Hill, Ca 90755

Intertek Caleb Brett

REPORT OF ANALYSIS

Vessel	:	SUBMITTED SAMPLE AND ANALYSIS
Port/Terminal	:	ORYXE ENERGY
Customer Reference	:	---
Our Reference	:	260-0004211
Date Sample Taken	:	---
Date Submitted	:	05/01/06
Date Tested	:	05/01-02/06
Sample Designated As	:	DIESEL FUEL
Drawn By	:	AS SUBMITTED
Representing	:	OFFICIAL TCEQ B-20 UNTREATED CANDIDATE FUEL
Lab Reference	:	06-27621

TEST	METHOD	RESULT	UNITS
Gravity, API	D 287	35.0 / 35.0 / 35.0	---
Viscosity @ 40°C	D 445	3.187 / 3.189 / 3.180	cSt
Flash Point (P.M.C.C.)	D 93A	186 / 186 / 186	°F
Cloud Point	D 2500	-15	°C
Water & Sediment	D 2709	0.005	Vol%
Ash Content	D 482	<0.001	Wt%
Total Aromatics by SFC	D 5186	9.0 / 9.0 / 9.0	Wt%
Total Aromatics by SFC	D 5186	9.5 / 9.6 / 9.6	Vol%
PNA by SFC	D 5186	0.6 / 0.6 / 0.6	Wt%
Carbon Residue, Ramsbottom (10% Btms)	D 524	0.09	Wt%
Copper Corrosion (3hrs @ 50°C)	D 130	1a	---
Cetane Number	D 613	48.0 / 48.1 / 47.8	---
Distillation			
-Initial Boiling Point	D 86	414 / 413 / 416	°F
-5% Recovered	D 86	442 / 443 / 445	°F
-10% Recovered	D 86	458 / 456 / 458	°F
-20% Recovered	D 86	480 / 479 / 479	°F
-30% Recovered	D 86	500 / 498 / 500	°F
-40% Recovered	D 86	518 / 517 / 518	°F
-50% Recovered	D 86	533 / 533 / 534	°F
-60% Recovered	D 86	550 / 550 / 551	°F
-70% Recovered	D 86	569 / 569 / 570	°F
-80% Recovered	D 86	593 / 593 / 594	°F
-90% Recovered	D 86	621 / 621 / 623	°F
-95% Recovered	D 86	640 / 637 / 640	°F
-End Point	D 86	646 / 645 / 649	°F
-%Recovered	D 86	97.9 / 98.2 / 97.6	Vol%
-%Residue	D 86	1.4 / 1.4 / 1.3	Vol%
-%Loss	D 86	0.7 / 0.4 / 1.1	Vol%
Sulfur Content	D 5453	12 / 12 / 12	ppm
Nitrogen Content	D 4629	5 / 5 / 5	ppm

INTERTEK Caleb Brett

Los Angeles Laboratory
1941 Freeman Ave, Suite A Signal Hill, Ca 90755

Intertek Caleb Brett

REPORT OF ANALYSIS

Vessel	:	SUBMITTED SAMPLE AND ANALYSIS
Port/Terminal	:	ORYXE ENERGY
Customer Reference	:	---
Our Reference	:	260-0004211
Date Sample Taken	:	---
Date Submitted	:	05/01/06
Date Tested	:	05/01-02/06
Sample Designated As	:	DIESEL FUEL
Drawn By	:	AS SUBMITTED
Representing	:	B-100 SOY BIODIESEL
Lab Reference	:	06-27622

TEST	METHOD	RESULT	UNITS
Viscosity @ 40°C	D 445	4.138	cSt
Flash Point (P.M.C.C.)	D 93A	320	°F
Cloud Point	D 2500	+3	°C
Water & Sediment	D 2709	0.005	Vol%
Sulfated Ash	D 874	<0.001	Wt%
Carbon Residue, MCRT	D 524	0.01	Wt%
Phosphorous Content	D 4951	<0.00001	Wt%
Acid Number	D 664	0.49	mgKOH/g
Copper Corrosion (3hrs @ 50°C)	D 130	1a	---
Cetane Number	D 613	47	---
Vacuum Distillation			
-Initial Boiling Point	D 1160	653	°F
-5% Recovered	D 1160	656	°F
-10% Recovered	D 1160	658	°F
-20% Recovered	D 1160	659	°F
-30% Recovered	D 1160	660	°F
-40% Recovered	D 1160	661	°F
-50% Recovered	D 1160	662	°F
-60% Recovered	D 1160	663	°F
-70% Recovered	D 1160	664	°F
-80% Recovered	D 1160	666	°F
-90% Recovered	D 1160	668	°F
-95% Recovered	D 1160	690	°F
-End Point	D 1160	828	°F
-%Recovered	D 1160	97.3	Vol%
-%Residue + %Loss	D 1160	2.7	Vol%
Sulfur Content	D 5453	<1	ppm
Free Glycerin	D 6584	<0.001	Wt%
Total Glycerin	D 6584	0.137	Wt%

INTERTEK Caleb Brett

Los Angeles Laboratory
1941 Freeman Ave, Suite A Signal Hill, Ca 90755

West Central
PO Box 68
406 1st Street
Ralston, IA 51459
Phone: 712-667-3380
Fax: 712-667-3679
www.soypower.net

Certificate of Analysis

Order Number:			
Shipping Date:		**Rail/Truck Number:**	
Customer:		**Attention:**	
Street Number:		**Post Office Box:**	
City:		**State:**	**Zip Code:**

Lot Number:	060308106

Test	Results	Limits	Units	ASTM Test Method
Free Glycerin:	0.000	0.020 max.	% Mass	D 6584
Total Glycerin:	0.090	0.240 max.	% Mass	D 6584
Cloud point:	1	n/a	°C	D 2500
Flashpoint:	146	130 min.	°C	D 93
Water & Sediment:	0.03	0.050 max.	% Vol.	D 2709
Acid Number:	0.62	0.80 max.	mg KOH/g	D 664
Visual Inspection:	1	2 max	Haze	D 4176, Procedure 2
Carbon Residue:	0.012*	0.050 max.	% Mass	D 4530
Sulfated Ash:	0.0030*	0.020 max.	% Mass	D 874
Kinematic Viscosity at 40 °C:	4.184*	1.9-6.0	mm^2/sec.	D 445
Sulfur:	0.001*	0.050 max.	% Mass	D 5453
Cetane Number:	52*	47 min.	n/a	D 613
Copper Corrosion:	1A*	No. 3 max.	n/a	D 130
Phosphorus:	0.0008*	0.001 max.	% Mass	D 4951
Distillation at 90% Recovered:	353*	360 max.	°C	D 1160

Prepared by: *Rob Knoll* 3/9/2006

Rob Knoll, Laboratory Technologist, West Central

* Typical result