NTRD Program Disclaimers

1. Disclaimer of Endorsement:

The posting herein of progress reports and final reports provided to TCEQ by its NTRD Grant Agreement recipients does not necessarily constitute or imply an endorsement, recommendation, or favoring by TCEQ or the State of Texas. The views and opinions expressed in said reports do not necessarily state or reflect those of TCEQ or the State of Texas, and shall not be used for advertising or product endorsement purposes.

2. Disclaimer of Liability:

The posting herein of progress reports and final reports provided to TCEQ by its NTRD Grant Agreement recipients does not constitute by TCEQ or the State of Texas the making of any warranty, express or implied, including the warranties of merchantability and fitness for a particular purpose, and such entities do not assume any legal liability or responsibility for the accuracy, completeness, or usefulness of any information,apparatus, product, or process disclosed, or represent that its use would not infringe privately owned rights.

New Technology Research & Development Program
Grant Contract 582-5-70807-T032

Task 1 Deliverable 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.

ORYXE ENERGY INTERNATIONAL, INC.
6 Thomas Avenue
Irvine, California 92618 USA
Telephone: (949) 452-9272
Facsimile: (949) 452-9274

November 18, 2005

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

Dear Mr. Shankle:

ORYXE Energy International, Inc. (ORYXE Energy) intends to begin official testing of OR-LED2 (an updated formulation of our current OR-LED additive), pursuant to Section 114.312 (f) and 114.315 (c) of Title 30 of the Texas Administrative Code (Alternative Diesel Fuel Formulations). 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. It is ORYXE Energy's understanding that successful completion of this program will enable transition of our approval status under the TxLED regulation from the current "alternative plan" to the "alternative fuel formulation" category, and that this transition is consistent with TCEQ's future vision of the direction of the TxLED program.

Attached to this document, Exhibit A, you will find 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. This is Alternative 4. We request your approval of the protocol as official testing will begin **December 5, 2005** at West Virginia University (WVU).

Exhibit A also discusses the reference and candidate fuels selected for this test. The reference fuel has been analyzed for its required fuel properties in triplicate by Caleb Brett in Signal Hill, California and is attached. What should be a representative sample of the candidate fuel has been analyzed for its required fuel properties once by Caleb Brett in Signal Hill, California and is attached. The required triplicate fuel property analysis of the actual candidate fuel to be used in our testing program shall be completed by Caleb Brett in Signal Hill, California by next week and forwarded to you by no later than Friday, November 25, 2005 -- well within the seven days notice period now required by the TCEQ.

Detailed instructions on how to blend/mix the OR-LED2 additive into the candidate fuel, the chemical composition of OR-LED2, and the treat rate of OR-LED2 are all listed on Exhibit B that is attached to this letter but found within a separate brown envelope and marked "Confidential/Proprietary: inform applicant and seek AG opinion before releasing." For your information, OR-LED2 uses the same

ingredients as our current OR-LED additive but at different ratios and is used at an overall lower treat rate. OR-LED's U.S. Environmental Protection Agency's registration number is 192220004. The procedure used to identify the OR-LED2 additive concentration has been developed in-house and is attached hereto as Exhibit C.

The test data set generated from this testing program will be reviewed by WVU personnel for outliers using ASTM E178, "Recommended Practice for Dealing with Outlying Observations."

After you and your staff have had the opportunity to review the enclosed information, please contact either myself or our consultant, Gregg Cooke, 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,

FOR

Nick Economides
Executive Vice President,
Commercial Products and Technology Management

Enclosures (3): Exhibit A – Proposed Test Protocol
Exhibit B – Chemical Composition of OR-LED2
Exhibit C – Analytical Procedure to Identify the Concentration of OR-LED2

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-LED2) can be used to produce Texas Low Emission Diesel (TxLED) 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-LED2 via 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-LED2-treated 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, representative ultra low sulfur U.S. Environmental Protection Agency diesel with an additive (OR-LED2) to qualify as TxLED and help the State of Texas maximize both on-road and off-road mobile NO_X reductions. Full adoption of this product could provide annual NO_X reductions in excess of 8,100 tons in the 110 noncompliant counties requiring TxLED.

Success of this project will demonstrate the applicability of ORYXE Energy International, Inc.'s OR-LED2 product to achieve cost effective TxLED emission reduction compliance in diesel fuel 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. 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 such as 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 (post-1990 model year) is the engine specified in the TAC §114.315(c)(4)(A) for carrying out the test program being proposed in this application.

(3) Testing Fuels

The two fuels ORYXE Energy proposes to use at WVU using the DDC-60 is a reference fuel and a candidate, each of which is discussed in further detail, below.

Reference Fuel

ORYXE Energy proposes to use a low total aromatics reference fuel. The attached fuel property analysis was carried out on the reference fuel in triplicate 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).

Candidate Fuel

ORYXE Energy proposes to use a high total aromatics candidate fuel. The attached fuel property analysis (untreated with OR-LED2) was carried out (once) on a representative sample of the candidate fuel by Caleb Brett in Signal Hill, California and is attached. In accordance with TAC §114.315(c)(2)(A), a triplicate fuel property analysis of the actual candidate fuel to be used during this test program shall be conducted and forwarded by no later than Friday, November 25, 2005, well within the TCEQ required seven day notice period. The final candidate fuel to be used shall conform to the diesel fuel standards listed in the American Society for Testing and Materials (ASTM) D-975 method, except for lubricity. The final candidate fuel properties will also satisfy TAC §114.315(c)(1)(C).

(4) Testing Protocol

The testing protocol ORYXE Energy proposes to run at WVU using the DDC-60 and the final 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 treated candidate fuel FTP). Details are discussed and described below.

Overview

Proposed Testing Protocol
Day 1 – R R
Days 2-3 – C [Conditioning the engine (not to exceed 48 total hours)] – Untreated
Day 4 – C C – Treated
Day 5 – C – Treated
Days 6- R R

Key

R	Reference fuel meeting specifications in TAC §114.315(c)(3)(A-I) (3 "hot" starts)
C	Candidate fuel meeting specifications in TAC §114.315(c)(1)(C) (3 "hot" starts). Depending on the day, this fuel will either be treated with ORYXE Energy's OR-LED2 additive or not.

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 the TAC §114.315(c)(4)(C)(iv).

On Day 2, ORYXE Energy proposes to initiate the conditioning of the DDC-60 engine using the untreated candidate fuel for no more than 48 total hours (2 days) of engine operation. This conditioning period will be run in accordance with and will represent normal engine operation.

On Day 4, ORYXE Energy proposes to carry out six FTP "hot" start tests using the OR-LED2-treated candidate fuel.

On Day 5, ORYXE Energy proposes to carry out only three FTP "hot" start tests using the OR-LED2-treated candidate fuel.

On Day 6, 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 6 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 OR-LED2-treated candidate fuel run on Day 4 and Day 5 (this includes the six "hots" on Day 4 and the three "hots" on Day 5) 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 be run with the untreated candidate fuel on Day 2 and Day 3 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 OR-LED2-treated 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).

* * * *

November 25, 2005

Via Federal Express

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

Re: Supplemental Information Provided for Approval to Conduct Texas Low Emission Diesel Alternative Fuel Formulation Testing

Dear Mr. Shankle:

This letter is being sent as a supplement to the letter sent to you dated November 18, 2005. In this letter, ORYXE Energy International, Inc. (ORYXE Energy) indicates its desire to begin official testing of OR-LED2 pursuant to Section 114.312(f) and 114.315(c) of Title 30 of the Texas Administrative Code (TAC) (Alternative Diesel Fuel Formulations) by December 5, 2005 (if possible). Therefore, in order to make our official application complete, enclosed you will find the following three (3) supplemental pieces of information:

- Candidate Fuel Analysis – In accordance with TAC §114.315(c)(2)(A), a triplicate fuel property analysis of the candidate fuel to be used during our testing program (untreated with OR-LED2) is attached. This 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); and

- Reference Fuel Analysis - In accordance with TAC §114.315(c)(3), a triplicate fuel property analysis of the reference fuel to be used during our testing program is attached. This reference 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)(D). The extra tests performed in this analysis while not required were added to demonstrate compliance with ASTM D-975; and

- Exhibit C -- Analytical Procedure – In accordance with TAC §114.315(c)(2)(B), a analytical test method to detect the identity and concentration of OR-LED2 in the candidate fuel is attached but within a separate brown envelope 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 either myself or our consultant, Gregg Cooke, 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

c.c. David Schanbacher (via FedEx)
Morris Brown (via FedEx)

Enclosures (2): Candidate Fuel Analysis
Exhibit C – Analytical Procedure to Identify the Concentration of OR-LED2

Intertek Caleb Brett

REPORT OF ANALYSIS

Vessel	: SUBMITTED SAMPLE AND ANALYSIS
Port/Terminal	: ORYXE ENERGY
Customer Reference	: ---
Our Reference	: 260-0003109
Date Sample Taken	: ---
Date Submitted	: 11/23/05
Date Tested	: 11/23-24/05
Sample Designated As	: DIESEL
Drawn By	: AS SUBMITTED
Representing	: CANDIDATE FUEL
Lab Reference	: 05-19090

TEST	METHOD	RESULT	UNITS
Gravity, API	D 4052	38.5	---
Sulfur Content	D 5453	2 / 2 / 2	ppm
Flash Point	D 93A	164	°F
Viscosity @ 40°C	D 445	2.458	cSt
Total Aromatics by SFC	D 5186	29.4 / 29.5 / 29.4	Wt%
Total Aromatics by SFC	D 5186	28.3 / 28.3 / 28.3	Vol%
PNA by SFC	D 5186	2.8 / 2.8 / 2.8	Wt%
Cetane Number (1^{st},2^{nd},3^{rd})	D 613	47.0 / 47.6 / 47.6	---
Distillation			
-Initial Boiling Point	D 86	352	°F
-5% Recovered	D 86	389	°F
-10% Recovered	D 86	405	°F
-20% Recovered	D 86	430	°F
-30% Recovered	D 86	457	°F
-40% Recovered	D 86	487	°F
-50% Recovered	D 86	512	°F
-60% Recovered	D 86	528	°F
-70% Recovered	D 86	540	°F
-80% Recovered	D 86	551	°F
-90% Recovered	D 86	569	°F
-95% Recovered	D 86	596	°F
-End Point	D 86	613	°F
-%Recovered	D 86	97.6	Vol%
-%Residue	D 86	1.8	Vol%
-%Loss	D 86	0.6	Vol%
Nitrogen Content	D 4629	<1 / <1 / <1	ppm
Water and Sediment	D 2709	0.005	Wt%
Ash Content	D 482	<0.001	Wt%
Copper Corrosion 3hrs @ 50°C	D 130	1a	---
Cloud Point	D 2500	-10	°C
Carbon Residue (10% Ramsbottom)	D 524	<0.1	Wt%

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	: 260-0003109
Date Sample Taken	: ---
Date Submitted	: 11/14, 16/05
Date Tested	: 11/14, 16-18, 21/05
Sample Designated As	: DIESEL FUEL
Drawn By	: AS SUBMITTED
Representing	: REFERENCE FUEL
Lab Reference	: 05-18690, 05-18567

TEST	METHOD	RESULT	UNITS
Gravity, API	D 287	36.8 / 36.8 / 36.8	---
Viscosity @ 40°C	D 445	3.283/3.284/3.263	cSt
Flash Point (P.M.C.C.)	D 93A	188 / 188 / 190	°F
Water & Sediment	D 2709	0.005	Vol%
Ash Content	D 482	0.004	Wt%
Total Aromatics by SFC	D 5186	9.3 / 9.5 / 9.3	Wt%
Total Aromatics	D 5186	9.8 / 10.0 / 9.8	Vol%
PNA by SFC	D 5186	0.7 / 0.8 / 0.6	Wt%
Carbon Residue, Ramsbottom (10% Btms)	D 524	< 0.1	Wt%
Cetane Number	D 613	49.3 / 49.3 / 49.1	---
Cloud Point	D 2500	-16	°C
Copper Corrosion (3hrs @ 50°C)	D 130	1a	---
Distillation			
-Initial Boiling Point	D 86	408 / 408 / 403	°F
-5% Recovered	D 86	437 / 438 / 440	°F
-10% Recovered	D 86	454 / 454 / 454	°F
-20% Recovered	D 86	473 / 474 / 474	°F
-30% Recovered	D 86	488 / 487 / 489	°F
-40% Recovered	D 86	501 / 502 / 502	°F
-50% Recovered	D 86	514 / 514 / 515	°F
-60% Recovered	D 86	526 / 525 / 527	°F
-70% Recovered	D 86	537 / 537 / 538	°F
-80% Recovered	D 86	550 / 550 / 550	°F
-90% Recovered	D 86	571 / 570 / 571	°F
-95% Recovered	D 86	597 / 594 / 601	°F
-End Point	D 86	624 / 622 / 624	°F
-%Recovered	D 86	98.3 / 98.4 / 98.1	Vol%
-%Residue	D 86	1.2 / 1.2 / 1.4	Vol%
-%Loss	D 86	0.5 / 0.4 / 0.5	Vol%
Nitrogen Content	D 4629	5 / 4 / 5	ppm
Sulfur Content	D 5453	16 / 14 / 14	ppm
Carbon Content	D 5291	86.40/86.41/86.40	Wt%
Hydrogen Content	D 5291	13.58/13.50/13.56	Wt%

INTERTEK Caleb Brett

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

EXHIBIT B

CHEMICAL COMPOSITION OF OR-LED2

The following are detailed instructions on how to blend/mix the OR-LED2 additive into the candidate fuel, the chemical composition of OR-LED2, and the treat rate of OR-LED2.

Blending Instructions

Mix the OR-LED2 additive into the candidate fuel as follows:

Step 1: Measure the requisite amount of OR-LED2 additive and pour it into a graduated cylinder
Step 2: Pour off the graduated cylinder into the "feed" drum containing the candidate fuel.
Step 3: Seal the 16-gallon drum tight.
Step 4: Mix the treated candidate fuel thoroughly to ensure that the OR-LED2 additive and the candidate fuel are homogeneously mixed. This can be accomplished by any reasonable and/or traditional means, including, but not limited to, mechanical mixer(s), drum roller(s), stirrer(s) or manual drum rotating/rolling.

In general, each of the steps above shall be done so that the additive is minimally exposed to light and air. Nitrogen "blanketing," or use of another inert gas in order to create an oxygen-free atmosphere is necessary when storing OR-LED2.

Chemical Composition

The chemical composition of OR-LED2 is as follows:

- Isomixtene (a vitamin precursor to beta-carotene),
- Santoquin (an antioxidant used in the feed industry),
- 2 Ethylhexyl Nitrate (a common cetane improver used in diesel fuel), and
- Toluene (a component compatible with diesel fuel).

Treat Rate

The treat rate of OR-LED2 is 12 mls/gallon of diesel fuel treated.

ORYXE Energy International, Inc. seeks confidentiality of all this information due to the fact that the above listed blending instructions, components and treat rate are not publicly known and are in fact confidential/proprietary information. ORYXE Energy International, Inc. therefore requires all of this information to be kept as trade secrets.

* * * *

EXHIBIT C

ANALYTICAL PROCEDURE TO IDENTIFY THE CONCENTRATION OF OR-LED2

See the attached analytical procedure used to detect the identity and concentration of OR-LED2 in the candidate fuel.

ORYXE Energy International, Inc. seeks confidentiality of all of the attached information due to the fact that it is not publicly known and is in fact confidential/proprietary information. ORYXE Energy International, Inc. therefore requires all of this information to be kept as a trade secret.

Standard Test Method
For Isomixtene in Treated Diesel Fuels

This method is approved by ORYXE Energy International for the detection of Isomixtene in the OR-LED.

1. Scope

1.1 This method describes a procedure for determining the amount of Isomixtene present in treated diesel fuels. It is applicable to diesel fuels over the concentration range of 2 – 10 mg/L of Isomixtene. Higher concentrations may also be determined by adjusting the sample dilution to the appropriate range.

Note 1: This method is suitable for determining the total of cis plus trans isomers of β-carotene in diesel fuels. The method is not suitable for determining the presence of the amount of cis or trans isomers of β-carotene in treated diesel fuels. A High Performance Liquid Chromatography method should be used for determining the isomers of β-carotene present in treated diesel fuels.

1.2 *This method does not address all of the safety concerns associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of the Test Method

2.1 A treated diesel fuel is diluted with cyclohexane and the absorbance of the diluted fuel is measured at 458 and 484 nm using the diluted untreated fuel as the reference blank. The amount of Isomixtene is calculated using the absorptivity of Isomixtene in diluted diesel fuel as determined at the Oryxe Energy Laboratory.

Note 2: A sample of the untreated diesel fuel is required for each different treated fuel to be analyzed. The untreated fuel must be used for the reference blank in this analysis. Failure to use the appropriate untreated fuel as the reference blank will lead to erroneous results for Isomixtene content of treated diesel fuels. If a sample of the untreated fuel is not available, an alternate method of analysis such as HPLC must be used for the analysis of Isomixtene.

3. Significance and Use

3.1 This method can be used to determine the amount of Isomixtene in treated diesel fuels. The method is applicable to diesel fuels which have been treated with Oryxe OR series additives. This procedure can be used to verify that diesel fuels have been dosed correctly and to monitor the stability of Isomixtene in diesel fuels.

4. Interferences

4.1 Foreign substances present in diesel fuels, which absorb at 455 or 483 nm can interfere with the method. If contamination of a diesel fuel is suspected, acquire a complete absorption spectrum over the range of 350 – 600 nm and examine for unexpected absorption maxima. Should unexpected peaks appear contact laboratory staff at Oryxe Energy International, Inc.

5. Apparatus

5.1 *Double Beam UV-VIS Spectrophotometer,* capable of recording an absorption spectrum over the range of 350 – 600 nm.

5.2 *Quartz Absorption Cells,* matched pair, 1-cm with stoppers.

5.3 *Suitable Micropipettes*, for transferring fuel to absorption cells.

5.4 *Lint Free Wipes,* for cleaning windows of the absorption cells.

5.5 *Volumetric Flasks*, 25 mL, low-actinic glassware only.

5.6 *Volumetric Pipettes*, 10 mL, Class A only.

6. Reagents

6.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

6.2 *Acetone*, reagent grade for cleaning absorption cells.

6.3 *Compressed Nitrogen Gas*, instrument grade for drying absorption cells, compressed air may also be used provided that it is instrument grade and oil free.

6.4 *Cyclohexane.*

7. Sampling

7.1 Samples shall be collected in accordance with Practice D 4057.

7.2 Use of metallic or brown glass containers for samples is recommended.

7.3 Samples should be protected from entrained air, light and heat. Rinse container with sample and discard. Fill container to the top to minimize air in the headspace of the container and analyze immediately.

8. Instrument Settings and Preparation

8.1 Turn on power to the spectrophotometer and insure that the tungsten lamp is turned on. Allow the lamp to warm up for 15 minutes before starting measurements.

8.2 Obtain a sample of the untreated diesel fuel for each treated fuel to be analyzed. Using a volumetric pipet transfer 10 mL of the untreated fuel to a 25 mL low actinic volumetric flask and dilute to the mark with cyclohexane.

8.2 After the spectrophotometer has warmed up for 15 minutes, place a 1-cm quartz cell containing the diluted untreated diesel fuel (Sec 8.2) in the sample side of the sample compartment. Wipe the clear windows of the absorption cell with a lint free wipe to remove any residual liquid or fingerprints from the cell. Clean and wipe the windows of a second 1 cm quartz cell containing the diluted untreated diesel fuel (Sec 8.2) and place in the reference side of the sample compartment. Ensure that the clear windows of the cells are aligned with the light beam.

8.3 Adjust the zero level of the instrument at 458 and 484 nm. Remove the quartz cell from the sample compartment, dispose of the contents, clean cell with acetone and dry with compressed nitrogen gas.

9. Procedure

9.1 Using a volumetric pipet, transfer 10 mL treated diesel fuel to a 25 mL low-actinic volumetric flask and dilute to the mark with Cyclohexane.

9.2 Using a micropipette transfer diluted fuel into a clean and dry 1-cm quartz absorption cell. Fill to approximately 80% of the cell capacity and secure stopper.

9.3 If diluted diesel fuel has spilled on to the quartz windows of the cell, clean with cyclohexane and wipe dry with lint free wipe.

9.4 Place cells containing diluted sample in a dark storage area until ready to measure absorbance.

9.5 Set the spectrophotometer to measure absorbance of samples at 458 and 484 nm. Measure each sample three times using an integration time of three seconds for each measurement.

9.6 After the measurements and made, remove the quartz cells from the cell holders, pour the contents into an appropriate waste container, clean the cells with acetone and dry with compressed air/nitrogen.

10. Calculations

10.1 Obtain a hardcopy of the absorbance for the samples and calculate Isomixtene concentration using the following equation:

$$[ISO]_{mg/L} = \frac{(A + 0.050)\ x\ 10{,}000\ x\ D}{2{,}643} \qquad \text{Eq. 1}$$

Where:

A = Absorbance of sample at 458 nm
D = Dilution factor for sample, 2.5 (Sec. 9.1)
10,000 = Conversion factor for absorptivity from % to mg/L
2,643 = Absorptivity for Isomixtene $E_{1\%,1cm}$ in diluted diesel fuel
0.050 = Calibration curve intercept

Equation 1 can be simplified to the following providing the dilution factor is 2.5 as noted above:

$$[ISO]_{mg/L} = (A + 0.050)\ x\ 9.459 \qquad \text{Eq. 2}$$

Where:

A = Absorbance of sample at 458 nm

11. Precision and Bias

11.1 *Repeatability* - Within the same laboratory, duplicate results are expected to differ from each other by no more than 10% relative.

11.2 *Bias* – The accuracy of this method is based upon the absorptivity of Isomixtene in diesel fuel. Provided that the procedure outlined above is followed closely, the accuracy of this method is estimated to be better than 20% relative.

12. Key Words

12.1 Isomixtene, diesel fuels, treated.