

Final Technical Report

Project Title: Optimized Co-Processing of Algae Bio-Crude through a Petroleum Refinery
Award Number: DE-EE0006065

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Reporting Period: January 13th through February 28, 2014
Date of Report: March 14, 2014
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Project goal/ Objectives

- Sapphire produces an algae bio-crude that is similar to petroleum crude oil
- This project will establish the optimal refinery operating conditions required to upgrade algae bio-crude produced via the Sapphire process
- Identifying the operating conditions required for algae crude upgrading is critical for bringing algae bio-crude to market and to establish the sales price and full value chain economics of the algae derived biofuels
- Alignment with DE-FOA-0000686:
- Algae bio-crude oil is produced in a sustainable manner from algae biomass produced in open ponds from captured CO₂ and sunlight
 - Algae bio-crude is not a finished transportation fuel and must be upgraded
 - through insertion into a petroleum refinery downstream of the crude unit
- Algae bio-crude is produced by thermochemical liquefaction (hydrothermal treatment) of algae biomass

Technical Approach:

- Evaluate the yield and process conditions for each of the refinery processes using Sapphire Algae bio-crude produced at the IABR in New Mexico and blended with refinery intermediate streams
- Verify product quality and that process conditions found in typical refineries can be used to upgrade Sapphire algae bio-crude at greater than 5% bio-crude composition

- Hydrotreating – lab scale (6cm³ reactor), pilot scale (4 liter reactor, 175 gallons of product)
- Coking – pilot scale (8 gallon coke drum)
- Hydrocracking – lab scale (6cm³ reactor)
- Fluid Catalytic Cracking – small lab scale
- Experimental set up and monitoring done by experienced
Sapphire researchers with oil industry and catalysis
background

Relevance:

- Verifying that bio-crude oils produced from the thermochemical conversion of algae can be upgraded in existing refiners without equipment modification is critical to the future purchase of bio-crude oil by petroleum refiners and ultimately end users
- Renewable fuels that are compatible with the existing petroleum infrastructure
- will spur the creation of a domestic bio industry
- Bio-crude oil will be able to be sold at parity with petroleum derived feed if:
- It is compatible with existing refinery process conditions (temp, pressure,
 - catalyst, metallurgy)
- Has product yield that is similar to or better than petroleum feedstock
- Upgrading data is available to refiners to review prior to sale
- This project will establish the process conditions and yield for each upgrading process option and then use refinery techno-economic analysis (LP's) to determine the economics of each insertion point

1. Task Summaries		
PMP ITEM	DESCRIPTION	STATUS
A	Studies, Testing & Upgrading	
A.1.1	<u>Identify and Contact Candidate Refiners</u> A detailed ranking of US refining companies was conducted and several refiners were contacted and the benefits of partnering with Sapphire were discussed with them. Initial offtake agreement with Tesoro was announced in 2Q 2013. Weekly discussions with Tesoro evaluated the potential for co-processing in their refineries and the challenges with obtaining Part 79 certification especially with Tesoro's lack of lab/pilot facilities. Evaluation of other refiners led to development of an oil upgrading partnership with P66.	Complete
A.1.2	<u>Brief Potential Refiners and Align Roles</u> Weekly meetings are held with both of our partners, Tesoro and P66. We have established a detailed upgrading development program with P66 and continue to explore co-processing options with Tesoro.	Complete
A.1.3	<u>Present Upgrading Study Data & Receive Feedback</u> We have presented our upgrading data to P66 and to Tesoro. Since the announcement of the partnership with P66 we have held two face to face meetings with P66 to brief them on our work to date. We have also coordinated a call between P66 and Baker Hughes to transfer the results of the Baker Hughes work we contracted for to P66.	Complete
A.1.4	<u>Secure Initial Refining Partner</u> Off take agreement with Tesoro announced, upgrading technology development partnership with P66 announced.	Complete
A.ML.1	<u>Initial Refining Partner Announced</u> Off take agreement with Tesoro announced, upgrading technology development partnership with P66 announced.	Complete
A.2.1	<u>Collect 100 gallons of Oil from Sapphire Las Cruces Test Site</u> Sapphire has produced over 1500 gallons of algae biocrude. We have upgraded 40+gallons at Southwest Research and sent 60 gallons to P66 for further testing and lab and pilot scale testing at their facility in Bartlesville, OK	Complete
A.2.2	<u>Test Transportability, Storability and Oil Blending</u> Oil produced over the past two years has been analyzed for viscosity, chemical composition and distillation. This testing has shown that	Complete

there is no appreciable difference in oil quality as a result of storage. Oil samples had been stored in heated tanks, room temperature and ambient conditions in Las Cruces, NM to demonstrate the effect of different temperature conditions in addition to the impact of time. Initially this project

Baker Hughes has completed an initial blending study to identify the compatibility of Sapphire bio-crude with petroleum feedstocks. These studies have showed that Sapphire bio-crude does not increase the deposition of asphaltenes when Sapphire bio-crude is mixed with petroleum feedstocks. The deposition of asphaltenes is a major constraint on feedstock blending for traditional refinery feedstocks and confirmation that Sapphire bio-crude oil is the same or better as petroleum feedstocks is a major development. Further work in this area is underway at Baker Hughes and P66 to validate these initial results. (See Appendix Figure 1)

The investigation of pipeline transport of algae bio-crude was not progressed as the changes in oil transportation dynamics in the US have led to a reduction in the importance of pipeline transport as most refiners now have rail offloading capability. Discussions with our refining partners, Tesoro and P66, have indicated that rail will be the preferred delivery method for our feedstock.

A.ML.2	<u>Oil Blending, Transport and Storage Tests Complete</u> Project milestone.	Complete
A.2.3	<u>Collect up to 40 bbl. of Oil from Sapphire Las Cruces Test Site</u> Ongoing operations at the Sapphire IABR have produced over 40bbls of algae bio-crude oil and this oil is stored on site at the Las Cruces, NM facility.	Complete
A.3.1	<u>Identify and Secure Vendors to Conduct Studies</u> All vendors have been selected and work is progressing and/or has been completed.	Complete
A.3.2	<u>Establish Final Scope and Work Plan for Studies</u> All work plans have been finalized and work is progressing and/or has been completed in all areas.	Complete
A.3.3	<u>Secure Agreement for Petroleum Co-processing</u> All agreements are in place and work is progressing and/or has been completed in all areas.	Complete
A.3.4	<u>Determine Process Conditions and Catalyst for Tests</u> These activities have Complete.	Complete

A.3.5	<u>Perform Pilot Scale Hydrotreating Testing</u> These activities have Complete.	Complete
A.ML.3	<u>Large Pilot Scale Hydrotreating Study Complete</u> The pilot scale hydrotreating runs at SwRI were a middle distillate algal oil blend and red diesel algal oil blend from Sapphire Energy, Inc. were hydrotreated and distilled. The middle distillate feedstock blend was 8.0 wt.% biocrude and 92.0 wt.% middle distillate. The red diesel feedstock blend was 12.6 wt.% biocrude and 87.4 wt.% red diesel. During steady state with reactor conditions of 1800psig and 370°C, 151.4 kilograms of hydrotreated middle distillate/algal oil blend product was collected. During steady state, 312.6 kilograms of red diesel/algal oil blend hydrotreated product was collected. From the liquid product of the hydrotreated middle distillate/algal oil blend, 9.75 wt.% of the jet fuel cut is estimated to be from the algal oil. From the liquid product of the hydrotreated red diesel/algal oil blend, 11.3 wt.% of the diesel cut is estimated to be from the algal oil. The jet fuel cut of the middle distillate algal oil blend hydrotreated liquid product was analyzed using ASTM D1655, Standard Specification for Aviation Turbine Fuels. The diesel cut of the red diesel algal oil blend hydrotreated liquid product was analyzed using ASTM D975, Standard Specification for Diesel Fuel Oils. Both product streams met all of standard ASTM specifications met by refineries running petroleum feedstocks. The diesel fuel from this large scale pilot is being used as part of the EPA Part 79 application process.	Complete
A.3.6	<u>Perform Pilot Scale Hydrocracking, Cat Cracking or MAT Testing</u> These activities have Complete.	Complete
A.ML.4	<u>Screening Hydrocracking Studies Complete</u> Initial studies were not promising due to high nitrogen content in the fuel. Options for nitrogen removal in a two stage process are being explored. Some refineries have two stage units that could be used to hydrocrack our fuel. However, this is not being explored at this time as our test partners do not have the capability of simulating this two stage hydrocracking arrangement. This work will be continued with P66 in 2H 2014.	Complete
A.ML.5	<u>FCC MAT Testing Complete</u> Tests have commenced at the University of Kentucky.	In Progress
A.3.7	<u>Perform Pilot Scale Coking Testing</u> Tests were complete on a pilot scale coker have been completed. The results of this test were encouraging. The coke produced by co-processing algae bio-crude met anode grade (highest grade and price) quality. Algae bio-crude produced slightly more coke than petroleum	Complete

	feedstock but also yielded more high value distillate product and less low value gas product. The liquid products contained oxygen and would have to be hydrotreated before being blended into finished transportation fuels. The need to hydrotreat coker liquid products is consistent with what refiners currently need to do as coker liquid product are typically high in olefins (gum precursors) and sulfur that must be removed by hydrotreating to meet fuel specifications. (See Appendix Figure 2)	
A.ML.6	<u>Coker Testing Complete</u> Project milestone.	Achieved
A.3.8	<u>Convert Feedstocks to Finished Fuels</u> Finished jet and diesel fuel has been produced.	Complete
B	Analysis and Results	
B.1.1	<u>Compile Results from Testing and Start Technoeconomic Model</u> The Sapphire Techno-Economic Model (TEM) has been updated to reflect the data collected on coking and hydrotreating. Once FCC MAT data is obtained the Sapphire TEM will be updated. An oil upgrading module is being developed to allow stand-alone analysis of the different oil upgrading options.	In Progress
B.1.2	<u>Develop an Updated LCA of Process</u> A peer reviewed paper was published in BioResource Technology using data from the Sapphire IABR and this upgrading study. The paper showed that Algae Bio-Crude produced from hydrothermal liquefaction and open pond cultivation has a better EROI and lower GHG footprint than current petroleum and biofuels options. <i>Bioresource Technology 148 (2013) 163–171</i> <i>Pilot-scale data provide enhanced estimates of the life cycle energy and emissions profile of algae biofuels produced via hydrothermal liquefaction</i> <i>Xiaowei Liu, Benjamin Saydah, Pragnya Eranki, Lisa M. Colosi, B. Greg Mitchell, James Rhodes, Andres F. Clarens</i>	Complete
B.ML.1	<u>Technoeconomic and LCA Modeling Complete</u> LCA Modeling complete TEM modeling in progress.	Not Achieved
B.1.3	<u>Produce Jet Fuel for ASTM Testing</u> These activities have Complete.	Complete
B.ML.2	<u>On-Spec Fuel Produced</u> On-spec Jet fuel and Diesel fuel have been produced. This fuel is being used to obtain EPA Part 79 and to progress certification of jet fuel.	Achieved

B.1.4	<u>Initial Testing for EPA Title 40 Part 79 Testing</u> Initial screening tests have been completed and Sapphire is progressing an option to obtain a small business waiver to avoid the \$500+k cost of engine testing required for large companies.	Complete
B.ML.3	<u>Part 79 Screening Analysis Complete</u> Screening analysis has been completed and final analysis and data preparation are being completed and a Part 79 application will be presented to the EPA in 1H 2014.	Not Achieved
B.2.1	<u>Present Results and Analysis to External Organizations</u> Sapphire has presented results from the work funded by this Grant at the April 2013 AIChE meeting, May 2013 DOE Peer Review, October 2013 Algae Biomass Summit, December 2013 Pacific Rim Summit on Industrial Biotechnology and Bioenergy and the March 2014 Food and Fuel for the 21 st Century Symposium.	Complete
B.ML.4	<u>Hydrotreating Results Presented at AIChE Meeting</u> Neil Osterwalder presented the results of the hydrotreating work at the Spring 2013 AIChE meeting.	Complete
B.2.2	<u>Secure Refining Partner for Next Phase Studies</u> These activities have Complete.	Complete
B.ML.5	<u>Refining Partner for Post Dec 2013 Upgrading Announced</u> A partnership has been announced with P66 and work has progressed since the Dec 2013 partnership announcement.	Complete

2. Summary of any critical project decisions (affecting scope, schedule, budget, etc)

Large scale FCC pilot testing has been removed from this grant and has been replaced with FCC MAT testing. Large scale FCC testing was not possible because of a lack of access to suitable facilities. This large scale FCC testing work will be conducted with P66 as part of the partnership with P66.

3. Summary of any changes in financing arrangements or budget adjustments between categories

No material changes.

4. Summary of any technical matters or scope changes

Experiments have progressed as planned with viable upgrading routes identified and the viability of these routes have been confirmed. Specifically, hydrotreating and coking are deemed viable routes for co-processing of algae bio-crude. The confirmation of these results with lab and pilot scale testing is consistent with our internal analysis of the oil quality and demonstrates a major breakthrough in the understanding of hydrothermally produced oils and the ability of refiners to co-process hydrothermally produced oils. The oil blending work has also confirmed that bio-crude can be mixed with petroleum feedstocks confirming that the bio-crude can be fed to a refinery with out any logistics issues related to asphaltene deposition.

The one technical set back was the high nitrogen levels limiting the amount of work conducted in the area of hydrocracking. This was anticipated and work will progress with P66 after this grant is completed to identify specific two stage hydrocrackers that could be utilized to remove nitrogen in a first stage and then hydrocrack the bio-crude in a second stage.

Large scale FCC pilot plant testing has been delayed until P66 has open pilot plant capacity in 2015. This work has been replaced with MAT testing (small scale FCC testing) at the University of Kentucky.

5. Summary of any non-technical issues that affect may affect the project scope, schedule or budget (contracts, permits, the project site, etc)

None during this reporting period.

6. Summary of any significant project schedule changes

None during this reporting period.

Hydrogenation and Distillation of Biocrude Blend, Phase II

December 30, 2013

Southwest Research Institute[®] (SwRI[®])
Chemistry and Chemical Engineering Division
Project 19277

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1. Executive Summary

A middle distillate algal oil blend and red diesel algal oil blend from Sapphire Energy, Inc. were hydrotreated and distilled. The middle distillate feedstock blend was 8.0 wt.% biocrude and 92.0 wt.% middle distillate. The red diesel feedstock blend was 12.6 wt.% biocrude and 87.4 wt.% red diesel.

During steady state, 151.4 kilograms of hydrotreated middle distillate/algal oil blend product was collected. During steady state, 312.6 kilograms of red diesel/algal oil blend hydrotreated product was collected. From the liquid product of the hydrotreated middle distillate/algal oil blend, 9.75 wt.% of the jet fuel cut is estimated to be from the algal oil. From the liquid product of the hydrotreated red diesel/algal oil blend, 11.3 wt.% of the diesel cut is estimated to be from the algal oil.

The jet fuel cut of the middle distillate algal oil blend hydrotreated liquid product was analyzed using ASTM D1655, *Standard Specification for Aviation Turbine Fuels*. The diesel cut of the red diesel algal oil blend hydrotreated liquid product was analyzed using ASTM D975, *Standard Specification for Diesel Fuel Oils*.

2. Introduction

The hydrotreater in the Alternative Energy Fuels Center at the Southwest Research Institute can be operated with various feedstocks to remove heteroatoms such as sulfur, nitrogen, and oxygen. Figure 1 shows a process flow diagram of the setup that was used to process the biocrude blends provided by Sapphire Energy, Inc.

A continuous distillation column was then used to separate the diesel fraction from the red diesel/algal oil blend product and jet fuel fraction from the middle distillate/algal oil blend product.

3. Equipment Setup

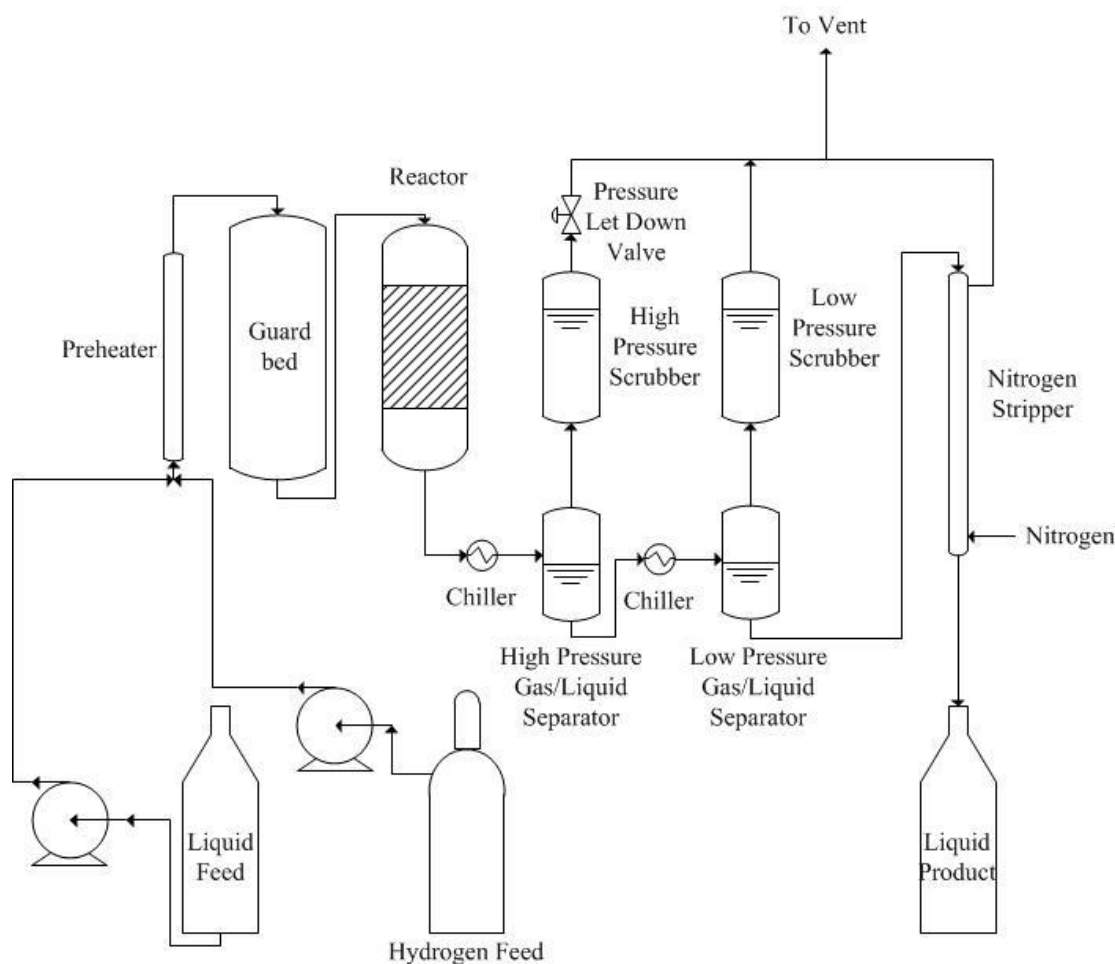


Figure 1. Process flow diagram of hydrotreater used for Phase II work.

SwRI operates a 4.0 liter reactor for hydrotreating fuels and oils, located within a building specially designed for hydrotreating. The building has ventilation and safety sensors throughout. At the request of Sapphire Energy, Inc., the guard bed was not used. The reactor operated at a

pressure of 1800 psig and a temperature of 370°C. Operators controlled the system using iFix process control software and only entered the high bay with the hydrotreater system as needed to perform walkthroughs and take samples.

The heaters on the reactor and guard bed are connected to a computer interface. The software program, iFix, allows for control and historical monitoring of temperatures, flow rates, and pressures in the hydrotreater system. The historical monitoring data is available by request. .

Hydrogen fed to the reactor was delivered using a mass flow controller. The effluent gas leaving the product collection vessel was measured through a rotameter. The remaining reactor product vapors were sent through a 7 wt.% sodium hydroxide in water solution before venting to atmosphere.

The flow of liquid feedstock to the reactor was measured using a mass flow meter. Gas samples were taken at the same time that weights were recorded for the mass balance, every 4 hours. In this way, the mass balance is time averaged, in addition to the overall start and end mass balance. The product was sent through a water knock out before being sent to the liquid product collection vessel. The liquid collected in the knockout was weighed and analyzed for hydrocarbon content to determine the amount of water and hydrocarbons collected.

Once collected, the liquid product from the hydrotreater was sent through a distillation column at approximately one gallon per hour. The jet and diesel fractions were processed separately. The first pass through the distillation column for the jet fuel from the middle distillate/algae oil blend hydrotreated product removed liquid with boiling points under 302°F. The second pass through the distillation column removed the jet fuel cut, 302 – 572°F. Using the red diesel/algae oil blend hydrotreated product, the first pass through the fractionator for the diesel removed liquid with boiling points under 455°F. The second pass for the diesel through the fractionator removed the diesel fuel cut, 455 – 650°F, leaving the vacuum gas oil residuum. Each cut was verified using simulated distillation and the mass recorded.

4. Operation Summary

The operation summaries for the hydrogenation and distillation processes are outlined below.

4.1 Hydrogenation

On Sunday, June 9, 2013, the reactor was filled with 4 kilograms (4 liters) of GoBioHDO Sapphire 1 neat.

After completing the final pressure check with hydrogen and completing the final system checks, the sulfiding procedure began at 09:01 AM on June 11, 2013. Once the sulfiding procedure was complete, the feed was switched to middle distillate neat. At 12:04 AM on June 13, 2013, middle distillate neat was introduced into the system. At 11:20 AM, the system reached steady state and a hydrotreated middle distillate neat sample was taken. At 1:10 PM, the middle distillate algal oil blend was introduced into the system. At 8:30 PM on June 13, 2013, steady state was reached for the middle distillate algal oil blend. At 11:01 PM on June 15, 2013, the middle distillate algal oil run was completed after collecting 151.4 kilograms of hydrotreated product.

After completing the middle distillate algal oil blend run, the red diesel algal oil blend was fed into the system. At 3:00 AM on June 17, 2013, steady state was reached for the red diesel algal oil blend. At 7:35 PM on June 21, 2013, the red diesel algal oil run was completed after collecting 312.6 kilograms of hydrotreated product. Afterwards, red diesel neat was fed through the system. Once the red diesel neat reached steady state and samples were collected, the system was shut down.

Once the system had cooled, the reactor catalyst was removed from the system and collected. Based on visual observations, no unusual coking occurred.

4.2 Distillation

On June 27, 2013 at 10:27 PM, the hydrotreated middle distillate algal oil blend was processed through the distillation column at atmospheric pressure in order to separate the light ends cut (less than 302°F) from the feed. The first pass of the hydrotreated middle distillate algal oil blend was completed at 12:49 PM on June 28, 2013.

On June 28, 2013 at 1:40 PM, the hydrotreated red diesel algal oil blend was processed through the fractionator column at atmospheric pressure in order to separate the light ends from the feed. At 12:15 PM on June 29, 2013, the first pass of the hydrotreated algal oil blend was completed.

At 3:00 PM on June 29, 2013, the bottoms product from the first cut of the hydrotreated middle distillate algal oil blend was processed through the fractionator column under vacuum in order to collect the jet fuel cut (302 – 572°F). During the run, the distillation had to be stopped due to a restriction at the bottom of the kettle which limited flow of the bottoms products. Once the restriction was cleared, the fractionation was restarted with a higher percent bottoms cut in order to facilitate flow. At 9:00 PM on July 3, 2013, fractionation of the hydrotreated middle distillate

algal oil blend was completed. The bottoms material was fractionated again using a potstill distillation in order to recover the remainder of the jet fuel (302 – 572°F) cut.

At 12:20 AM on July 4, 2013, the bottoms product from the first pass of the hydrotreated red diesel algal oil blend was processed through the fractionator column under vacuum in order to remove the material with a boiling point less than 455°F. At 1:50 PM, the second pass of the hydrotreated diesel algal oil blend was completed. After the completion of the second pass, the bottoms product was fed back into the distillation column and the temperature of the column increased in order to separate the diesel cut (455 – 650°F) during the third pass. At 6:18 PM on July 6, 2013, the third pass was stopped due a restriction at the bottom of the column, preventing the bottoms pump from draining the column. The remainder of the feed and bottoms material was processed using a batch distillation to recover the remainder of the diesel (455 – 650°F) cut.

5. Biocrude Blend Feedstocks

A middle distillate/algal oil and red diesel/algal oil blend feedstocks were created using Sapphire Energy's biocrude. The blend components for the middle distillate/algal oil blend are shown in Table 1 below. The weighted simulated distillation data for each component is shown below in Figure 2.

Table 1. Total biocrude/middle distillate blend.

Initial Blend	Weight (kg)	Weight %
Biocrude	29.7	10.02
DMDS	0.12	0.04
Middle Distillate	266.7	89.94
Total	296.52	100.00
Adjustments		
Water in feed ¹	5.86	1.98
Residual Solids	0.8	0.27
Feed blend		
Biocrude	23.04	7.95
DMDS	0.12	0.04
Middle Distillate	266.7	92.01
Total	289.86	100

¹ Based on ASTM D4928

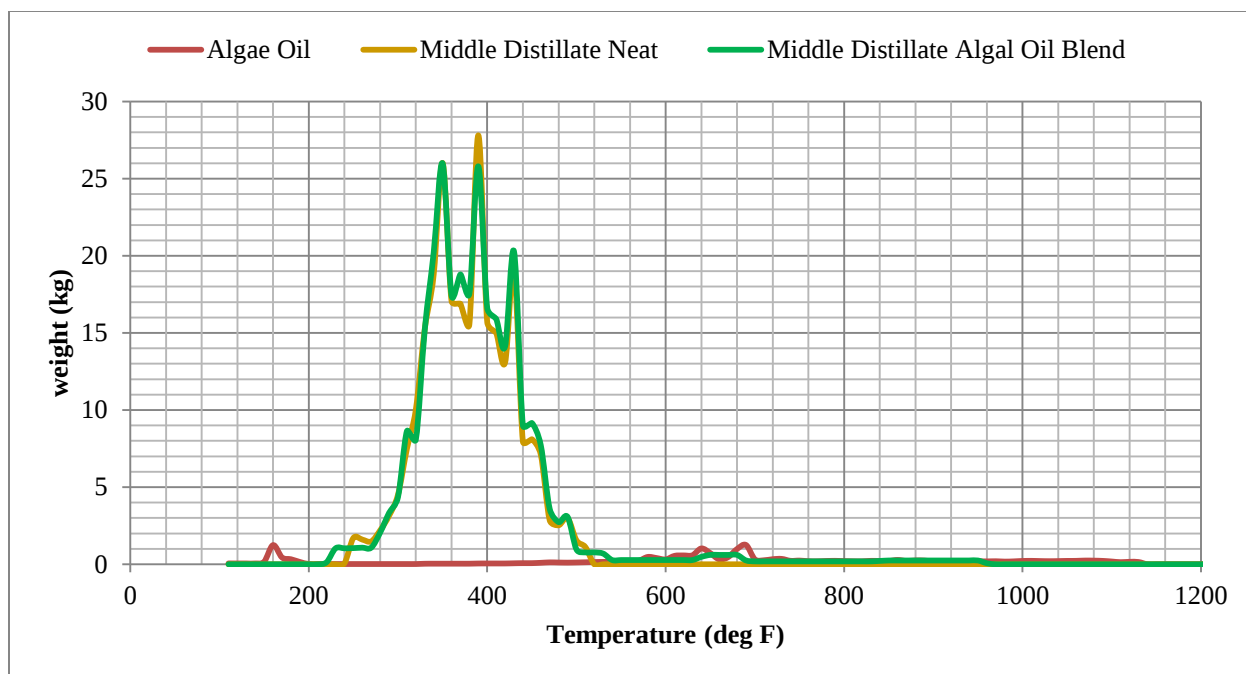


Figure 2. Weighted simulated distillation data of middle distillate biocrude blend.

The blend components for the red diesel/algal oil blend are shown in Table 2 below. The weighted simulated distillation data for each component is shown below in Figure 3.

Table 2. Total biocrude/red diesel blend.

Initial Blend	Weight (kg)	Weight %
Biocrude	72.00	15.00
DMDS	0.19	0.04
Red Diesel	407.80	84.96
Total	479.99	100.00
Adjustments		
Water in feed ²	13.28	2.77
Residual Solids	1.20	0.25
Feed blend		
Biocrude	57.52	12.36
DMDS	0.19	0.04
Red Diesel	407.80	87.60
Total	465.51	100.00

² Based on ASTM D4928

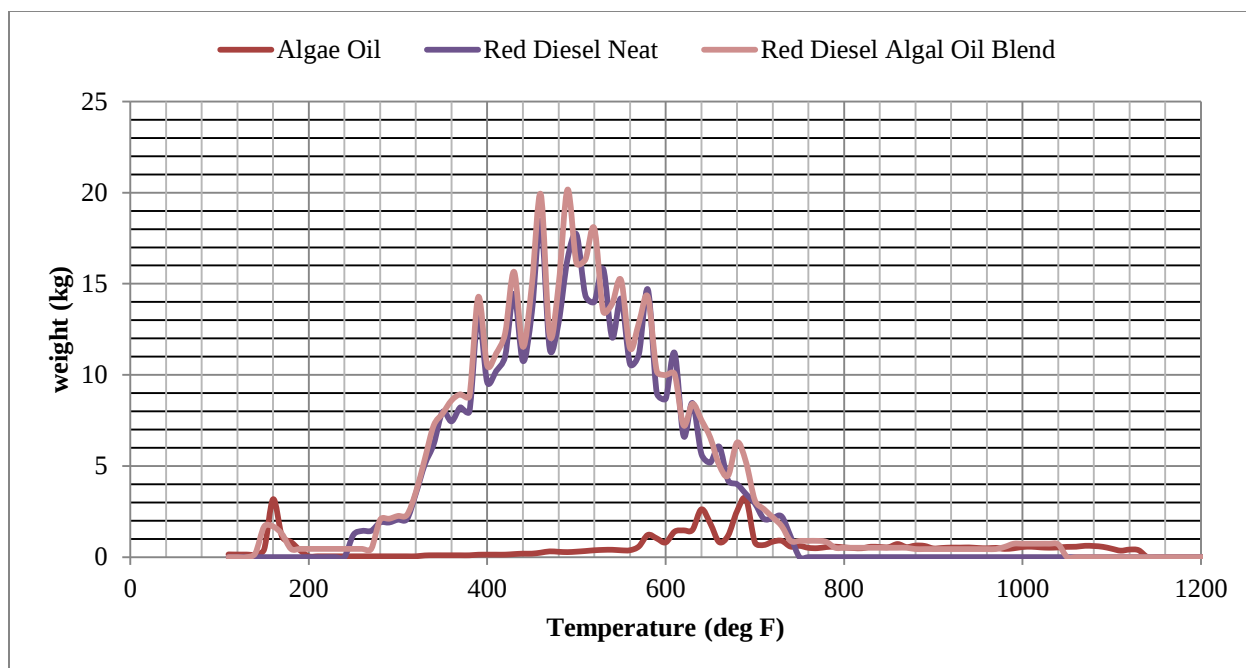


Figure 3. Weighted simulated distillation data of red diesel biocrude blend.

6. Mass Balance

The mass balance is calculated in two sections: hydrogenation and distillation.

6.1 Hydrogenation

During hydrogenation, the mass balance was performed for the steady state portion of the run. Steady state was established when clear liquid product was collected with a consistent API and a nitrogen and sulfur content of less than 10ppm. The overall mass balance, which takes into consideration both steady and non-steady state regimes, was also calculated.

The liquid feed and product mass is shown below in Table 3. The liquid product is a sum of the liquid products collected, including samples.

Table 3. Mass balance for steady state regime.

	Liquid Feed (kg)	Liquid Product (kg)	Wt. %
Middle Distillate Algal Oil Blend	150.6	151.6	100.5
Red Diesel Algal Oil Blend ³	296.2	312.6	105.5

6.2 Algae oil in Jet Fuel Cut

Figure 4 below shows the simulated distillation curve for the middle distillate neat feed and product from hydrotreating. Figure 5 below shows the difference in boiling point distribution of the middle distillate neat before and after hydrotreating.

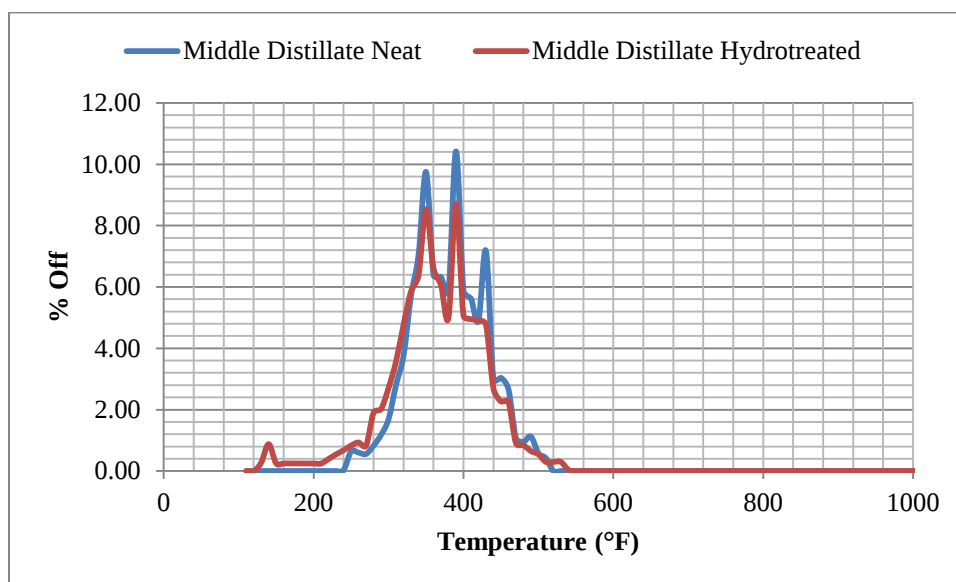


Figure 4. Middle distillate neat before and after hydrotreating.

³Product collection vessels were not completely drained once steady state was reached. As a result, overall material balance is slightly higher than 105 wt%.

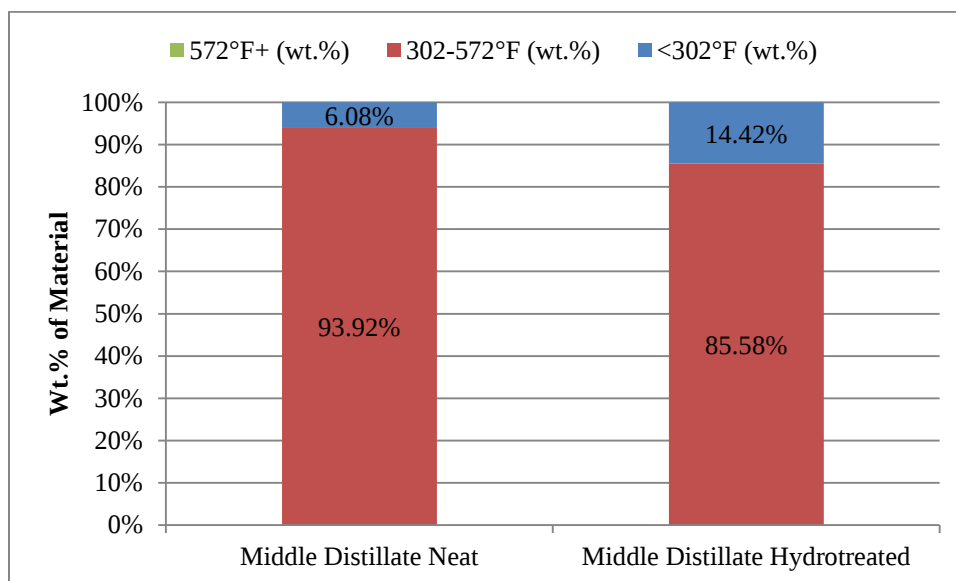


Figure 5. Change in boiling point distribution for middle distillate neat before and after hydrotreating.

After hydrotreating the middle distillate neat feed, the amount of material with boiling points less than 302°F increased. The amount of material with boiling points greater than 302°F decreased.

Figure 6 shows the simulated distillation curve for the middle distillate/algal oil blend before and after hydrotreating. Figure 7 below the difference in boiling point distribution of the middle distillate algal oil blend before and after hydrotreating.

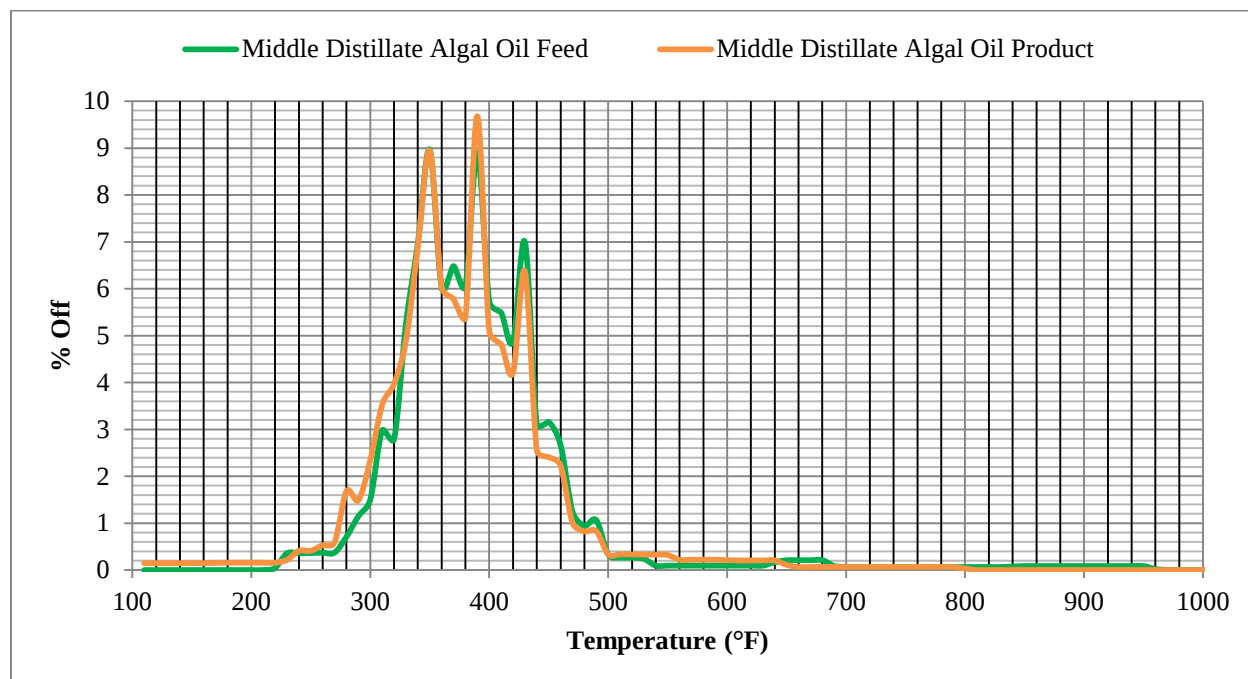


Figure 6. Middle distillate algal oil blend before and after hydrotreating.

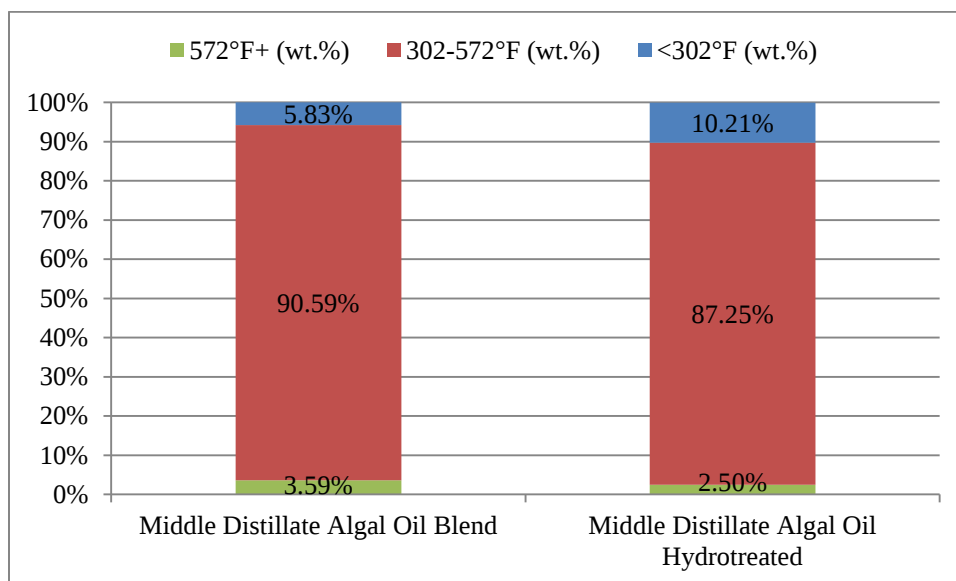


Figure 7. Change in boiling point distribution for middle distillate algal oil blend before and after hydrotreating.

After hydrotreating the middle distillate/algal oil blend, the amount of material with boiling points less than 302°F increased, and the amount of material with boiling points greater than 302°F decreased. The cuts for the middle distillate neat, middle distillate/algal oil blend, algae oil, and hydrotreated middle distillate/algal oil blend product are shown below in Table 4.

Table 4. Cuts for middle distillate neat⁴, middle distillate/algal oil blend⁴, algal oil feed⁵, and hydrotreated product⁴

	Middle Distillate Neat	Middle Distillate Algal Oil Blend	Algal Oil Feed	Middle Distillate Algal Oil Blend Hydrotreated
<302°F (wt.%)	6.08	5.83	2.84	10.21
302-572°F (wt.%)	93.92	90.58	51.96	87.29
572°F+ (wt.%)	0.00	3.59	45.20	2.50
Total Mass ⁶				
<302°F (kg)	8.43	8.77	0.34	15.38
302-572°F (kg)	130.20	136.42	6.22	131.46
572°F+ (kg)	0.00	5.41	5.41	3.76

⁴ Based on ASTM D2887.

⁵ Calculated by weight difference between middle distillate neat and middle distillate algal oil blend. Wt.% distribution of algal oil feed is not representative of ASTM D2887 results. Algal wt.% distribution likely changed due to heating while blending biocrude feedstock.

⁶ Based on 150.6 kg feed.

Based on Table 4, hydrotreating the middle distillate/algal oil blend decreased the overall jet fuel cut in the product. In order to calculate the amount of the jet fuel cut (302-572°F) that is from the algae oil, it is necessary to determine whether the amount of change in jet fuel that is due to the middle distillate neat or the algae oil. Since hydrotreating the middle diesel neat indicated significant change in the jet fuel cut, it is assumed that changes to the jet fuel cut are from both the middle distillate neat and the algae oil.

The wt. % of the algae oil jet fuel in the final hydrotreated middle distillate algal blend product can be calculated as outlined below⁷:

$$\text{wt. \% algae oil in jet} = \frac{\text{Jet from algae oil feed} + \text{change in jet fuel due to algae oil}}{\text{Total jet in middle distillate algal blend product}} \times 100 \text{ wt. \%}$$

Therefore, the weight percent of the algae oil in the jet fuel cut of the final distilled hydrotreated liquid product is 9.75 wt.%.

$$\text{wt. \% algae oil in jet} = \frac{6.22 \text{ kg} + 6.60 \text{ kg}}{131.46 \text{ kg}} \times 100 \text{ wt. \%} = 9.75 \text{ wt. \%}$$

6.3 Algae oil in Diesel Cut

Figure 8 below shows the simulated distillation curve for the red diesel neat before and after hydrotreating. Figure 9 below shows the difference in boiling point distribution of the red diesel neat before and after hydrotreating.

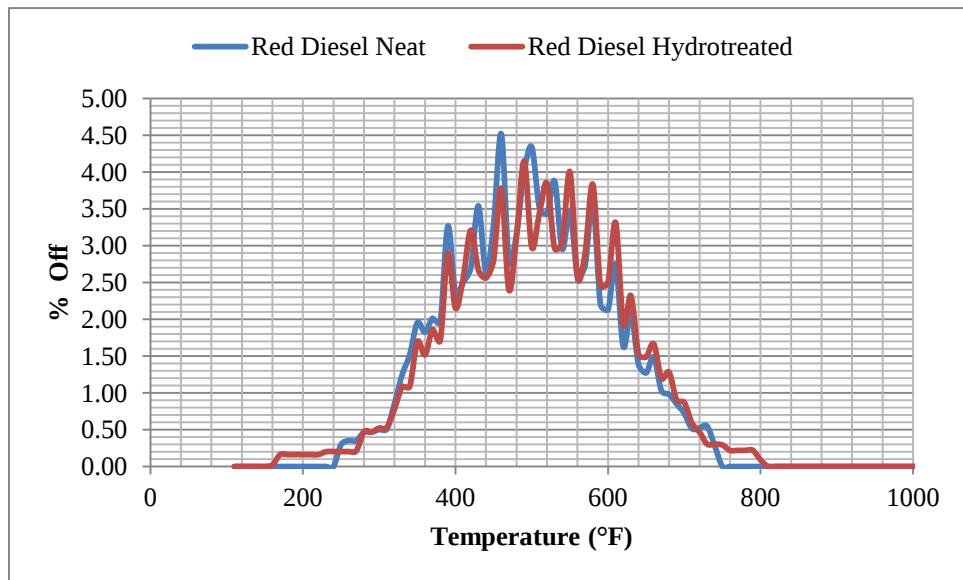


Figure 8. Red diesel neat before and after hydrotreating.

⁷ Detailed calculations can be found in Appendix B.

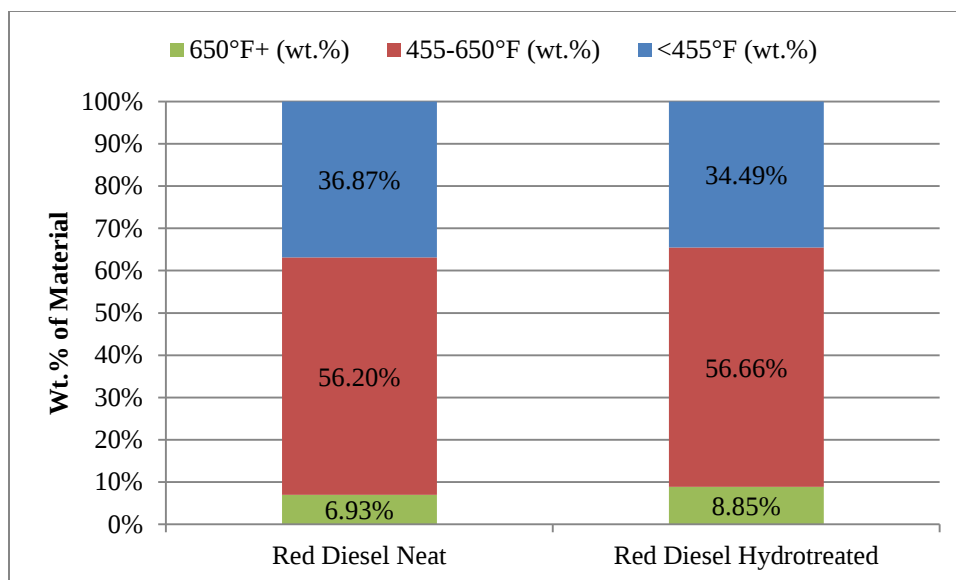


Figure 9. Change in boiling point distribution for red diesel neat before and after hydrotreating.

After hydrotreating the red diesel neat feed, the diesel content did not change by more than 0.50wt%.

Figure 10 below shows the simulated distillation curve for the red diesel/algal oil blend before and after hydrotreating. Figure 11 below shows the difference in boiling point distribution for the red diesel/algal oil blend before and after hydrotreating.

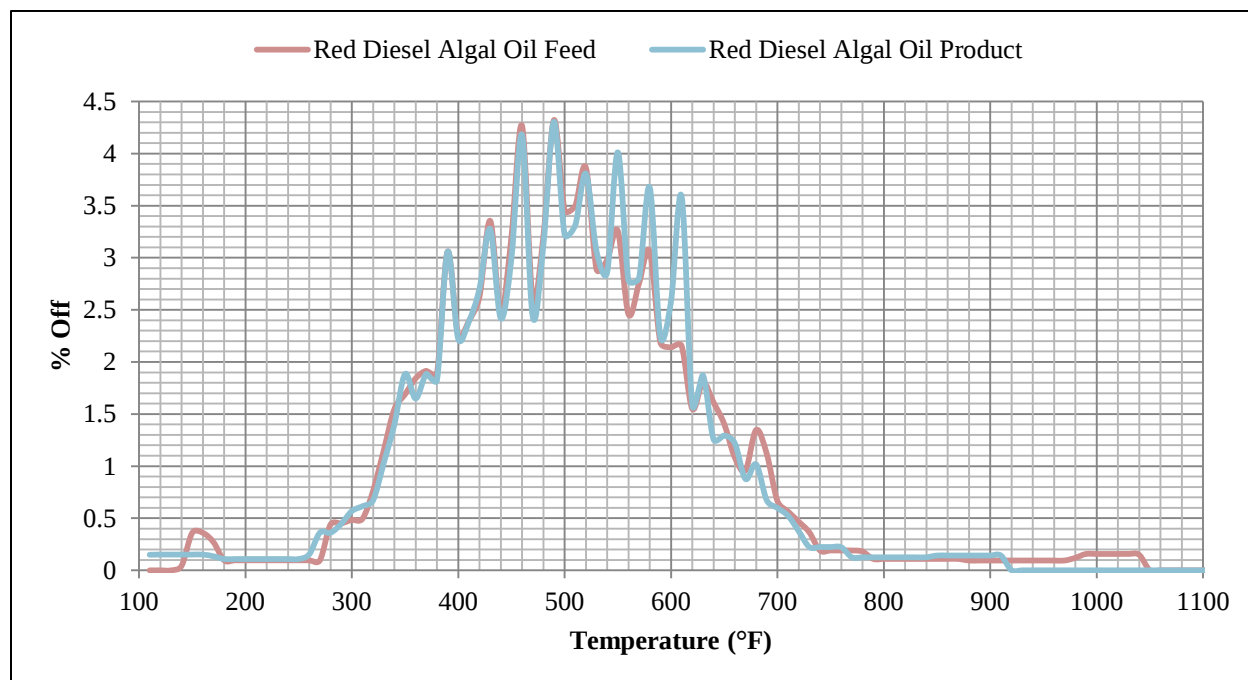


Figure 10. Red Algal Oil Blend before and after hydrotreating.

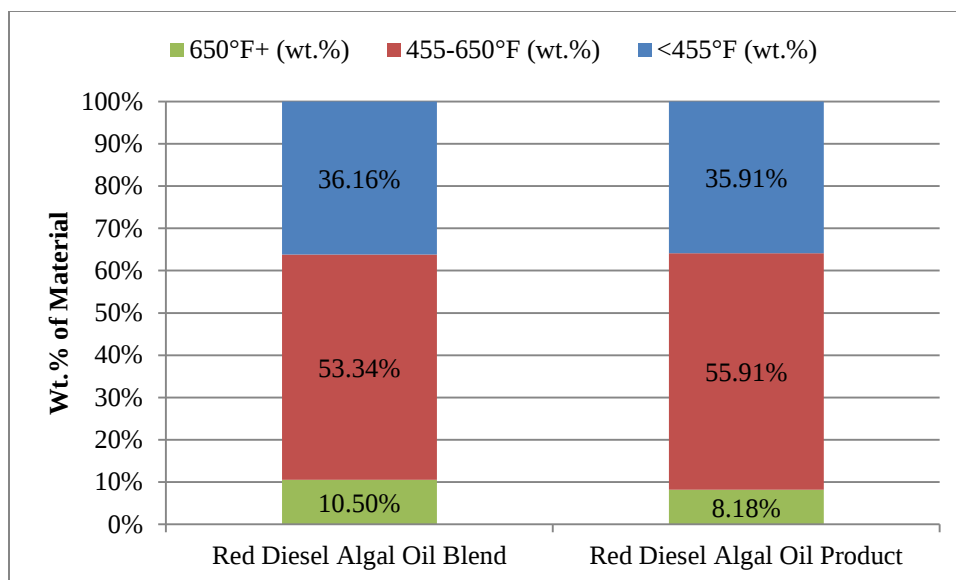


Figure 11. Difference in wt.% off for red diesel algal oil blend before and after hydrotreating.

The cuts based on simulated distillation for red diesel neat, the red diesel/algal oil blend, and the hydrotreated biocrude blend product are shown below in Table 5. The mass of each cut is calculated based on weights of feed and product during the steady state regime of hydrotreating.

Table 5: Cuts for red diesel neat⁸, red diesel algal oil blend⁸, algal oil feed⁹, and red diesel algal oil hydrotreated product⁸.

	Red Diesel neat	Red Diesel Algal Oil Blend	Algal Oil Feed	Red Diesel Algal Oil Hydrotreated Product
<455°F (wt.%)	36.00	35.70	33.59	35.59
455-650°F (wt.%)	57.07	53.80	31.10	56.24
650°F+ (wt.%)	6.93	10.50	35.31	8.18
Total Mass ¹⁰				
<455°F (kg)	93.22	105.74	12.52	105.42
455-650°F (kg)	147.77	159.36	11.59	166.58
650°F+ (kg)	17.94	31.10	13.16	24.23

Based on Table 5, hydrotreating the red diesel/algal oil blend increased the overall diesel cut in the hydrotreated product. In order to calculate the amount of the diesel cut (302-572°F) that is from the algae oil, it is necessary to determine whether the diesel production is due to the red

⁸ Based on ASTM D2887.

⁹ Calculated by weight difference between red diesel neat and red diesel algal oil blend. Wt.% distribution of algal oil feed is not representative of ASTM D2887 results. Algal wt.% distribution likely changed due to heating while blending biocrude feedstock.

¹⁰ Based on 296.2 kg feed.

diesel neat or the algae oil. Since hydrotreating the red diesel neat indicated little change in the diesel cut, it is assumed that changes to the diesel cut are from the algae oil.

Using the data in Table 5 above, the wt.% of the algae oil diesel in the final diesel product can be calculated as outlined below¹¹:

$$\text{wt. \% algae oil in diesel} = \frac{\text{Diesel from algae oil feed} + \text{change in diesel due to algae}}{\text{Total diesel in product}(kg)} \times 100\text{wt. \%}$$

Therefore, the wt.% of the algae oil in the diesel cut of the final distilled hydrotreated liquid product is 11.3 wt.%:

$$\text{wt. \% algae oil in diesel} = \frac{11.59 \text{ kg} + 7.22 \text{ kg}}{166.58 \text{ kg}} \times 100\text{wt. \%} = 11.3 \text{ wt. \%}$$

¹¹ Detailed calculations can be found in Appendix B.

6.4 Distillation

The hydrotreated middle distillate/algal oil blend was initially passed through the continuous distillation twice in order to separate out the jet fuel cut. The remaining bottom cut was distilled again using a potstill distillation to separate out the remainder of the jet fuel. The combined totals of each cut are shown below in Table 9.

Table 6: Jet fuel distillation cuts.

CUT	weight (kg)	weight%
IBP – 302°F	11.8	11.3%
302 – 572°F	86.6	82.8%
572°F+	6.2	5.9%
TOTAL	104.6	100.0%

The hydrotreated red diesel/algal oil blend was first passed through the continuous distillation column in order to remove the light ends (in the first pass) and the material with a boiling point below 455°F. During the third pass to remove the diesel cut from the bottoms, part of the 455 – 650°F cut was removed, but much of the cut had to be distilled using the potstill distillation due to a restriction that formed at the bottom of the kettle, preventing the bottoms pump from moving the product¹². The combined cuts are shown below in Table 10.

Table 7. Diesel distillation cuts.

CUT	weight (kg)	weight %
Cold traps + first pass	36.2	12.9%
IBP – 455°F	81.2	29.0%
455 – 650°F	127.2	45.4%
650°F+	35.4	12.6%
TOTAL	280	100.0%

¹² It is likely still possible to run the material through a continuous distillation column; a pump rated for operating temperatures greater than the pour point of the material would be required.

7. ASTM D975 results

After hydrotreating and distilling the biocrude blend, the diesel cut of the product was analyzed using ASTM D975. The results for the biocrude blend are shown below in Table 7.

Table 8. ASTM D975 results.

Method	Test	Units	ASTM specification	Sapphire Energy Diesel Blend
D130	Copper		No.3	3A
D1319	Aromatics	%	Max 35	11.8
	Olefins	%	none	3.2
	Saturates	%	none	85
D2500	Cloud Point	°C	none	-10.8
D2624	Conductivity	pS/m	Min 25	2
	Temperature	°C	none	21.6
D2709	Water and Sediment	vol%	Max 0.05	< 0.005
D445 40c	Viscosity	mm ² /S	Min 1.9, max 2.4	2.816
D482	Percent Ash	mass %	Max 0.01	<0.001
D524_10%	Ramsbottom carbon residue on 10% distillation residue	wt. %	Max 0.15	0.06
D5453	Sulfur	ppm	Max 15	12.4
D6079	Lubricity Major Axis	mm	Max 520	0.624
	Lubricity Minor Axes	mm	none	0.599
	Lubricity Wear Scar	mm	none	0.612
	Description of Scar		none	Circular Evenly Abraded
	Fuel Temperature	°C	none	60
D613	Cetane No		Min 40	61.6
D86	Initial Boiling Point (IBP)	°F	none	443.2
	Evaporation of 5 wt. %	°F	none	466.8
	Evaporation of 10 wt. %	°F	none	472.9
	Evaporation of 15 wt. %	°F	none	478.3
	Evaporation of 20 wt. %	°F	none	481.8
	Evaporation of 30 wt. %	°F	none	491.6
	Evaporation of 40 wt. %	°F	none	502.3
	Evaporation of 50 wt. %	°F	none	512.9
	Evaporation of 60 wt. %	°F	none	523.9
	Evaporation of 70 wt. %	°F	none	536.6
	Evaporation of 80 wt. %	°F	none	551.5
	Evaporation of 90 wt. %	°F	Min 540, max 640	570.5
	Evaporation of 95 wt. %	°F	none	584.4
	Final Boiling Point (FBP)	°F	none	600.1
	Recovered	mL	none	98.9

Method	Test	Units	ASTM specification	Sapphire Energy Diesel Blend
	Residue	mL	none	0.9
	Loss	mL	none	0.2
	Corrected ¹³ IBP	°F	none	443.2
	Corrected ¹ FBP	°F	none	600.1
	Corrected ¹ Evaporation of 10 wt.%	°F	none	473
	Corrected ¹ Evaporation of 50 wt.%	°F	none	513
	Corrected ¹ Evaporation of 90 wt.%	°F	none	570.7
	Corrected ¹ Recovered	mL	none	99
	Corrected ¹ Loss	mL	none	0.1
D93	Flash Point	°F	none	213
	Flash Point	°C	Min 38	101

¹³ Corrected value for barometric pressure, per ASTM D86

8. ASTM D1655 Results

After hydrotreating and distilling the biocrude blend, the jet cut of the product was analyzed using ASTM D1655. The results for the biocrude blend are shown below in Table 7.

Table 9. ASTM D1655 Results.

Method	Test	Units	ASTM specification	Sapphire Jet Fuel Blend
D1319	Aromatics	%	Max 25	7.5
	Olefins	%	none	2.4
	Saturate	%	none	90.1
D130	Corrosion (Copper Strip)		Max No.1	1A
D4052	Density	kg/m ³	775 to 840	791.3
	API at 60°F		none	47.3
	Specific Gravity at 60°F		none	0.7915
	Density at 15°C	g/ml	none	0.7913
D2887	Distillation		none	
	Initial Boiling Point (IBP)	°C	none	128.6
	Evaporation of 5 wt.%	°C	none	152.3
	Evaporation of 10 wt.%	°C	Max 205	160.1
	Evaporation of 15 wt.%	°C	none	166.7
	Evaporation of 20 wt.%	°C	none	171.9
	Evaporation of 25 wt.%	°C	none	174.8
	Evaporation of 30 wt.%	°C	none	179.1
	Evaporation of 35 wt.%	°C	none	183.2
	Evaporation of 40 wt.%	°C	none	188.4
	Evaporation of 45 wt.%	°C	none	192.9
	Evaporation of 50 wt.%	°C	Report	196.1
	Evaporation of 55 wt.%	°C	none	198
	Evaporation of 60 wt.%	°C	none	202.6
	Evaporation of 65 wt.%	°C	none	208
	Evaporation of 70 wt.%	°C	none	212.5
	Evaporation of 75 wt.%	°C	none	216.4
	Evaporation of 80 wt.%	°C	none	219
	Evaporation of 85 wt.%	°C	none	226.4
	Evaporation of 90 wt.%	°C	none	236.1
	Evaporation of 95 wt.%	°C	none	252.2
	Final Boiling Point (FBP)	°C	Max 300	353.1
D2624	Electrical conductivity	pS/m	none	1
	Temperature	°C	none	22.8
D381	Existent Gum	mg/100mL	Max 7	5
D56	Flash Point	°C	Min 38	48
		°F	none	118

D5972	Freezing point	°C	Max -40 ¹⁴	-59
D1840	Naphthalene Content	Vol. %	none	0.06
D4809	Net heat of combustion	MJ/kg	Min 42.8	43.308
		BTU/lb	none	18619
		cal/g	none	10343.9
D1322	Smoke Point 1	mm	Min 25	27.5
	Smoke Point 2	mm	Min 25	27.0
	Smoke Point 3	mm	Min 25	27.0
	Smoke Point A	mm	Min 25	27.2
D3227	Sulfur, Mercaptan	Mass %	Max 0.003	<0.0003
D2622	Sulfur, Total	Mass %	Max 0.30	0.00102
D3241	Thermal Stability		none	Pass at 260°C
	Filter pressure drop	mm Hg	Max 25	0
	Tube deposits less than		3	1
D3242	Total Acidity	mg KOH/g	Max 0.10	0.007
D445	Viscosity at -20°C	mm ² /s	Max 8.0	3.81
	Viscosity at -20°C	cSt	none	3.81
D3948	Water Separation		Min 85	100

¹⁴ Max -40°C for Jet A. Max -47°C for Jet A-1.

APPENDIX A – Reactor Catalyst Inspection

At the completion of the run, the catalyst was removed from the reactor and inspected. There was no unusual coking on the catalyst, as shown below.



Figure A 1. Reactor catalyst after Sapphire Energy run.

Appendix B: Detailed Calculations of Algae Contribution to Jet Fuel and Diesel Cuts

B1. Middle Distillate Neat Hydrotreated Product Calculation

The middle distillate neat contribution to the hydrotreated middle distillate/algal oil blend is determined using the middle distillate neat hydrotreated results.

The results used for this determination are shown below:

Table B 1: Change in cut distribution for middle distillate neat before and after hydrotreating.

	Middle Distillate Neat	Middle Distillate Hydrotreated	Change in Middle Distillate after Hydrotreating
<302°F (wt.%)	6.08	14.42	8.34
302-572°F (wt.%)	93.92	85.58	-8.34
572°F+ (wt.%)	0.00	0.00	0.00

From these results, it can be assumed that for every 100 kg of middle distillate feed, 8.34 kg of jet fuel will be cracked in the hydrotreated product.

On this basis, for a feed amount of 138.43 kg of middle distillate neat, the amount of jet fuel in the hydrotreated product is 118.64 kg. Table B2 below shows the calculated contribution of the middle distillate neat for the middle distillate algae oil blend hydrotreating process.

Table B 2: Cut distribution for middle distillate neat before and after hydrotreating.

	Middle Distillate Neat	Middle Distillate Hydrotreated
<302°F (wt.%)	6.08	14.42
302-572°F (wt.%)	93.92	85.58
572°F+ (wt.%)	0.00	0.00
Total Mass ¹⁵		
<302°F (kg)	8.43	19.99
302-572°F (kg)	130.20	118.64
572°F+ (kg)	0.00	0.00

¹⁵ Based on 150.6 kg feed.

An example calculation to determine the amount of material in a specific cut of the middle distillate hydrotreated product is shown below:

$$\text{Jet fuel in hydrotreated product} = \text{Jet fuel in feed} - \left(\text{Feed amount (kg)} \times \frac{8.34 \text{ kg of jet fuel lost}}{100 \text{ kg of feed}} \right)$$

$$\text{Jet fuel in hydrotreated product} = 130.20 \text{ kg} - \left(138.63 \text{ kg} \times \frac{8.34 \text{ kg of jet fuel lost}}{100 \text{ kg of feed}} \right)$$

$$\text{Jet fuel in hydrotreated product} = 130.20 \text{ kg} - 11.56 \text{ kg}$$

$$\text{Jet fuel in hydrotreated product} = 118.64 \text{ kg}$$

B2. Algae Oil in Jet Fuel of Hydrotreated Middle Distillate Algae Oil Blend

The values shown in Table B3 will be used to calculate the wt.% algae oil in the jet fuel of the hydrotreated middle distillate algal oil blend.

Table B 3: Cut distribution for middle distillate neat, middle distillate algal oil blend, algae oil feed, middle distillate hydrotreated, and middle distillate algal oil blend.

	Middle Distillate Neat	Middle Distillate Algal Oil Blend	Algal Oil Feed	Middle Distillate Hydrotreated	Middle Distillate Algal Oil Blend Hydrotreated
<302°F (wt.%)	6.08	5.83	2.84	14.42	10.21
302-572°F (wt.%)	93.92	90.58	51.96	85.58	87.29
572°F+ (wt.%)	0.00	3.59	45.20	0.00	2.50
Total Mass ¹⁶					
<302°F (kg)	8.43	8.77	0.34	19.99	15.38
302-572°F (kg)	130.20	136.42	6.22	118.64	131.46
572°F+ (kg)	0.00	5.41	5.41	0.00	3.76

The weight percent of algae oil in jet fuel is defined by the following equation:

$$\text{wt. \% algae oil in jet product} = \frac{\text{Jet from algae oil feed} + \text{change in jet due to algae oil}}{\text{Total jet in middle distillate algal oil product}} \times 100 \text{ wt. \%}$$

Where:

$$\text{Jet from algae oil feed} = 6.22 \text{ kg}$$

$$\text{Total jet in middle distillate algal oil product} = 131.46 \text{ kg}$$

$$\text{Change in jet due to algae oil} = \text{overall change in jet} - \text{jet change due to middle distillate neat}$$

¹⁶ Based on 150.6 kg feed.

The change in jet due to algae oil can be calculated once the overall change in jet and jet change due to middle distillate neat are determined. These values are determined as follows:

$$\text{Overall change in jet} = \text{jet in middle distillate algal oil product} - \text{jet in middle distillate algal oil feed}$$

$$\text{Overall change in jet} = 131.46 \text{ kg} - 136.42 \text{ kg}$$

$$\text{Overall change in jet} = -4.96 \text{ kg}$$

$$\text{Jet change due to middle distillate neat} = \text{jet in middle distillate hydrotreated} - \text{jet in middle distillate neat}$$

$$\text{Jet change due to middle distillate neat} = 118.64 \text{ kg} - 130.20 \text{ kg}$$

$$\text{Jet change due to middle distillate neat} = -11.56 \text{ kg}$$

As a result, the change in jet due to algae oil is calculated as follows:

$$\text{Change in jet due to algae oil} = -4.96 \text{ kg} - (-11.56 \text{ kg})$$

$$\text{Change in jet due to algae oil} = 6.60 \text{ kg}$$

The wt.% of algae oil in jet product is determined as follows:

$$\text{wt. \% algae oil in jet product} = \frac{6.22 \text{ kg} + 6.60 \text{ kg}}{131.46 \text{ kg}} \times 100 \text{ wt. \%}$$

$$\text{wt. \% algae oil in jet product} = \frac{12.82 \text{ kg}}{131.46 \text{ kg}} \times 100 \text{ wt. \%}$$

$$\text{wt. \% algae oil in jet product} = 9.75 \text{ wt. \%}$$

B3. Algae Oil in Diesel of Hydrotreated Red Diesel Algae Oil Blend

The values shown in Table B4 will be used to calculate the wt.% algae oil in the diesel cut of the hydrotreated red diesel algal oil blend.

Table B 4: Cut distribution for the red diesel neat, red diesel algal oil blend, algal oil feed, and red diesel algal oil hydrotreated product

	Red Diesel neat	Red Diesel Algal Oil Blend	Algal Oil Feed	Red Diesel Algal Oil Hydrotreated Product
<455°F (wt.%)	36.00	35.70	33.59	35.59
455-650°F (wt.%)	57.07	53.80	31.10	56.24
650°F+ (wt.%)	6.93	10.50	35.31	8.18
Total Mass ¹⁷				
<455°F (kg)	93.22	105.74	12.52	105.42
455-650°F (kg)	147.77	159.36	11.59	166.58
650°F+ (kg)	17.94	31.10	13.16	24.23

The weight percent of algae oil in the diesel cut is defined by the following equation:

$$\text{wt. \% algae oil in diesel} = \frac{\text{Diesel from algae oil feed} + \text{diesel produced}}{\text{Total diesel in product}} \times 100\text{wt. \%}$$

Where:

$$\text{Initial diesel from algae oil} = \text{Red diesel algal oil blend diesel cut} - \text{red diesel neat diesel cut}$$

$$\text{Diesel produced} = \text{Hydrotreated product diesel} - \text{red diesel algal oil blend diesel}$$

The initial diesel from algae oil is calculated as follows:

$$\text{Initial diesel from algae oil} = 159.36 \text{ kg} - 147.77 \text{ kg}$$

$$\text{Initial diesel from algae oil} = 11.59 \text{ kg}$$

The diesel produced is calculated as follows:

$$\text{Diesel produced} = 166.58 \text{ kg} - 159.36 \text{ kg}$$

$$\text{Diesel produced} = 7.22 \text{ kg}$$

¹⁷ Based on 296.2 kg feed.

The wt.% of algae oil in the diesel product is determined as follows:

$$\text{wt. \% algae oil in diesel product} = \frac{11.59 \text{ kg} + 7.22 \text{ kg}}{166.58 \text{ kg}} \times 100 \text{ wt. \%}$$

$$\text{wt. \% algae oil in diesel product} = \frac{18.81 \text{ kg}}{166.58 \text{ kg}} \times 100 \text{ wt. \%}$$

$$\text{wt. \% algae oil in diesel product} = 11.3 \text{ wt. \%}$$

7. ASTM D975 results

After hydrotreating and distilling the biocrude blend, the diesel cut of the product was analyzed using ASTM D975. The results for the biocrude blend are shown below in Table 7.

Table 8. ASTM D975 results.

Method	Test	Units	ASTM specification	Sapphire Energy Diesel Blend
D130	Copper		No.3	3A
D1319	Aromatics	%	Max 35	11.8
	Olefins	%	none	3.2
	Saturates	%	none	85
D2500	Cloud Point	°C	none	-10.8
D2624	Conductivity	pS/m	Min 25	2
	Temperature	°C	none	21.6
D2709	Water and Sediment	vol%	Max 0.05	< 0.005
D445 40c	Viscosity	mm ² /S	Min 1.9, max 2.4	2.816
D482	Percent Ash	mass %	Max 0.01	<0.001
D524 10%	Ramsbottom carbon residue on 10% distillation residue	wt. %	Max 0.15	0.06
D5453	Sulfur	ppm	Max 15	12.4
D6079	Lubricity Major Axis	mm	Max 520	0.624
	Lubricity Minor Axes	mm	none	0.599
	Lubricity Wear Scar	mm	none	0.612
	Description of Scar		none	Circular Evenly Abraded
	Fuel Temperature	°C	none	60
D613	Cetane No		Min 40	61.6
D86	Initial Boiling Point (IBP)	°F	none	443.2
	Evaporation of 5 wt. %	°F	none	466.8
	Evaporation of 10 wt. %	°F	none	472.9
	Evaporation of 15 wt. %	°F	none	478.3
	Evaporation of 20 wt. %	°F	none	481.8
	Evaporation of 30 wt. %	°F	none	491.6
	Evaporation of 40 wt. %	°F	none	502.3
	Evaporation of 50 wt. %	°F	none	512.9
	Evaporation of 60 wt. %	°F	none	523.9
	Evaporation of 70 wt. %	°F	none	536.6
	Evaporation of 80 wt. %	°F	none	551.5
	Evaporation of 90 wt. %	°F	Min 540, max 640	570.5
	Evaporation of 95 wt. %	°F	none	584.4
	Final Boiling Point (FBP)	°F	none	600.1
	Recovered	mL	none	98.9

Method	Test	Units	ASTM specification	Sapphire Energy Diesel Blend
	Residue	mL	none	0.9
	Loss	mL	none	0.2
	Corrected ¹³ IBP	°F	none	443.2
	Corrected ¹ FBP	°F	none	600.1
	Corrected ¹ Evaporation of 10 wt.%	°F	none	473
	Corrected ¹ Evaporation of 50 wt.%	°F	none	513
	Corrected ¹ Evaporation of 90 wt.%	°F	none	570.7
	Corrected ¹ Recovered	mL	none	99
	Corrected ¹ Loss	mL	none	0.1
D93	Flash Point	°F	none	213
	Flash Point	°C	Min 38	101

¹³ Corrected value for barometric pressure, per ASTM D86

8. ASTM D1655 Results

After hydrotreating and distilling the biocrude blend, the jet cut of the product was analyzed using ASTM D1655. The results for the biocrude blend are shown below in Table 7.

Table 9. ASTM D1655 Results.

Method	Test	Units	ASTM specification	Sapphire Jet Fuel Blend
D1319	Aromatics	%	Max 25	7.5
	Olefins	%	none	2.4
	Saturate	%	none	90.1
D130	Corrosion (Copper Strip)		Max No.1	1A
D4052	Density	kg/m ³	775 to 840	791.3
	API at 60°F		none	47.3
	Specific Gravity at 60°F		none	0.7915
	Density at 15°C	g/ml	none	0.7913
D2887	Distillation		none	
	Initial Boiling Point (IBP)	°C	none	128.6
	Evaporation of 5 wt.%	°C	none	152.3
	Evaporation of 10 wt.%	°C	Max 205	160.1
	Evaporation of 15 wt.%	°C	none	166.7
	Evaporation of 20 wt.%	°C	none	171.9
	Evaporation of 25 wt.%	°C	none	174.8
	Evaporation of 30 wt.%	°C	none	179.1
	Evaporation of 35 wt.%	°C	none	183.2
	Evaporation of 40 wt.%	°C	none	188.4
	Evaporation of 45 wt.%	°C	none	192.9
	Evaporation of 50 wt.%	°C	Report	196.1
	Evaporation of 55 wt.%	°C	none	198
	Evaporation of 60 wt.%	°C	none	202.6
	Evaporation of 65 wt.%	°C	none	208
	Evaporation of 70 wt.%	°C	none	212.5
	Evaporation of 75 wt.%	°C	none	216.4
	Evaporation of 80 wt.%	°C	none	219
	Evaporation of 85 wt.%	°C	none	226.4
	Evaporation of 90 wt.%	°C	none	236.1
	Evaporation of 95 wt.%	°C	none	252.2
	Final Boiling Point (FBP)	°C	Max 300	353.1
D2624	Electrical conductivity	pS/m	none	1
	Temperature	°C	none	22.8
D381	Existent Gum	mg/100mL	Max 7	5
D56	Flash Point	°C	Min 38	48
		°F	none	118

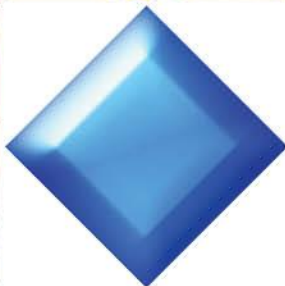
D5972	Freezing point	°C	Max -40 ¹⁴	-59
D1840	Naphthalene Content	Vol. %	none	0.06
D4809	Net heat of combustion	MJ/kg	Min 42.8	43.308
		BTU/lb	none	18619
		cal/g	none	10343.9
D1322	Smoke Point 1	mm	Min 25	27.5
	Smoke Point 2	mm	Min 25	27.0
	Smoke Point 3	mm	Min 25	27.0
	Smoke Point A	mm	Min 25	27.2
D3227	Sulfur, Mercaptan	Mass %	Max 0.003	<0.0003
D2622	Sulfur, Total	Mass %	Max 0.30	0.00102
D3241	Thermal Stability		none	Pass at 260°C
	Filter pressure drop	mm Hg	Max 25	0
	Tube deposits less than		3	1
D3242	Total Acidity	mg KOH/g	Max 0.10	0.007
D445	Viscosity at -20°C	mm ² /s	Max 8.0	3.81
	Viscosity at -20°C	cSt	none	3.81
D3948	Water Separation		Min 85	100

¹⁴ Max -40°C for Jet A. Max -47°C for Jet A-1.

Acknowledgement similar to the following: “This material is based upon work supported by the Department of Energy under Award Number EE0006065.”

Disclaimer similar to the following: “This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.”

Growing the World's Fuels



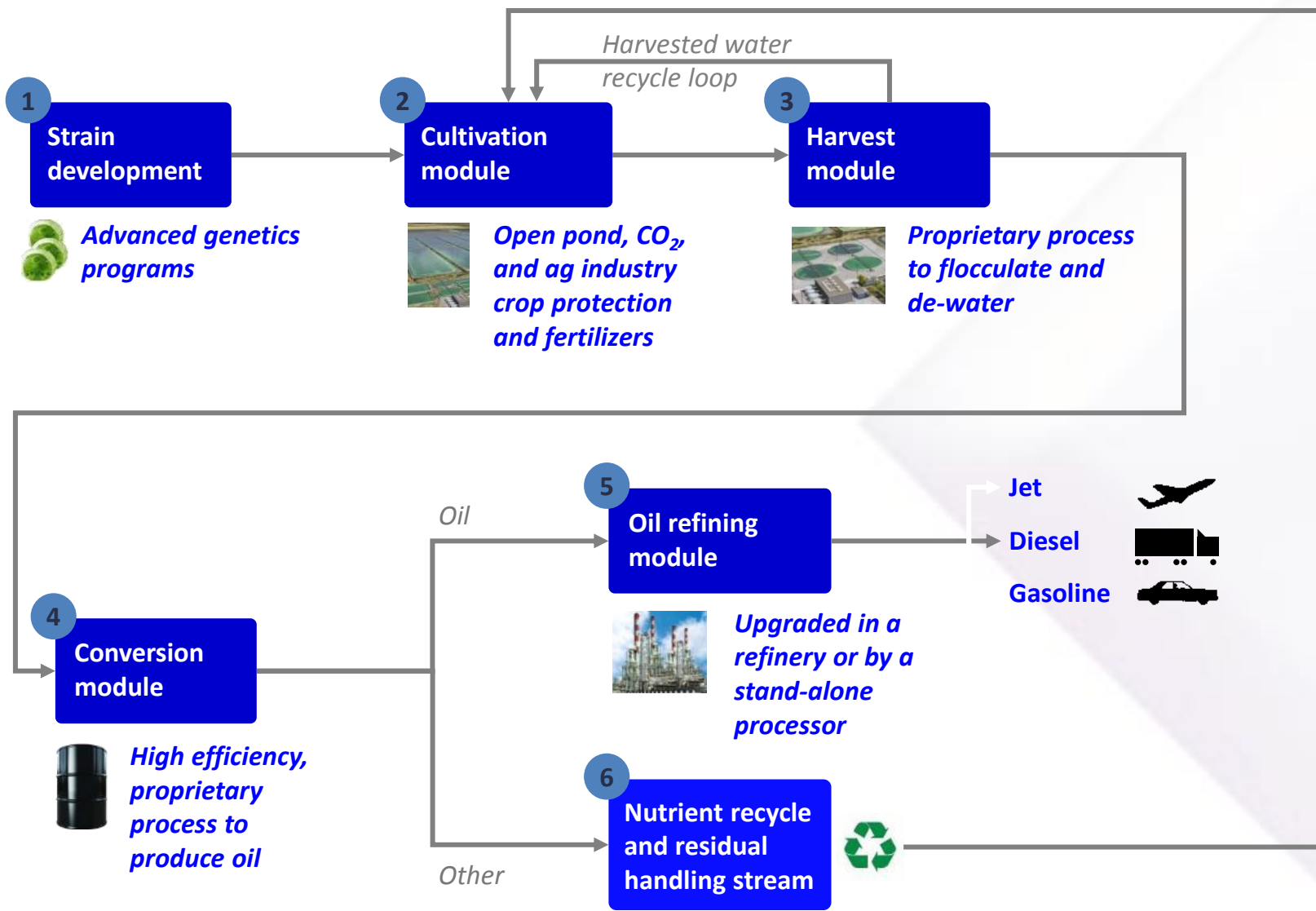
Sapphire
Energy®



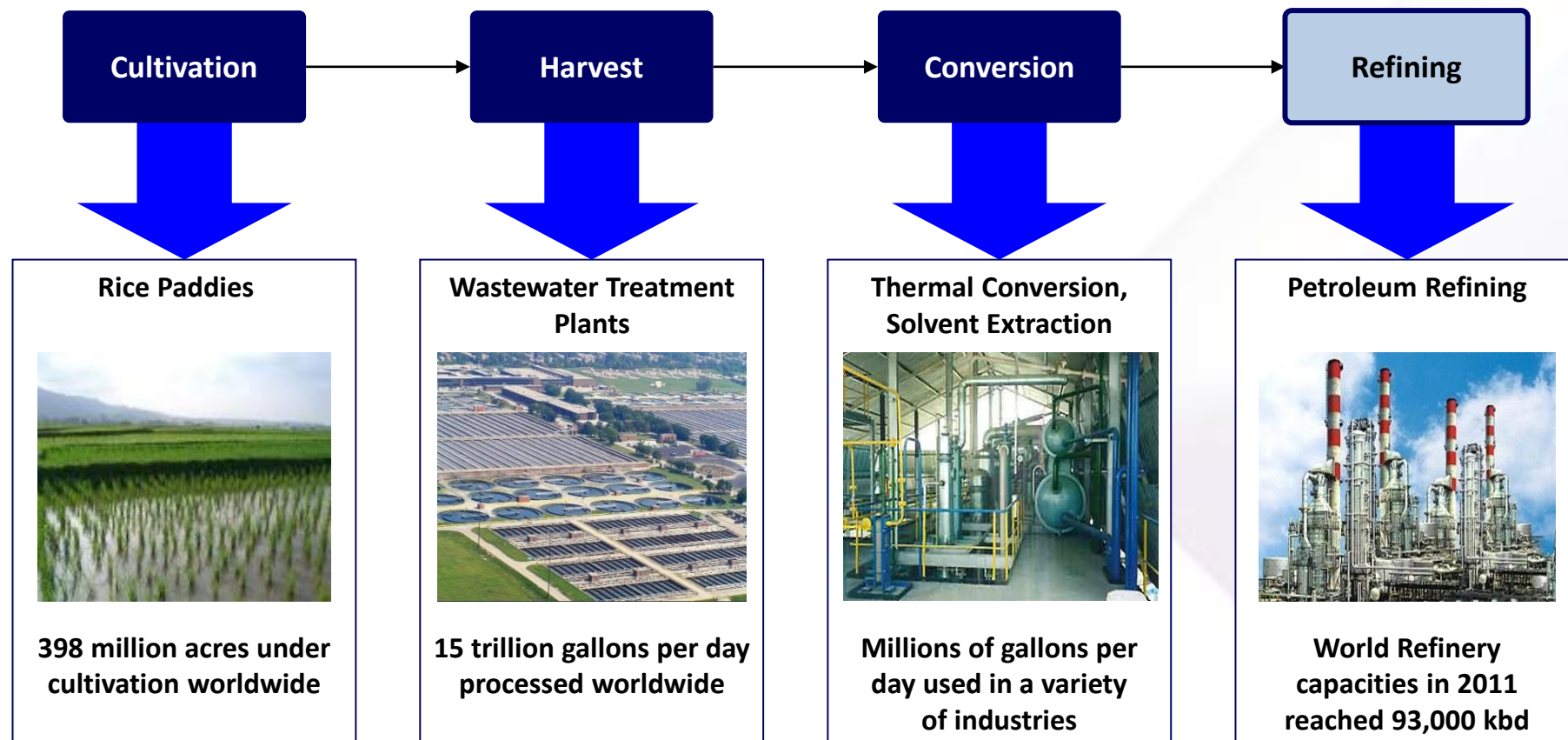
Co-Processing of Green Crude in Existing Petroleum Refineries

Algae Biomass Summit
1 – October - 2014

Overview of Sapphire's process for making algae-derived fuel



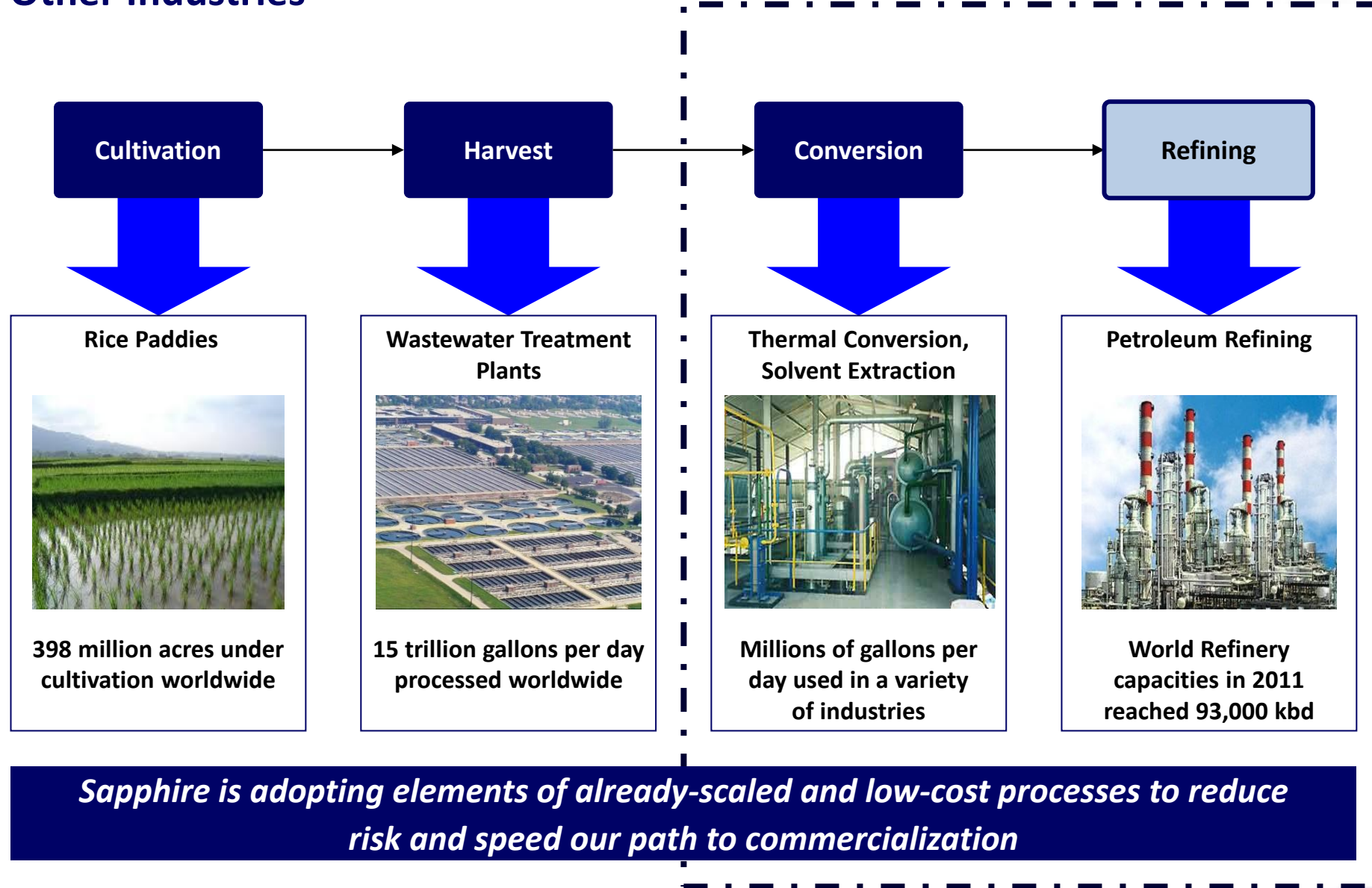
Scalable Technology: Leveraging Existing Techniques and Best Practices from Other Industries



Sapphire is adopting elements of already-scaled and low-cost processes to reduce risk and speed our path to commercialization

Source: UN FAO; BP statistical review 2012

Scalable Technology: Leveraging Existing Techniques and Best Practices from Other Industries



Source: UN FAO; BP statistical review 2012

Conversion: Proprietary Wet Conversion Process Maximizes Oil Yield & Avoids Costly Drying



Sapphire uses a proprietary, innovative, hydrothermal conversion system that is being scaled up with assistance from Linde



Concentrated algae enters the converter as a slurry



Slurry undergoes chemical reactions

- **Heat and pressure:** the slurry is exposed to heat and pressure, releasing lipids and converting algae biomass to Green Crude oil
- **Chemicals:** solvents are added to complete separation process

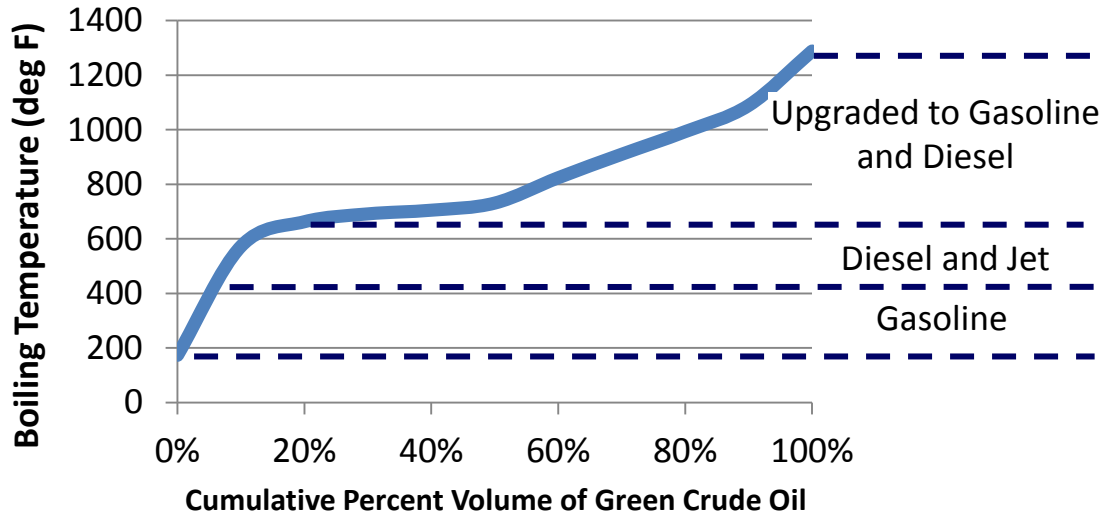
Conversion process creates refinable crude oil



Concentrated algae is processed using proprietary technology to convert biomass into oil

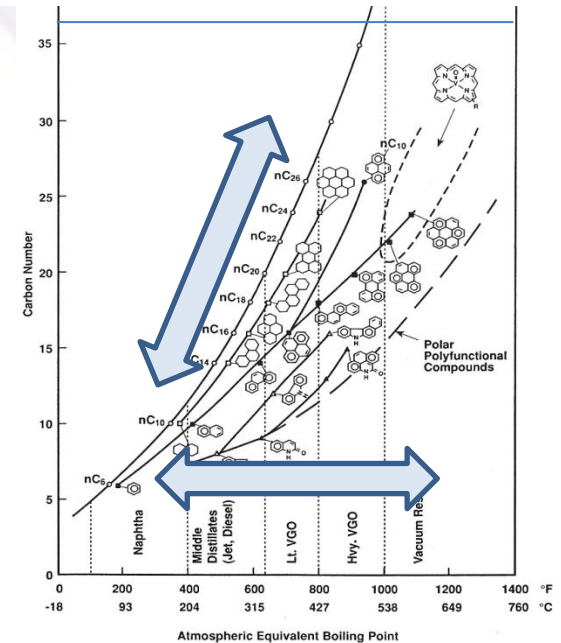
Sapphire Green Crude oil has properties that are similar to a petroleum refinery feedstocks making it attractive for refinery co-processing

Sapphire Green Crude oil reports a full boiling range in high temperature simulated distillation

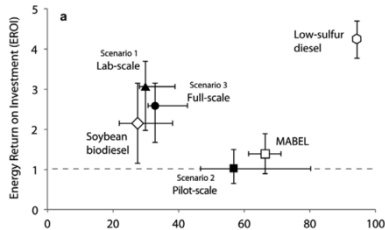


Sapphire Green Crude is highly paraffinic

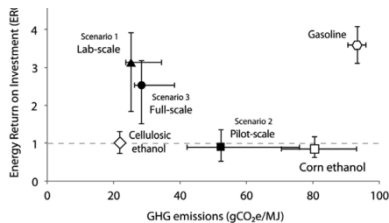
Boduszynski Plot: C# vs. atmospheric equivalent boiling point



Diesel: 65% reduction in GHG vs. petroleum

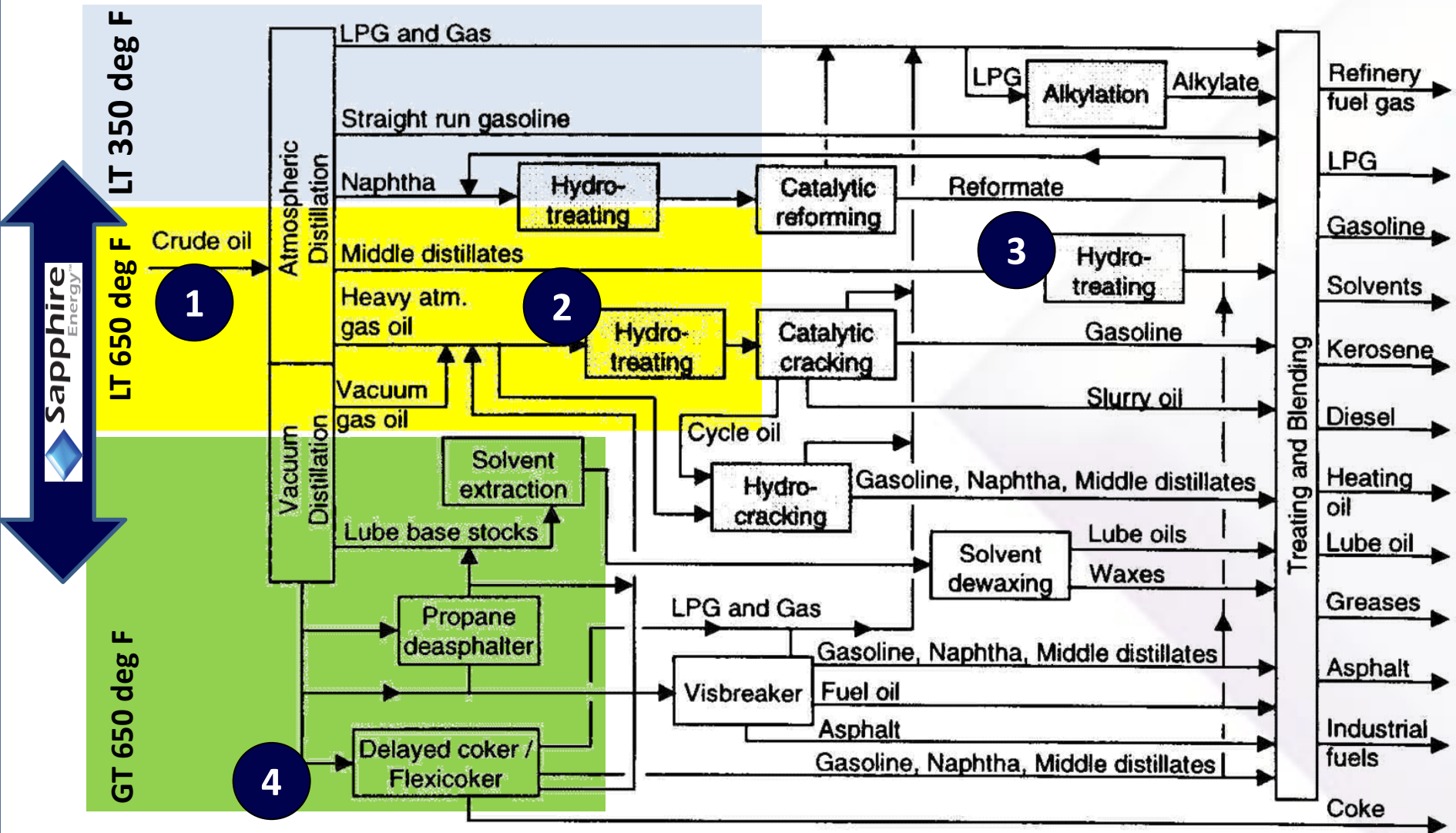


Gasoline 70% reduction in GHG vs. petroleum

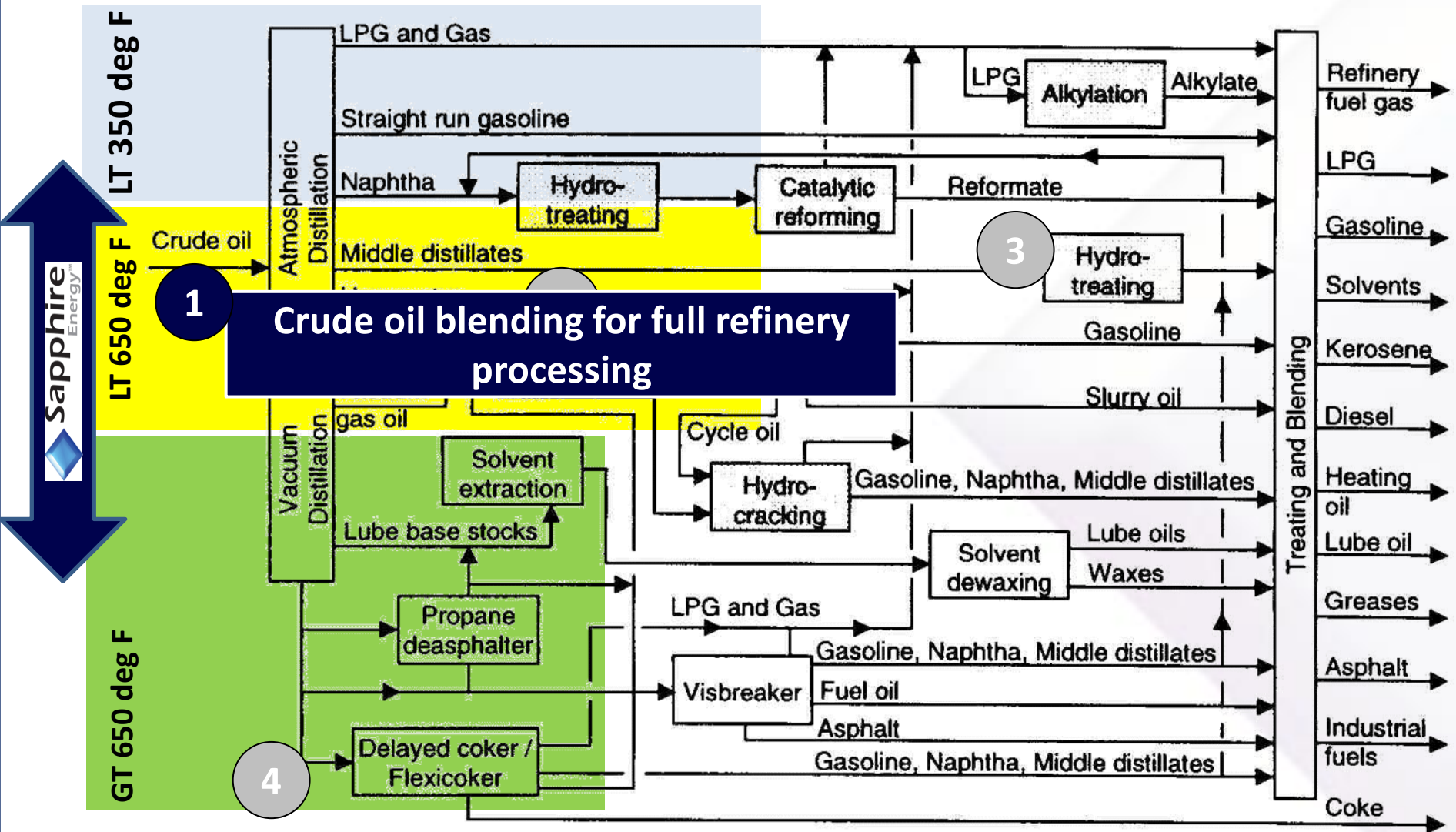


Attribute	Sapphire Green Crude oil compared to petroleum crude
Boiling range	Similar
API Gravity	Similar to a heavy crude oil
Sulfur	Lower
Total Acid Number	Similar to high TAN crudes
Asphaltenes	Lower
Yield of Transportation Fuels	Similar
Carbon Footprint	Lower
Nitrogen and Oxygen	Higher
Metals	Higher

Sapphire has tested of many options for refinery co-processing of Green Crude oil



Sapphire has tested of many options for refinery co-processing of Green Crude oil



Crude blending – why does it matter?

Incompatible crude oils can cause refinery upsets when blended together

- Refiners run multiple crude oils at the same time to optimize overall refinery utilization and maximize profitability
- The co-mingling of incompatible crude oils can cause unplanned refinery outages
 - Mixing incompatible crude oils can lead to heat exchanger fouling and desalter upsets
 - Most crude unit fouling is caused by destabilization of asphaltenes
- Refiners test crude oil compatibility prior to co-processing different crudes
- Baker Hughes Field ASIT Services™ technology is a leading technology to study asphaltene stability in crude blends



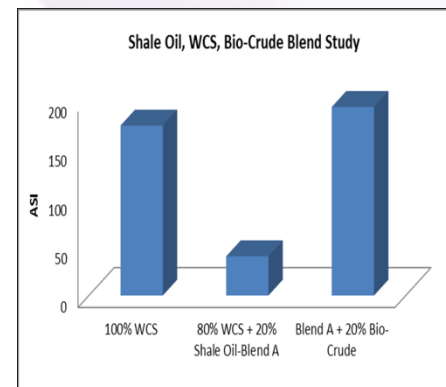
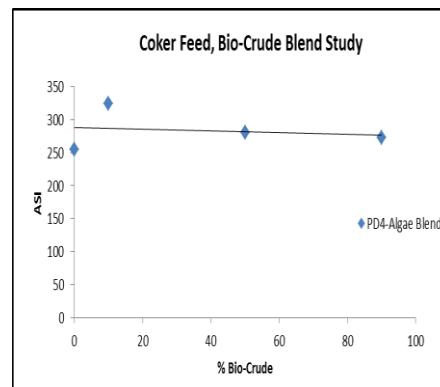
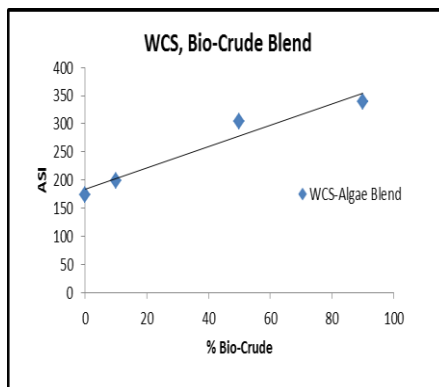
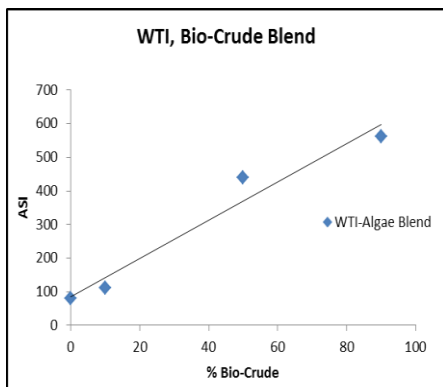
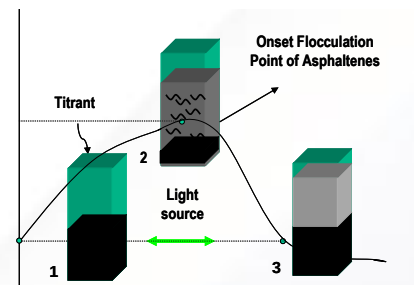
Testing using Baker Hughes ASIT™ shows Sapphire Green Crude oil helps to stabilize asphaltenes in petroleum crude oils



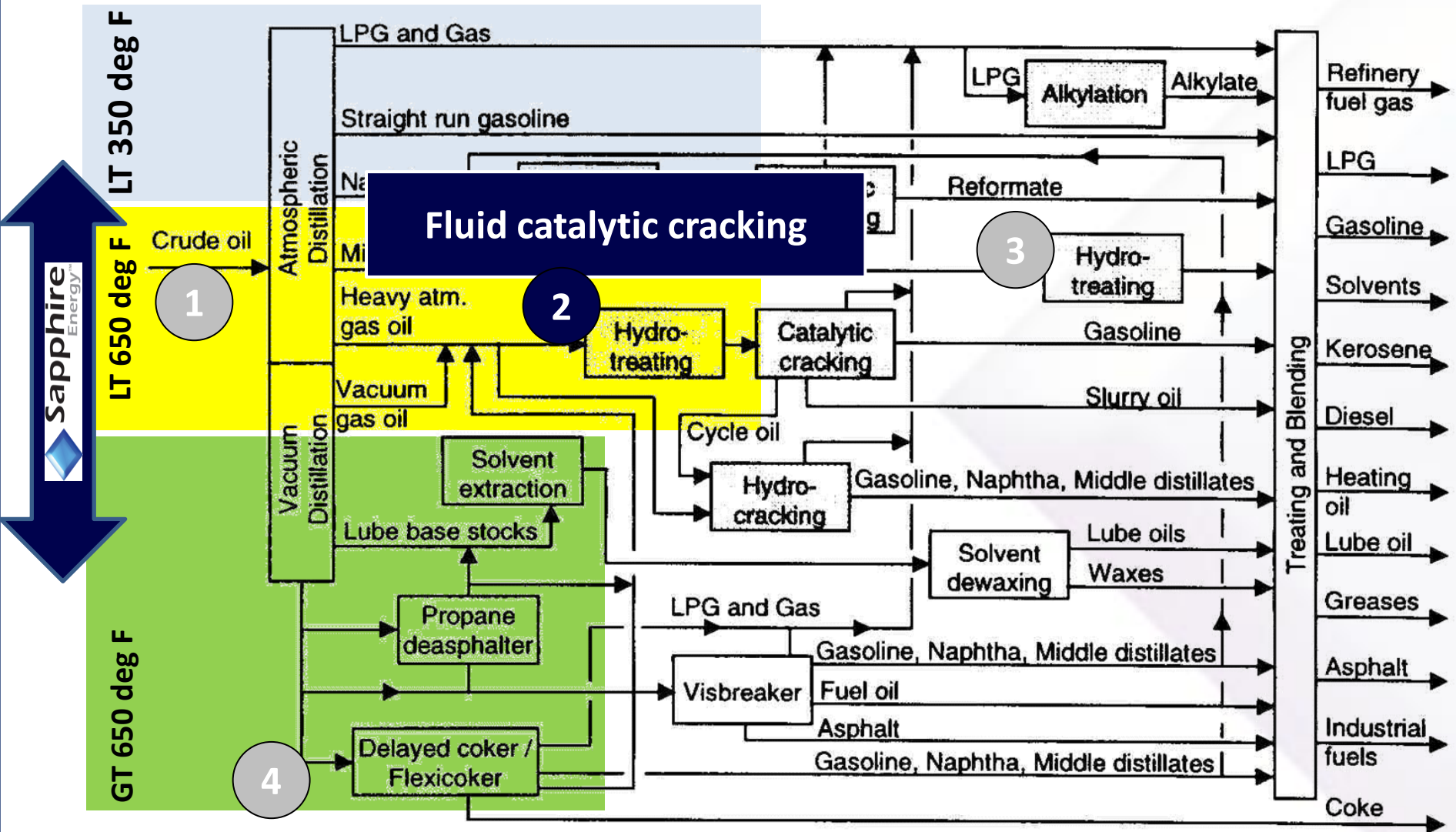
- Preliminary blend studies show that blending Green Crude into conventional crude oils has a positive effect on asphaltenes stability
- Coker feed asphaltenes stability increases up to 10% addition of Green Crude but with any higher blend ratios there is a decreasing trend
- Stability of Shale oil, WCS and Bio crude blend was 5x greater than stability of shale oil/ WCS blend alone

Baker Hughes Field ASIT Services™ Operation Principle

Coherent light is employed measuring the transmittance to determine

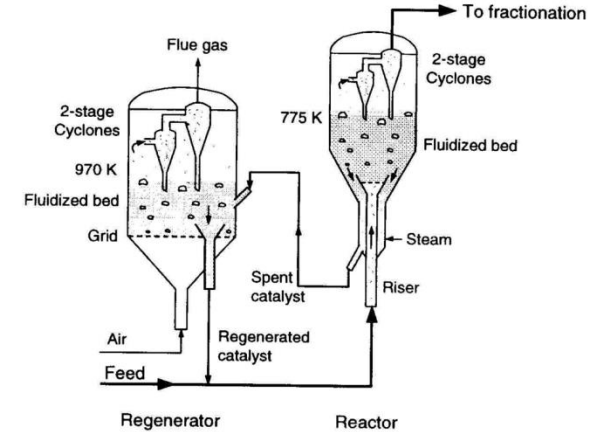


Sapphire has tested of many options for refinery co-processing of Green Crude oil



Fluid Catalytic Cracking: converts high molecular weight gasoils to gasoline, distillate and petrochemical feedstock

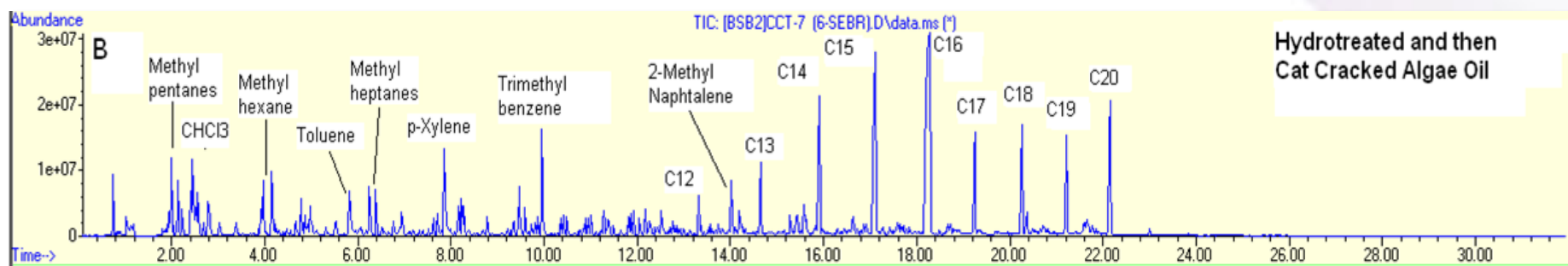
- Key conversion unit in most US refineries
- Heavy gasoils are converted with a fluidized catalyst to crack the carbon carbon bonds
- Coke is deposited on the catalyst in the reactor and burned off in the regenerator to provide heat to the process
 - Robust process design enables new catalyst to be added without a shutdown
- Cracking lowers the average molecular weight and produces a high yield of gasoline and diesel precursors
- FCC products typically need to be processed downstream to produce transportation fuels
 - C4's alkylated to improve gasoline anti-knock properties
 - Gasoline and diesel range material hydrotreated to remove sulfur



Sapphire Green Crude has been tested through refinery fluid catalytic cracker at lab scale and will have a higher margin than typical refinery feed

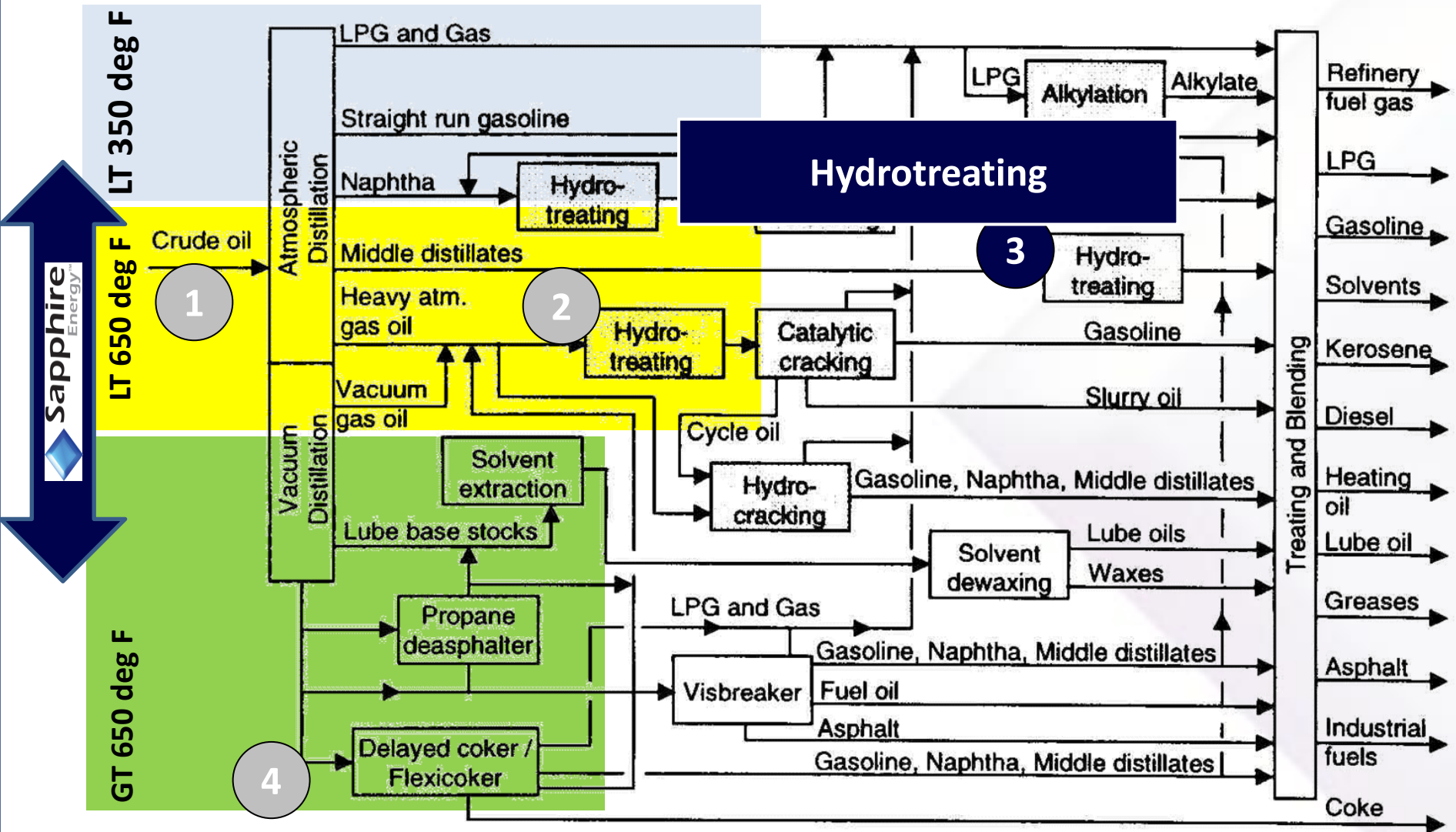
- Product quality from hydrotreating algae Green Crude are consistent with petroleum derived feed
- Products from Fluid Catalytic Cracking algae Green Crude are of higher value than traditional vacuum gasoil feed (VGO)
- Coke formation was the same for petroleum VGO and Green Crude
- Higher nitrogen and oxygen had no impact on yield or product quality
- Higher metals can cause increased catalyst deactivation

FCC Conversion	Green Crude compared to petroleum VGO
C/O	Lower
Conversion (wt%)	Same
C2- (wt%)	50% Lower
C3	30% Higher
C4	30% Higher
Gasoline	Lower
Light Cycle Oil (diesel)	50% Higher
Decant Oil	75% Lower
Coke on Catalyst	Same



NOTE: FCC yield demonstrated using MAT Testing (ASTM D3907)

Sapphire has tested of many options for refinery co-processing of Green Crude oil



Hydrotreating: uses hydrogen to remove sulfur from transportation fuels

- Used to remove sulfur from gasoline and diesel range refinery streams
- Found in almost every refinery
- Fixed bed catalytic reaction in the presence of hydrogen at elevated temperature (600 to 900 °F) and pressure (300 to 2500 psi)
- Removes about 95+% of contaminants such as nitrogen, sulfur, oxygen, and metals from liquid petroleum fractions
- Hydrotreating catalyst typically lasts 24 to 48 months and requires a unit shutdown to change out



Fresh Catalyst



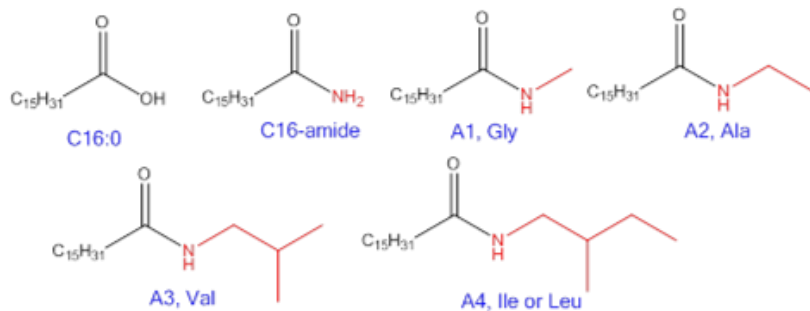
Used Catalyst



Diesel from 10% Sapphire Green Crude/ 90% petroleum distillate produced a higher quality product than 100% petroleum distillate feed

- Hydrotreating was done at typical refinery conditions (2000psi) with a commercial hydrotreating catalyst
- Product has lower aromatic content, lower sulfur, higher cetane
- Negligible catalyst deactivation and coking
- Higher feed oxygen and easy to convert nitrogen produced manageable exotherm at 10% feed with no adverse downstream impacts
- 75 to 85 wt% of the algae oil ended up in the diesel product¹
- 82 to 88 wt% of the algae carbon ended up in the diesel product²
- Hydrotreated over 400 gallons of 10% bio-crude blends with no detrimental impact on catalyst

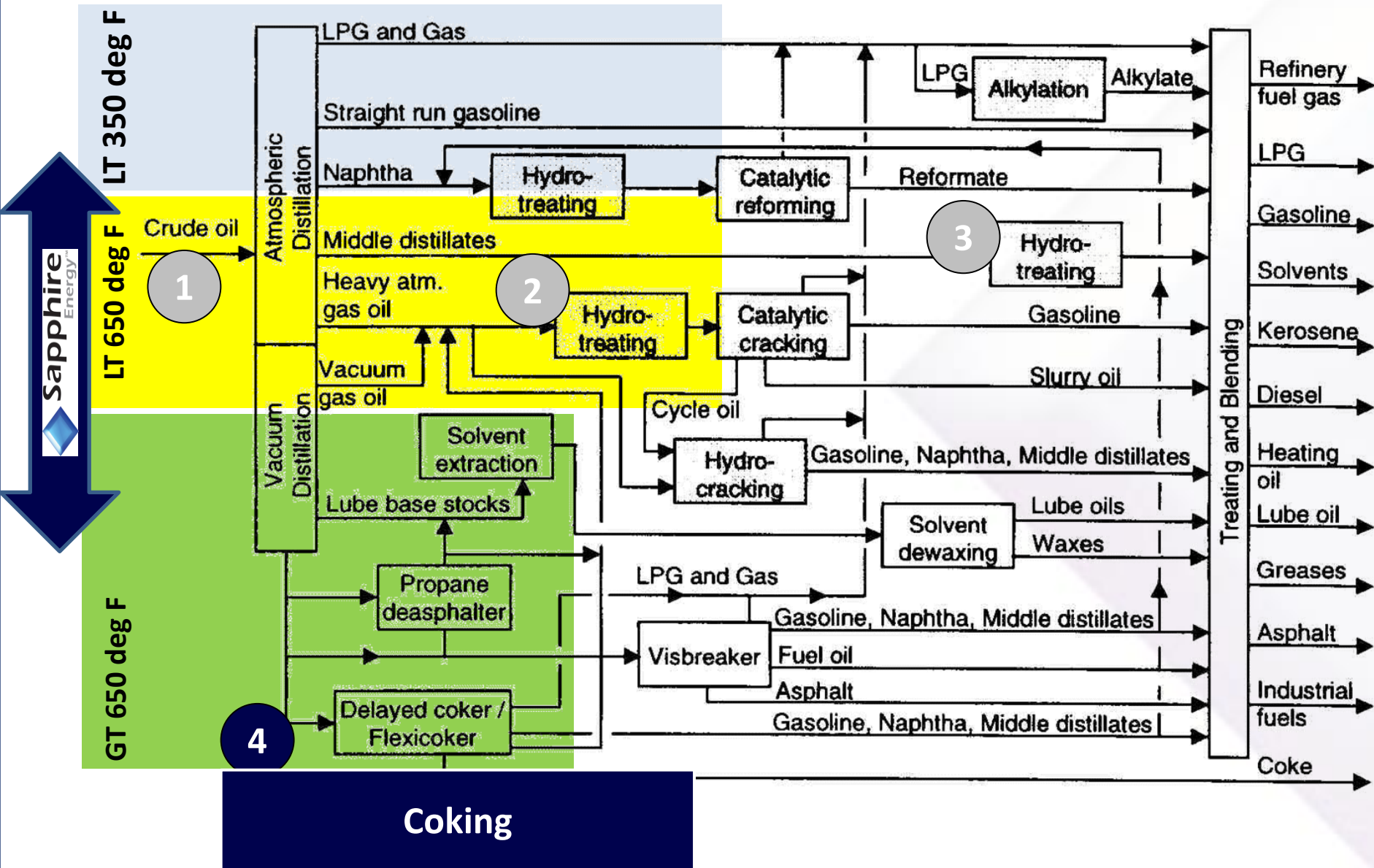
Nitrogen is 90+% easy to convert nitriles and amides



Hydrotreated product quality	100% Petroleum Distillate Feed	10% SEI Green Crude / 90% Petroleum Distillate Feed
Cetane	56	64
Sulfur (ppm)	7.6	2.6
Aromatics (%)	16.4	10.1
Saturates (%)	80.9	88.3
Olefins (%)	2.7	1.6

Notes: (1) Based on pilot plant mass balance, (2) Based on C14 carbon dating

Sapphire has tested of many options for refinery co-processing of Green Crude oil



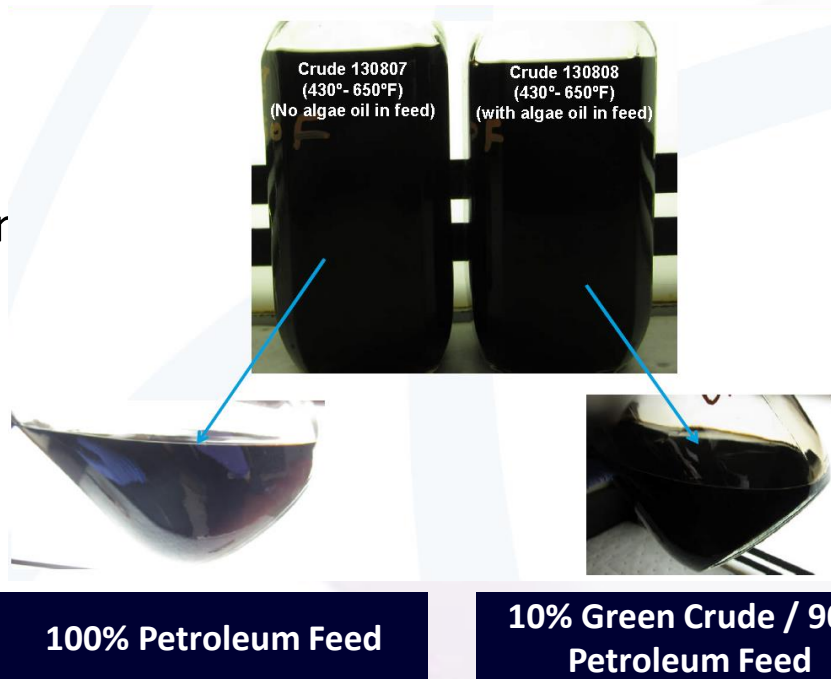
Coking: converts the bottom of the barrel to higher value intermediates

- A coker converts low value high molecular weight vacuum resid to higher value gasoline, jet fuel and diesel blending components
- Coking process can be explained as “Thermal decomposition of high molecular weight hydrocarbon molecules to smaller molecules”
- The principle of coking is simple, requiring only time and temperature
- All the metals and a significant portion of the feed sulphur and nitrogen are rejected in the petroleum coke
- Petroleum coke yield is about 20 to 30 wt% of feed and is essentially solid carbon
 - Cokers produce sponge fuel grade, shot fuel grade or anode grade coke
 - Anode grade coke is sold at a premium while fuel grade is sold at its heating value



Green Crude coker yield is essentially the same as petroleum feed stock and coking provides the lowest risk entry point into a refinery

- Green Crude coker liquid products still contained higher levels of nitrogen and oxygen
- Liquid product yield is the same between Green Crude and petroleum based feed
- Metals were concentrated in petroleum coke
- Green Crude coker product did not adversely effect coke quality



	Petroleum Feed (wt%)	Green Crude (wt%)
Fuel Gas / LPG	24	25
Gasoline Range	19	19
Diesel Range	19	18
Gasoil Range	29	28
Coke	9	10

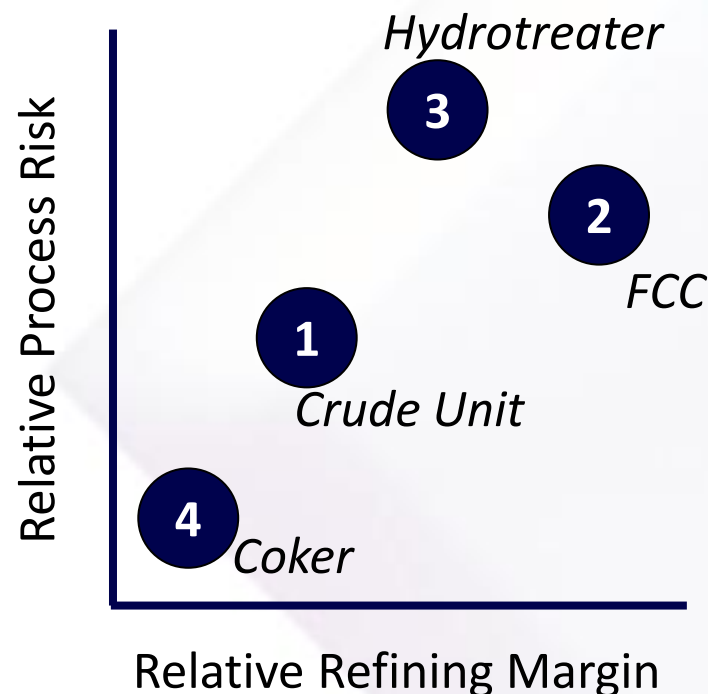


THE UNIVERSITY OF TULSA
Delayed Coking Project

Sapphire Green Crude can be inserted into multiple refinery locations generating similar yield and margin to standard petroleum feedstock

- The testing completed to date has produced data to reduce the perceived risk to processing Green Crude by demonstrating:
 - Co-processing Green Crude with petroleum crude reduces the chance of asphaltene deposition
 - FCC has higher transportation fuels yield than petroleum VGO and similar coke laydown
 - High levels of nitrogen in Green Crude are easily removed via hydrotreating
 - 82+% of the carbon in Green Crude ends up in the diesel product after hydrotreating
 - Metals are concentrated in the coke in the coking process and Green Crude yields are similar to petroleum feeds
- Further testing and larger scale testing is required to provide refiners with the confidence needed to process Green Crude in existing refineries

Refinery insertion points have different process risk and margin



Margin based on 2014 YTD average USGC prices and lab/pilot scale yield data

1. Crude Unit, 2. FCC, 3. Hydrotreater, 4. Coker