

Lignin-derived diesel fuel for rail application (Phase II)

Final Report

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EXECUTIVE SUMMARY

The Transport Canada-sponsored project entitled “Lignin-Derived Drop-in Renewable Diesel Fuels for Rail Applications” has been designed by CanmetENERGY-Ottawa (CE-O, Natural Resources Canada) and its industrial partner CRB Innovations Inc. to assess the feasibility of using lignin-derived diesel fuels as a drop-in biofuel for locomotives for reducing the emissions of non-biogenic greenhouse gases and criteria air contaminants from the rail sector. These “drop-in” biofuels are hydrocarbon-based blending stocks fully compatible with conventional diesel fuels. CE-O and CRB Innovations Inc. have been working together on the development of technologies to convert lignin (a major component of wood) into renewable fuels under a multi-year task-shared agreement. CE-O develops and carries out the catalytic hydrotreatment whereas CRB deconstructs and fractionates lignocellulosic biomass and catalytically depolymerizes the lignin-rich fraction producing clean “oligomeric lignin feedstock” used by CE-O. In this project, the focus is to verify whether diesel fuel blends, containing lignin-derived diesel, can meet CAN/CGSB-3.18-2010 (reaffirmed 2016) specifications for locomotive fuel.

In phase I of the project on lignin-derived diesel fuel for rail applications, CE-O demonstrated that the 5 vol% blend met all 10 CGSB-3.18-2010 locomotive fuel specifications, while the 10 vol% diesel blend met 9 specifications. The total acid number (TAN) was slightly off the CGSB-3.18-2010 specification (0.10 mg KOH/g). Phase I also included a diesel exhaust emission test at Environment and Climate Change Canada using 5 vol% L-diesel blend and a preliminary techno-economic analysis performed by CRB Innovations.

In Phase II of this project (2019-22), CE-O continued to validate that lignin-derived diesel fuels can be blended in a broad range of concentrations with typical fossil diesel fuels for rail applications and that such blends can meet the CGSB-3.18-2010 locomotive fuel specifications. CE-O has improved the quality of L-diesel by designing, assessing and scaling-up an innovative process configuration. CE-O demonstrated that the innovative catalytic hydrotreating process configuration that CE-O developed for the lignin oligomers produced by CRB Innovations Inc. has dramatically improved the oil product quality as evidenced by the lower oxygen content in the final hydrocarbon products and the diesel fraction. CE-O evaluated higher blending ratios and

demonstrated that 100% lignin-derived diesel fuel met 9 CGSB-3.18-2010 and the same 9 CGSB-3.517-2020 specifications, including flash point, kinematic viscosity, distillation temperature @ 90% recovery, sediment and water, total acid number, sulfur content, copper corrosion, carbon residue on 10% bottoms and ash in petroleum products. The low cloud point of 100% L-diesel (155-345 °C) [-36 °C by ASTM D5773] indicates that L-diesel has a fairly good low-temperature operability.

The low value of electrical conductivity suggests that an additive known as an electrical conductivity improver may be needed as it is currently the case for commercial ultra-low sulfur diesel. In terms of lubricity, the wear scar diameter evaluated using the high-frequency reciprocating rig (HFRR) at 60 °C (ASTM D6079) is 590 µm for 100% L-diesel, which is higher than the specification, indicating that the 100% L-diesel or a high concentration blend would require a lubricity additive as it is for the ultra-low sulfur diesel to increase the lubricity thus avoiding excessive wear. Thus the low electrical conductivity of L-diesel and its relatively poor lubricity characteristic are not limiting factors for mixing and blending, as ultra-low sulfur diesel also requires a lubricity additive and an electrical conductivity improver.

The derived cetane number for 100% L-diesel is 39.14 (ASTM D6890-18), which is slightly lower than the minimum cetane number requirement of 40 (CGSB-3.517-2020). A cetane enhancer additive would be needed to boost the ignition quality of 100% L-diesel for its application as locomotive fuel.

Overall, the results indicate that there is no blending limit for the application of L-diesel as locomotive fuel, contingent on adding a cetane improver to 100% L-diesel.

RÉSUMÉ

Le projet parrainé par Transports Canada intitulé « Carburants diesel renouvelables dérivés de la lignine pour les applications ferroviaires » a été conçu par CanmetÉNERGIE-Ottawa (CÉ-O, Ressources naturelles Canada) et son partenaire industriel CRB Innovations Inc. (CRB) pour évaluer la faisabilité d'utiliser des carburants diesel dérivés de la lignine (diesel-L) comme biocarburants pour locomotives afin de réduire les émissions de gaz à effet de serre non biogéniques et celles des principaux contaminants atmosphériques du secteur ferroviaire. Ces biocarburants de remplacement sont des stocks de mélange à base d'hydrocarbures entièrement compatibles avec les carburants diesel conventionnels. CÉ-O et CRB Innovations Inc. ont travaillé ensemble sur le développement de technologies pour convertir la lignine (un composant majeur du bois) en carburants renouvelables dans le cadre d'un accord pluriannuel de partage des tâches. CÉ-O développe et réalise l'hydrotraitement catalytique tandis que CRB déconstruit et fractionne la biomasse lignocellulosique et dépolymérise catalytiquement la fraction riche en lignine produisant des oligomères de lignine qui sont utilisés par CÉ-O comme matière première. Dans ce projet, l'objectif est de vérifier si les mélanges de carburant diesel, contenant du diesel dérivé de la lignine (diesel-L), peuvent répondre aux spécifications CAN/ONGC-3.18-2010 (confirmée en 2016) pour le carburant de locomotive.

Dans la phase I du projet sur le carburant diesel dérivé de la lignine pour les applications ferroviaires, CÉ-O a démontré que le mélange à 5 % en volume répondait aux 10 spécifications de la norme ONGC-3.18-2010 pour carburant de locomotives, tandis que le mélange à 10 % en volume répondait à 9 spécifications. La phase I comprenait également un test d'émissions de moteur diesel à Environnement et Changement climatique Canada utilisant un mélange de diesel-L à 5 % ainsi qu'une analyse technico-économique préliminaire effectuée par CRB.

Au cours de la phase II de ce projet (2019-22), CÉ-O a continué de valider que les carburants diesel dérivés de la lignine peuvent être mélangés dans une large gamme de concentrations avec des carburants diesel fossiles pour les applications ferroviaires et que ces mélanges peuvent répondre à la norme ONGC-3.18-2010. CÉ-O a aussi amélioré la qualité du diesel-L en concevant, évaluant et développant une nouvelle configuration du procédé de conversion. CÉ-O a démontré

que cette nouvelle configuration du procédé d'hydrotraitement catalytique que CÉ-O a développée pour les oligomères de lignine produits par CRB a considérablement amélioré la qualité des hydrocarbures produits, comme en témoigne la faible teneur en oxygène dans les produits d'hydrocarbures finaux et dans la fraction diesel. CÉ-O a évalué des rapports de mélange plus élevés et a démontré que le diesel-L à 100 % répondait à 9 spécifications de la norme ONGC-3.18-2010 et à 9 spécifications de la norme ONGC-3.517-2020, y compris le point d'éclair, la viscosité cinématique, la température de distillation à 90% de récupération, les teneurs en sédiments et en eau, l'indice d'acidité, la teneur en soufre, la corrosion de bandes de cuivre, la teneur en résidus de carbone dans la fraction à 10% la plus lourde, et la teneur en cendres dans les produits pétroliers. Le faible point de trouble du diesel-L à 100 % [-36 °C selon ASTM D5773] indique que le diesel-L a une assez bonne opérabilité à basse température.

La faible valeur de la conductivité électrique suggère qu'un additif connu sous le nom d'améliorant de la conductivité électrique peut être nécessaire comme c'est actuellement le cas pour le diesel commercial à ultra-faible teneur en soufre. En termes d'onctuosité, le diamètre de la cicatrice d'usure évalué à l'aide du banc alternatif haute fréquence (HFRR) à 60 °C (ASTM D6079) est de 590 µm pour 100 % diesel-L, ce qui est supérieur à la spécification, indiquant que 100 % diesel-L ainsi qu'un mélange à haute concentration de diesel-L nécessiteraient un additif de lubrification comme c'est le cas pour le diesel à ultra-faible teneur en soufre pour augmenter le pouvoir lubrifiant, évitant ainsi une usure excessive. Ainsi, la faible conductivité électrique du diesel-L et son pouvoir de lubrification relativement médiocre ne sont pas des facteurs limitants pour la préparation de mélanges, car le diesel à ultra-faible teneur en soufre nécessite également un additif de lubrification et un améliorant de conductivité électrique.

L'indice de cétane dérivé pour le diesel-L à 100 % est de 39,14 (ASTM D6890-18), ce qui est légèrement inférieur à l'indice de cétane minimum requis de 40 (CGSB-3.517-2020). Un additif améliorant l'indice de cétane serait nécessaire pour l'application du diesel-L à 100 % comme carburant de locomotive.

Dans l'ensemble, les résultats indiquent qu'il n'y a pas de limite de mélange pour l'application du diesel-L comme carburant de locomotive, sous réserve de l'ajout d'un additif améliorant l'indice de cétane du diesel-L à 100 %.

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1. INTRODUCTION

The Government of Canada is committed to reducing greenhouse gas (GHG) emissions in line with the targets set out in the Strengthened Climate Plan (SCP) [1]. In 2017, the transportation sector was responsible for about 24% of Canada's overall GHG emissions making it the economic sector with the second highest influence on Canada's GHG emissions after only the oil and gas industry which accounted for 27% of Canada's emissions. Transportation is also a major source of air pollution (e.g. nitrogen oxides, sulfur oxides, particulate matter, volatile organic compounds and carbon monoxide), or criteria air contaminants (CACs). Action is required to address the significant emissions that remain and to align with emerging global and United States standards.

Canada's current rail industry is largely powered by diesel fossil fuels. In accordance with the Strengthened Climate Plan, there is a need for greener fuel alternatives for the rail industry. One option that exists for this objective is blending sustainable biofuels with existing diesel fuels. With this method, the net CO₂ emissions are reduced while keeping the existing infrastructures and diesel engines in operation.

Biodiesel and renewable diesel are two commercially available alternative diesel fuels. However, the cold flow properties of biodiesel greatly limit the volume that can be blended in petroleum-derived diesel and its utilization in cold temperatures. Renewable diesel fuels are produced by hydrotreating vegetable oils, used cooking oils and animal fats, followed by hydroisomerization (HEFA process). However, the high cost of these feedstocks may be an issue for expanding the utilization of renewable diesel.

Lignocellulosic materials are potential lower-cost feedstocks for the production of transportation biofuels from non-food sources. Canada has large quantities of lignocellulosic feedstocks available, including forest biomass and agricultural residues. Depending on plant species, lignin accounts for 15 to 30 wt% of lignocellulosic biomass. The lignin content generally ranges from 5 to 12% in non-woody monocotyledons, 25 to 35% in softwoods, and 15 to 30% in hardwoods [2]. In terms of chemical structure, lignin is a polymer composed of cross-linked phenylpropanoid units

(or monomers) joined by ether bonds (C-O-C) and carbon-to-carbon bonds (C-C). Currently, a massive amount of lignin is generated worldwide as a residue from chemical pulp mills where it is most often burned to produce process heat and power for the mill [3]. Lignin can be converted to lignosulfonates but the markets for these chemicals, such as concrete plasticizers, are small. Furthermore, it is anticipated that future fermentation-based cellulosic ethanol plants will generate large volumes of lignin as a by-product because lignin cannot be converted to fermentable sugars contrary to cellulose and hemicellulose, the other major constituents of trees and plants [4].

CanmetENERGY-Ottawa (CE-O), a R&D centre that is a part of Natural Resources Canada, has been working together with CRB Innovations Inc. to develop a process to convert lignin into “drop-in” biofuels, i.e. renewable fuels that are fully compatible with current petroleum-derived transportation fuels. As indicated in Figure 1, CRB Innovations Inc. is focusing on the lignin fractionation technology using a base-catalyzed depolymerization approach to convert lignin into smaller oxygenated compounds (monomers and oligomers) [5]. CE-O investigates the second processing step where oxygen is removed from oligomers by catalytic hydrotreating to produce “drop-in” biofuels [6, 7].

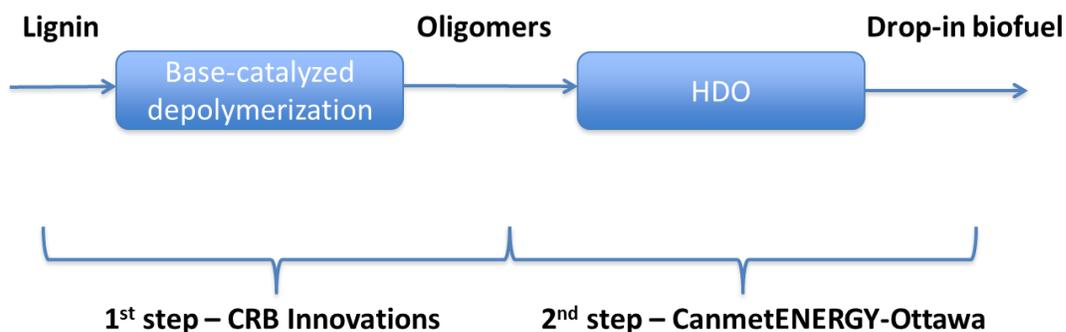


Figure 1 Flow diagram of the process from lignin to drop-in biofuel

In [Phase I](#) of the Transport Canada-sponsored project on lignin-derived diesel fuel (L-diesel) for rail application (see Figure 2), CE-O produced diesel fuels from lignin-derived feedstocks supplied by CRB Innovations Inc. using a catalytic hydrotreating approach in a bench-scale continuous reactor system followed by distillation of the oil product. CE-O prepared two homogeneous blends containing respectively 5 vol% and 10 vol% lignin-derived diesel in petroleum-derived diesel

fuels. CE-O demonstrated that the 5 vol% blend met all CAN/CGSB-3.18-2010 (reaffirmed 2016) locomotive fuel specifications, while the 10 vol% diesel blend met 9 out of 10 specifications. Only the total acid number (TAN) was slightly off the CGSB-3.18-2010 specification, being higher than 0.10 mg KOH/g (0.10 to 0.15 depending on the methods used) [8]. Phase I also included diesel exhaust emission tests at Environment and Climate Change Canada using a 5 vol% L-diesel blend, and a preliminary techno-economic analysis performed by CRB Innovations.

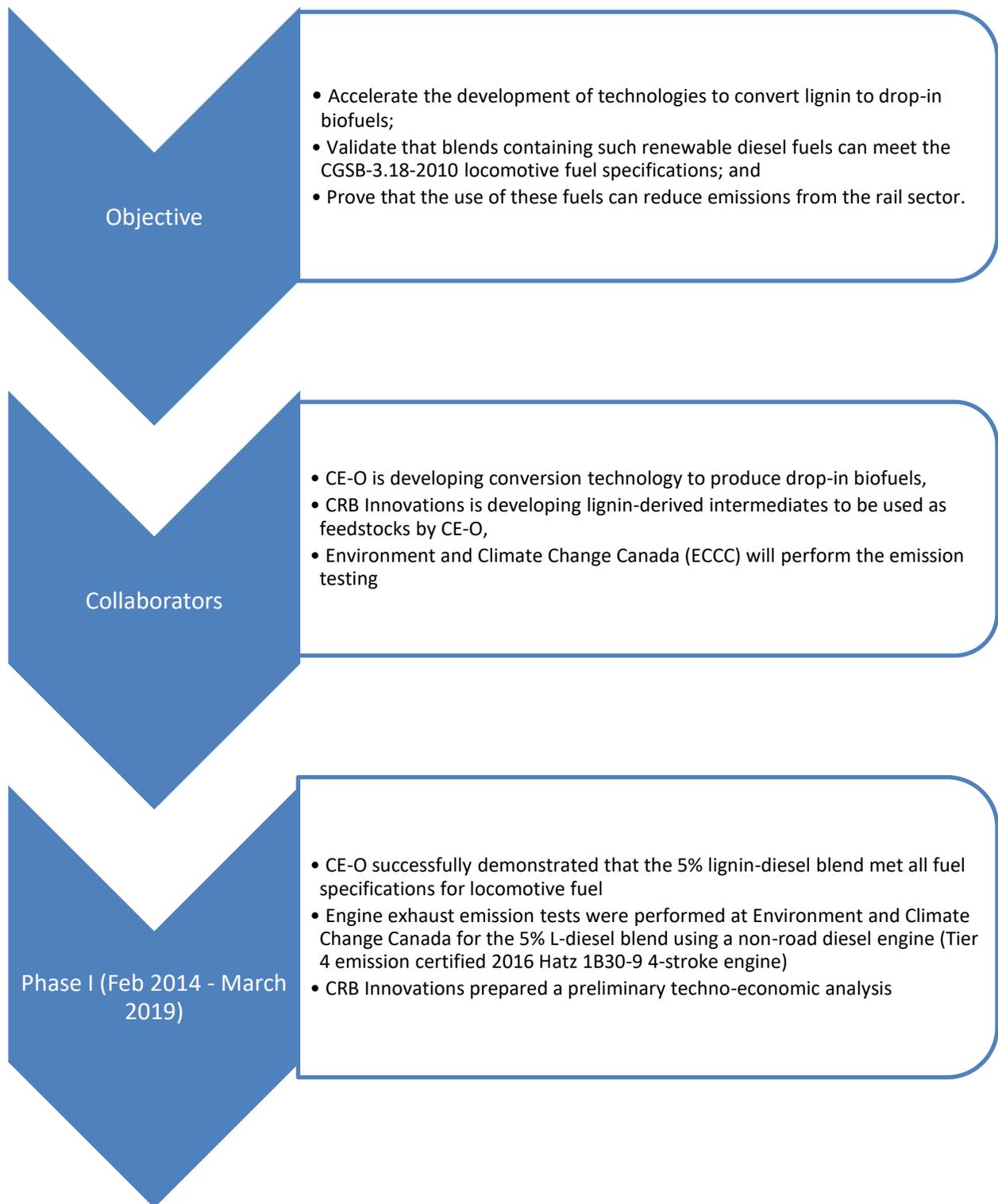


Figure 2 Project flow diagram

Following Phase I, CE-O is continuing to accelerate the development of the technology to convert lignin-derived feedstocks to “drop-in” biofuels (renewable hydrocarbons). The goal of the Phase II project (2019-2022) is to validate that lignin-derived diesel fuels can be blended in a broad range of concentrations with conventional fossil diesel fuels for rail applications and that such blends can meet the CGSB-3.18-2010 locomotive fuel specifications, which are presented in Table 1. The Phase II plan, which has been modified due to the COVID-19 pandemic, is presented in Figure 3. CE-O will produce sufficient volumes of higher-quality hydrotreated oil products, distill them to produce the L-diesel fraction, and subsequently blend the L-diesel at various concentrations with certified diesel for characterization against CGSB-3.18-2010 locomotive fuel specifications. The volume of L-diesel required is dependent on the blending ratio and a total volume of 1.5 L is needed for each blend for full characterization.

Table 1 CGSB-3.18-2010 locomotive fuel specifications

Properties	Test	Specifications
Flash point	ASTM D93	Min: 40.0 °C
Kinematic viscosity @ 40 °C	ASTM D445	Min: 1.70 cSt; Max: 5.0 cSt
Distillation T, 90% recovered	ASTM D86	Max: 360 °C
Sediment and water	ASTM D2709	Max: 0.05 vol%
Total acid number	ASTM D664	Max: 0.10 mg KOH/g
Sulphur	ASTM D7039	Max: 500 mg/kg
Copper corrosion @ 50 °C for 3 hrs	ASTM D130	Max: No.1
Carbon residue on 10% bottoms	ASTM D4530	Max: 0.4 wt%
Ash in petroleum products	ASTM D482	Max: 0.010 wt%
Electrical conductivity	ASTM D2624	Min: 25 pS/m

In Year 1 of Phase II (2019-20), CE-O prepared sufficient volumes of oil products to generate a 1.5-L blend of 10 vol% L-diesel, distill the oil products, prepare the blend with the diesel fraction and characterize it against CGSB-3.18-2010 locomotive fuel specifications.



Figure 3 Project phase II flow chart

In Year 2 of this project (2020-21), CE-O continued improving and scaling-up the catalytic conversion process to further reduce the residual oxygenates and improve the quality of L-diesel. CE-O produced enough oil products for distillation and use L-diesel for preparing blends at higher levels than 10 vol% (20% and higher). These blends are to be characterized against the CGSB-3.18-2010 specifications for viscosity and acidity.

In Year 3 of this project (2021-22), CE-O continued optimizing and scaling-up the conversion process to produce more of the high-quality L-diesel and increase the L-diesel concentration in the blends to determine the maximum blending ratio that meets the specifications. CE-O continued to use acidity and viscosity as the indicators for the initial evaluation of these L-diesel blends against CGSB-3.18-2010 specifications and determine the blending concentration limit at which these parameters are not met. CE-O then performed a full characterization of properties against CGSB-3.18-2010 for the highest successful blending achieved.

2. EXPERIMENTAL

2.1 Catalytic hydrotreating of lignin-derived feedstocks

The CE-O team has conducted extensive experimental tests on the conversion of lignin oligomers into hydrocarbons. A major development in Phase II is that CE-O designed, assessed and scaled-up an innovative processing configuration as a part of its work with CRB Innovations. This new processing configuration has significantly reduced the oxygen content in the output oil product, thus improving the product quality. In addition, to produce a sufficiently large volume of oil products from lignin oligomers for distillation and blending, CE-O scaled up the process to a 200-mL (reactor volume) continuous reactor system in 2020-21 from a smaller 70-mL bench-scale continuous system that was used in previous years. This corresponds to a scale-up factor of about 3, thus increasing the output capacity. The process was further scaled up to a 1-L reactor in 2021-22. Figure 4 shows the pictures of the automated 70-mL, 200-mL and 1-L continuous reactor systems used by CE-O for this work. CRB Innovations provided several batches of lignin oligomers using their 1 kg/h pilot-scale unit for lignin depolymerization.

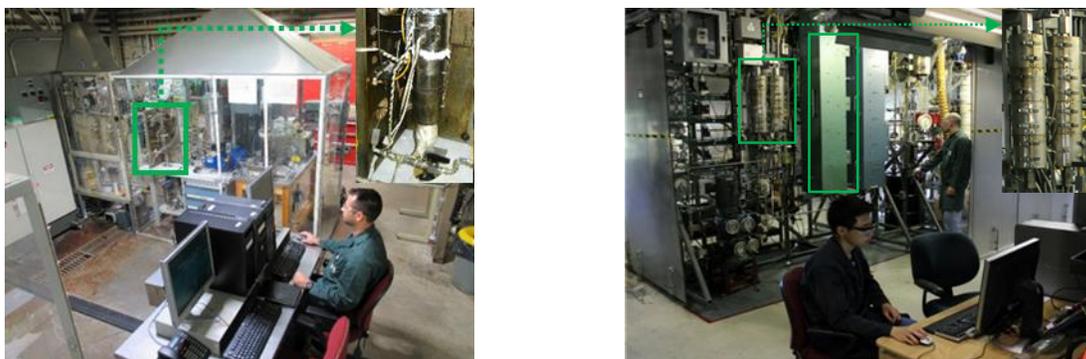


Figure 4 CE-O's reactor systems used for catalytic hydrotreating of lignin-derived feedstocks to drop-in transportation fuels (lignin-derived diesel fuels) (70-mL bench-scale continuous reactor system on the left, 200-mL and 1-L continuous reactor system on the right)

2.2 Vacuum distillation of organic products

The liquid products from catalytic hydrodeoxygenation (HDO) of lignin oligomers process introduced in the previous section usually consisted of two phases (organic and aqueous) which were separated by decantation and/or centrifugation. The organic (oil) phase was mostly made of

hydrocarbons. Vacuum distillation was used to fractionate the organic phase into naphtha (Initial Boiling Point [IBP] to 155 °C), jet fuel (155-250 °C), heavy middle distillates (250-345 °C) and heavy gas oil (345 °C-Final Boiling Point [FBP]). In 2020-21, the lignin-derived heavy middle distillates (250-345 °C) were used for preparing the L-diesel blends, instead of the full diesel range (155-345 °C). The reason for using heavy middle distillates was because CRB Innovations required the 155-250 °C fraction of the oil product for another project. The full diesel range was tested in 2021-22. No product post-treatment was applied in 2020-21 and 2021-22.

The L-diesel fraction analyses include CHN elemental analysis (ASTM D5291), trace sulfur (ASTM D5453), trace nitrogen (ASTM D5762), simulated distillation (ASTM D2887, SIMDIS), and GC-MS search to identify the residual oxygenates. The L-diesel fraction was sent for radiocarbon analysis to confirm its biocarbon content compared with the F¹⁴C of lignin-derived feedstock. Radiocarbon analyses were performed using a 3MV tandem accelerator mass spectrometer (AMS) built by High Voltage Engineering Europa B.V. (HVE) at University of Ottawa. Measurements were normalized with respect to the reference material Oxalic II and carbon ages were calculated using the Libby ¹⁴C half-life of 5568 years.

In 2020-21, CE-O purchased a spinning band vacuum distillation unit (Figure 5). This unit increased CE-O's distillation capacity from 1-L to 5-L while also improving the distillation efficiency. The system was commissioned in 2020-21 and was used for product fractionation in 2021-22.



Figure 5 CE-O's vacuum distillation units (5-L spinning band distillation unit on the left, 1-L short-path vacuum distillation unit on the right)

2.3 Lignin-derived diesel blending and analysis

The diesel fractions were obtained by vacuum distillation of the HDO oil products and blended with petroleum-derived certified diesel fuel at predetermined volumetric ratios to prepare three different diesel blends for characterization according to the CGSB-3.18-2010 locomotive fuel specifications. The L-diesel and petroleum-derived certified diesel were mixed in the same bottle that was vigorously shaken to ensure that the L-diesel blends were homogeneous. CE-O obtained the certified diesel fuel (US EPA 2007 Tier 2 Diesel) from the Air Quality Research Division of Environment and Climate Change Canada. Table 2 lists some characteristics of the certified diesel fuel with more detailed information available in Appendix A. For the L-diesel blending, CE-O prepared blends containing 10, 20, 30 and 40 vol% lignin-derived diesel respectively. These L-diesel blends were characterized against CGSB-3.18-2010 specifications.

Table 2 Characteristics of certified diesel fuel used as blending stock

Property	ASTM	Value
Density @ 15 °C, kg/m ³	D4052	859.3
Specific Gravity, 60/60 °F	D4052	0.860
Elemental analysis		
Carbon	D5291 modified	86.8 wt%
Hydrogen	D5291 modified	13.2 wt%
Nitrogen	D5291 modified	n.a.
Sulphur	D5453	7.0 ppm

3. PROPERTIES OF LIGNIN-DERIVED DIESEL BLENDS

The characteristics of various L-diesel blends were evaluated against the CGSB-3.18-2010 locomotive fuel specifications in Years 1 and 2 of Phase II (2019-2021). The 100% L-diesel without blending was characterized against the CGSB-3.18-2010 locomotive fuel specifications and CAN/CGSB-3.517-2020 diesel fuel specifications in Year 3 of Phase II project.

3.1 Characterization of 10 vol% lignin-derived diesel blend

In Year 1 of Phase II (2019-20), CE-O demonstrated that the 10 vol% diesel blend meets all the tested locomotive fuel specifications, including the total acid number (TAN). Although in Phase I, TAN values fell either on the limit or very close to the limit of the TAN specification (0.10 mg KOH/g) depending on the characterization method, in Phase II, the TAN value is less than 0.05 mg KOH/g, well below the maximum value of 0.10 mg KOH/g specified in CGSB-3.18-2010 (Table 3). In order to reduce the acidity in 2019-20, more severe processing conditions were tested in the hydrotreating process along with a post-treatment step to remove the oxygenate residual [9]. The low TAN value suggested that proceeding with L-diesel concentrations higher than 10 vol% was possible. It should be noted that not all the properties were tested, i.e., carbon residue, ash in petroleum products and electrical conductivity were not measured in 2019-20 due to the limited sample volume.

Table 3 Properties of 10 vol% lignin-derived diesel blends vs. CGSB-3.18-2010 locomotive fuel specifications

Property, unit	ASTM Test	Specifications	Phase I*			Years 1 of Phase II**	
			Commercial Diesel	5% blend	10% blend	10% blend	Certified Diesel
Flash point, °C	D93	Min: 40.0	49.5	49.0	53.0	68.0	72.2
Kinematic viscosity @ 40°C, cSt	D445	Min: 1.70 Max: 5.0	1.969	1.964	2.028	2.641	2.6
Distillation T, 90% recovered, °C	D86	Max: 360	302.6	300.1	300.1	313.6 ^d	312
Sediment and water, vol%	D2709	Max: 0.05	<0.005	<0.005	<0.01	<0.01	n/a
Total acid number, mg KOH/g	D664	Max: 0.10	<0.10	0.06	0.10 ^a 0.15 ^b 0.14 ^c	<0.05 ^e	n/a
Sulphur, mg/kg	D7039	Max: 500	4.1	6.1	10.6	6.4	7.0
Copper corrosion @ 50 °C for 3 hrs	D130	Max: No.1	1a	1a	1a	1a	n/a
Carbon residue on 10% bottoms, wt%	D4530	Max: 0.4	<0.01	<0.01	<0.1	n/a	n/a
Ash in petroleum products, wt%	D482	Max: 0.010	0.001	0.002	0.001	n/a	n/a
Electrical conductivity, pS/m	D2624	Min: 25	505 ^f	62 ^f	60 ^f	n/a	n/a

* Commercial diesel was used for blending in Phase I; ** Certified diesel was used for blending in Phase II.

^a: D664 EQP; ^b: D664 Buffer; ^c: D974; ^d: SIMDIS; ^e: result<0.05; ^f: at 21.6 °C;

3.2 Improved oil product quality (reduced oxygen contents)

In Year 2 of Phase II (2020-21), CE-O designed an innovative catalytic hydrotreating configuration to convert lignin oligomers supplied by CRB Innovations Inc. into hydrocarbons and demonstrated the effectiveness of this process configuration by significantly reducing the oxygen contents in both the oil product and the L-diesel fraction after distillation. Table 4 shows the elemental analysis of two final hydrocarbon products before distillation and that of the heavy middle distillates (250-345°C) and L-diesel (155-345°C) after vacuum distillation. The oxygen contents of oil products and heavy middle distillates have been reduced to 0.65 wt% or less in 2021. The oil product sample generated in 2022 included less than 0.10 wt% oxygen, while L-diesel contained 0.28 wt% oxygen.

The nitrogen content of the heavy middle distillates is less than 1 mg/kg. Heavy middle distillates and L-diesel have sulphur levels of 1.4 mg/kg and 0.6 mg/kg, respectively, which are much lower than the CGSB-3.18-2010 maximum limit of 500 mg/kg and the ultra-low sulfur diesel (ULSD) maximum limit of 15 mg/kg. Therefore, L-diesel blends prepared at any ratio will always meet the CGSB-3.18-2010 sulfur specification.

Table 4 Elemental analysis of oil products, heavy middle distillates (250-345 °C) and L-diesel (150-345 °C).

	ASTM	Oil product 2021	HMD ^a (250-345 °C) 2021	Oil product 2022	L-diesel (150-345 °C) 2022
Carbon, wt%	D5291 modified	87.1	86.7	86.6	86.7
Hydrogen, wt%	D5291 modified	13.5	13.1	13.7	13.2
Nitrogen, wt%	D5291 modified	<0.15	<0.15	<0.15	<0.15
Sulfur, mg/kg	D5453	n/a	1.4	<1.0	0.6 ^b
Total nitrogen, mg/kg	D5762	n/a	<1	<1.0	n/a
Oxygen (wet), wt%	in-house by Elementar	0.62	0.51	<0.1	0.28
Density @15 °C, kg/m³	D4052	873.7	908.9 ^c	839.5	866.6
Water Content by Karl Fisher, mg/kg	D6304	20	13 ^c	13.3	n/a

^a: Heavy middle distillates; ^b: ASTM D7039; ^c: approximate value from a 250 °C+ sample

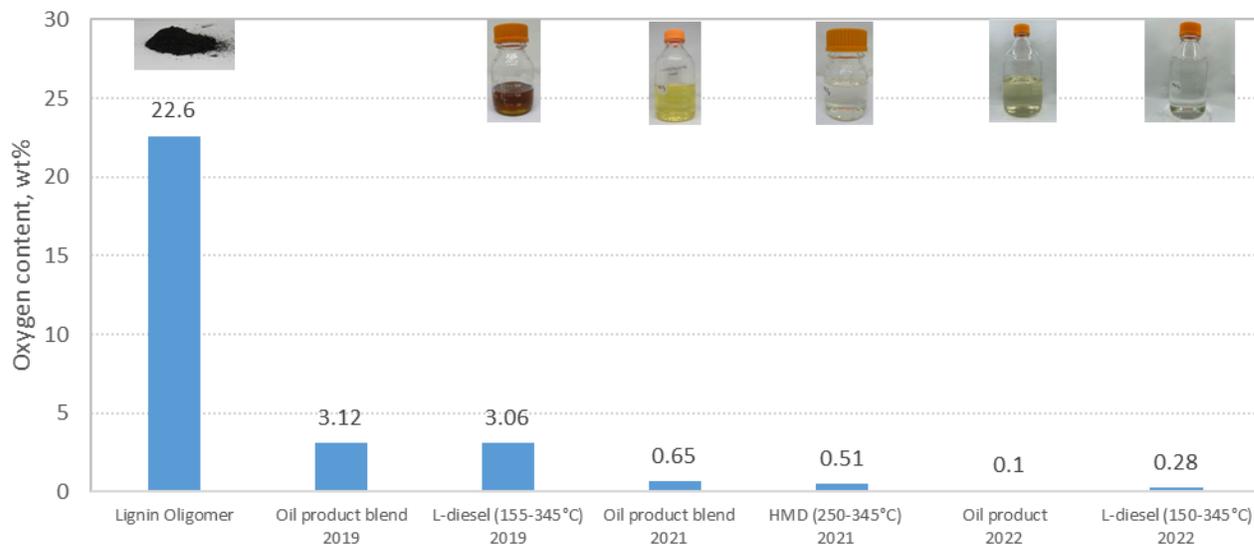


Figure 6 Oxygen contents and pictures of oil product samples and corresponding heavy middle distillates (250-345 °C) and diesel (155-345 °C).

Figure 6 compares the oxygen contents in lignin oligomers, hydrocarbon products, and L-diesel fractions, along with sample pictures. The oxygen content in the starting solid lignin-derived materials is 22.6 wt%. After the hydrotreating process, the oxygen content was reduced to ~3 wt% in 2019-20. With CE-O's improved processing configuration, the oxygen contents in the oil product were significantly reduced to 0.62 wt% or less. The final heavy middle distillates (HMD) fraction contained 0.51 wt% oxygen content and the oxygen content of the L-diesel (155-345 °C) was 0.28 wt%, as shown in Figure 6. In addition, as the oxygen content was reduced, the samples became colorless or with a slight yellow tint similar to conventional fossil diesel. The heavy middle distillates (HMD) fraction (250-345 °C) generated in 2020-21 and the L-diesel (155-345°C) were clear and transparent.

3.3 Characterization of 20, 30 and 40 vol% lignin-derived diesel blend

In Year 2 of Phase II (2020-21), CE-O demonstrated that diesel blends containing 20, 30 and 40 vol% lignin-derived diesel fuel met the CGSB-3.18-2010 acidity and viscosity specifications, as indicated in Table 5. In 2020-21, the heavy fraction of L-diesel (250-345 °C) was used for blending and not all the properties were tested but the focus was rather on acidity and viscosity. The acidity was selected as an evaluation indicator because it was found to be the most challenging specification to meet for the 10 vol% blends in the past. Knowing that the CGSB-3.18-2010 standard has a maximum viscosity limit, the viscosity was characterized because our previous studies showed that it increased with increasing L-diesel content (Table 3) and because heavy middle distillates, which were expected to be somewhat more viscous than the full diesel range (155-345 °C), were used for blending.

With the newly designed, assessed and scaled-up process configuration employed in 2020-21, the quality of the oil product was significantly improved. The Total Acid Number (TAN) values of diesel blends, containing 20, 30, and 40 vol% lignin-derived diesel, are less than 0.05 mg KOH/g, well below the spec limit without applying any post-treatment step [10]. These positive results eliminate the needs for post-treatment of the diesel fraction. The kinematic viscosities of 10%, 20%, 30%, and 40% L-diesel blends are well below the maximum limit of 5.0 cSt and tend to increase with the concentration of L-diesel, from 2.641, to 3.354 cSt.

Table 5 Properties of lignin-derived diesel blends vs. CGSB-3.18-2010 locomotive fuel specifications

Property, unit	ASTM Test	Specifications	Commercial Diesel	Certified Diesel	Years 1 and 2 of Phase II**			
					10% blend	20% blend ^a	30% blend ^a	40% blend ^a
Kinematic viscosity @ 40°C, cSt	D445	Min: 1.70 Max: 5.0	1.969	2.6	2.641	2.928	3.116	3.354
Total acid number, mg KOH/g	D664	Max: 0.10	<0.10	n/a	<0.05	<0.10 ^b (<0.05)	<0.10 ^b (<0.05)	<0.10 ^b (<0.05)

** Certified diesel was used for blending in Phase II.

^a: lignin-derived heavy middle distillates (250-345 °C) were used for blending.; ^b: result<0.05.

3.4 Biogenic carbon content in L-oligomer diesel

Both L-diesel and fossil diesel consist of similar hydrocarbon structures. The key distinction between the two is that the L- diesel should comprise solely biogenic carbons due to its biomass origin and should not include any fossil carbon. ^{14}C analysis has been used as a method to determine the biocarbon content in biofuel blends. CE-O used this technique to confirm the biocarbon content in the final lignin-derived diesel fractions in 2020-21. Table 6 lists the results of lignin-derived feedstocks (oligomers) from CRB Innovations and the L-diesel fraction derived after CE-O's catalytic processing. The fraction modern carbon, $F^{14}\text{C}$, is based on the $^{14}\text{C}/^{12}\text{C}$ isotope ratio for the sample compared to that of the Oxalic II standard. The $F^{14}\text{C}$ measured for lignin-derived feedstocks was 1.1703 ± 0.0037 , and was 1.1765 ± 0.0043 for the HMD fraction (250-345 °C) of L-diesel. These radiocarbon dating analysis results confirmed that the L-diesel HMD fraction contained 100% biocarbon with no contamination of fossil carbon resulting from the processing.

Table 6 Radiocarbon results of lignin-derived feedstock and HMD (250-345 °C)

Material	Material Code	^{14}C yr BP	$F^{14}\text{C}$	Biocarbon content, %
Lignin-derived feedstocks	D	>Modern	1.1703 ± 0.0037	
HMD (250-345 °C)	D	>Modern	1.1765 ± 0.0043	100.5%

3.5 Characterization of 100% L-diesel

The low TAN values obtained in 2020-21 for 20, 30 and 40 vol% blends suggest that a 100% L-diesel has the potential to meet the acidity specification. In 2021-22 (Year 3 of Phase II), CE-O continued to use acidity and viscosity as indicators for the initial evaluation of 100% L-diesel against CGSB-3.18-2010 locomotive fuel specifications. A sample of heavy middle distillates (250-345 °C) without blending with fossil diesel was submitted for total acidity (TAN) (ASTM D664) and kinematic viscosity at 40 °C (ASTM D445). Results are presented in Table 7 under Phase II. The acidity of a sample consisting of 100% heavy middle distillates (HMD) meets the specification. The kinematic viscosity value of 5.530 cSt measured at 40 °C is slightly higher than

the requirement. However, the kinematic viscosity of the full boiling range of L-diesel (155-345 °C) is expected to be lower because the HMD is the heavy portion of diesel. In addition, both the sulfur content and the distillation temperature at 90% recovery of the L-diesel fraction met the CGSB-3.18-2010 specifications prior to blending, i.e. at 100% blending ratio. The distillation temperature at 90% recovery was 309.5 °C, which is much lower than the specification of 360 °C maximum. The sulfur content of the heavy middle distillates was 1.4 mg/kg, far lower than ULSD specification of 15 mg/kg and CGSB-3.18-2010 specifications of 500 mg/kg. These preliminary results indicate that 100% L-diesel has great potential to meet the CGSB-3.18-2010 locomotive fuel specifications.

Table 7 Properties of pure Heavy middle distillates (250-345 °C) and how they relate to CGSB-3.18-2010 standard

Property, unit	ASTM Test	Specification	Commercial Diesel	Certified Diesel	100% L-diesel (250-345 °C)
Appearance					Colorless
Kinematic viscosity @ 40°C, cSt	D445	Min: 1.70 Max: 5.0	1.969	2.6	5.530
Distillation T, 90% recovered, °C	D86	Max: 360	302.6	312	309.5 ^a
Total acid number, mg KOH/g	D664	Max: 0.10	<0.10	n/a	<0.10 ^b (<0.05)
Sulphur, mg/kg	D7039	Max: 500	4.1	7.0	1.4

^a: SIMDIS; ^b: result<0.05.

In the Fall of 2021 (Year 3 of Phase II), CE-O produced more than 1.5 L of L-diesel (155-345 °C) to evaluate its properties against the CGSB-3.18-2010 locomotive fuel specifications. Figure 7 presents a picture of 100% L-diesel, which is a clear and colorless liquid (water-like) at room temperature.



Figure 7 Appearance of pure L-diesel (155-345 °C, samples of 2021-22)

Results in Table 8 demonstrate that the 100% L-diesel meets 9 CGSB-3.18-2010 locomotive fuel specifications, including flash point, kinematic viscosity, distillation temperature @ 90% recovery, sediment and water, total acid number, sulfur content, copper corrosion, carbon residue on 10% bottoms and ash in petroleum products. The flash point is well above the minimum limit of 40 °C whereas kinematic viscosity, sediment and water, and carbon residue are well below the maximum limits. The sulfur content is extremely low, being 0.6 ppm. The acidity, the distillation temperature @ 90% recovery and ash in petroleum products are below the maximum limits.

The value of electrical conductivity is 1 pS/m at 21.7 °C, which is less than the minimum requirement of 25 pS/m. It should be noted that for ultra-low sulfur diesel (ULSD), the electrical conductivity is also very low at 1-2 pS/m [11]. For both fuels, the low conductivity is due to the heteroatomic species (sulfur-containing compounds or oxygenates), that promote electrical conductivity, being removed by hydrotreating. The low electrical conductivity of L-diesel suggests that an additive, known as static dissipator additive or anti-static additive or electrical conductivity improver, is needed, as it is for commercial ULSD, to increase the conductivity of the fuel, thus dissipating static charges.

Table 8 Properties of pure L-diesel (155-345 °C) and how they relate to CGSB-3.18-2010 standard

Property, unit	ASTM Test	Specification	Commercial Diesel	Certified Diesel	100% L-diesel (250-345 °C)	100% L-diesel (155-345 °C)
Appearance					Colorless	Colorless
Flash point, °C	D93	Min: 40.0	49.5	72.2		61.5
Kinematic viscosity @ 40°C, cSt	D445	Min: 1.70 Max: 5.0	1.969	2.6	5.530	2.174
Distillation T, 90% recovered, °C	D86	Max: 360	302.6	312	309.5 ^a	278.6
Sediment and water, vol%	D2709	Max: 0.05	<0.005			0.01
Total acid number, mg KOH/g	D664	Max: 0.10	<0.10		<0.10 ^b (<0.05)	<0.1
Sulphur, mg/kg	D7039	Max: 500	4.1	7.0	1.4	0.6
Copper corrosion @ 50 °C for 3 hrs	D130	Max: No.1	1a			1b
Carbon residue on 10% bottoms, wt%	D4530	Max: 0.4	<0.01			<0.1
Ash in petroleum products, wt%	D482	Max: 0.010	0.001			<0.010
Electrical conductivity, pS/m	D2624	Min: 25	505 ^c at 21.6 °C			1 at 21.7 °C

^a SIMDIS, ^b result<0.05, ^c static dissipator additive or anti-static additive is used.

3.6 Evaluation of 100% L-diesel against CAN/CGSB-3.517-2020 standard

In September 2021, with the approval of the CGSB Committee on Middle Distillate Fuels, the CAN/CGSB-3.18-2010 (reaffirmed 2016) Standard was withdrawn. The fuel meeting standard CAN/CGSB-3.517-2020 may be used for applications that were formerly covered by CAN/CGSB-3.18-2010. The evaluation of 100% L-diesel against CGSB-3.517-2020 is presented in Table 9 [12]. The CGSB-3.517-2020 specifications are more stringent than CGSB-3.18-2010 in terms of kinematic viscosity, water and sediment, sulfur, and carbon residue while the specifications for flash point, distillation temperature at 90% recovery, acid number, copper strip corrosion and ash in petroleum products remain unchanged.

Results presented in Table 9 demonstrate that the 100% L-diesel still meets 9 CGSB-3.517-2020 specifications for both Type A and Type B, including flash point, kinematic viscosity, distillation

temperature @ 90% recovery, sediment and water, total acid number, sulfur content, copper corrosion, carbon residue on 10% bottoms, and ash in petroleum products.

Table 9 Evaluation of 100% L-diesel against CGSB-3.517-2020

Property	CGSB-3.517-2020		Test method ASTM	100% L-diesel (155-345 °C)	CGSB-3.18- 2010
	Type A	Type B			
Flash point, °C	Min: 40.0	Min: 40.0	D93	61.5	Min: 40.0
Kinematic viscosity at 40°C, mm ² /s (cSt)	Min: 1.30 Max: 3.60	Min: 1.70 Max: 4.10	D445	2.174	Min: 1.70 Max: 5.0
Distillation T, 90% recovered, °C	Max: 290	Max: 360	D86	278.6	Max: 360
Water and sediment, % by volume	Max: 0.02	Max: 0.02	D2709	0.01	Max: 0.05
Acid number, mg KOH/g	Max: 0.10	Max: 0.10	D664	<0.1	Max: 0.10
Sulphur, mg/kg	Max: 15	Max: 15	D7039	0.6	Max: 500
Copper strip corrosion, 3 h at a minimum test temperature of 50°C	Max: No. 1	Max: No. 1	D130	1b	Max: No.1
Carbon residue on 10% bottoms, % by mass	Max: 0.1	Max: 0.2	D4530	<0.1	Max: 0.4
Ash, % by mass	Max: 0.010	Max: 0.010	D482	<0.010	Max: 0.010
Low-temperature flow properties, Cloud point, °C	Varies with the period and location of intended use		D5773	-36	
Electrical conductivity, at point, time and temperature of delivery to the purchaser, pS/m	Min: 25	Min: 25	D2624	1 at 21.7 °C	Min: 25
Lubricity, HFRR at 60 °C, WSD	Max: 460 µm	Max: 460 µm	D6079	590	
Ignition quality, derived cetane number (DCN)	Min: 40.0	Min: 40.0	D6890-18	39.14	Min: 37

Note: Type A is intended for use in selected applications such as urban buses and underground mining or when ambient temperatures require better low-temperature properties than Type B. Type B is seasonally adjusted to meet the low-temperature operability requirements for the period and location of intended use for most locations in Canada. This adjustment affects the other properties of the fuel. Type A diesel fuel is applicable for use where the low-temperature operability of Type B is insufficient. The maximum 90% distillation temperature limits of 290°C for Type A and 360°C for Type B are based on Canadian experience with petroleum-derived diesel fuel.

The cloud point of 100% L-diesel (155-345 °C) was measured as -36 °C by ASTM D5773, which is lower than that of certified diesel -28.9 °C (Appendix A). The lower cloud point indicates that L-diesel has a good cold operability characteristic.

In terms of lubricity, the wear scar diameter evaluated using the high-frequency reciprocating rig (HFRR) at 60 °C (ASTM D6079) was 590 µm for 100% L-diesel, which exceeded the maximum

of 460 μm WSD limit in CAN/CGSB-3.517-2020. It should be noted that the ultra-low sulfur diesel (ULSD) typically has poor natural lubricity as well, (roughly from 484 to 551 μm WSD) [13] which is due to the desulfurization process altering the chemistry of the final ULSD product and removing some of its organic compounds responsible for lubrication. The low lubricity of 100% L-diesel suggests that a lubricity additive may be needed to increase the lubricity and avoid excessive wear, as it is the case for ULSD. Lubricity additives generally used in fossil diesel fuels are expected to function with L-diesel.

To assess the ignition quality of pure L-diesel, cetane index values were initially calculated according to the commonly used ASTM D976 and D4737 methods, which are based on experimental values of density and distillation temperatures. The density measurement was conducted using the ASTM D4052 method whereas the distillation profile of L-diesel was obtained using the ASTM D86 test method (Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure). Table 10 lists the cetane index values of L-diesel blends (10, 20, 30 vol%) and 100% L-diesel. For the 10, 20 and 30 vol% L-diesel blends, the cetane index determined with the ASTM D976 two-variable equation was 43.7, 41.3, and 40.0, respectively, whereas the ASTM D4737 four-variable equation gave 41.9, 40.2, and 39.0 respectively. These cetane index values show that the 10% and 20% L-diesel blends meet the minimum cetane number specified in CGSB-3.18-2010 and CGSB-3.517-2020. In the case of the 30% L-diesel blend, the cetane index values are 40.0 or 39.0, which are on the borderline.

Table 10 Cetane index of L-diesel blends (155-345 °C)

Property, unit	ASTM Test	Certified Diesel	10% L-diesel blend	20% L-diesel blend	30% L-diesel blend	100% L-diesel
Density @ 15 °C, kg/m ³	D4052	859.3	860.0	859.3	860.3	866.6
Distillation temperature, °C						
T10		215	209.6 ^a	204.8 ^a	202.3 ^a	194
T50		266	265.3 ^a	254.3 ^a	250.5 ^a	233.4
T90		312	313.6 ^a	307.3 ^a	303.5 ^a	278.6
Cetane Index ^b	D976	44.1	43.7	41.3	40.0	33.3
Cetane Index ^b	D4737 A	42.6	41.9	40.2	39.0	32.7

a: ASTM D 86 correlation values from SIMDIS D 2887 exta; b: calculated values.

The calculated cetane index for 100% L-diesel is in the range of 32.7 to 33.3 (Table 10), which is lower than the minimum cetane number requirement of 40.0 (CGSB-3.517-2020). However, it should be noted that the calculated cetane index is an approximation of the cetane number. Compared to conventional diesel, L-diesel has a narrow boiling range, with a final boiling point (FBP) of 298 °C, thus resulting in a low T_{50} of 233.4 °C (vs. 266 °C for the certified diesel). Based on the D976 correlation equation, a lower T_{50} value has a negative impact on the cetane index.

To validate the calculated cetane index values, a sample of 100% L-diesel was evaluated according to ASTM D6890-18, which gives derived cetane number (DCN). The derived cetane number was measured as 39.14 using an ignition quality tester (Table 9, Appendix K). The DCN value of 39.14 is only slightly lower than the minimum cetane number requirement of 40 (CGSB-3.517-2020). This result demonstrates that the cetane index correlations from SIMDIS underestimated the ignition property of 100% L-diesel, however, a cetane enhancer additive would still be needed to boost the ignition quality of 100% L-diesel for its use as locomotive fuel. For 10, 20 and 30% L-diesel blends, the derived cetane numbers could also be higher than the cetane index values reported in Table 10.

4. CONCLUSIONS

In Phase II (2019-2022), CanmetENERGY-Ottawa continued to validate that lignin-derived diesel fuels can be blended in a broad range of concentrations with typical fossil diesel fuels for rail applications and that such blends can meet the CAN/CGSB-3.18-2010 locomotive fuel and CGSB-3.517-2020 diesel standards. CE-O has kept on improving the quality of L-diesel by designing, assessing and scaling-up an innovative process configuration. CE-O demonstrated that the innovative catalytic hydrotreating process configuration that CE-O developed for the lignin oligomers produced by CRB Innovations Inc. has dramatically improved the oil product quality as evidenced by the lower oxygen content in the final hydrocarbon products and diesel cut. CE-O evaluated higher blending ratios (10, 20, 30 and 40 vol%), and demonstrated that 100% pure lignin-derived diesel fuel met 9 CGSB-3.18-2010 locomotive fuel specifications and the same 9 CGSB-3.517-2020 specifications for Types A and B diesel fuels, including flash point, kinematic viscosity, distillation temperature @ 90% recovery, sediment and water, total acid number, sulfur content, copper corrosion, carbon residue on 10% bottoms and ash in petroleum products. The

low cloud point of 100% L-diesel (155-345 °C) (-36 °C by ASTM D5773) suggests that L-diesel would have fairly good low-temperature operability.

The low value of electrical conductivity implies that an electrical conductivity improver may be needed as it is currently the case for commercial ultra-low sulfur diesel (ULSD). In terms of lubricity, the wear scar diameter evaluated using ASTM D6079 is 590 µm for 100% L-diesel, which is higher than permitted in the specification. This indicates that, similar to commercial ULSD, the 100% L-diesel or a high concentration blend requires a lubricity additive to increase the lubricity and prevent excessive wear. Thus the low electrical conductivity of L-diesel and its relatively poor lubricity characteristic are not limiting factors for mixing and blending, as ultra low sulfur diesel also requires a lubricity additive and an electrical conductivity improver.

The derived cetane number (DCN) for 100% L-diesel is 39.14 (ASTM D6890-18), which is only slightly lower than the minimum cetane number requirement of 40 (CGSB-3.517-2020). A cetane enhancer additive would be needed to boost the ignition quality of 100% L-diesel for its use as locomotive fuel.

Overall, the results indicate that there is no blending limit for the application of L-diesel as locomotive fuel, contingent on adding a cetane improver to 100% L-diesel.

5. NEXT STEPS

CE-O is planning to continue the optimization, integration and scale-up of the catalytic hydroprocessing of the lignin oligomers produced by the CRB Fractionation process. Larger quantities of L-diesel will thus be produced to do the engine tests in order to determine the exhaust emissions. Furthermore, an engine emission test using blends with a higher renewable content (10 or 20 vol%) will be conducted to better understand the impact of blending of lignin-derived diesel on the diesel exhaust emissions. Our main industrial partner, CRB Innovations, is developing the plan along with Enerkem and other partners, participating in the scale-up effort on feedstock procurement and preparation as well as on hydroprocessing of the intermediate oligomers derived from CRB's Biomass Fractionation process.

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7. APPENDICES

APPENDIX A - Properties of certified diesel fuel

Baseline Fuel Specifications	USEPA 2007 Tier 2 Diesel
ERMD #	CertDiesel-2019-1
Specification	CFR 1065-703
Analysis Report #	COA
Density (kg/m ³ @15 °C)	859.3
Specific Gravity (60/60°F)	0.8600
Gravity deg API=141.5/SG-131.5	33.0
Cetane Number (ASTM D613)	40.8
Cetane Index	41.3
Sum H%+C%	100.0
Carbon (wt%)	86.8
Hydrogen (wt%)	13.20
Nitrogen (ppm)	na
Total Sulfur %	0.0007%
Total Sulfur (ppm)	7.0
Volume % Aromatics	30.0
% Olefins	na
% Saturates	na
Flashpoint, min °C	72.2
Cloud Point max °C	-28.9
Kinematic Viscosity, Centistokes	2.600
Net Heating Value (btu/lb)	18158
Net Heating Value MJ/kg	42.21
Distillation Range, % Evap	
IBP (initial boiling point) °C	186
10%	215
50%	266
90%	312
End Point	335
Volatility ° F=(P10+P50+P90)/3	501
IBP (initial boiling point) °F	347.0
10%	407.0
50%	505.0
90%	591.0
End Point	636.0
Volatility ° F=(P10+P50+P90)/3	501

APPENDIX B – Analysis reports of 10% L-diesel blend



Report of Analysis

Natural Resources Canada
Jillian Gawlik
1 Haanel Drive
Ottawa, ON K1A1M1
Canada

Our Reference Number: CA120-0023957
Lab Reference Number: 2020-HAML-000348
Customer Reference Number: PO #: 3000871561

Sample ID: 2020-HAML-000348-001	Drawn By: Client
Vessel: Natural Resources Canada	Date Taken: 05-Mar-2020
Sample Representing: Sample ID: 20087-01	Date Submitted: 05-Mar-2020
Sample Designated As: 10 vol% L-diesel in certified diesel	Date Tested: 05-Mar-2020
Job Location: Hamilton, ON Canada, Stoney Creek, Ontario, Canada	

Method	Property	Result	Units
ASTM D2709	Sediment and Water	< 0.01	Vol %
ASTM D130	Copper Corrosion @ 50°C (122°F)/3 hr	1a	
Seal Condition	: Good (Seal Intact)		

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The sample was submitted for testing purposes. Intertek cannot accept liability for the representativeness of the sample.

Signed: _____

On behalf of Abraham Kim, Laboratory Supervisor

Iftikhar Chughtai,
Laboratory Manager

Date: 05-Mar-2020



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2020-03-10

Page 1 of 1

Client Name:	PEC J. Monnier	Group Number:	20087
Project Code:	P-003013.001	Job Number:	
Due Date:	2020-03-10	Submitted by:	Jacques Monnier
Project Title:	Bioenergy - Conversion of Lignin-Derived F	Index Date:	2020-02-25
Project Info:		Approved by:	Jillian_Gawlik

SampleNumber: 20087-01

CustomerSampleNumber: 10 vol% L-diesel in certified diesel

Submission Date: 2020-02-25

Comment:

Test Results

Parameter	Method		
Contract_fee	in-house	Done	
Flash Point, Pensky-Marten Closed Cu	ASTM D93	68.0	°C
Kinematic viscosity @ 40°C	ASTM D445	2.641	CST
SIMDIS_2887EXT	ASTM D2887	Done	
Sulphur	ASTM D7039	6.4	mg/kg

Test Comments:

CONTRACT_fee Contract_fee in-house
Copper Corrosion & Sediment & Water results sent to client 2020-03-06



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2020-02-19

Page 1 of 1

Client Name:	PEC J. Monnier	Group Number:	20070
Project Code:	P-003013.001	Job Number:	
Due Date:	2020-02-26	Submitted by:	Jacques Monnier
Project Title:	Bioenergy - Conversion of Lignin-Derived F	Index Date:	2020-02-12
Project Info:		Approved by:	Jillian_Gawlik

SampleNumber: 20070-01

CustomerSampleNumber: 10 vol% methanol-washed diesel in certifie

Submission Date: 2020-02-12

Comment:

Test Results

Parameter	Method		
Total Acid Number	ASTM D664	<0.05	mg KO

APPENDIX C - Elemental analysis reports of oil products and heavy middle distillates (250-345 °C)



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2021-05-06

Page 1 of 6

Client Name:	PEC J. Monnier	Group Number:	21024
Project Code:	P-004034.001	Generated by:	Nada_Khalil
Index Date:	2021-01-27	Verification by:	SantAnna, Silvia
Project Title:	Bioenergy - Conversion of Lignin-Derived F		
Submitted by:	Christine MacFarlane		

SampleNumber: 21024-01
CustomerSampleNumber: HDL20210120-T
Submission Date: 2021-01-27
Comment:

Elemental Results

Parameter	Method		
Carbon	ASTM D5291 modified	87.1	wt%
Hydrogen	ASTM D5291 modified	13.5	wt%
Nitrogen	ASTM D5291 modified	<0.15	wt%
Elementar Oxygen	in-house	0.62	wt%

Physical Results

Parameter	Method		
Density @ 15°C	ASTM D4052	873.7	kg/m3

Chemical Results

Parameter	Method		
Water Content by Karl Fisher	ASTM D6304	20.0	mg/kg



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2021-02-24

Page 2 of 3

Client Name:	PEC J. Monnier	Group Number:	21028
Project Code:	P-004034.001-NBB1	Generated by:	Nada_Khalil
Index Date:	2021-02-03	Verification by:	
Project Title:	Bioenergy - Conversion of Lignin-Derived F		
Submitted by:	Christine MacFarlane		

SampleNumber: 21028-02
 CustomerSampleNumber: HDL20210120-20-22-T Blend 250-345
 Submission Date: 2021-02-03
 Comment:

Elemental Results

Parameter	Method		
Carbon	ASTM D5291 modified	86.7	wt%
Hydrogen	ASTM D5291 modified	13.1	wt%
Nitrogen	ASTM D5291 modified	<0.15	wt%
Sulfur	ASTM D5453	1.4	mg/kg
Total Nitrogen	ASTM D5762	<1	mg/kg
Elementar Oxygen	in-house	0.51	wt%

Chromatographic Results

Parameter	Method
SIMDIS_2887EXT	ASTM D2887



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2021-04-13

Page 1 of 1

Client Name: PEC J. Monnier
 Project Code: P-004034.001-NBA1
 Index Date: 2021-01-12
 Project Title: Bioenergy - Production of Fungible Biofuel
 Submitted by: Christine MacFarlane

Group Number: 21009
 Generated by: Nada_Khalil
 Verification by: Corbett, Huena
 Digitally signed by Corbett, Huena
 Date: 2021.04.13 07:55:38 -0400

SampleNumber: 21009-01
 CustomerSampleNumber: HDL20201216-18-T Blend 250+q
 Submission Date: 2021-01-12
 Comment:

Elemental Results

Parameter	Method		
Carbon	ASTM D5291 modified	87.6	wt%
Hydrogen	ASTM D5291 modified	12.6	wt%
Nitrogen	ASTM D5291 modified	<0.15	wt%
Sulfur	ASTM D5453	15	mg/kg
Total Nitrogen	ASTM D5762	1.3	mg/kg
Elementar Oxygen	in-house	0.62	wt%

Physical Results

Parameter	Method		
Density @ 15°C	ASTM D4052	908.9	kg/m3

Chemical Results

Parameter	Method		
Water Content by Karl Fisher	ASTM D6304	13.0	mg/kg

APPENDIX D Characteristics reports of 20 vol%, 30vol%, and 40 vol% L-diesel blends



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2021-03-30

Page 1 of 3

Client Name:	PEC J. Monnier	Group Number:	21049
Project Code:	P-003013.001-NBA1	Generated by:	Nada_Khalil
Index Date:	2021-02-22	Verification by:	Corbett, Huena
Project Title:	TC Lignin-derived diesel fuel for rail (Trans		<small>Digitally signed by Corbett, Huena Date: 2021.03.31 08:57:54 -04'00'</small>
Submitted by:	Christine MacFarlane		

SampleNumber: 21049-01
 CustomerSampleNumber: 20% DB HDL20210120-20-22 250-345
 Submission Date: 2021-02-22
 Comment:

Physical Results

Parameter	Method		
Kinematic viscosity @ 40°C	ASTM D445	2.928	CST

Chemical Results

Parameter	Method		
Total Acid Number	ASTM D664	<0.1	mg KO

Test Comments:

TAN Total Acid Number ASTM D664
 Result <0.05.



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2021-03-30

Page 2 of 3

Client Name:	PEC J. Monnier	Group Number:	21049
Project Code:	P-003013.001-NBA1	Generated by:	Nada_Khalil
Index Date:	2021-02-22	Verification by:	
Project Title:	TC Lignin-derived diesel fuel for rail (Trans		
Submitted by:	Christine MacFarlane		

SampleNumber: 21049-02
 CustomerSampleNumber: 30% DB HDL20210120-20-22 250-345
 Submission Date: 2021-02-22
 Comment:

Physical Results

Parameter	Method		
Kinematic viscosity @ 40°C	ASTM D445	3.116	CST

Chemical Results

Parameter	Method		
Total Acid Number	ASTM D664	<0.1	mg KO

Test Comments:

TAN Total Acid Number ASTM D664
 Result <0.05.



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2021-03-30

Page 3 of 3

Client Name:	PEC J. Monnier	Group Number:	21049
Project Code:	P-003013.001-NBA1	Generated by:	Nada_Khalil
Index Date:	2021-02-22	Verification by:	
Project Title:	TC Lignin-derived diesel fuel for rail (Trans		
Submitted by:	Christine MacFarlane		

SampleNumber: 21049-03

CustomerSampleNumber: 40% DB HDL20210120-20-22 250-345

Submission Date: 2021-02-22

Comment:

Physical Results

Parameter	Method		
Kinematic viscosity @ 40°C	ASTM D445	3.354	CST

Chemical Results

Parameter	Method		
Total Acid Number	ASTM D664	<0.1	mg KO

Test Comments:

TAN Total Acid Number ASTM D664
 Result <0.05.

APPENDIX E Analysis report of 100% L-diesel (250-345 °C)



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2021-07-05

Page 1 of 1

Client Name:	PEC J. Monnier	Group Number:	21144
Project Code:	P-003081.001-NBL1	Generated by:	Jessica_Kennedy
Index Date:	2021-06-15	Verification by:	SantAnna, Silvia
Project Title:	Bioenergy - Enerkem		
Submitted by:	Christine MacFarlane		

Digitaly signed by SantAnna,
 Silvia
 Date: 2021.07.05 10:46:42 -0400

SampleNumber: 21144-01
 CustomerSampleNumber: HDL20210120-20-22-T (250-345)
 Submission Date: 2021-06-15

Comment:

Physical Results

Parameter	Method		
Kinematic viscosity @ 40°C	ASTM D445	5.530	CST

Chemical Results

Parameter	Method		
Total Acid Number	ASTM D664	<0.1	mg KO

Test Comments:

TAN Total Acid Number ASTM D664
 Result was less than 0.05 mg KOH/g.

APPENDIX F Analysis Report of 100% L-diesel (155-345 °C) Part 1



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2022-02-17
 Page 1 of 2

Client Name:	PEC J. Monnier	Group Number:	22014
Project Code:	P-003013.001-NBL1	Generated by:	Katrina_Campbell
Index Date:	2022-01-28	Verification by:	Bulsink, Philip
Project Title:	TC Lignin-derived diesel fuel for rail		Digitally signed by Bulsink, Philip
Submitted by:	Christine MacFarlane		Date: 2022.02.17 13:14:15 -0500
SampleNumber:	22014-01		
CustomerSampleNumber:	HDL20211214 Blend 1+2 (155-310)		
Submission Date:	2022-01-28		
Comment:			

Physical Results

Parameter	Method		
Kinematic viscosity @ 40°C	ASTM D445	2.174	CST
Flash Point, Pensky-Marten Closed Cu	ASTM D93	61.5	°C
Ash	ASTM D482	<0.010	wt%

Chemical Results

Parameter	Method		
Total Acid Number	ASTM D664	<0.1	mg KO

Contracted Analysis

Parameter	Method	
Samples Sent	N/A	Done
Results Received	N/A	Done

Test Comments:

ContractingSent Samples Sent N/A
 Sample sent to Intertek 2022-02-08

ContractingReceived Results Received N/A
 Results received from Intertek 2022-02-10

APPENDIX G Analysis Report of 100% L-diesel (155-345 °C) Part 2



Report of Analysis

Natural Resources Canada
Jillian Gawlik
1 Haanel Drive
Ottawa, ON K1A1M1
Canada

Our Reference Number: CA120-0027245
Lab Reference Number: 2022-HAML-000170
Customer Reference Number: PO#3000671561

Sample ID: 2022-HAML-000170-002	Drawn By: Client
Vessel: Natural Resources Canada	Date Taken: 08-Feb-2022
Sample Representing: Sample ID:22014-01	Date Submitted: 10-Feb-2022
Sample Designated As: Diesel	Date Tested: 10-Feb-2022
Job Location: Hamilton, ON Canada, Stoney Creek, Ontario, Canada	

Method	Property	Result	Units
ASTM D86	Initial Boiling Point	173.7	°C
	5% Recovery	189.7	°C
	10% Recovery	194.5	°C
	20% Recovery	203.3	°C
	30% Recovery	212.5	°C
	40% Recovery	223.0	°C
	50% Recovery	233.4	°C
	60% Recovery	245.2	°C
	70% Recovery	255.1	°C
	80% Recovery	266.3	°C
	90% Recovery	278.6	°C
	95% Recovery	289.0	°C
	Final Boiling Point	298.0	°C
	Recovery	98.3	Vol %
	Residue	1.3	Vol %
	Total Recovery	99.6	Vol %
	Loss	0.4	Vol %
	Corrected Loss	0.4	Vol %
	Corrected Recovery	98.3	Vol %
	Corrected Total Recovery	99.6	Vol %
ASTM D4530	Micro Method Carbon Residue 10% Distillation Residue	<0.1	%(m/m)
ASTM D2709	Sediment and Water	0.01	Vol %
ASTM D130	Copper Corrosion @ 50°C (122°F)/3 hr	1b	
ASTM D2624	Temperature	21.7	°C
	Electrical Conductivity	1	pS/m
Seal Condition	: Good (Seal Intact)		



AT-2072.14

710 South Service Rd., Unit 1, Stoney Creek Ontario, Canada L8E 5S7
Tel.: +1 (905) 529-0090 Fax.: +1 (905) 529-5999 Email: HamLab@intertek.com

Report Generated: 10-Feb-2022 6:22 pm

Page 1 of 2



Report of Analysis

Our Reference Number: CA120-0027245
Lab Reference Number: 2022-HAML-000170
Customer Reference Number: PO#3000671561

f - Denotes analysis results which are ISO/IEC 17025 accredited by ANSI National Accreditation Board.

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The sample was submitted for testing purposes. Intertek cannot accept liability for the representativeness of the sample.

Signed By: Syed
On behalf of Syed Gardezi, Laboratory Technician
Iftikhar Chughtai,
Laboratory Manager

Date: February 10, 2022



710 South Service Rd., Unit 1, Stoney Creek Ontario, Canada L8E 5S7
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APPENDIX H Analysis Report of 100% L-diesel (155-345 °C) Part 3



ISO 9001:2015
ABS-QE Certificate # 30701

Report of Analysis

Canmet Energy
Jessica Kennedy
Natural Resources
1 Haanel Drive
Ottawa

Our Reference Number: CA170-0011506
Lab Reference Number: 2022-SARN-000063
Customer Reference Number: NA

Customer Product: ULSD	Sample ID: 2022-SARN-000063-001
CL ID: HDL20211214 Blend1+2 (155-310)A	Date Sampled: 03-Feb-2022
Sample ID: 22014-01A	Date Submitted: 09-Feb-2022
Submitted By: Client	Date Tested: 10-Jan-2022

Method	Property	Result	Units	Min	Max	P/F
ASTM D7039	Sulfur Content	0.6	mg/kg			

Material conforms to the requirements on above relevant point(s) of testing

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Signed By:


Roger Archambault, Laboratory Manager

Date: February 10, 2022

APPENDIX I Analysis Report of 100% L-diesel (155-345 °C) Part 4



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2022-03-09

Page 1 of 1

Client Name:	PEC J. Monnier	Group Number:	22033
Project Code:	P-004034.001.NBL1	Generated by:	Katrina_Campbell
Index Date:	2022-02-23	Verification by:	Bulsink, Philip
Project Title:	Biofuels from lignin-derived feedstocks		Digitally signed by Bulsink, Philip Date: 2022.03.09 12:10:54 -0500
Submitted by:	Christine MacFarlane		

SampleNumber: 22033-01
 CustomerSampleNumber: HDL20211214 Blend 1+2 (155-310)
 Submission Date: 2022-02-23
 Comment:

Elemental Results

Parameter	Method		
Carbon	ASTM D5291 modified	86.7	wt%
Hydrogen	ASTM D5291 modified	13.2	wt%
Nitrogen	ASTM D5291 modified	<0.15	wt%
Elementar Oxygen	In-house	0.28	wt%

Physical Results

Parameter	Method		
Density @ 15°C	ASTM D4052	866.6	kg/m ³
Specific Gravity 60/60F	ASTM D4052	0.8674	GRAV

Calculated Results

Parameter	Method	
Cetane Index-D976	ASTM	33.3
Cetane Index-D4737 A	ASTM	32.7
Cetane Index-D4737 B	ASTM	30.2

Test Comments:

CI_CALC Cetane Index-D976 ASTM
 Cetane Index calculated using client supplied D86 results.

APPENDIX J Cetane index of 20% and 30% L-diesel blends



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2022-04-11

Page 1 of 2

Client Name:	PEC J. Monnier	Group Number:	22061
Project Code:	P-004034.001	Generated by:	Katrina_Campbell
Index Date:	2022-03-31	Verification by:	Bulsink, Philip
Project Title:	Bioenergy - Conversion of Lignin-Derived F		Digitally signed by Bulsink, Philip
Submitted by:	Yi Zhang		Date: 2022.04.11 15:01:46 -04'00'

SampleNumber: 22061-01
 CustomerSampleNumber: 20% DB HDL20211214 Blend 1 + 2 (155-3)
 Submission Date: 2022-03-31
 Comment:

Physical Results

Parameter	Method		
Density @ 15°C	ASTM D4052	859.3	kg/m3
Specific Gravity 60/60F	ASTM D4052	0.8608	GRAV

Chromatographic Results

Parameter	Method	
SIMDIS_2887EXT	ASTM D2887	Done

Calculated Results

Parameter	Method	
Cetane Index-D976	ASTM	41.3
Cetane Index-D4737 A	ASTM	40.2
Cetane Index-D4737 B	ASTM	38.6

Test Comments:

CI_CALC Cetane Index-D976 ASTM
 Cetane Index calculated using D2887 correlation to D86 distillation points.



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2022-04-11
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Client Name:	PEC J. Monnier	Group Number:	22061
Project Code:	P-004034.001	Generated by:	Katrina_Campbell
Index Date:	2022-03-31	Verification by:	
Project Title:	Bioenergy - Conversion of Lignin-Derived F		
Submitted by:	Yi Zhang		

SampleNumber: 22061-02
 CustomerSampleNumber: 30% DB HDL20211214 Blend 1 + 2 (155-3)
 Submission Date: 2022-03-31
 Comment:

Physical Results

Parameter	Method		
Density @ 15°C	ASTM D4052	860.3	kg/m3
Specific Gravity 60/60F	ASTM D4052	0.8619	GRAV

Chromatographic Results

Parameter	Method	
SIMDIS_2887EXT	ASTM D2887	Done

Calculated Results

Parameter	Method	
Cetane Index-D976	ASTM	40.0
Cetane Index-D4737 A	ASTM	39.0
Cetane Index-D4737 B	ASTM	37.2

Test Comments:

CI_CALC Cetane Index-D976 ASTM
 Cetane Index calculated using D2887 correlation to D86 distillation points.

APPENDIX K Derived cetane number report for 100% L-diesel (155-345 °C)

Canmet Energy**Derived Cetane Number (DCN) Report Form**

for Sample: 22055-01C
CFR # 220622-1

Derived Cetane Number: **39.14**
Date Tested: 22-Jun-22
Location: CFR
Machine Used: IQT-LAH-AJD-LM
Operator: OR

Summary of D6890-18 Constant Volume Combustion Chamber Test Data

DCN (ASTM D6890-18)	39.14
Ignition Delay, ms	5.380
Charge air O ₂ content, vol.-%	20.9
Charge air temperature, °C	544.0

Quality Control Testing

QC Sample Tested Prior to Fuel Sample Test

Lot No. CF20
DCN ARV 49.2
DCN (test result) 49.4



CFR Engines Canada ULC
17 Fitzgerald Rd., Suite 102
Ottawa, Ontario
K2H 9G1

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