Quantification of the Cold Flow Properties of Biodiesels Blended with ULSD

Donald A. Heck*, Jordan Thaeler[§], Steve Howell[§] and Joshua A. Hayes*

***Iowa Central Community College**

[§]National Biodiesel Board

Michigan Soybean and Promotion Council

Iowa Biodiesel Board

ABSTRACT

Cloud point, cold filter plugging point (CFPP) and low temperature flow test (LTFT) methods were used to assess the cold-flow properties for seven different biodiesel fuels blended with four different ULSD fuels representing the span of the market in 2009. The pour point of the neat biodiesel and B50 blends were also analyzed. This intent of this project was to provide the industry with an independently generated set of cold flow information on a variety of fuels in the market today with the new Ultra Low Sulfur Diesel Fuel (ULSD).

The neat fuels were tested in addition to biodiesel blends with ratios of 2% biodiesel (B2), 5% biodiesel (B5), 11% biodiesel (B11), 20% biodiesel (B20) and 50% biodiesel (B50) for cloud point, CFPP, and LTFT. Pour point was tested for the neat biodiesels and B50 blends only.

The petrodiesel and biodiesel collected were actual market fuels and were targeted to encompass the range of fuels available in the market in 2009. Three petrodiesel fuels with cloud points of -53.5F, +3.2F, and +12.2 F were used to produce petrodiesel having target cloud points of -40F, -30F, -15F, and +10F. Seven biodiesel (B100) samples were selected. Three B100's of low cloud point (i.e. vegetable oil base, in this case soybean oil) from various manufacturing processes (distilled biodiesel, non distilled biodiesel from hexane extracted oil, non distilled biodiesel from extruder-expeller oil) were collected from commercial biodiesel producers. Four additional biodiesels of mid to mid-high to high cloud points from commercial biodiesel producers representing mixed saturation levels (in this case blends of vegetable, animal, and recycled oil based biodiesel). The cloud point values of the various B100 samples were +27.5F, +28.4F, +29.3F, +33.8F, + 44.6F, +46.4F, and +53.6F.

The reader is cautioned not to associate the individual cloud point values of the fuels used in this study as any sort of 'average' or 'accepted' value for those fuels. Actual values for #1 or #2 fuels or their blends, or actual values for biodiesel made by various processes and oils or their blends, will vary in the market. The user is advised to utilize actual test data on the fuels they plan to use rather than assume some sort of generic value.

METHODS and MATERIALS

Fuel Procurement:

The study utilized four different ULSD fuels with cloud point temperatures of -40°F, -30°F, -15°F and +10°F. The four blends were created from three different ULSD fuels sent in bulk shipments of 6, 55-gallon drums of #1 ULSD with a reported cloud point temperature of -49.9°F; 6, 55-gallon drums of #2 ULSD with a reported cloud point temperature of +4.1°F; and 4, 55-gallon drums of #2 ULSD with a reported cloud point temperature of +17.6°F. Cloud points for all three bulk fuels were determined in-house using a Lawler model OL-14/DR-2L automated cloud and plug point bath and were found to be -53.5°F, +3.2°F and +12.2°F; respectively (Table 1, average of 2 or more results). All fuel samples were blanketed with nitrogen upon receipt and every time the fuel container was opened thereafter. Before obtaining an aliquot of fuel, each barrel was re-circulated for 10 minutes using a standard electric fuel

pump to assure homogeneity. Stock ULSD fuel blends of -40°F, -30°F and -15°F cloud points were created by blending appropriate amounts of the #1 ULSD and #2 ULSD fuels from the original shipment as determined by constructing a blend curve using #2 ULSD with increasing amounts of #1 ULSD (see below).

Table 1. Fuel properties for ULSD stocks. The reported values were taken from the certificates of analysis supplied with each fuel and are assumed to be from a single reading. The tested cloud points were obtained in-house and represent the average of 2 independent determinations. The standard deviation of in-house measurements was less than 2°F in all cases.

Fuel	Reported Cloud (°F)	Tested Cloud (°F)	Density (g/mL)
#1 ULSD	-49.9	-53.5	0.8039
#2 ULSD	+4.1	+3.2	0.8394
High Cloud #2	+17.6	+12.2	0.8296

The study utilized seven different biodiesel stocks. Three B100's of low cloud point (i.e. vegetable oil base, in this case soybean oil) from various manufacturing processes (distilled biodiesel, non distilled biodiesel from hexane extracted oil, non distilled biodiesel from extruder-expeller oil) were collected from commercial biodiesel producers. Four additional biodiesels of mid to mid-high to high cloud points from commercial biodiesel producers representing mixed saturation levels (in this case blends of vegetable, animal, and recycled oil based biodiesel). The cloud point values of the various B100 samples were +27.5°F, +28.4°F, +29.3°F, +33.8°F, + 44.6°F, +46.4°F, and +53.6°F.

Table 2. Fuel properties for biodiesel stocks. The reported values were taken from the certificates of analysis supplied with each fuel and are assumed to be from a single reading. The tested cloud points were obtained in-house and represent the average of 2 or 3 determinations. The standard deviation of in-house measurements was less than 2°F in all cases. NA = not available.

Biodiesel	Reported Cloud Point (°F)	Tested Cloud Point (°F)	*n*	Density (g/mL)
Low Cloud, Non-distilled	NA	+27.5	2	0.8792
Low Cloud, Extruded	+32.0	+28.4	2	0.8793
Low Cloud, Distilled	+32.0	+29.3	2	0.8775
Mid Cloud	+53.6	+33.8	2	0.8783
Mid-High Cloud A	NA	+44.6	2	0.8711
Mid-High Cloud B	+50.0	+46.4	2	0.8699
High Cloud	+57.2	+53.6	3	0.8686

All fuel samples were blanketed with nitrogen upon receipt and every time the fuel container was opened thereafter. The reported cloud points, along with in-house measurements are recorded in Table 2.

Fuel Blending:

A blend curve was constructed to determine the appropriate blend ratio of #1 and #2 ULSD for the -40°F, -30°F and -15°F stock ULSD fuels (Figure 1). Based on this curve, the appropriate blend ratio for the -40°F stock fuel was 90% #1 ULSD with 10% #2 ULSD, the -30°F stock fuel was 80% #1 and 20% #2, and the -15°F stock fuel was 57% #1 and 43% #2 (Table 3). For the +10°F cloud point ULSD a blend of 10% #2 and 90% "high cloud" ULSD was found to give the appropriate ratio; a full blend curve was not generated for this stock fuel.

Table 3. Blend ratios for stock ULSD fuels.

Fuel	% Blend for -40°F	% Blend for -30°F	% Blend for -15°F	% Blend for +10°F
#1 ULSD	90%	80%	57%	--
#2 ULSD	10%	20%	43%	10%
High Cloud #2	--	--	--	90%

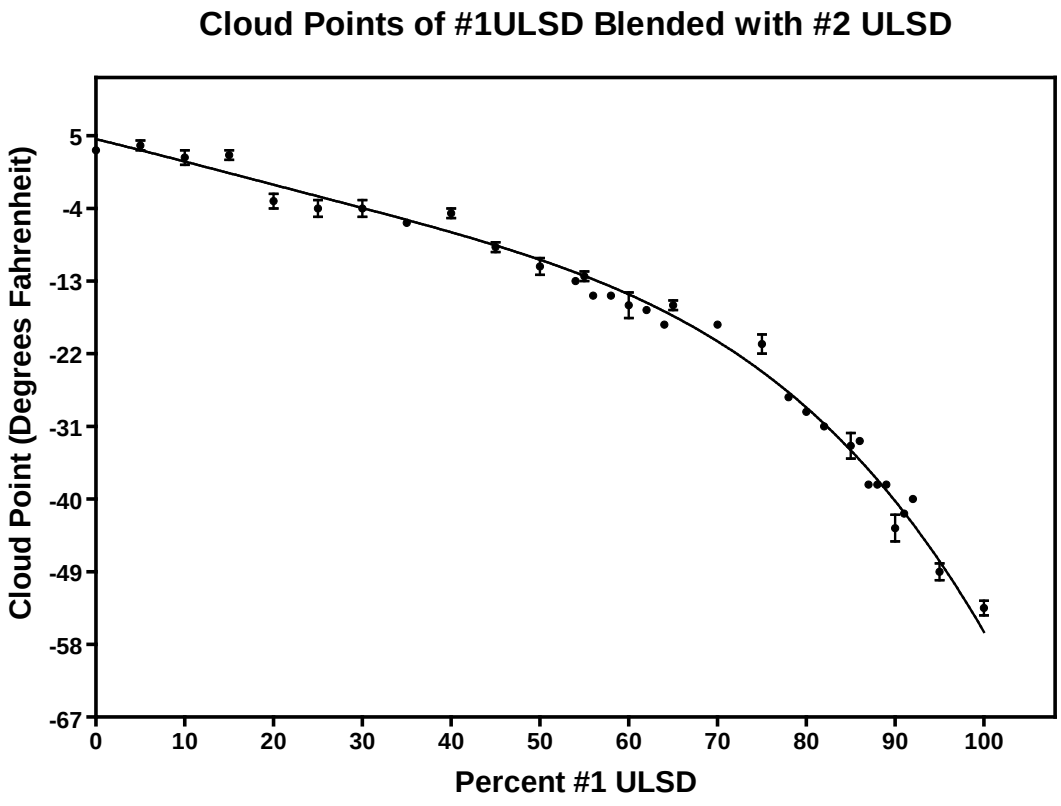


Figure 1. Blend curve for ULSD fuels. #1 ULSD was blended with #2 ULSD in 2 or 5 degree increments. Error bars represent the average ± SD of 2 to 4 determinations.

The -40°F, -30°F, -15°F, and +10°F ULSD blends were made in approximately 20 L aliquots. The density of the #1 and #2 ULSD stock fuels was taken by ASTM D 4052 and used to determine the appropriate mass of each fuel for each blend with a mass precision of 0.1g. The blended ULSD fuels were then placed into approved 20 L containers and blanketed with nitrogen initially and again every time after use.

The biodiesel blends were filtered through Whatman filters and prepared fresh daily in 92 mL aliquots for the cloud point and CFPP tests, or 142 mL aliquots when doing the cloud point, CFPP and pour point tests. The density of the ULSD fuel and biodiesel fuel was taken and used to determine the appropriate mass of each fuel for each blend to the nearest 0.0001g. Mixing was assured by pouring the blend back and forth several times between two containers. The blend was then poured into the appropriate test jars and used immediately. The biodiesel blends used for the LTFT method were prepared in 800 mL aliquots in the same manner but using mass precision to 0.1g. These blends were stored in 1 L amber glass bottles and typically used within a day or two.

Instrument methods and accuracy:

Cloud point and CFPP determinations were performed according to ASTM D 2500 and D 6371, respectively, using a Lawler model OL-14/DR-2L automated cloud and plug point bath (Lawler Manufacturing Corporation, Edison, New Jersey). Briefly, fuel samples were poured into a test jar up to the line indicating sample height (approximately 45 mL). For cloud point determinations, the samples were cooled to approximately 18 degrees F above the anticipated cloud point and detection of the cloud was performed by optical reflection. Sample cooling was continued and the procedure was repeated at every 1.8 degree Fahrenheit increment until the detection of a cloud. Cloud point determinations were done manually with the High Cloud biodiesel product because of interference with the optical sensor due to the formation of crystalline structures.

For CFPP determinations, samples were cooled to approximately 9 degrees F above the anticipated plugging point and drawn into a pipette under controlled vacuum through a standardized wire mesh filter. Sample cooling was continued and the test procedure repeated until the sample could no longer fill the pipette in 60 seconds.

Pour point determinations were performed according to ASTM D 97 using a Koehler model K46100 manual refrigerated bath (Koehler Instrument Company, Bohemia, New York). Briefly, fuel samples were poured into a test jar up to the line indicating sample height (approximately 45 mL). For samples with an expected pour point temperature above -27.4°F, the samples were heated in a water bath to +113°F and then placed into the refrigerated bath. Beginning at 48.2°F above the expected pour point, and at every 5.4°F below that, the samples were tested for pour point by briefly removing the sample from the bath, tilting to the side, and observing any movement of sample. The test was repeated until no movement of sample was observed upon holding the sample horizontally for 5 seconds.

LTFT determinations were performed according to ASTM D 4539 using a Lawler model 265-12L refrigerated bath with accessories. Briefly, fuel samples in 200 mL aliquots were filtered through dry, lintless filter paper and placed into a refrigerated bath at a temperature no less than 9°F above the cloud point of the sample. The sample temperature was lowered at a rate of +1.8°F per hour to the desired test

temperature at which time the sample was filtered through the apparatus. The minimum LTFT pass temperature was determined as outlined in ASTM D4539 section 8.14 where subsequent aliquots of the same sample were tested repeatedly at incrementally lower temperatures until a failing result was obtained. The reported value represents the lowest passing temperature before a failed test was obtained.

Commercial reference standards were used to determine instrument accuracy for the cloud point, pour point and CFPP methods. The cloud point reference standards were purchased from Koehler Instrument Company (Houston, Texas) and have a reported analytical uncertainty of ± 3.6°F. The CFPP and pour point reference standards were purchased from Spectrum Quality Standards (Sugarland, Texas) and have reported analytical uncertainties of ± 2.7°F and ± 5.4°F, respectively. A reference standard for the LTFT method was not available. In all cases, the reported average result for each test is within two degrees F of the stated reference value with a standard deviation less than one (Table 4).

Table 4. Instrument accuracy data for the cloud point, CFPP and pour point methods.

	Reference Value (°F)	Result (°F)	*n*
Cloud Point			
	+39.2°F	+39.2 ± 0	4
	+10.4°F	+9.5 ± 1.1	4
	-5.8°F	-7.6 ± 0	4
CFPP			
	+1.4°F	+3.2 ± 0	2
	-25.6°F	-23.8 ± 0	2
Pour Point			
	21.2°F	+21.2 ± 0	2
	5°F	+5 ± 0	2

RESULTS

Figure 2. Cloud point temperatures.

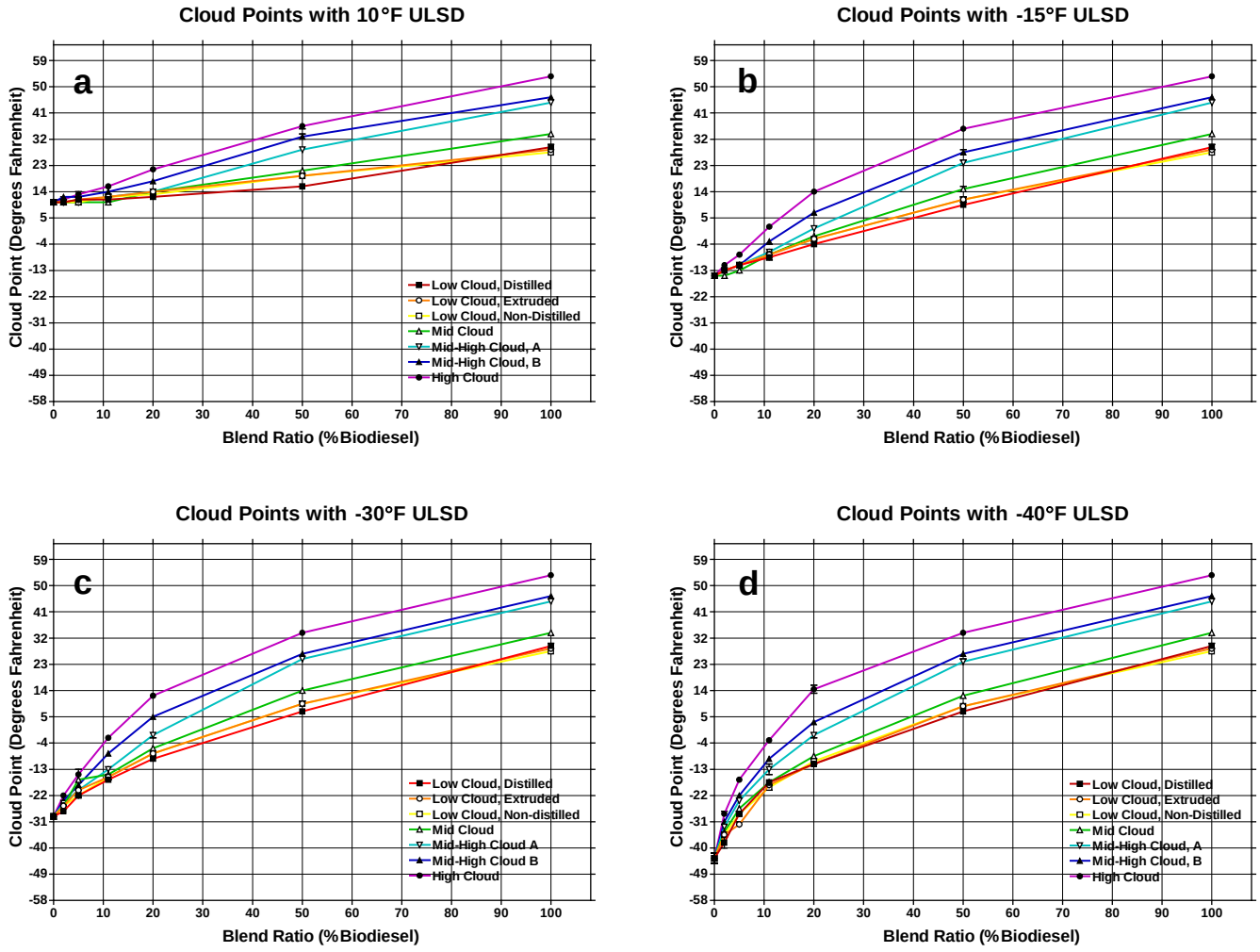


Figure 3. CFPP temperatures.

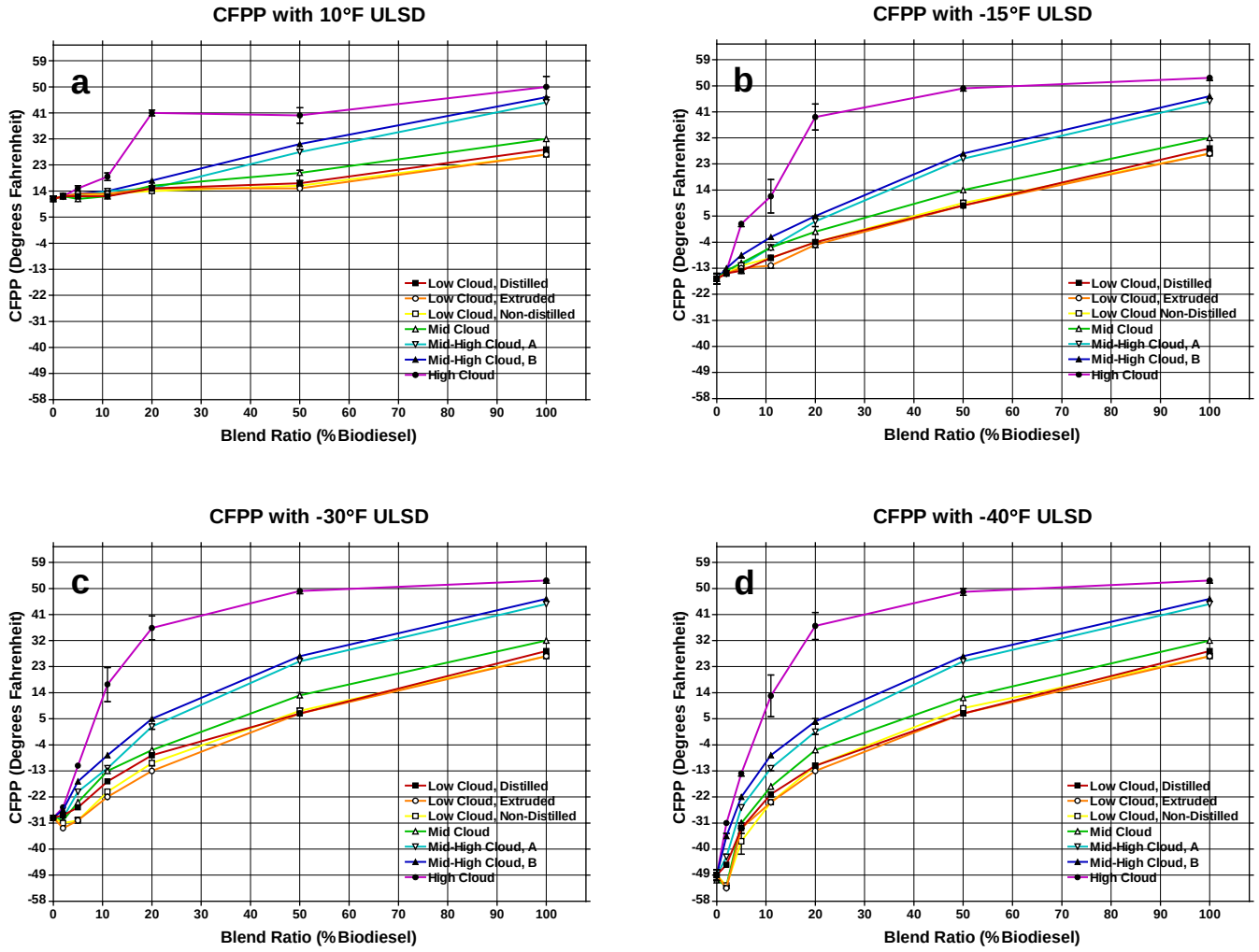


Figure 4. LTFT temperatures.

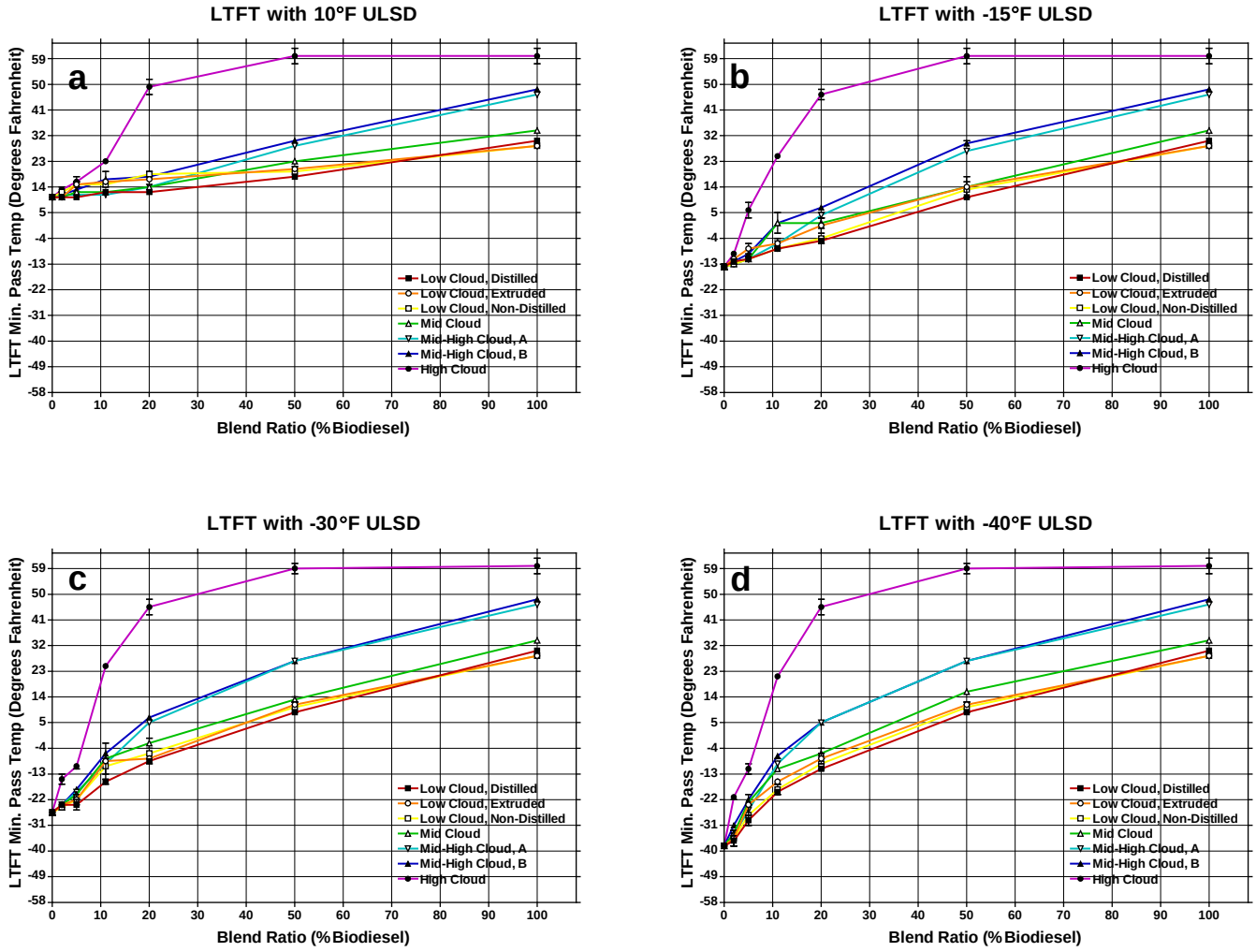


Figure 5. Pour points of neat ULSD and biodiesel fuels.

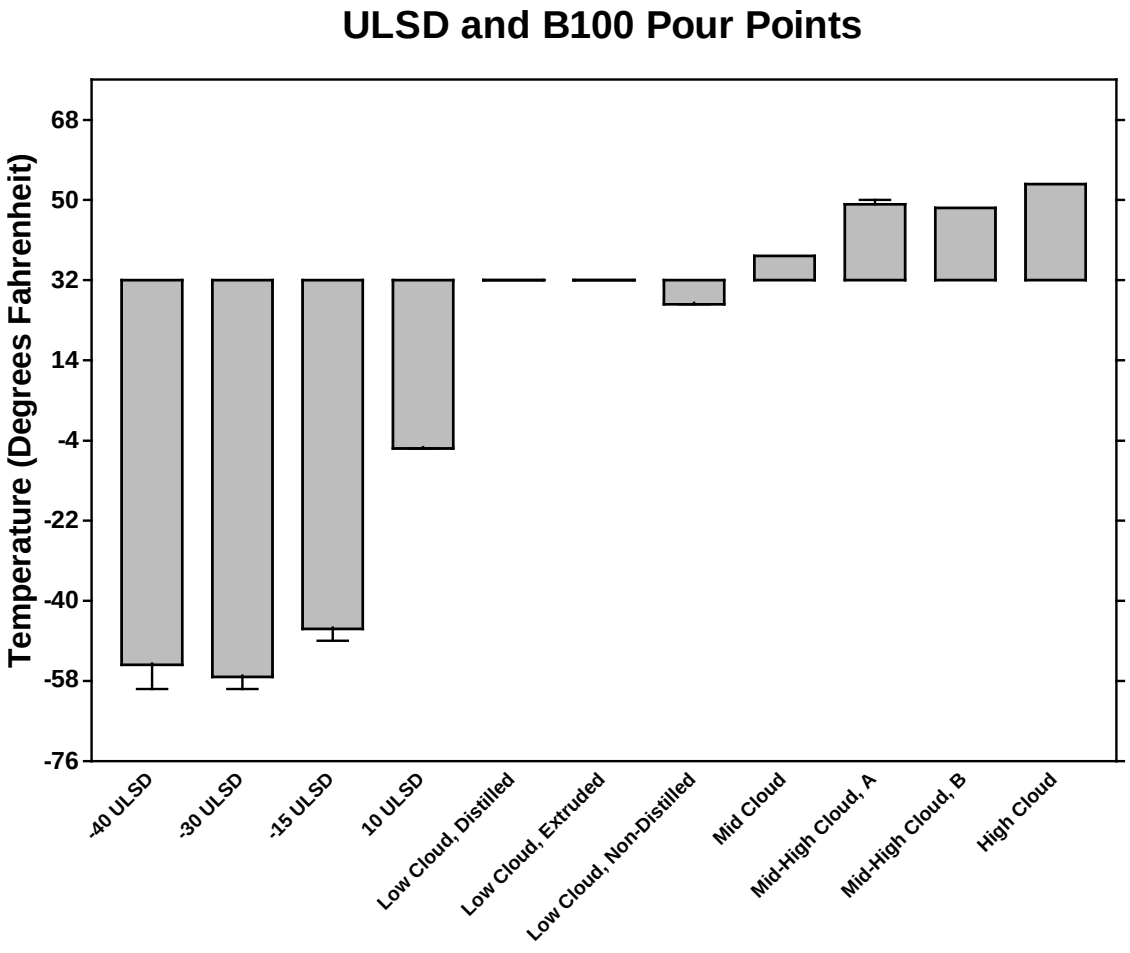


Figure 6. Pour points of biodiesel B50 blends.

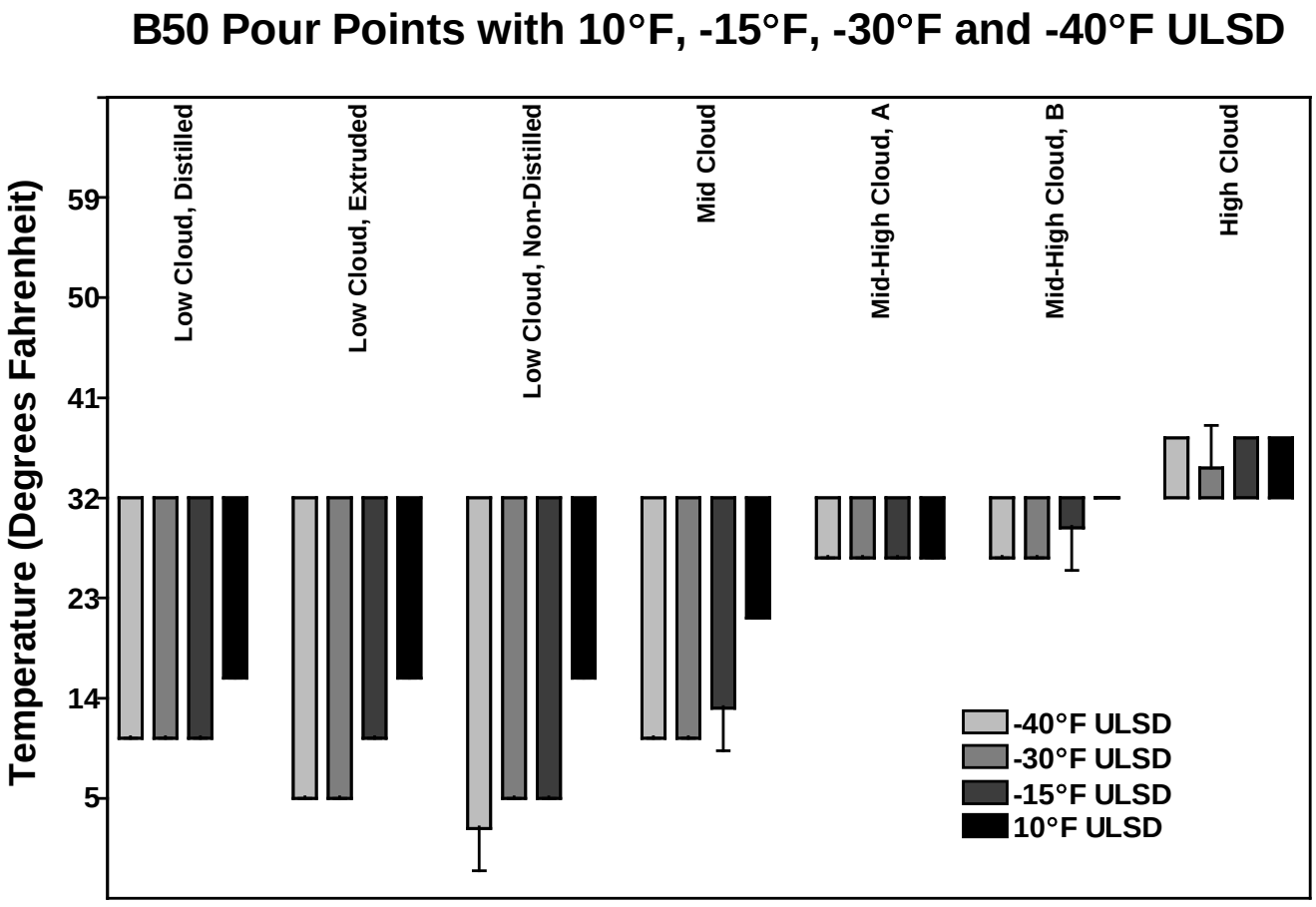


Figure 7. Methods comparison for Low Cloud, Distilled product.

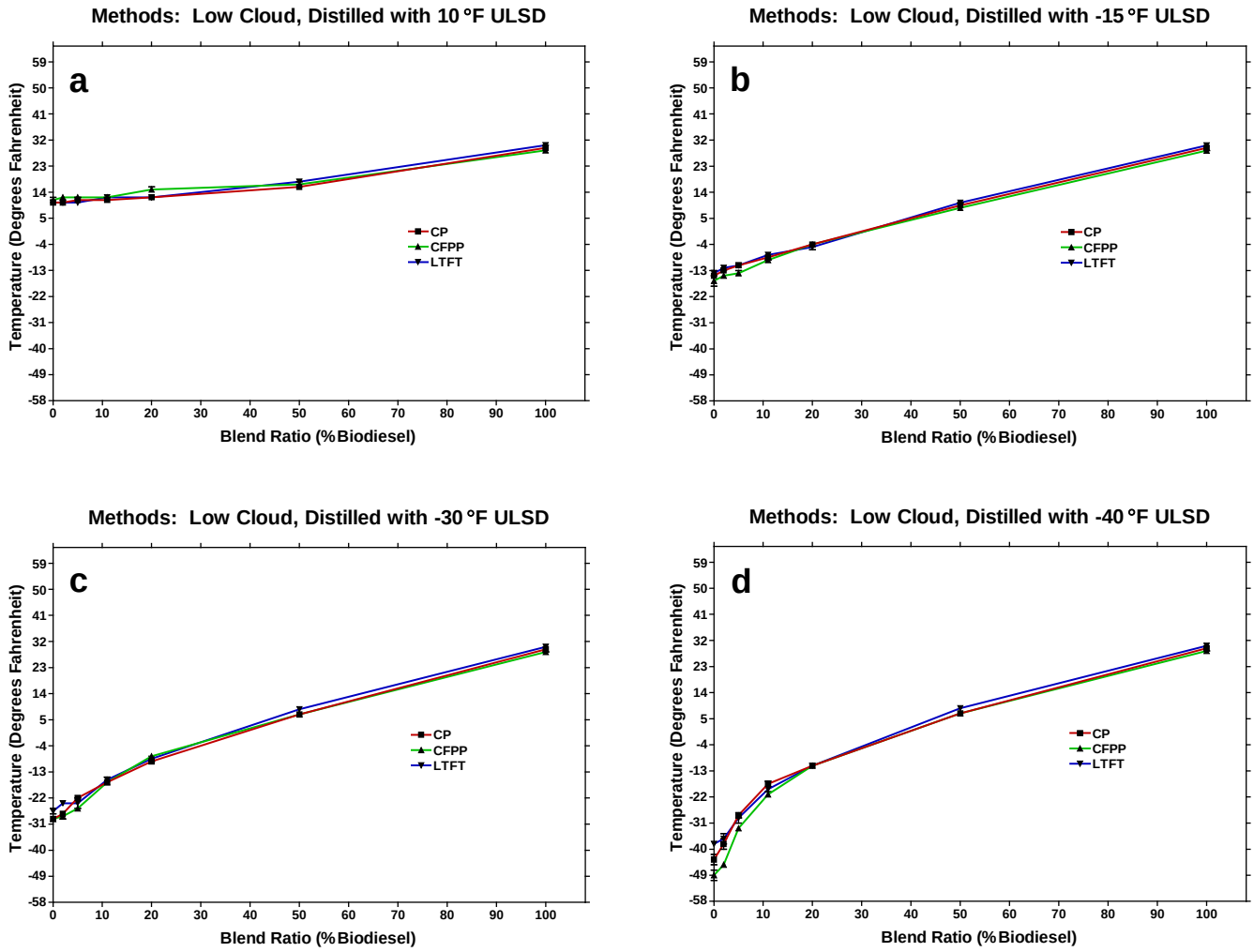


Figure 8. Methods comparison for Low Cloud, Extruded product.

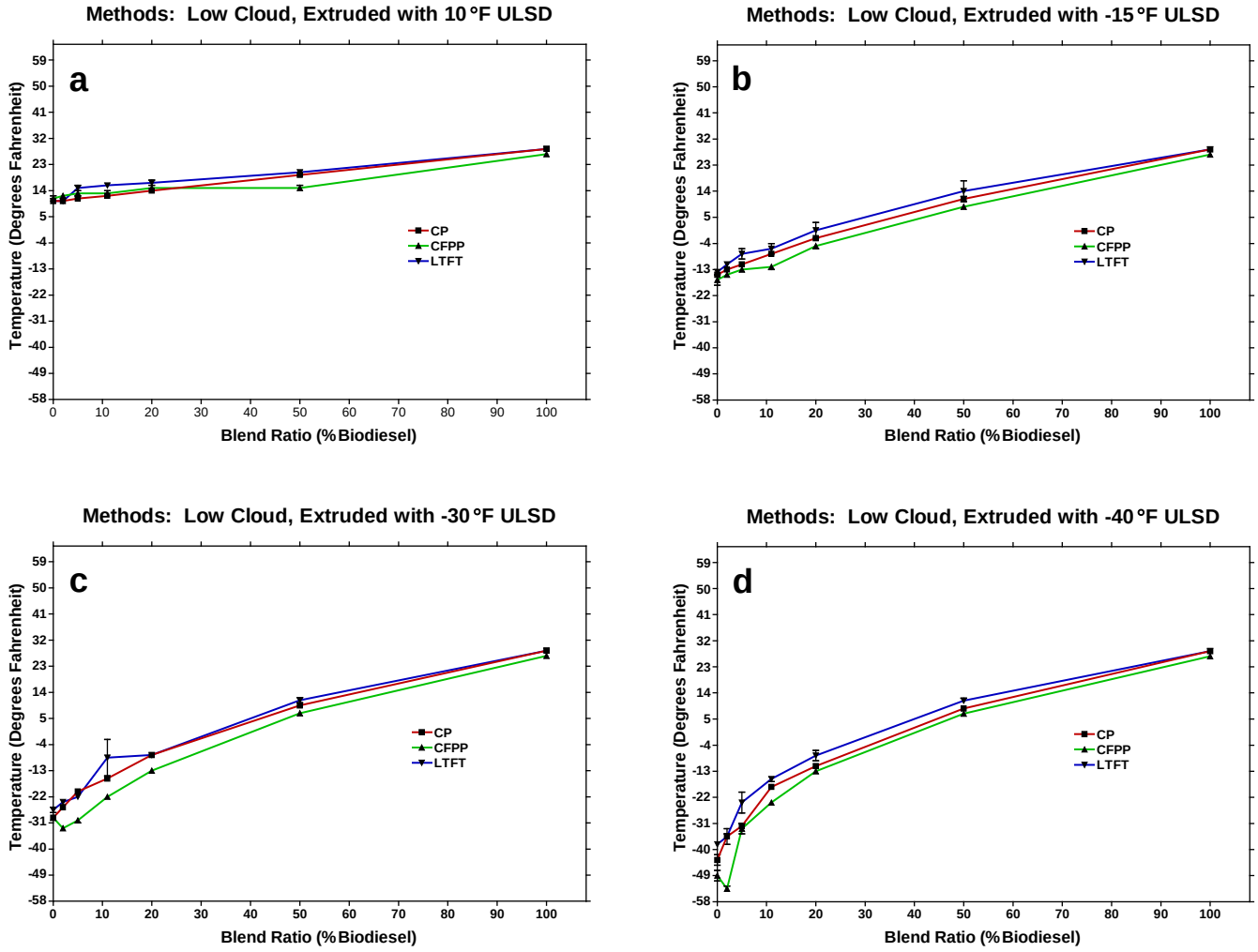


Figure 9. Methods comparison for Low Cloud, Non-Distilled product.

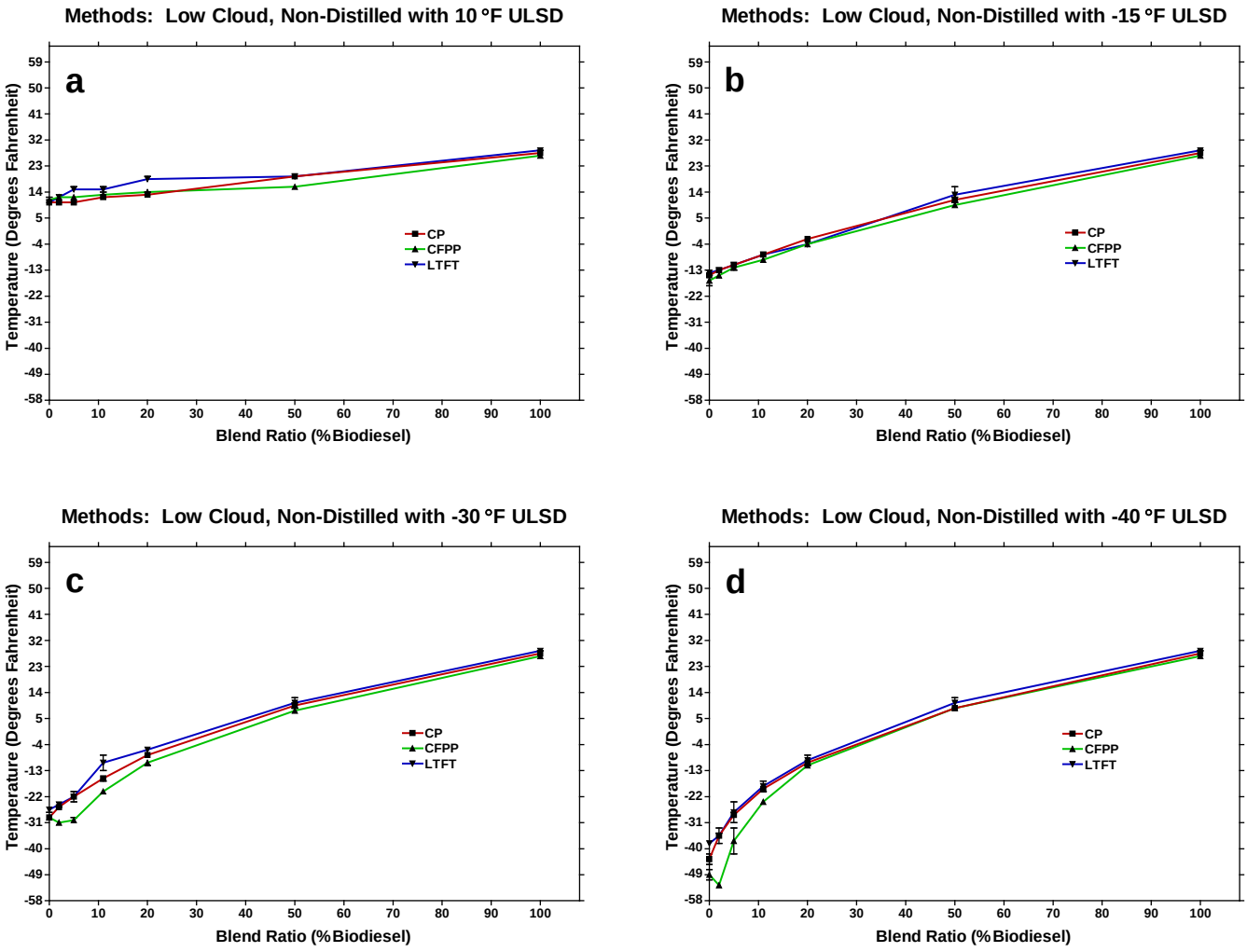


Figure 10. Methods comparison for Mid Cloud product.

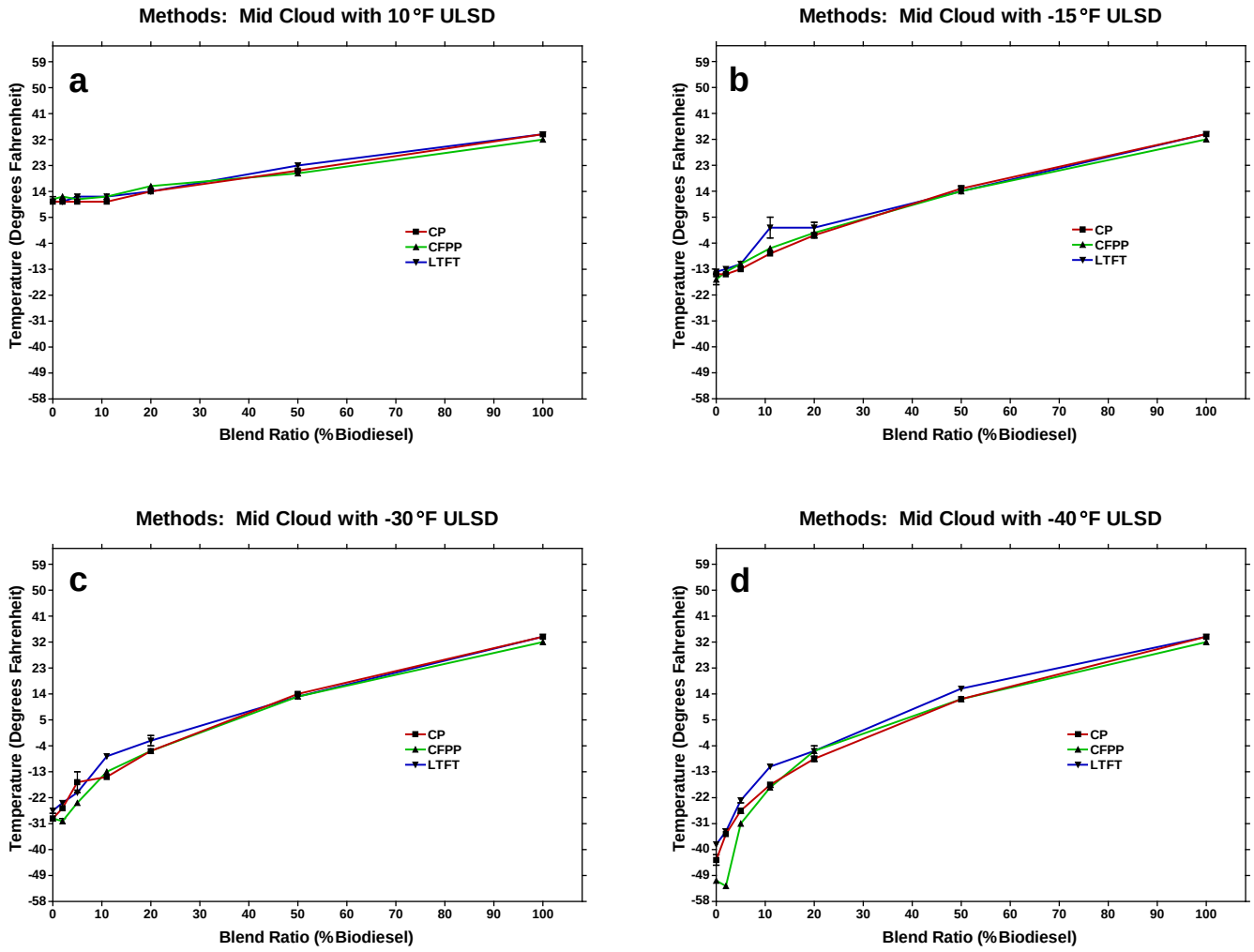


Figure 11. Methods comparison for Mid-High Cloud, A product.

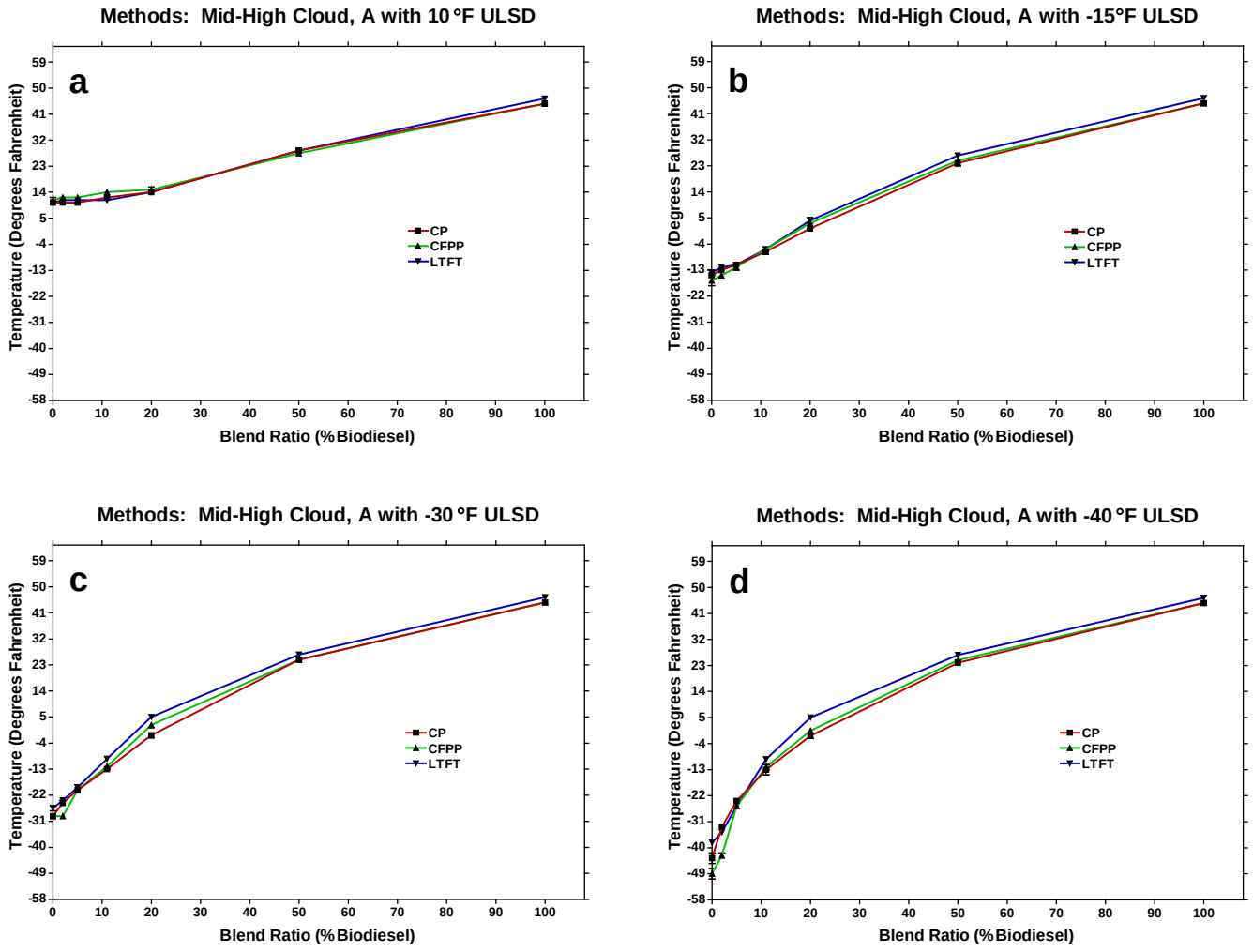


Figure 12. Methods comparison for Mid-High Cloud, B product.

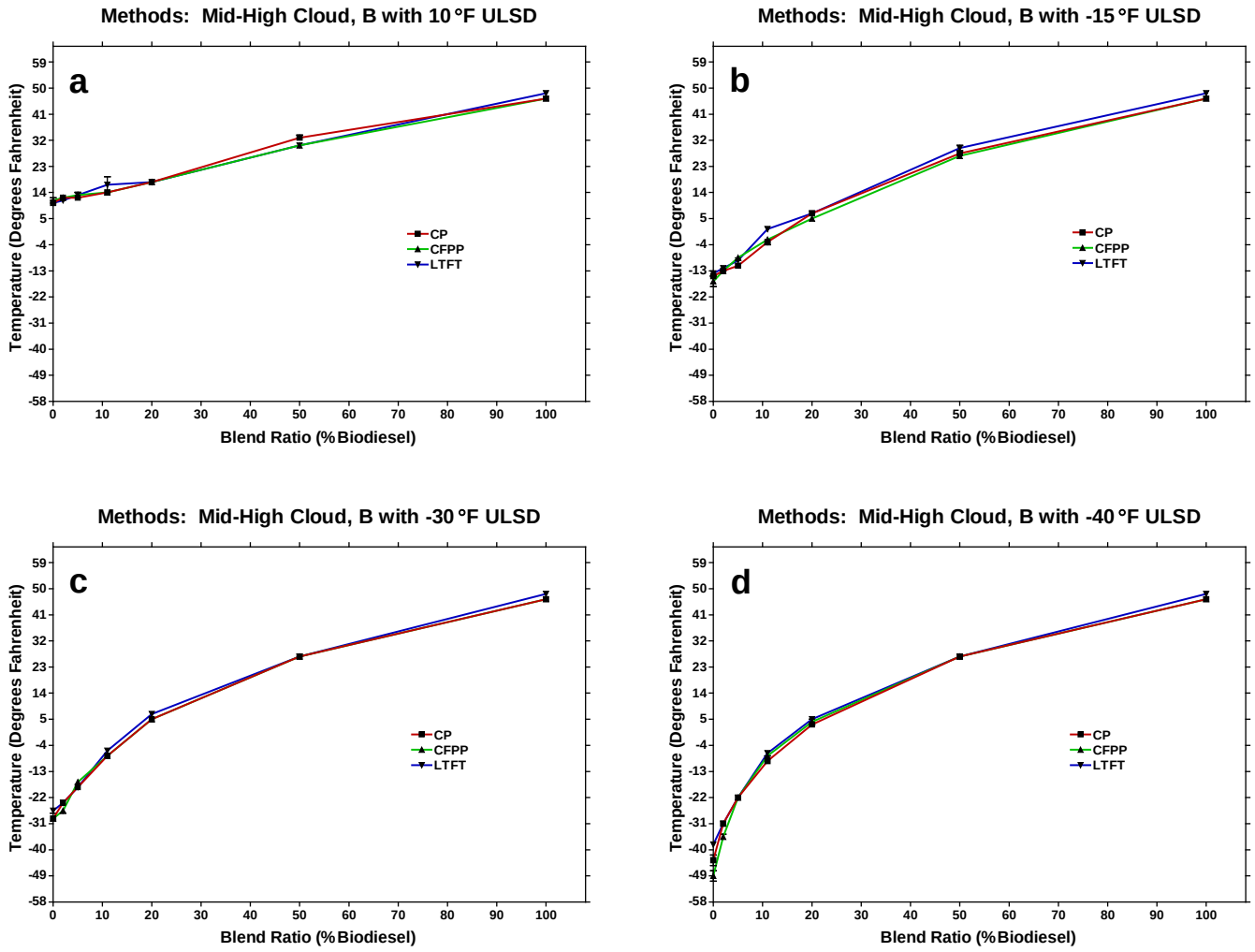
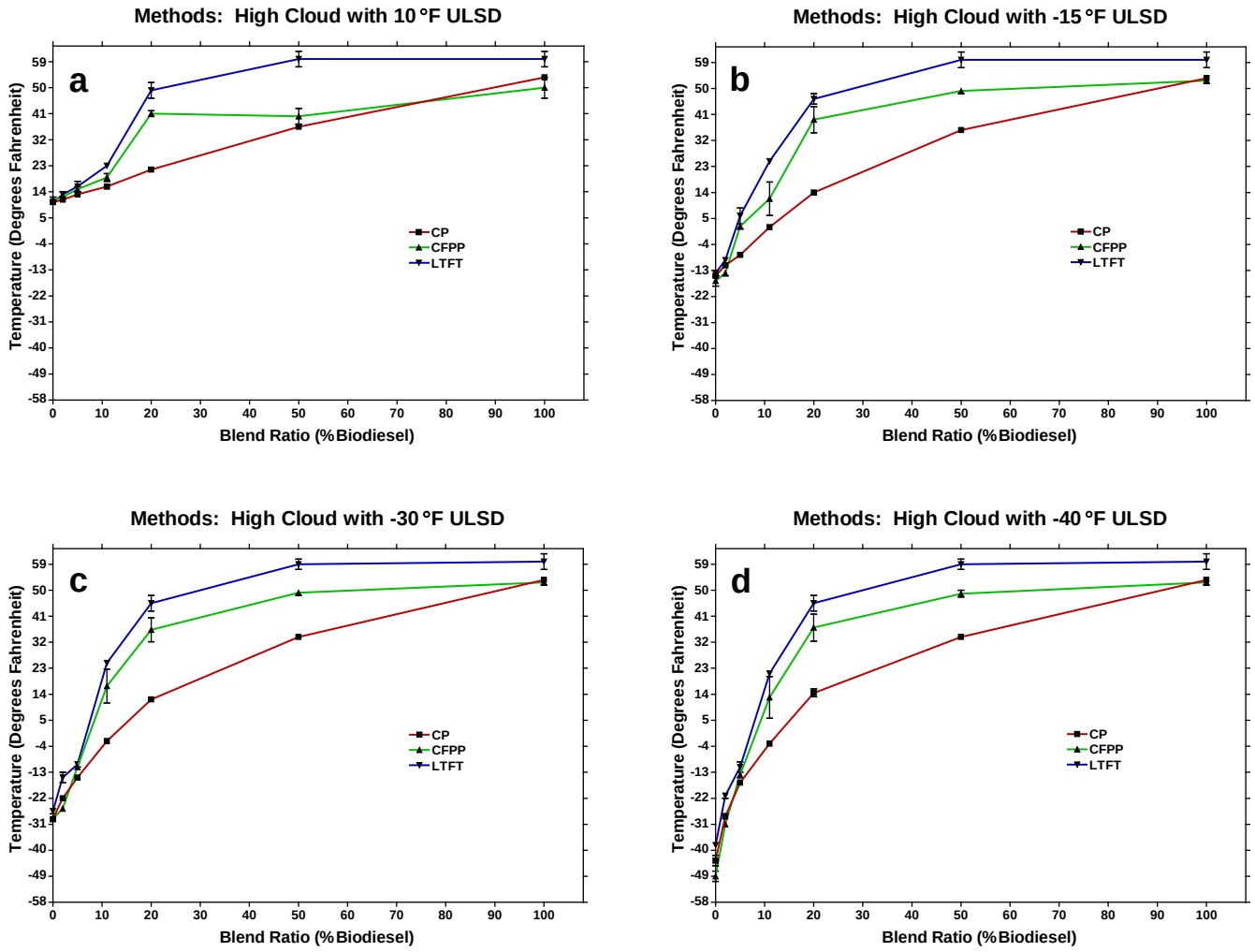


Figure 13. Methods comparison for High Cloud product.



SUMMARY and CONCLUSIONS

Samples of commercially available petrodiesel and biodiesel representing the range of cloud points of products in the market in 2009 were collected, blended and analyzed for cold flow properties.

Three petrodiesel samples were collected and ranged from -53.5°F to +12.2F in cloud point. This represents a range of petrodiesel cloud points of over 65F in the market. The petrodiesel samples collected were used to produce 4 petrodiesel samples with cloud points of approximately -40F, -30F, -15F, and +10F. These were used for subsequent cold flow analysis and blending with biodiesel.

Seven biodiesel samples (B100) were collected and ranged from +27.5°F to +53.6°F in cloud point. This represents a range of only 26.1°F. This is less than half the variation in cloud point than petrodiesel, although the range is higher than that of petrodiesel. These were used for subsequent cold flow analysis and blending with petrodiesel.

The neat fuels and blends of B2, B5, B11, B20 and B50 were analyzed and reported for Cloud Point, Cold Filter Plugging Point (CFPP), and Low Temperature Flow Test (LTFT). The pour point of the neat biodiesel and B50 blends were also analyzed. In some cases the impact on cold flow properties of blending biodiesel with petrodiesel appeared to be mostly linear, while in others the impact was curvilinear. In all cases except a few with low blends of biodiesel where the CFPP of the blend was slightly below that of the petrodiesel, the blended fuel values fell between the pure petrodiesel and pure biodiesel values for all three cold flow measurements.

This information was developed to provide an independent, updated database on ULSD and biodiesel blends and cold flow impacts of the addition of biodiesel. The report does not provide data on the beneficial impact of cold flow additives, but a second phase has been planned to provide independent testing on the impact of cold flow improving additives on CFPP and LTFT of some commercial additives.

The reader is cautioned not to associate the individual cloud point values of the fuels or blends used in this study as any sort of 'average' or 'accepted' value for those fuels. Actual values for #1 or #2 fuels or their blends, or actual values for biodiesel made by various processes and oils or their blends, will vary in the market. The user is advised to utilize actual test data on the fuels they plan to use rather than assume some sort of generic value.

APPENDIX A

Table A1: Raw data for neat ULSD fuels.

	Cloud Point °F		Plug Point °F		LTFT °F		Pour Point °F	
Neat ULSD °F	N=1	N=2	N=1	N=2	N=1	N= 2	N= 1	N= 2
-40	-41.8	-45.4	-50.8	-47.2	-38.2	-38.2	-59.8	-49.0
-30	-29.2	-29.2	-29.2	-29.2	-25.6	-27.4	-54.4	-59.8
-15	-14.8	-14.8	-14.8	-18.4	-13.0	-14.8	-49.0	-43.6
10	10.4	10.4	12.2	10.4	10.4	10.4	-5.8	-5.8

Table A2: Raw data for neat biodiesel fuels.

	Cloud Point °F		Plug Point °F		LTFT °F		Pour Point °F	
Neat Biodiesel	N=1	N=2	N=1	N=2	N=1	N= 2	N= 1	N= 2
Low Cloud, Non-Distilled	26.6	28.4	26.6	26.6	28.4	28.4	26.6	26.6
Low Cloud, Extruded	28.4	28.4	26.6	26.6	28.4	28.4	32.0	32.0
Low Cloud, Distilled	28.4	30.2	28.4	28.4	30.2	30.2	32.0	32.0
Mid Cloud	33.8	33.8	32.0	32.0	33.8	33.8	37.4	37.4
Mid-High Cloud A	44.6	44.6	44.6	44.6	46.4	46.4	50.0	48.2
Mid-High Cloud B	46.4	46.4	46.4	46.4	48.2	48.2	48.2	48.2
High Cloud	53.6	53.6	53.6	51.8	57.2	62.6	53.6	53.6

Table A3: Raw data for biodiesel blends using -40°F ULSD fuel.

-40°F ULSD		Cloud Point °F		Plug Point °F		LTFT °F		Pour Point °F	
Biodiesel	Blend	N= 1	N= 2	N= 1	N= 2	N= 1	N= 2	N= 1	N= 2
Low Cloud, Non-Distilled	B2	-36.4	-34.6	-52.6	-52.6	-38.2	-32.8		
	B5	-29.2	-27.4	-32.8	-41.8	-31.0	-23.8		
	B11	-20.2	-18.4	-23.8	-23.8	-20.2	-16.6		
	B20	-9.4	-11.2	-11.2	-11.2	-11.2	-7.6		
	B50	8.6	8.6	8.6	8.6	8.6	12.2	5.0	-0.4
Low Cloud, Extruded	B2	-34.6	-36.4	-52.6	-54.4	-38.2	-32.8		
	B5	-31.0	-32.8	-31.0	-34.6	-27.4	-20.2		
	B11	-18.4	-18.4	-23.8	-23.8	-16.6	-14.8		
	B20	-11.2	-11.2	-13.0	-13.0	-9.4	-5.8		
	B50	8.6	8.6	6.8	6.8	10.4	12.2	5.0	5.0
Low Cloud, Distilled	B2	-36.4	-40.0	-45.4	-45.4	-38.2	-34.6		
	B5	-29.2	-27.4	-32.8	-32.8	-31.0	-27.4		
	B11	-18.4	-16.6	-20.2	-22.0	-18.4	-20.2		
	B20	-11.2	-11.2	-11.2	-11.2	-11.2	-11.2		
	B50	6.8	6.8	6.8	6.8	8.6	8.6	10.4	10.4
Mid Cloud	B2	-34.6	-34.6	-52.6	-52.6	-32.8	-34.6		
	B5	-27.4	-25.6	-31.0	-31.0	-23.8	-22.0		
	B11	-18.4	-16.6	-18.4	-18.4	-11.2	-11.2		
	B20	-9.4	-7.6	-5.8	-5.8	-7.6	-4.0		
	B50	12.2	12.2	12.2	12.2	15.8	15.8	10.4	10.4
Mid-High Cloud A	B2	-32.8	-32.8	-41.8	-43.6	-34.6	-34.6		
	B5	-23.8	-23.8	-25.6	-25.6	-25.6	-25.6		
	B11	-14.8	-11.2	-13.0	-11.2	-9.4	-9.4		
	B20	-0.4	-2.2	1.4	-0.4	5.0	5.0		
	B50	23.0	24.8	24.8	24.8	26.6	26.6	26.6	26.6
Mid-High Cloud B	B2	-31.0	-31.0	-36.4	-34.6	-31	-31.0		
	B5	-22.0	-22.0	-22.0	-22.0	-22.0	-22.0		
	B11	-9.4	-9.4	-7.6	-7.6	-7.6	-5.8		
	B20	3.2	3.2	3.2	5.0	5.0	NA		
	B50	26.6	26.6	26.6	26.6	26.6	NA	26.6	26.6
High Cloud	B2	-29.2	-27.4	-31.0	-31.0	-22.0	-20.2		
	B5	-16.6	-16.6	-13.0	-14.8	-13.0	-9.4		
	B11	10.4	-4.0	6.8	-0.4	15.8	NA		
	B20	44.6	17.6	21.2	41.0	59.0	42.8		
	B50	48.2	33.8	50.0	50.0	57.2	60.8	37.4	37.4

Table A4: Raw data for biodiesel blends using -30°F ULSD fuel.

-30°F ULSD		Cloud Point °F		Plug Point °F		LTFT °F		Pour Point °F	
Biodiesel	Blend	N= 1	N= 2	N= 1	N= 2	N= 1	N= 2	N= 1	N= 2
Low Cloud, Non-Distilled	B2	-25.6	-25.6	-31.0	-31.0	-25.6	-23.8		
	B5	-22.0	-22.0	-29.2	-31.0	-23.8	-20.2		
	B11	-14.8	-16.6	-20.2	-20.2	-13.0	-7.6		
	B20	-7.6	-7.6	-11.2	-9.4	-5.8	-5.8		
	B50	8.6	10.4	6.8	8.6	8.6	12.2	5.0	5.0
Low Cloud, Extruded	B2	-25.6	-25.6	-32.8	-32.8	-23.8	-23.8		
	B5	-20.2	-20.2	-29.2	-31.0	-22.0	-22.0		
	B11	-14.8	-16.6	-22.0	-22.0	-14.8	-2.2		
	B20	-7.6	-7.6	-13.0	-13.0	-7.6	-7.6		
	B50	8.6	10.4	6.8	6.8	10.4	12.2	5.0	5.0
Low Cloud, Distilled	B2	-27.4	-27.4	-29.2	-27.4	-23.8	-23.8		
	B5	-22.0	-22.0	-25.6	-25.6	-25.6	-22.0		
	B11	-16.6	-16.6	-16.6	-16.6	-16.6	-14.8		
	B20	-9.4	-9.4	-7.6	-7.6	-7.6	-9.4		
	B50	6.8	6.8	6.8	6.8	8.6	8.6	10.4	10.4
Mid Cloud	B2	-25.6	-25.6	-31.0	-29.2	-23.8	-23.8		
	B5	-13.0	-20.2	-23.8	-23.8	-20.2	-20.2		
	B11	-14.8	-14.8	-13.0	-13.0	-7.6	-7.6		
	B20	-5.8	-5.8	-5.8	-5.8	-4.0	-0.4		
	B50	14.0	14.0	12.2	14.0	14.0	12.2	10.4	10.4
Mid-High Cloud A	B2	-25.6	-23.8	-29.2	-29.2	-23.8	-23.8		
	B5	-20.2	-20.2	-20.2	-20.2	-18.4	-20.2		
	B11	-13.0	-13.0	-13.0	-11.2	-9.4	-9.4		
	B20	-0.4	-2.2	3.2	1.4	5.0	5.0		
	B50	24.8	24.8	24.8	24.8	26.6	26.6	26.6	26.6
Mid-High Cloud B	B2	-23.8	-23.8	-27.4	-25.6	-23.8	-23.8	-23.8	
	B5	-18.4	-18.4	-16.6	-16.6	-18.4	-18.4	-18.4	
	B11	-7.6	-7.6	-7.6	-7.6	-5.8	-5.8	-5.8	
	B20	5.0	5.0	5.0	5.0	6.8	6.8	NA	
	B50	26.6	26.6	26.6	26.6	26.6	26.6	26.6	26.6
High Cloud	B2	-22.0	-22.0	-25.6	-25.6	-16.6	-13.0		
	B5	-14.8	-14.8	-11.2	-11.2	-11.2	-9.4		
	B11	23.0	-2.2	30.2	28.4	24.8	NA		
	B20	46.4	12.2	42.8	21.2	59.0	42.8		
	B50	51.8	50.0	48.2	50.0	57.2	60.8	32.0	37.4

Table A5: Raw data for biodiesel blends using -15°F ULSD fuel.

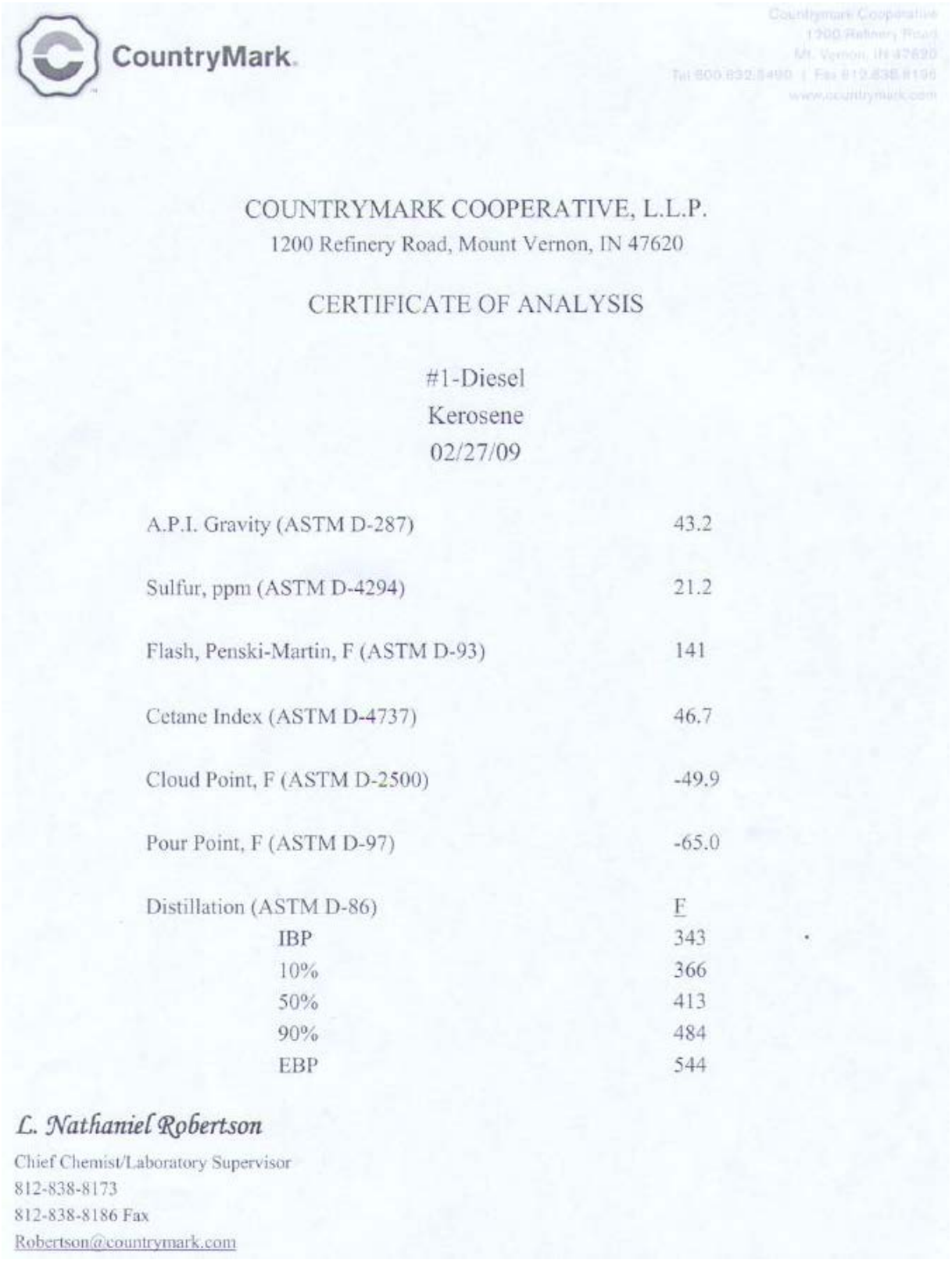
-15°F ULSD		Cloud Point °F		Plug Point °F		LTFT °F		Pour Point °F	
Biodiesel	Blend	N= 1	N= 2	N= 1	N= 2	N= 1	N= 2	N= 1	N= 2
Low Cloud, Non-Distilled	B2	-13.0	-13.0	-14.8	-14.8	-13.0	-13.0		
	B5	-11.2	-11.2	-13.0	-11.2	-11.2	-11.2		
	B11	-7.6	-7.6	-9.4	-9.4	-7.6	-7.6		
	B20	-2.2	-2.2	-4.0	-4.0	-4.0	-4.0		
	B50	10.4	12.2	8.6	10.4	10.4	15.8	5.0	5.0
Low Cloud, Extruded	B2	-13.0	-13.0	-14.8	-14.8	-11.2	-11.2		
	B5	-11.2	-11.2	-13.0	-13.0	-9.4	-5.8		
	B11	-7.6	-7.6	-13.0	-11.2	-7.6	-4.0		
	B20	-2.2	-2.2	-5.8	-4.0	-2.2	3.2		
	B50	10.4	12.2	8.6	8.6	10.4	17.6	10.4	10.4
Low Cloud, Distilled	B2	-13.0	-13.0	-14.8	-14.8	-11.2	-13.0		
	B5	-11.2	-11.2	-13.0	-14.8	-11.2	-11.2		
	B11	-9.4	-7.6	-9.4	-9.4	-7.6	-7.6		
	B20	-4.0	-4.0	-4.0	-4.0	-4.0	-5.8		
	B50	8.6	10.4	8.6	8.6	10.4	10.4	10.4	10.4
Mid Cloud	B2	-14.8	-14.8	-14.8	-13.0	-13	-13.0		
	B5	-1.0	-13.0	-11.2	-11.2	-11.2	-11.2		
	B11	-7.6	-7.6	-5.8	-5.8	-2.2	5.0		
	B20	-2.2	-0.4	-0.4	-0.4	-0.4	3.2		
	B50	15.8	14.0	14.0	14.0	14.0	14.0	10.4	15.8
Mid-High Cloud A	B2	-13.0	-13.0	-14.8	-14.8	-11.2	-13.0		
	B5	-11.2	-11.2	-11.2	-13.0	-11.2	-11.2		
	B11	-7.6	-5.8	-5.8	-5.8	-5.8	-5.8		
	B20	1.4	1.4	1.4	5.0	3.2	5.0		
	B50	24.8	23.0	24.8	24.8	26.6	26.6	26.6	26.6
Mid-High Cloud B	B2	-13.0	-13.0	-13.0	-13.0	-11.2	-13.0		
	B5	-11.2	-11.2	-9.4	-7.6	-9.4	-9.4		
	B11	-2.2	-4.0	-2.2	-2.2	1.4	NA		
	B20	6.8	6.8	5.0	5.0	6.8	NA		
	B50	28.4	26.6	26.6	26.6	28.4	30.2	32.0	26.6
High Cloud	B2	-11.2	-11.2	-14.8	-13.0	-9.4	-9.4		
	B5	-7.6	-7.6	1.4	3.2	3.2	8.6		
	B11	1.4	1.4	3.2	17.6	24.8	NA		
	B20	41.0	14.0	42.8	21.2	60.8	44.6		
	B50	53.6	51.8	48.2	50.0	57.2	62.6	37.4	37.4

Table A6: Raw data for biodiesel blends using +10°F ULSD fuel.

+10°F ULSD		Cloud Point °F		Plug Point °F		LTFT °F		Pour Point °F	
Biodiesel	Blend	N= 1	N= 2	N= 1	N= 2	N= 1	N= 2	N= 1	N= 2
Low Cloud, Non-Distilled	B2	10.4	10.4	12.2	12.2	12.2	12.2		
	B5	10.4	10.4	12.2	12.2	14.0	15.8		
	B11	12.2	12.2	14.0	12.2	14.0	15.8		
	B20	14.0	12.2	14.0	14.0	19.4	17.6		
	B50	19.4	19.4	15.8	15.8	19.4	19.4	15.8	15.8
Low Cloud, Extruded	B2	10.4	10.4	12.2	12.2	10.4	10.4		
	B5	12.2	10.4	14.0	12.2	14.0	15.8		
	B11	12.2	12.2	14.0	12.2	15.8	15.8		
	B20	14.0	14.0	14.0	15.8	15.8	17.6		
	B50	19.4	19.4	14.0	15.8	21.2	19.4	15.8	15.8
Low Cloud, Distilled	B2	10.4	10.4	12.2	12.2	10.4	10.4		
	B5	12.2	10.4	12.2	12.2	10.4	10.4		
	B11	12.2	10.4	12.2	12.2	12.2	12.2		
	B20	12.2	12.2	14.0	14.0	12.2	12.2		
	B50	15.8	15.8	17.6	17.6	17.6	19.4	15.8	15.8
Mid Cloud	B2	10.4	10.4	12.2	12.2	10.4	10.4		
	B5	10.4	10.4	12.2	10.4	12.2	12.2		
	B11	10.4	10.4	12.2	12.2	12.2	12.2		
	B20	14.0	14.0	15.8	15.8	14.0	14.0		
	B50	21.2	21.2	21.2	19.4	23.0	23.0	21.2	21.2
Mid-High Cloud A	B2	10.4	10.4	12.2	12.2	12.2	10.4		
	B5	10.4	10.4	12.2	12.2	12.2	10.4		
	B11	12.2	12.2	14.0	14.0	12.2	10.4		
	B20	14.0	14.0	14.0	15.8	14.0	14.0		
	B50	28.4	28.4	28.4	26.6	28.4	28.4	26.6	26.6
Mid-High Cloud B	B2	12.2	12.2	12.2	12.2	12.2	10.4		
	B5	12.2	10.4	12.2	14.0	14.0	12.2		
	B11	14.0	14.0	14.0	14.0	14.0	19.4		
	B20	17.6	17.6	17.6	17.6	17.6	17.6		
	B50	32.0	33.8	30.2	30.2	30.2	30.2	32.0	32.0
High Cloud	B2	12.2	10.4	12.2	12.2	14.0	12.2		
	B5	14.0	12.2	14.0	15.8	14.0	17.6		
	B11	15.8	15.8	17.6	17.6	23.0	23.0		
	B20	21.2	21.2	37.4	41.0	62.6	46.4		
	B50	35.6	37.4	42.8	37.4	62.6	57.2	37.4	37.4

APPENDIX B

Figure B1. Certificate of Analysis for #1 ULSD.



CountryMark.

Countrymark Cooperative
1200 Refinery Road
Mt. Vernon, IN 47620
Tel 800.632.5490 | Fax 812.838.8196
www.countrymark.com

COUNTRYMARK COOPERATIVE, L.L.P.
1200 Refinery Road, Mount Vernon, IN 47620

CERTIFICATE OF ANALYSIS

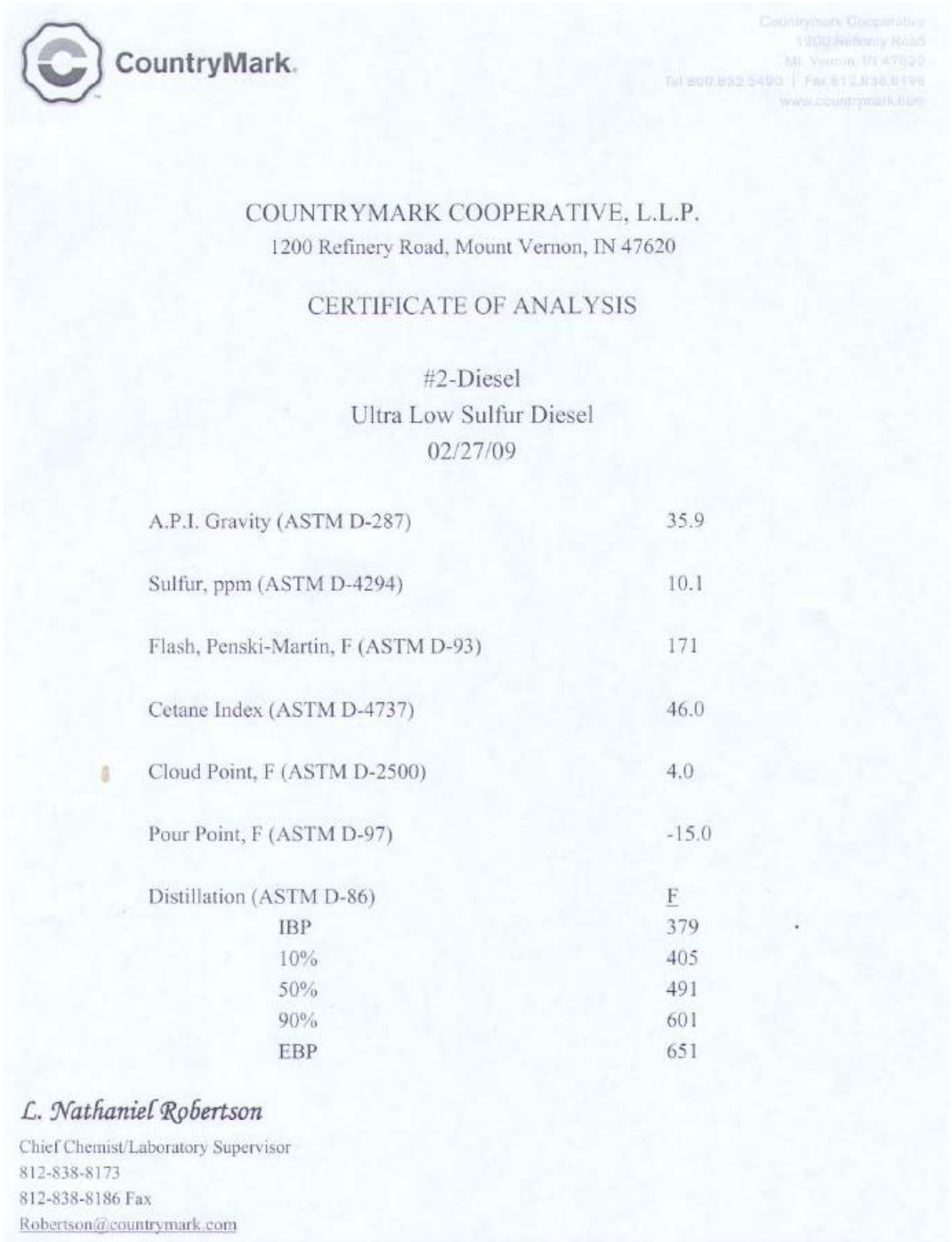
#1-Diesel
Kerosene
02/27/09

A.P.I. Gravity (ASTM D-287)	43.2
Sulfur, ppm (ASTM D-4294)	21.2
Flash, Penski-Martin, F (ASTM D-93)	141
Cetane Index (ASTM D-4737)	46.7
Cloud Point, F (ASTM D-2500)	-49.9
Pour Point, F (ASTM D-97)	-65.0
Distillation (ASTM D-86)	F
IBP	343
10%	366
50%	413
90%	484
EBP	544

L. Nathaniel Robertson

Chief Chemist/Laboratory Supervisor
812-838-8173
812-838-8186 Fax
Robertson@countrymark.com

Figure B2. Certificate of Analysis for #2 ULSD.



CountryMark.

COUNTRYMARK COOPERATIVE, L.L.P.
1200 Refinery Road, Mount Vernon, IN 47620

CERTIFICATE OF ANALYSIS

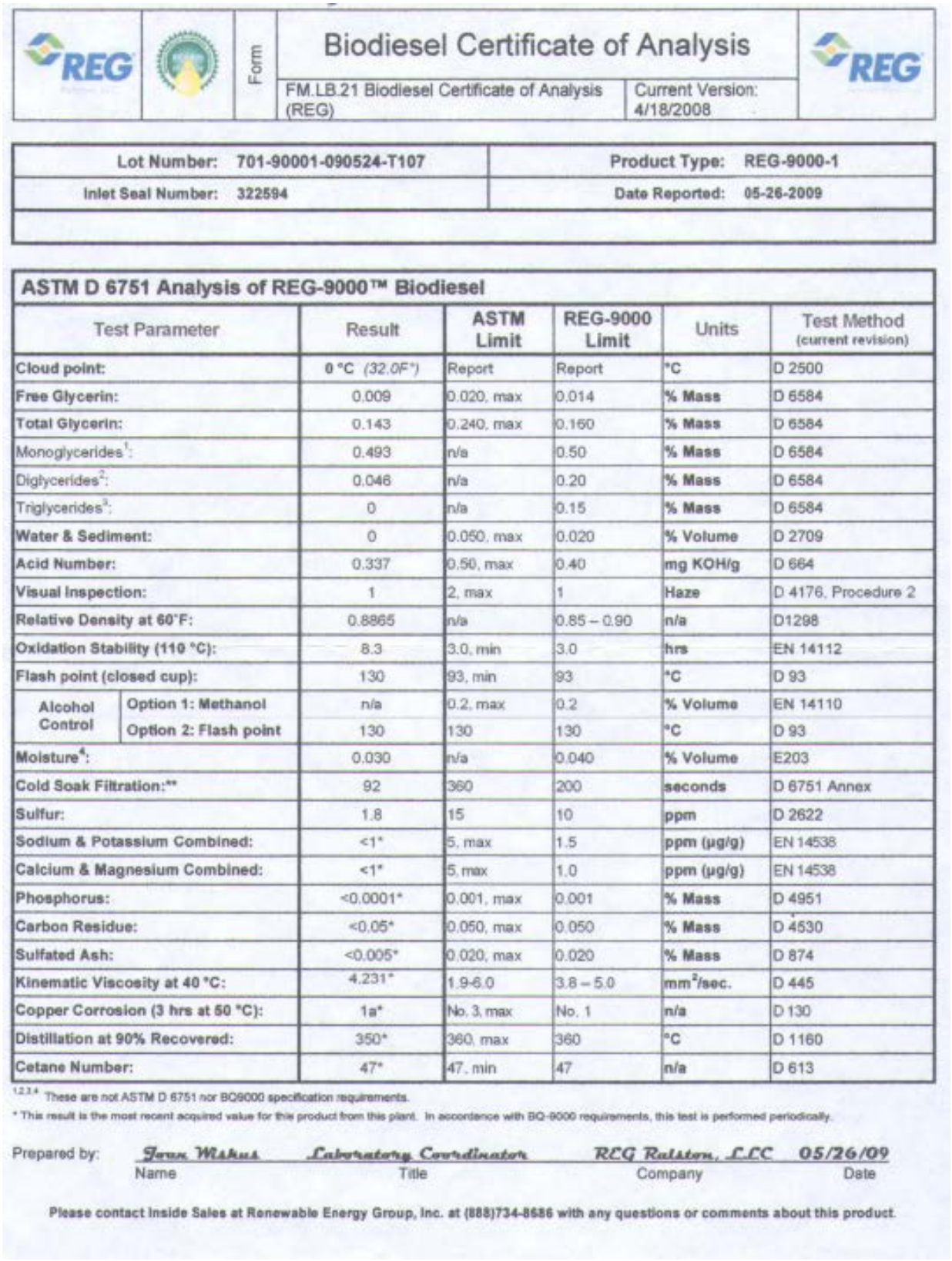
#2-Diesel
Ultra Low Sulfur Diesel
02/27/09

A.P.I. Gravity (ASTM D-287)	35.9
Sulfur, ppm (ASTM D-4294)	10.1
Flash, Penski-Martin, F (ASTM D-93)	171
Cetane Index (ASTM D-4737)	46.0
Cloud Point, F (ASTM D-2500)	4.0
Pour Point, F (ASTM D-97)	-15.0
Distillation (ASTM D-86)	F
IBP	379
10%	405
50%	491
90%	601
EBP	651

L. Nathaniel Robertson

Chief Chemist/Laboratory Supervisor
812-838-8173
812-838-8186 Fax
Robertson@countrymark.com

Figure B3. Certificate of Analysis for Low Cloud biodiesel.



Form	Biodiesel Certificate of Analysis	
	FM.LB.21 Biodiesel Certificate of Analysis (REG)	Current Version: 4/18/2008

Lot Number: 701-90001-090524-T107	**Product Type:** REG-9000-1
Inlet Seal Number: 322594	**Date Reported:** 05-26-2009

ASTM D 6751 Analysis of REG-9000™ Biodiesel

Test Parameter		Result	ASTM Limit	REG-9000 Limit	Units	Test Method (current revision)
Cloud point:		0 °C *(32.0F°)*	Report	Report	°C	D 2500
Free Glycerin:		0.009	0.020, max	0.014	% Mass	D 6584
Total Glycerin:		0.143	0.240, max	0.160	% Mass	D 6584
Monoglycerides[1]:		0.493	n/a	0.50	% Mass	D 6584
Diglycerides[2]:		0.046	n/a	0.20	% Mass	D 6584
Triglycerides[3]:		0	n/a	0.15	% Mass	D 6584
Water & Sediment:		0	0.050, max	0.020	% Volume	D 2709
Acid Number:		0.337	0.50, max	0.40	mg KOH/g	D 664
Visual Inspection:		1	2, max	1	Haze	D 4176, Procedure 2
Relative Density at 60°F:		0.8865	n/a	0.85 – 0.90	n/a	D1298
Oxidation Stability (110 °C):		8.3	3.0, min	3.0	hrs	EN 14112
Flash point (closed cup):		130	93, min	93	°C	D 93
Alcohol Control	**Option 1: Methanol**	n/a	0.2, max	0.2	% Volume	EN 14110
	Option 2: Flash point	130	130	130	°C	D 93
Moisture[4]:		0.030	n/a	0.040	% Volume	E203
Cold Soak Filtration:**		92	360	200	seconds	D 6751 Annex
Sulfur:		1.8	15	10	ppm	D 2622
Sodium & Potassium Combined:		<1*	5, max	1.5	ppm (µg/g)	EN 14538
Calcium & Magnesium Combined:		<1*	5, max	1.0	ppm (µg/g)	EN 14538
Phosphorus:		<0.0001*	0.001, max	0.001	% Mass	D 4951
Carbon Residue:		<0.05*	0.050, max	0.050	% Mass	D 4530
Sulfated Ash:		<0.005*	0.020, max	0.020	% Mass	D 874
Kinematic Viscosity at 40 °C:		4.231*	1.9-6.0	3.8 – 5.0	$mm^2/sec.$	D 445
Copper Corrosion (3 hrs at 50 °C):		1a*	No. 3, max	No. 1	n/a	D 130
Distillation at 90% Recovered:		350*	360, max	360	°C	D 1160
Cetane Number:		47*	47, min	47	n/a	D 613

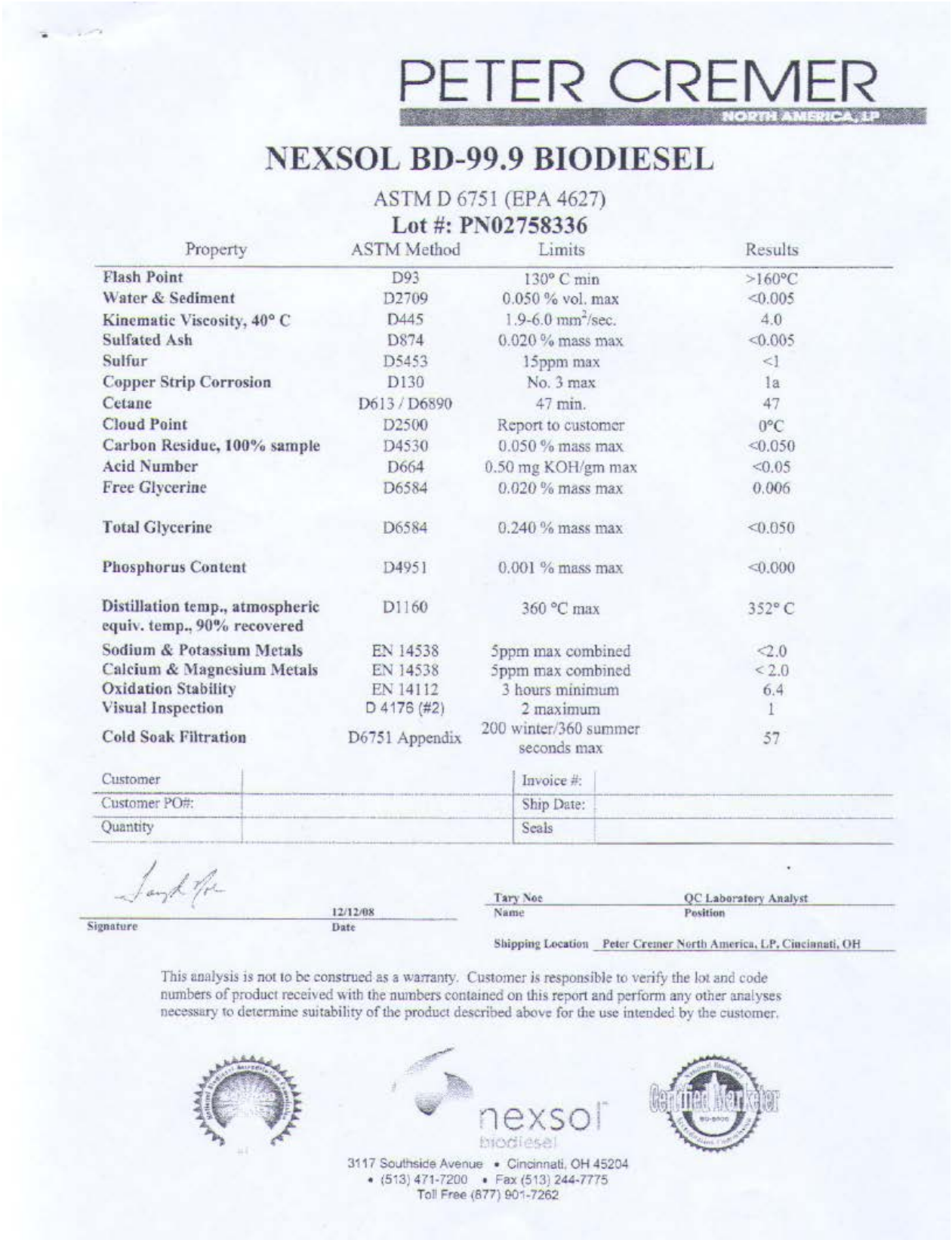
[1,2,3,4] These are not ASTM D 6751 nor BQ9000 specification requirements.

* This result is the most recent acquired value for this product from this plant. In accordance with BQ-9000 requirements, this test is performed periodically.

Prepared by: Jason Miskus (Name) Laboratory Coordinator (Title) REG Ralston, LLC (Company) 05/26/09 (Date)

Please contact Inside Sales at Renewable Energy Group, Inc. at (888)734-8686 with any questions or comments about this product.

Figure B4. Certificate of Analysis for Low Cloud, Distilled biodiesel first batch.



PETER CREMER
NORTH AMERICA, LP

NEXSOL BD-99.9 BIODIESEL

ASTM D 6751 (EPA 4627)

Lot #: PN02758336

Property	ASTM Method	Limits	Results
Flash Point	D93	130° C min	>160°C
Water & Sediment	D2709	0.050 % vol. max	<0.005
Kinematic Viscosity, 40° C	D445	1.9-6.0 $mm^2/sec.$	4.0
Sulfated Ash	D874	0.020 % mass max	<0.005
Sulfur	D5453	15ppm max	<1
Copper Strip Corrosion	D130	No. 3 max	1a
Cetane	D613 / D6890	47 min.	47
Cloud Point	D2500	Report to customer	0°C
Carbon Residue, 100% sample	D4530	0.050 % mass max	<0.050
Acid Number	D664	0.50 mg KOH/gm max	<0.05
Free Glycerine	D6584	0.020 % mass max	0.006
Total Glycerine	D6584	0.240 % mass max	<0.050
Phosphorus Content	D4951	0.001 % mass max	<0.000
Distillation temp., atmospheric equiv. temp., 90% recovered	D1160	360 °C max	352° C
Sodium & Potassium Metals	EN 14538	5ppm max combined	<2.0
Calcium & Magnesium Metals	EN 14538	5ppm max combined	< 2.0
Oxidation Stability	EN 14112	3 hours minimum	6.4
Visual Inspection	D 4176 (#2)	2 maximum	1
Cold Soak Filtration	D6751 Appendix	200 winter/360 summer seconds max	57

Customer		Invoice #:	
Customer PO#:		Ship Date:	
Quantity		Seals	

Signature — Date: 12/12/08

Tary Noe — Name — QC Laboratory Analyst — Position

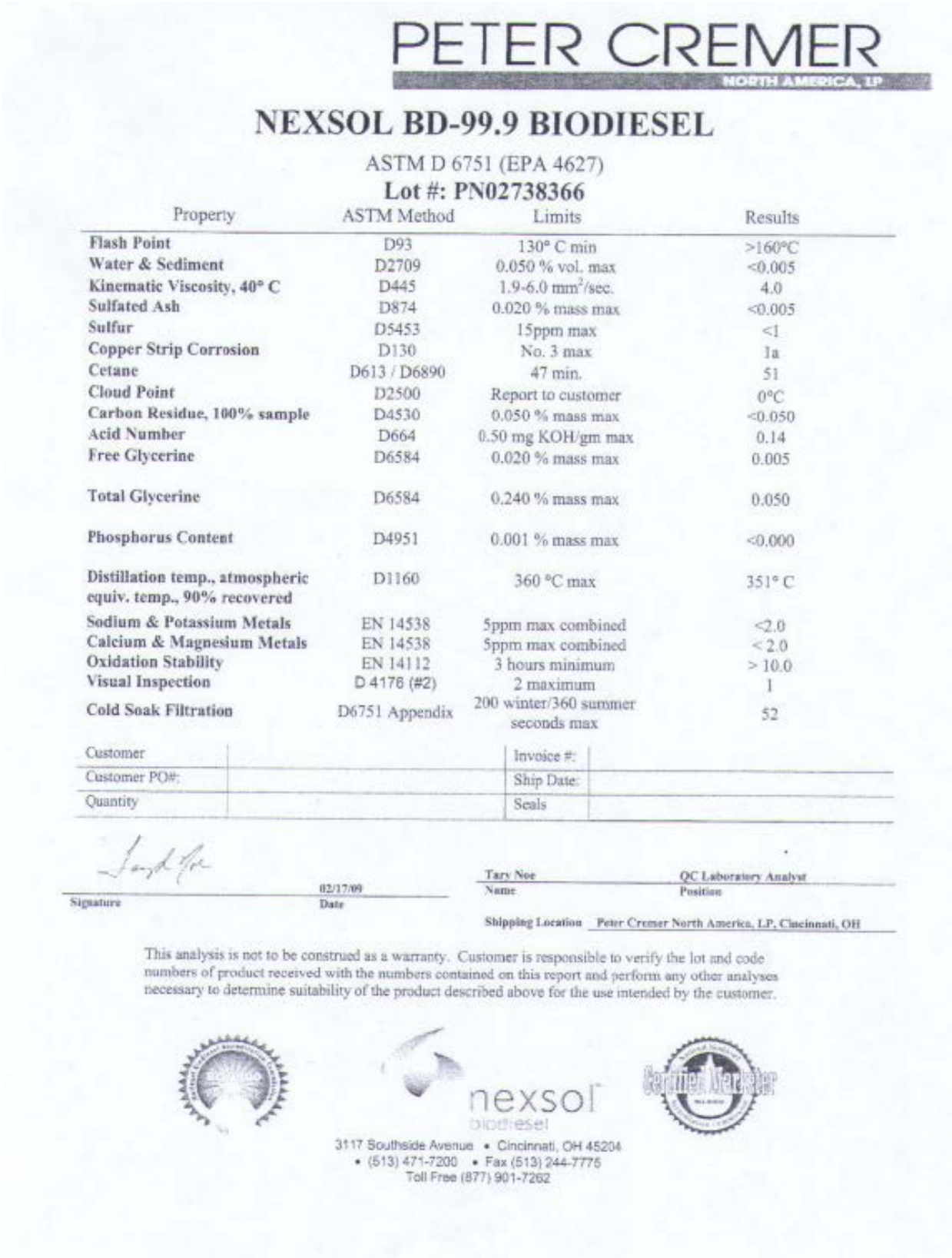
Shipping Location: Peter Cremer North America, LP, Cincinnati, OH

This analysis is not to be construed as a warranty. Customer is responsible to verify the lot and code numbers of product received with the numbers contained on this report and perform any other analyses necessary to determine suitability of the product described above for the use intended by the customer.

nexsol biodiesel

3117 Southside Avenue • Cincinnati, OH 45204
• (513) 471-7200 • Fax (513) 244-7775
Toll Free (877) 901-7262

Figure B5. Certificate of Analysis for Low Cloud, Distilled biodiesel second batch.



PETER CREMER
NORTH AMERICA, LP

NEXSOL BD-99.9 BIODIESEL

ASTM D 6751 (EPA 4627)

Lot #: PN02738366

Property	ASTM Method	Limits	Results
Flash Point	D93	130° C min	>160°C
Water & Sediment	D2709	0.050 % vol. max	<0.005
Kinematic Viscosity, 40° C	D445	1.9-6.0 $mm^2/sec.$	4.0
Sulfated Ash	D874	0.020 % mass max	<0.005
Sulfur	D5453	15ppm max	<1
Copper Strip Corrosion	D130	No. 3 max	1a
Cetane	D613 / D6890	47 min.	51
Cloud Point	D2500	Report to customer	0°C
Carbon Residue, 100% sample	D4530	0.050 % mass max	<0.050
Acid Number	D664	0.50 mg KOH/gm max	0.14
Free Glycerine	D6584	0.020 % mass max	0.005
Total Glycerine	D6584	0.240 % mass max	0.050
Phosphorus Content	D4951	0.001 % mass max	<0.000
Distillation temp., atmospheric equiv. temp., 90% recovered	D1160	360 °C max	351° C
Sodium & Potassium Metals	EN 14538	5ppm max combined	<2.0
Calcium & Magnesium Metals	EN 14538	5ppm max combined	< 2.0
Oxidation Stability	EN 14112	3 hours minimum	> 10.0
Visual Inspection	D 4176 (#2)	2 maximum	1
Cold Soak Filtration	D6751 Appendix	200 winter/360 summer seconds max	52

Customer		Invoice #:	
Customer PO#:		Ship Date:	
Quantity		Seals	

Signature — Date: 02/17/09

Tary Noe — Name — QC Laboratory Analyst — Position

Shipping Location: Peter Cremer North America, LP, Cincinnati, OH

This analysis is not to be construed as a warranty. Customer is responsible to verify the lot and code numbers of product received with the numbers contained on this report and perform any other analyses necessary to determine suitability of the product described above for the use intended by the customer.

nexsol biodiesel

3117 Southside Avenue • Cincinnati, OH 45204
• (513) 471-7200 • Fax (513) 244-7775
Toll Free (877) 901-7262

Figure B6. Certificate of Analysis for Mid Cloud biodiesel.

Central Iowa Energy

This biodiesel was produced at a BQ9000 certified facility

Form

Biodiesel Certificate of Analysis

FM.LB.21 Biodiesel Certificate of Analysis-REG 20090701

Replaces: FM.LB.14b

REG

Lot Number: 803-90010-090720-T5	Product Type: REG-9000-10
Inlet Seal Number: 287215	Date Reported: 7/21/2009

ASTM D 6751 Analysis of REG-9000™ Biodiesel

Test Parameter		Result	ASTM Limit	REG-9000 Limit	Units	Test Method (current revision)
Cloud point:		12°C (53.6°F)	Report	Report	°C	D 2500
Free Glycerin:		0.006	0.020, max	0.014, max	% Mass	D 6584
Total Glycerin:		0.095	0.240, max	0.160, max	% Mass	D 6584
Monoglycerides[1]:		0.289	n/a	0.50	% Mass	D 6584
Diglycerides[2]:		0.071	n/a	0.20	% Mass	D 6584
Triglycerides[3]:		0.030	n/a	0.20	% Mass	D 6584
Water & Sediment:		0	0.050, max	0.020, max	% Volume	D 2709
Acid Number:		0.24	0.50, max	0.40, max	mg KOH/g	D 664, Test Method A (Inflection Point)
Visual Inspection:		1 @ 73.9°F	2, max	1, max	Haze	D 4176, Procedure 2
Relative Density at 60°F:		0.8770	n/a	0.86 – 0.90	n/a	D1298
Oxidation Stability (110 °C):		13.0	3.0, min	3.0, min	hrs	EN 14112
Flash point (closed cup):		131.0	93, min	93, min	°C	D 93
Alcohol Control	Option 1: Methanol	n/a	0.2, max	0.2, max	% Volume	EN 14110
	Option 2: Flash point	131.0	130	130	°C	D 93
Moisture[4]:		0.032	n/a	0.040, max	% Volume	E203
Cold Soak Filtration:		130	360	200 / 360**	seconds	D 6751 Annex
Sulfur:		12	15	12	ppm	D 5453
Sodium & Potassium Combined:		<1.0*	5, max	1.5	ppm (µg/g)	EN 14538
Calcium & Magnesium Combined:		<1.0*	5, max	1.5	ppm (µg/g)	EN 14538
Phosphorus:		<0.0001*	0.001, max	0.001, max	% Mass	D 4951
Carbon Residue:		<0.05*	0.050, max	0.050, max	% Mass	D 4530
Sulfated Ash:		<0.005*	0.020, max	0.020, max	% Mass	D 874
Kinematic Viscosity at 40 °C:		4.539*	1.9-6.0	3.8 – 5.0	$mm^2/sec.$	D 445
Copper Corrosion (3 hrs at 50 °C):		1a*	No. 3, max	No. 1, max	n/a	D 130
Distillation at 90% Recovered:		351*	360, max	360, max	°C	D 1160
Cetane Number:		55.3*	47, min	47, min	n/a	D 613

[1,2,3,4] These are not ASTM D 6751 or BQ9000 specification requirements.

* This result is the most recent acquired value for this product from this plant. In accordance with BQ-9000 requirements, this test is performed periodically.
** REG-9000-1 and REG-9000-5 have a cold soak filtration limit of 200 sec. REG-9000-10 has a cold soak filtration limit of 360 sec.

Prepared by: *John Kiley, Quality Assurance Manager, Central Iowa Energy* *07/21/2009*

First Name Last Name , Title, Company

Please contact Inside Sales of Renewable Energy Group, Inc. at (888)734-8686 for all questions and comments about this product.

Figure B7. Certificate of Analysis for Mid-High Cloud B biodiesel.

Western Iowa Energy WIE

Form

Biodiesel Certificate of Analysis

FM.LB.21 Biodiesel Certificate of Analysis (REG) 20081014

REG

Lot Number: 802-90010-090402-T5	Product Type: REG-9000-10
Inlet Seal Number: 5524	Date Reported: 4-7-09
CFPP 8°C	

ASTM D 6751 Analysis of REG-9000™ Biodiesel

Test Parameter		Result	ASTM Limit	REG-9000 Limit	Units	Test Method (current revision)
Cloud point:		10°C (50°F)	Report	Report	°C	D 2500
Free Glycerin:		0.007	0.020, max	0.014	% Mass	D 6584
Total Glycerin:		0.092	0.240, max	0.160	% Mass	D 6584
Monoglycerides[1]:		0.276	n/a	0.50	% Mass	D 6584
Diglycerides[2]:		0.071	n/a	0.20	% Mass	D 6584
Triglycerides[3]:		0.024	n/a	0.15	% Mass	D 6584
Water & Sediment:		<0.005	0.050, max	0.020	% Volume	D 2709
Acid Number:		0.166	0.50, max	0.40	mg KOH/g	D 664
Visual Inspection:		1 @ 75.1	2, max	1	Haze	D 4176, Procedure 2
Relative Density at 60°F:		0.8775	n/a	0.85 – 0.90	n/a	D1298
Oxidation Stability (110 °C):		6.01	3.0, min	3.0	hrs	EN 14112
Flash point (closed cup):		151	93, min	93	°C	D 93
Alcohol Control	Option 1: Methanol	n/a	0.2, max	0.2	% Mass	EN 14110
	Option 2: Flash point	151	130	130	°C	D 93
Moisture[4]:		0.016	n/a	0.040	% Volume	E203
Cold Soak Filtration:		89	360	200	seconds	D 6751 Annex
Sulfur:		7.5	15	10	ppm	D 5453
Sodium & Potassium Combined:		<1.0	5, max	1.5	ppm (µg/g)	EN 14538
Calcium & Magnesium Combined:		<1.0	5, max	1.0	ppm (µg/g)	EN 14538
Phosphorus:		<0.0001	0.001, max	0.001	% Mass	D 4951
Carbon Residue:		<0.05	0.050, max	0.050	% Mass	D 4530
Sulfated Ash:		<0.005	0.020, max	0.020	% Mass	D 874
Kinematic Viscosity at 40 °C:		4.523	1.9-6.0	3.8 – 5.0	$mm^2/sec.$	D 445
Copper Corrosion (3 hrs at 50 °C):		1a	No. 3, max	No. 1	n/a	D 130
Distillation at 90% Recovered:		350	360, max	360	°C	D 1160
Cetane Number:		59.5	47, min	47	n/a	D 6890

[1,2,3,4] These are not ASTM D 6751 nor BQ9000 specification requirements.

* This result is an average of the 3 previous results for this product from this plant. In accordance with BQ-9000 requirements (Section 8), this test is performed periodically.

Prepared by:	Donnie R. Warehan	Laboratory Coordinator	Western Iowa Energy, LLC	4-7-09
	Name	Title	Company	Date

Please contact Inside Sales at Renewable Energy Group, Inc. at (888)734-8686 with any questions or comments about this product.

Figure B8. Certificate of Analysis for High Cloud biodiesel.

Form

Biodiesel Certificate of Analysis

REG

70790010090425T501

Replaces: NA

Lot Number: 70790010090425T501	Product Type: REG-9000-10
Inlet Seal Number: N/A	Date Reported: 042509

ASTM D 6751 Analysis of REG-9000™ Biodiesel

Test Parameter		Result	ASTM Limit	REG-9000 Limit	Units	Test Method (current revision)
Cloud point: (12)		14 °C (41°F)	Report	Report	°C	D 2500
Free Glycerin:		<0.005	0.020, max	0.014	% Mass	D 6584
Total Glycerin:		0.063	0.240, max	0.160	% Mass	D 6584
Monoglycerides[1]:		0.170	n/a	0.50	% Mass	D 6584
Diglycerides[2]:		0.078	n/a	0.20	% Mass	D 6584
Triglycerides[3]:		0.071	n/a	0.15	% Mass	D 6584
Water & Sediment:		<0.025	0.050, max	0.020	% Volume	D 2709
Acid Number:		0.35	0.50, max	0.40	mg KOH/g	D 664
Visual Inspection:		1	2, max	1	Haze	D 4176, Procedure 2
Relative Density at 60°F:		0.8755	n/a	0.85 – 0.90	n/a	D1298
Oxidation Stability (110 °C):		5.8	3.0, min	3.0	hrs	EN 14112
Flash point (closed cup):		>150	93, min	93	°C	D 93
Alcohol Control	Option 1: Methanol	n/a	0.2, max	0.2	% Volume	EN 14110
	Option 2: Flash point	>150	130	130	°C	D 93
Moisture[4]:		0.016	n/a	0.040	% Volume	E203
Cold Soak Filtration:		80	360	200	seconds	D 6751 Annex
Sulfur:		7.0	15	10	ppm	D 5453
Sodium & Potassium Combined:		<1	5, max	1.5	ppm (µg/g)	EN 14538
Calcium & Magnesium Combined:		<1	5, max	1.0	ppm (µg/g)	EN 14538
Phosphorus:		<0.001	0.001, max	0.001	% Mass	D 4951
Carbon Residue:		<0.05	0.050, max	0.050	% Mass	D 4530
Sulfated Ash:		<0.005	0.020, max	0.020	% Mass	D 874
Kinematic Viscosity at 40 °C:		4.662	1.9-6.0	3.8 – 5.0	$mm^2/sec.$	D 445
Copper Corrosion (3 hrs at 50 °C):		1a	No. 3, max	No. 1	n/a	D 130
Distillation at 90% Recovered:		354	360, max	360	°C	D 1160
Cetane Number:		65.8	47, min	47	n/a	D 613

[1,2,3,4] These are not ASTM D 6751 nor BQ9000 specification requirements.

Prepared by:	Jared Doiron	Lab Manager	REG-Houston	04-25-09
	Name	Title	Company	Date

Please contact Inside Sales at Renewable Energy Group, Inc. at (888)734-8686 with any questions or comments about this product.