Storage Stability of 50% Biodiesel Blends and Neat Biodiesel Produced in North America

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1 National Renewable Energy Laboratory
2 Enersol Resources

NREL is a national laboratory of the U.S. Department of Energy
Office of Energy Efficiency & Renewable Energy
Operated under Contract No. DE-AC36-08GO28308

Technical Report
NREL/TP-2A00-96381
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Suggested Citation
Fioroni, Gina M., Jennifer Cavaleri, Nimal Naser, Richard Nelson, and Robert L. McCormick. 2025. Storage Stability of 50% Biodiesel Blends and Neat Biodiesel Produced in North America. Golden, CO: National Renewable Energy Laboratory. NREL/TP-2A00-96381. https://www.nrel.gov/docs/fy26osti/96381.pdf.

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List of Acronyms

Bxx	Biodiesel blend containing xx volume percent biodiesel
IBP	Initial boiling point
ICN	Indicated cetane number
IP	Induction period
FBP	Final boiling point
NREL	National Renewable Energy Laboratory
TAN	Total acid number

Executive Summary

This study investigated aging of two petroleum diesel, seven B100, three B20 blends, and 14 B50 blends representing the full range of fuels in the North American market. Experimental measurement of fuel aging in storage was accomplished using 12-weeks at 43°C under the ASTM D4625 accelerated aging protocol. Twelve-weeks under D4625 aging represents approximately 12 months of storage time in clean, dry conditions. Samples were taken every two weeks for measurement of Rancimat induction period (IP), peroxide content, and TAN; and total insolubles were measured at the end of the 12-week test. As a baseline, the petroleum diesel samples showed no evidence of oxidation over the 12-week test.

B100 samples slowly oxidized over the 12-weeks to have reduced IP, below 2 h for the least stable samples. The three mid-range stability samples (initial IP 5-6 h) showed increased levels of peroxide but did not enter exponential growth. One sample with an initial induction period of roughly 3 h, showed levels of insoluble above the method reproducibility at the end of the test. These results show B100 samples with initial IP greater than 6 h do not exhibit significant acid number or insoluble formation over 12-weeks at D4625 conditions which represent 12 months in clean, dry storage.

B20 made from biodiesel sample number 31980 exhibited some peroxide formation over 12 weeks, as was also the case for this biodiesel when aged as B100. Neither of the other B20 samples evaluated showed peroxide formation and none of the samples showed increased TAN. None of these samples had measurable insoluble at the end of the test (<2 mg/100 mL). Consistent with our previous work, B20 blends with IP greater than 6 h are stable in D4625 storage for more than 12 weeks, indicating that real B20 fuels can be stored in a clean dry environment for at least 12 months.

For B50 blends, IP values decreased slowly over time, but in all cases remained above 2 h. Corresponding roughly to the B100 results, there was some growth in peroxides for the B50 samples made with lower IP B100, but with no significant change in acid number over time. One B50 sample showed insoluble formation at a value of 5.4±5.1 mg/100 mL, slightly above the repeatability of the test method. The B100 used for this B50 sample also produced a similar amount of insoluble when aged as B100. These results show B50 blends with initial IP greater than 6 h and made from a B100 with initial IP of over 6 h do not exhibit significant acid number or insoluble formation under D4625 conditions which represent 12 months in clean, dry storage.

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1. Introduction

Biodiesel produced from various fats, oils, and greases and consisting of fatty acid methyl esters is a significant contributor to diesel volumes in the United States, with consumption of nearly 2 billion gallons in 2024. ***(1)*** While there is some use of neat biodiesel (B100) the primary method of utilization is as a blend with petroleum diesel. Blend levels from 5 to 20 volume percent (B5 to B20) are common, ***(2)*** and many consumers have begun to request higher blend levels, such as B30 or B50. The quality of biodiesel is ensured by adherence to the ASTM D6751 Standard Specification for Biodiesel Fuel Blendstock (B100) for Middle Distillate Fuels, which is enabled by following quality guidelines of the BQ-9000 program. ***(3)*** Monitoring of B100 quality over many years demonstrates that quality is extremely high. ***(4)*** The quality of biodiesel blends up to B20 is ensured by adherence to the ASTM D7467 Standard Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20) and the B6-B20 grade in the ASTM D396 Standard Specification for Fuel Oils. The limit values for B100 in ASTM D6751 are based on those needed for successful blending of up to B20, but quality standards have not yet been set to cover the range from B21-B100 as finished fuels.

An important consideration for the use of blends over B20 is setting parameters that prevent formation of problematic degradation products over time. The oxidation stability specifications (often referred to as oxidation reserve or storage stability) have been put into place for B100 blendstock and for finished B20 blends as the primary control to prevent degradation over time in storage. Polyunsaturated fatty acid chains occur in biodiesel from nearly all common oils and fats used for biodiesel production. These can oxidize in the presence of air to form acids and insoluble gums in storage. ***(5,6,7,8)*** Antioxidant additives can significantly increase the oxidation reserve (the time where antioxidant is being consumed but the fuel is not oxidizing) and are commonly used in the market. ***(9,10)*** Oxidation is considered to occur in three phases as shown in Figure 1. ***(8)*** The goal of setting an oxidation stability standard for biodiesel is to maintain the fuel in Phase 1 where antioxidant protects the fuel from formation of deleterious degradation product (i.e., acids and non-volatile gums).

While studies have been conducted of the long-term storage of B20 and lower blends, as well as B100, ***(6,8,11)*** and were used as the primary data to set the current ASTM standards for B100 blendstock and B20 in the 2008-2015 timeframe, the present study extends this to cover B50 while also acquiring additional data on B100 and B20 on fuels representative of those in the current market.

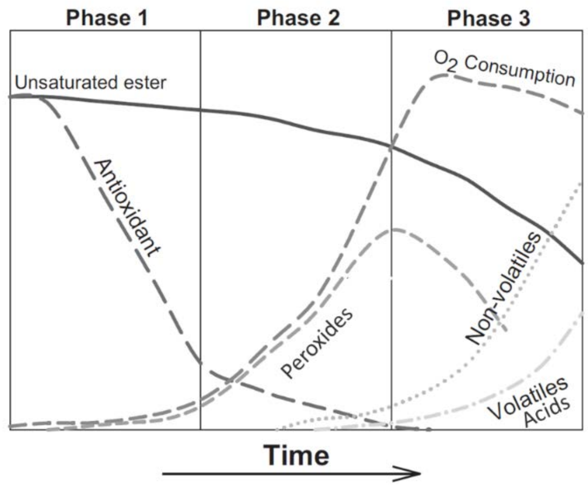

Figure 1. Phases of biodiesel oxidation in storage. *(8)*

1.1 Accelerated Measurements of Oxidation Stability

While changes in biodiesel or biodiesel blends in storage takes place over time (i.e. weeks or months) as the fuel is exposed to oxygen, measurement of oxidation stability for specification purposes must use accelerated tests that can be completed in hours. An accelerated test known as Rancimat induction period (or induction time, sometimes referred to as the Oil Stability Index, OSI) is used in ASTM and CEN specifications for biodiesel and biodiesel blends. In this test, the fuel is heated at 110°C while air is bubbled through the fuel at a high space velocity ensuring oxygen replete conditions. ***(12)*** The air then passes through deionized water in a separate tube. Electrical conductivity of the water is monitored and when all antioxidants are consumed and oxidation begins, volatile acids are formed and carried over to the water causing a sharp increase in electrical conductivity. The time between the start of the experiment and this sharp increase in conductivity is called the induction period (IP) and specifications require a minimum IP to help ensure adequate storage stability.

The Rancimat test is thought of as measuring oxidation reserve, defined in Equation (1):

$$Induction\ period \propto \frac{Antioxidant\ Concentration}{Radical\ Initiator\ Concentration + Bisallylic\ Site\ Concentration} \qquad (1)$$

The *bis*-allylic sites in polyunsaturated fatty acids (methylene groups between two double bonds) exhibit weaker C-H bonds that typical methylene groups making them more reactive for oxidation or free radical reactions. Radical initiators in the fuel include dissolved metals such as iron, copper and zinc. Because the test is conducted at much more aggressive conditions than the fuel will see in actual storage and handling (much higher temperature and saturated with oxygen), and because real-world storage conditions can vary, there is not a 1:1 correspondence between IP and storage life.

Current ASTM standards require B100 have a minimum 3 h induction time (see ASTM D6751) and that blends of B6 to B20 have a minimum 6 h induction time (see ASTM D7467). The 3 h minimum requirement for B100 is based on work done more than a decade ago showing that B100 with this level of stability will reliably produce B20 blends having a 6 h induction time as shown in Figure 2. ***(6)*** The IP specifications were set with the main controls for the blends being at the parent fuel level prior to blending and with the B20 specification values serving primarily as a means to check fuel quality of the blend if the parent fuel quality is not known. The B20 specifications require B100 to meet D6751 and the diesel fuel meet D975 prior to blending, except for diesel properties that biodiesel can be used to improve (i.e. lubricity, cetane, sulfur). This facilitates blending B100 into diesel fuel without a driving need to confirm the properties of the final blend before shipping to a customer, as is commonly done at fuel terminals in North America. This is an important consideration as specifications for finished blends over B20 are considered.

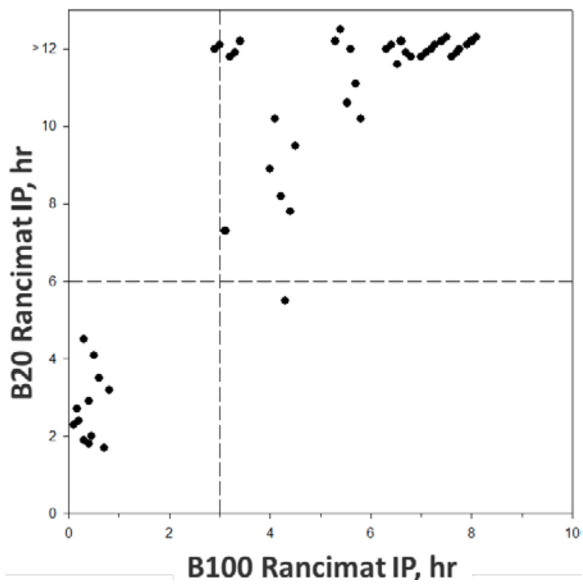

Figure 2. Relationship between B100 Rancimat induction time and induction time for B20 blends in petroleum diesel. *(6)*

1.2 Long-Term Stability Studies

The standard test for long-term stability studies is ASTM D4625 Standard Test Method for Middle Distillate Fuel Storage Stability at 43°C (110°F). Here a 400 mL sample of the fuel is held in a glass bottle open to the air via a "U" shaped tube to prevent ingress of dust or other particles. Thus, the test is not accelerated in terms of oxygen availability but is moderately accelerated by using a higher temperature than is typical of fuel storage conditions (which are typically 15 to 25°C). Evidence is presented in the published test method that one week at 43°C is equivalent to one month at 21°C, or a roughly fourfold acceleration. The sample is held for as many weeks as desired. The method as written for petroleum derived fuels is intended to measure the formation of gums and insolubles. For a 12-week test one might age 4 jars of the same fuel, harvesting one jar every three weeks for filtration to measure insolubles formation.

As implemented at NREL for biodiesel stability studies fuel is removed periodically during the test using a pipette to obtain sample for measurement of peroxide content, total acid number (TAN), and Rancimat IP. At the end of the aging period the remaining fuel is filtered to measure insolubles formation. ***(8,10,11)*** These studies have shown IP slowly decreasing during storage with slow growth in peroxide content. When IP is below approximately 2 h, peroxide formation can rapidly increase, followed by increasing TAN. Aged fuels failing the upper limit for TAN in D6751 or D7467 will sometimes show measurable levels of insolubles. B20 blends having at least the required 6 hr IP did not show high peroxide, TAN, or measurable insolubles for at least 12 weeks of aging, or a simulated one year of clean, dry storage. ***(8)***

In the present study, we age B50 blends made from market representative B100 for 12 weeks with measurement of IP, peroxide, and TAN at 2-week intervals, and total insolubles measured at the end of the test. The base diesel fuels, B100 samples and a few B20 blends are also aged to provide context.

2. Materials and Methods

2.1 Fuel Selection and Properties

Diesel Fuels

Two petroleum-derived No. 2 diesel fuels meeting the requirements of ASTM D975 Standard Specification for Diesel Fuel were acquired from pipeline terminals. The properties of these fuels are shown in Table 1. The fuels were selected to represent a range of base diesel fuel properties in the North American market of varying cloud point and aromaticity, as those properties are the most likely to impact the solubility of potential biodiesel blend aging products. A fuel representing wintertime fuels (cloud point -20°C) having total aromatics of 37 wt% was selected, along with a fuel representing summertime fuels (cloud point -8.7°C) having much lower aromatics of 21 wt%. These fuels are considered relatively stable with PetroOxy induction times of 100 min or longer.

Table 1. Properties of Petroleum diesel fuels used in this study

Property	Method	Units	Diesel 30902	Diesel 31956
Density at 15°C	D4052	g/ml	0.8596	0.8300
Flashpoint	D6450	°C	61.7	60.7
Kinematic Viscosity at 40°C	D7042	mm^2/s	2.639	2.413
Cloud point	D5773	°C	-20.3	-8.7
Cetane Number (ICN)	D8183		42	51.8
Total Aromatics	D5186	Mass%	37.2	21.0
MonoAromatics	D5186	Mass%	28.7	18.1
Polynuclear Aromatics	D5186	Mass%	8.5	2.9
Carbon Residue, 10% bottoms	D524	wt%	0.13	0.06
Sulfur	D5453	ppm	10.94	8.38
Distillation				
IBP	D86	°C	162.8	162.6
50% (Diesel)		°C	263.6	255.9
90% (Diesel)		°C	328.7	333.3
FBP		°C	359.9	360.4
Petroxy Induction time	D7545	min	120	100

Biodiesel Fuels

An important consideration for the ASTM specifications for B50 and B100 is to provide data covering fuels that span those meeting the current standards and that are representative of those in the commercial marketplace. NREL began publishing a report on the quality of the as-produced B100 in the North

American market in 2017. ***(4,13,14)*** The quality of the B100 has improved dramatically over the last 10 years ***(4)***, and the properties of B100 in the current market for acid number, induction period, and cloud point from 2017-2023 can be in Figures 3, 4, and 5.

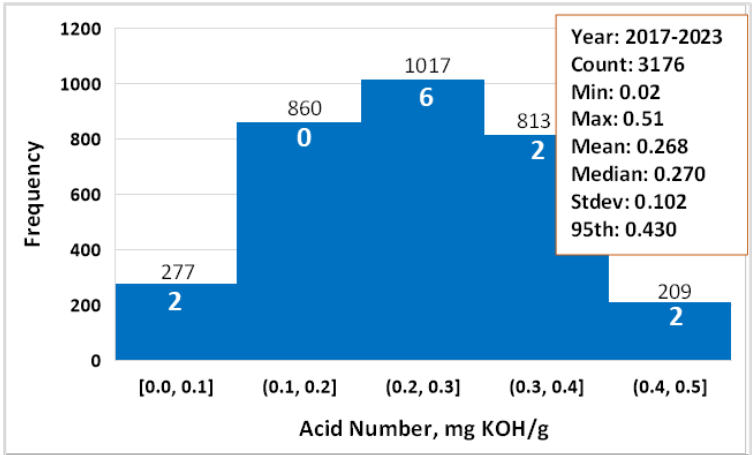

Figure 3. Acid number results from BQ-9000 survey data over seven years. *(4,13)*

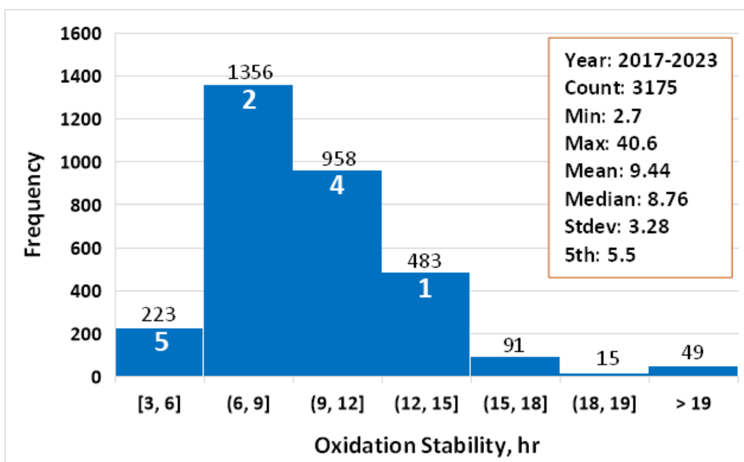

Figure 4. Rancimat IP results from BQ-9000 survey data over seven years. *(4,13)*

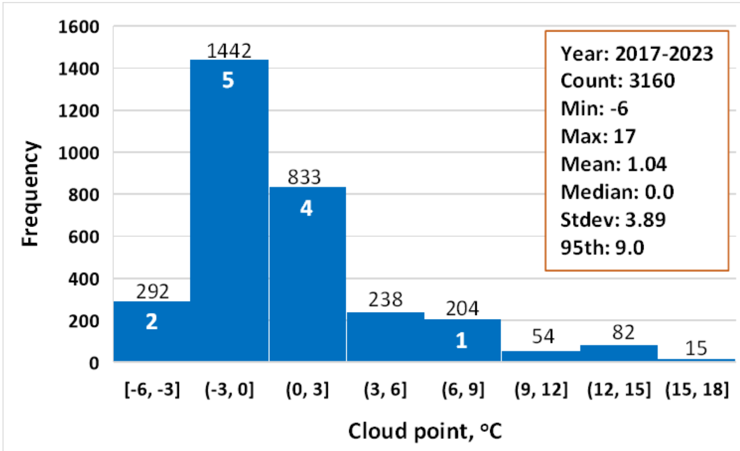

Figure 5. Cloud point results from BQ-9000 survey data over seven years. *(4,13)*

Twelve B100 samples were acquired from biodiesel producers as 10-gallon samples which were stored indoors at NREL in 10 x 1 gallon fully filled, nitrogen purged, spun aluminum bottles. Property data were measured by Iowa Central Fuel Testing Laboratory in Fort Dodge, Iowa and detailed properties of all 12 are shown in Appendix A; all met the ASTM D6751 specification. Figure 4, which shows the frequency distribution of IP for the U.S. market, also shows the distribution of IP for the original 12 samples as a white number on each bar, demonstrating that the B100s are representative for IP.

From this list of 12, seven samples were selected for aging in this study based on the concept of being representative of the current U.S. biodiesel market and production techniques. An abbreviated list of properties for these seven fuels is shown in Table 2. The samples have cloud points ranging from -4 to 7°C, suggesting that they encompass the primary feedstocks used in the U.S. market today. Total glycerin and monoglycerides for Sample 31985 are very low, suggesting it is a distilled biodiesel. Acid numbers range from 0.07 to 0.43 which span the BQ-9000 values for acid number. Selected fuels include two with high IP (11.4, 10.8), three with medium IP (9.2, 6.0, and 5.7) and two with low IP (4.8 and 2.8). NREL repeated TAN and IP measurements for these samples about one month after they arrived and these results are also shown in Table 2 and show good agreement with those measured when the samples were acquired, considering method reproducibility.

Blends of biodiesel into the two conventional diesel fuels were prepared volumetrically. All seven B100 samples were blended into both diesel fuels at 50 vol%. B100s 31980, 31981, and 31986 were also blended into diesel fuel 31956 at 20 vol%. Additionally, all B100 and both diesel fuels were aged.

Table 2. Properties of B100 samples used in this study (for additional details see Appendix A).

Sample#	31980	31981	31985	31986	31987	31989	32815
S, ppm	0.5	6.8	1.3	6.7	14.6	3	4.2
CSFT, s	95	118	99	96	101	123	107
Flashpoint, °C	115	153	177	165	157	175	149
Viscosity, cSt	4.028	4.498	4.248	4.097	4.585	4.305	4.354
Cloud Point, °C	-1	2	-4	2	1	7	0
TAN, mg KOH/g[a]	0.07±0.06	0.43±0.13	0.26±0.10	0.09±0.07	0.25±0.10	0.29±0.11	0.29±0.11
NREL TAN, mg KOH/g	0.05±0.05	0.26±0.10	0.19±0.09	0.02±0.04	0.22±0.10	0.16±0.09	0.18±0.09
Total Glycerin, wt%	0.098	0.129	0.018	0.024	0.111	0.028	0.136
Monoglycerides, wt%	0.349	0.312	0.048	0.068	0.349	0.098	0.388
IP, h[a]	5.7±1.5	10.8±2.4	11.4±2.5	2.8±0.9	4.8±1.3	9.2±2.1	6.0±1.5
NREL IP, h	5.6±1.4	10.6±2.4	11.1±2.5	3.5±1.0	5.4±1.4	7.8±1.9	6.2±1.6

[a] ASTM method reproducibility shown for TAN results, CEN method reproducibility is shown for Rancimat results.

2.2 Test Methods

Twelve weeks of D4625 aging at 43°C was accomplished in a Jeio Tech model ON-22 natural convection oven that can hold up to 48 jars. For this study we aged 26 jars in total. The jars used were borosilicate glass with bent glass tubes through the lid to ensure the samples are exposed to air. 400 mL of each B100, diesel fuel or blend was added to the jars. A photograph of the jars in the ovens is shown in Figure 6. At two-week intervals, 28 mL of fuel was removed from each jar via a glass pipet for characterization. This included Rancimat IP using EN 15751, TAN using ASTM D664 Method B, and peroxide content using AOCS Method AOCS Cd 8b-90 modified to be a potentiometric titration. PetroOxy induction time was measured for the diesel fuels using ASTM D7545 Standard Test Method for Oxidation Stability of Middle Distillate Fuels—Rapid Small Scale Oxidation Test (RSSOT). Filtration at the end of D4625 aging was performed according to the D4625 method using Whatman GFIF glass fiber filters, 47 mm diameter, catalog number 1825-047. In our experience the isooctane solvent called for in the method does not fully remove some biodiesel components from the insolubles on the filter. Isopropyl alcohol was used instead of isooctane. The total volume of fuel remaining in the jars was recorded prior to each filtration and was about 225 mL. The measured volume was used to calculate total insoluble in mg/100 mL.

Figure 6. Samples in D4625 jars in the aging oven.

3. Results

3.1 B100 and Diesel Fuel Aging Results

For the petroleum diesel samples, there was no development of peroxides, acids, and insolubles (<1 mg/100 mL – see Appendix B) were likely below detection at the end of the test. PetroOxy induction times for sample 30902 were 120 minutes at the start of aging and 109 minutes after 12 weeks. For sample 31956, the values were 100 minutes at the start of aging and 94 minutes after 12 weeks.

Aging results for the B100 samples are shown in Figure 7. Note that the colors and symbols in these charts are used consistently to identify the B100 (whether neat or in blend) throughout this report. For IP, we observe a slow decline, as seen previously in aging a more limited number of B100 samples. ***(8)*** All samples showed a slow loss in IP as they oxidized in Phase 1 (consumption of antioxidant). The four lowest stability samples (lowest initial IP: 31980, 31986, 31987, and 32815) show some growth in peroxide content, however, exponential peroxide growth indicating rapid oxidation did not occur, such that oxidation remained in Phase 1. Sample 31980 formed several hundred ppm of peroxide – even though its initial IP was over 5 h. While this level is not considered problematic and we cannot draw definitive conclusions as to the cause of this observation, a possible explanation is that the antioxidant in this sample was much less effective than that used in the other samples. Note that antioxidant additives, if used, were added to the B100 by the producer. There is a small TAN increase of almost 0.1 mg KOH/g for the two lowest initial stability samples.

Filtration results for 12 weeks of D4625 aging (representing 12 months of storage) are shown in Table 3 along with the D4625 method repeatability (r) and reproducibility (R). Only one sample (31986) showed total insolubles above the reproducibility (R) of the test method after 12 months of simulated aging while the remaining samples were low but over the repeatability (r) of the test method. The present results reveal that samples with IP > 6 h showed minimal formation of peroxides and insolubles were below the reproducibility of the test.

Table 3. Total insolubles results for B100 samples after 12-week aging.

Sample #	Total Insolubles, mg/100 mL	Repeatability, r	Reproducibility, R
31980	2.4	1.3	3.4
31981	2.3	0.9	3.3
31985	4.6	1.4	4.7
31986	5.2	1.4	5.0
31987	3.7	1.3	4.2
31989	2.8	1.0	3.7
32815	2.3	0.9	3.3

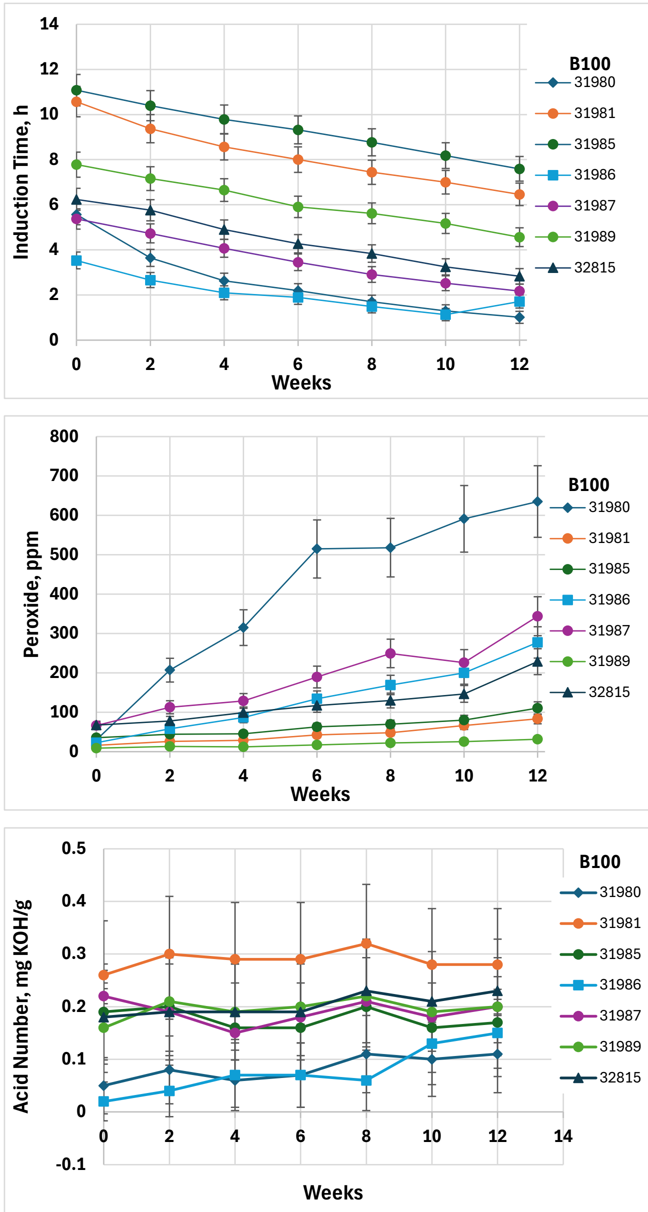

Figure 7. Results of aging B100 samples. Rancimat IP (top panel), peroxide content (middle panel), TAN (bottom panel). Error bars are ASTM method reproducibility.

3.2 Biodiesel Blend Aging Results

Initial induction times for the B20 and B50 blends are shown in Figure 8, along with B100 IP. As anticipated, the B20 blends have higher initial induction times than B50 blends. B50 blends in diesel 30902 (low cloud, high aromatics) have systematically slightly higher IP values than blends into diesel 31956 (high cloud, low aromatics) indicating the diesel fuel selection can have an impact, although the impact is much smaller than that of the biodiesel. We speculate that the higher aromatics content of fuel 30902 increases the available radical scavengers, increasing the measured IP. Time zero peroxide and TAN were very low for all blends.

Aging results for B20 samples prepared in diesel 31956 are shown in Figure 9. These are for B100 31980, 31981, and 31986 and intended to provide a tie-back to our previous work with B20. ***(8,11)*** The B20 made from biodiesel 31980 exhibited a small increase in peroxides over 12 weeks, as was also the case for this biodiesel when aged as B100. Neither of the other B20 samples showed peroxide formation and none of the samples showed increased TAN. None of these samples had total insolubles above the reproducibility (all <2 mg/100 mL – see Appendix B) at the end of the test. Consistent with our previous work, B20 blends with IP greater than 6 h do not show significant acid number or insolubles formation in D4625 storage for more than 12 weeks, indicating they can be stored in a clean dry environment for at least 12 months. ***(11)***

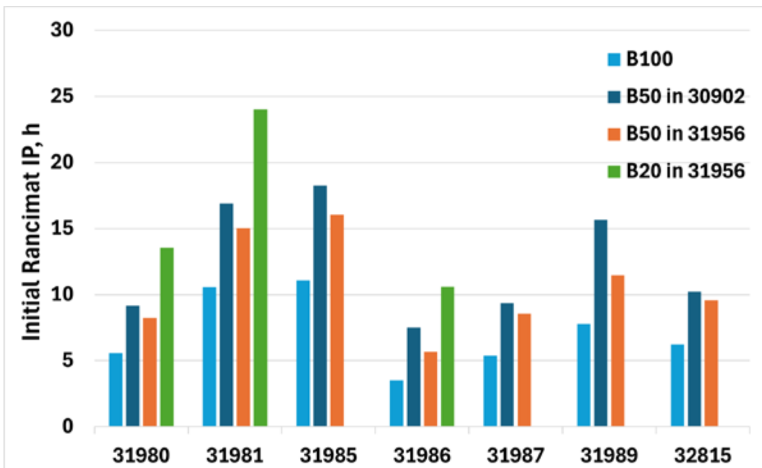

Figure 8. Initial Rancimat IP for B20 and B50 blends.

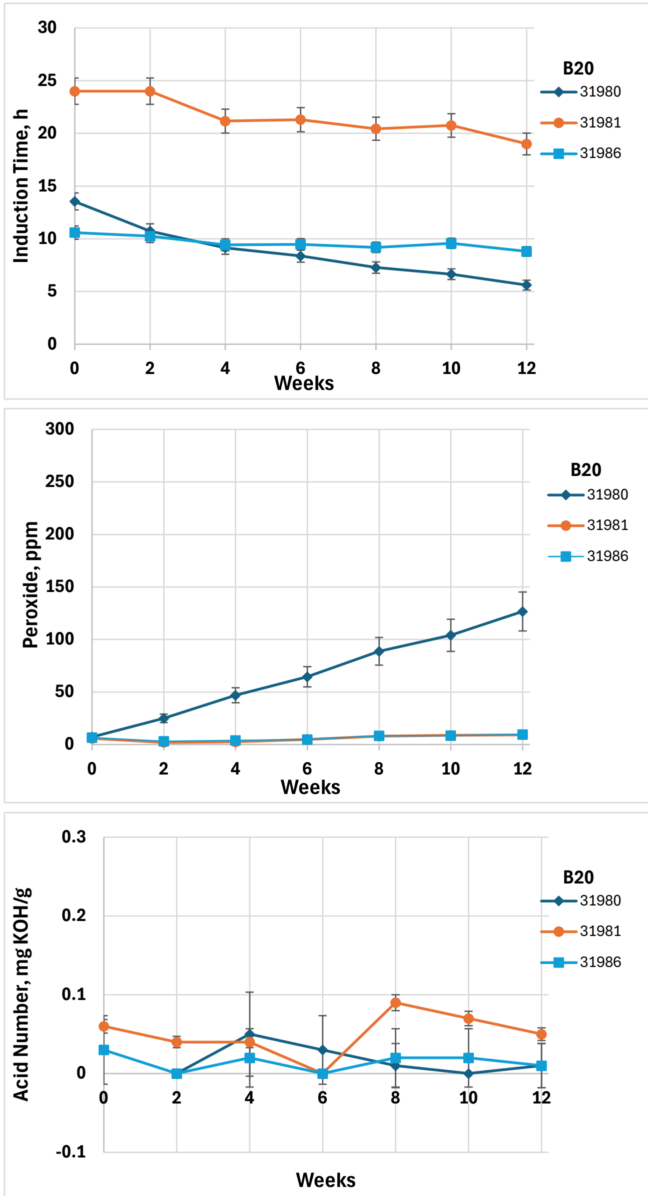

Figure 9. Aging results for B20 blends into diesel 31956. Rancimat IP (top panel), peroxide content (middle panel), TAN (bottom panel). Error bars are ASTM method reproducibility.

Figures 10 and 11 show aging results for B50 blends into diesel fuels 30902 and 31956, respectively. While initial IP values are slightly higher for blends into 30902, the results for the two petroleum blendstocks are very similar. IP values decrease slowly over the 12 weeks of D4625 aging, but in all cases remained above 2 h. There was a larger growth in peroxide for fuels containing B100 31980, and to a lesser extent for 31987, 31985, and 31986 in diesel 31956. None of the B50 samples showed significant change in acid number over time. One B50 sample, 31986 in diesel 30902 showed insolubles formation at a value of 5.4±5.1 mg/100 mL, slightly over the reproducibility of the test method. B100 31986 produced a similar amount of insolubles when aged as B100, and its initial induction period was roughly 3 h. For all other B50 samples, insoluble levels were less than approximately 3 mg/100 mL (see

Appendix B), all below method reproducibility. B100 31986 has the lowest initial IP at roughly 3 h, and its B50 blends did not show formation of harmful levels of peroxide or acids over the 12-week storage period. While insolubles formation slightly over reproducibility was observed for the B50 blend with B100 31986 in diesel 31956, levels were much lower than observed in previous work on B20 samples that had oxidized to fail TAN. ***(11)*** These results show B50 blends with initial IP greater than 6 h and made with an initial B100 IP of over 6 h do not exhibit significant acid number or insoluble formation under D4625 conditions which represent 12 months in clean, dry storage.

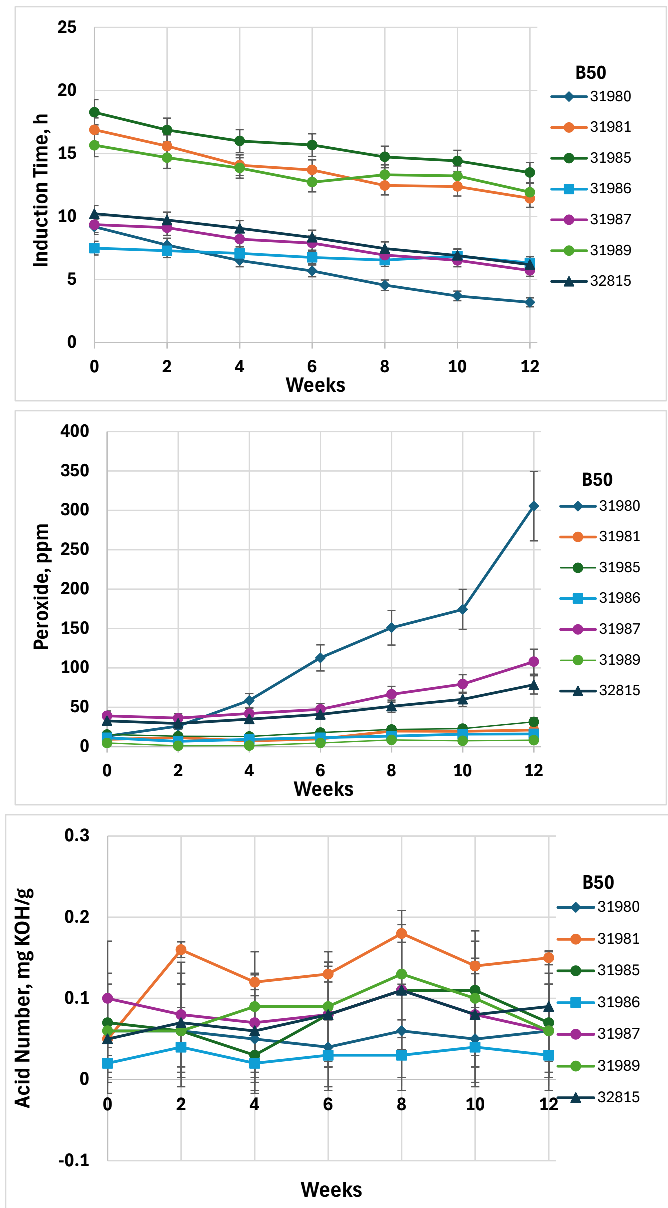

Figure 10. Aging results for B50 blends into diesel 30902. Rancimat IP (top panel), peroxide content (middle panel), TAN (bottom panel). Error bars are ASTM method reproducibility.

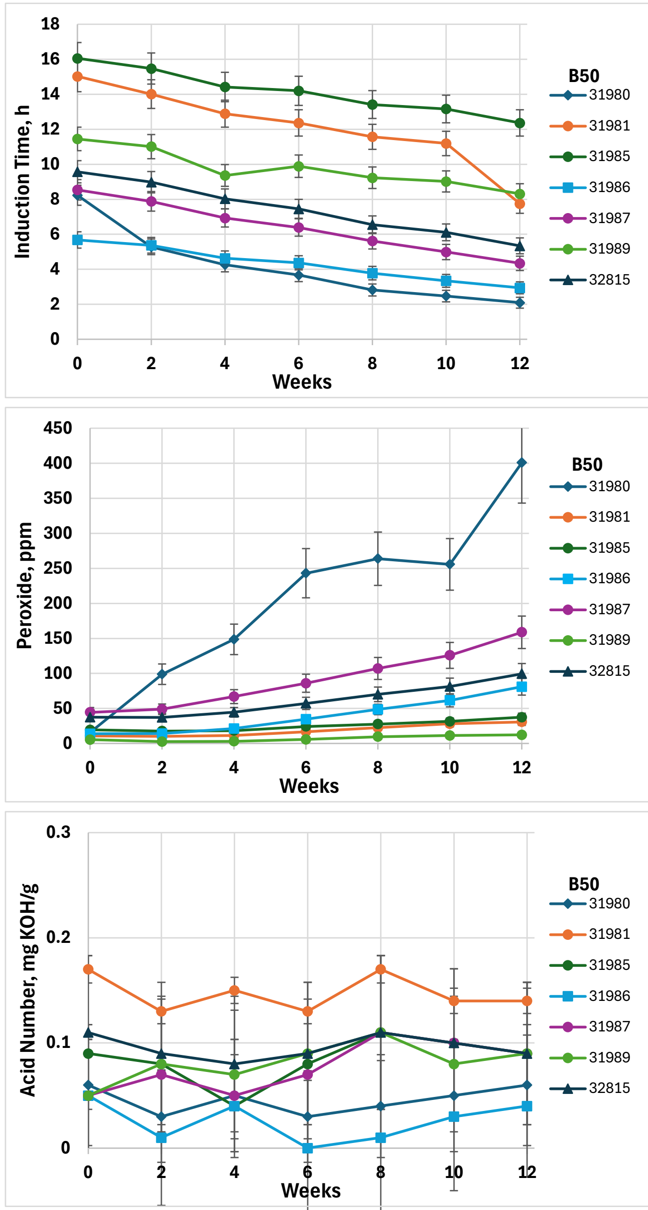

Figure 11. Aging results for B50 blends into diesel 31956. Rancimat IP (top panel), peroxide content (middle panel), TAN (bottom panel). Error bars are ASTM method reproducibility.

4. Summary and Conclusions

In order to provide data needed for consideration of ASTM specifications for biodiesel blends over 20% biodiesel by volume (B20), this study investigated aging of 2 petroleum diesel, 7 B100, 3 B20 blends, and 14 B50 blends representing the full range of fuels in the North American market. The fuels were aged for 12-weeks at 43°C using the ASTM D4625 accelerated aging protocol. Twelve-weeks under D4625 aging represents approximately 12 months of storage time in clean, dry conditions. Samples were taken every two weeks for measurement of IP, peroxide content, and TAN; and total insoluble were measured at the end of the 12-week test. As a baseline, the petroleum diesel samples showed no evidence of oxidation over the 12-weeks.

B100 samples slowly oxidized to have reduced IP, with the least stable samples having an IP below 2 h after 12-weeks. The three mid-range stability samples (initial IP 5-6 h) showed increased levels of peroxide but did not enter exponential growth (remained in Phase 1). One sample with an initial induction period of roughly 3 h, showed levels of insolubles above the method reproducibility at the end of the test. These results show B100 samples with initial IP greater than 6 h do not exhibit significant acid number or insolubles formation under D4625 conditions.

For B20 blends, the fuel made from biodiesel 31980 exhibited some peroxide formation over 12 weeks, as was also the case for this biodiesel when aged as B100. Neither of the other B20 samples showed peroxide formation and none of the samples showed increased TAN. None of these samples had measurable total insolubles at the end of the test (<2 mg/100 mL). Consistent with our previous work, B20 blends with IP greater than 6 h do not show significant acid number or insolubles formation in D4625 storage for more than 12 weeks, indicating they can be stored in a clean dry environment for at least 12 months. ***(11)***

For B50 blends, while initial IP values are slightly higher for blends into petroleum diesel 30902, the results for the two petroleum blendstocks are very similar. IP values decrease slowly over time, but in all cases remained above 2 h. Corresponding roughly to the B100 results, there was some growth in peroxides for the B50 samples made with lower IP B100, but no significant change in acid number over time. One B50 sample, 31986 in diesel 30902 showed insolubles formation at a value of 5.4±5.1 mg/100 mL, slightly above the reproducibility of the test method. B100 31986 produced a similar amount of insolubles when aged as B100. These results show B50 blends with initial IP greater than 6 h and made with an initial B100 IP of over 6 h do not exhibit significant acid number or insoluble formation under D4625 conditions representing 12 months in clean, dry storage.

5. References

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Appendix A: Properties of 12 B100 Samples

Table A-1. Properties of 12 B100 samples screened for use in this study.[a] Samples in bold font selected for aging.

#	S, ppm	CSFT[b], s	Flashpoint, °C	Viscosity, cSt	Cloud Point, °C	TAN, mg KOH/g	Total Glycerin, wt%	Monoglycerides, wt%	P, ppm	T90, °C	Na+K, ppm	IP, hr
31980	**0.5**	**95**	**115**	**4.028**	**-1**	**0.07**	**0.098**	**0.349**	**0.41**	**351**	**0.1**	**5.7**
31981	**6.8**	**118**	**153**	**4.498**	**2**	**0.43**	**0.129**	**0.312**	**0.36**	**353**	**0.8**	**10.8**
31982	1.9	103	127	4.437	-3	0.39	0.091	0.294	0.93	353	0	12
31984	0.4	89	165	4.018	-1	0.37	0.058	0.176	0.13	351	0.3	5.9
31985	**1.3**	**99**	**177**	**4.248**	**-4**	**0.26**	**0.018**	**0.048**	**0.23**	**352**	**0.3**	**11.4**
31986	**6.7**	**96**	**165**	**4.097**	**2**	**0.09**	**0.024**	**0.068**	**0.78**	**350**	**0**	**2.8**
31987	**14.6**	**101**	**157**	**4.585**	**1**	**0.25**	**0.111**	**0.349**	**0.43**	**354**	**0.8**	**4.8**
31988	0.5	156	157	4.066	-2	0.27	0.107	0.349	1.61	351	0.1	7.8
31989	**3.0**	**123**	**175**	**4.305**	**7**	**0.29**	**0.028**	**0.098**	**0.73**	**349**	**0.1**	**9.2**
32811	0.4	87	151	4.043	0	0.21	0.052	0.15	0.51	351	0	6.2
32812	11.0	109	145	4.474	1	0.41	0.096	0.330	0.27	354	0.6	14
32815	**4.2**	**107**	**149**	**4.354**	**0**	**0.29**	**0.136**	**0.388**	**0.42**	**354**	**0.6**	**6**

[a] For all samples: Ca+Mg ≤ 0.1 ppm, methanol ≤ 0.08 wt%, water & sediment ≤ 0.02 vol%, sulfated ash ≤ 0.005 wt%, Cu corrosion 1A, carbon residue ≤ 0.0028 wt%, and free glycerin ≤ 0.008 wt%.

[b] Cold soak filtration time

Appendix B: Total Insolubles Results for all Samples

Table A-2. Total insoluble results for all samples after 12-week aging.

Sample #	Total Insolubles, mg/100 mL	Repeatability, r	Reproducibility, R
31980 B100	2.4	1.4	3.4
31981 B100	2.3	0.9	3.3
31985 B100	4.6	1.4	4.7
31986 B100	5.2	1.4	5.0
31987 B100	3.7	1.3	4.2
31989 B100	2.8	1.0	3.7
32815 B100	2.2	0.9	3.3
30902 B0	0.7	0.5	1.8
31956B B0	0.2	0.3	1.0
31980/30902 B50	2.1	0.9	3.2
31980/31956B B50	0.3	0.3	1.2
31981/30902 B50	0.2	0.2	0.9
31981/31956B B50	0.4	0.4	1.5
31985/30902 B50	0.9	0.6	2.1
31985/31956B B50	0.3	0.4	1.3
31986/30902 B50	5.4	1.4	5.1
31986/31956B B50	3.3	1.1	4.0
31987/30902 B50	1.2	0.7	2.4
31987/31956B B50	1.3	0.7	2.5
31989/30902 B50	1.0	0.6	2.2
31989/31956B B50	2.1	0.9	3.2
32815/30902 B50	0.9	0.6	2.1
32815/31956B B50	0.5	0.4	1.5
31980/31956B B20	1.4	0.7	2.6
31981/31956B B20	1.8	0.8	2.9
31986/31956B B20	1.2	0.7	2.4