Effect of in Vehicle Storage
On B10 Diesel Quality

A Joint Industry Study

Sponsored by

The Petroleum Industry, Automotive
and Fuel Injection System Industries

1 December 2010

ISBN 978-2-87567-009-0

9 782875 670090 >

Effect of in Vehicle Storage on B10 Diesel Quality

Paul Lacey
Delphi Diesel Systems

Paul Greening
ACEA

Ken Rose
CONCAWE

Toshihiko Omori
DENSO Corporation

Theo Kweekel
Kuwait Petroleum Research and Technology BV

Emmanuel Jean
Intertek Caleb Bret

I. ABSTRACT

The oxidation stability of diesel fuel containing Fatty Acid Methyl Ester (FAME) is considerably reduced in comparison to conventional diesel. In general, a minimum stability requirement is defined for biodiesel fuels at the point of sale after which aging may occur during storage and use, affecting quality when consumed. Such changes can result in formation of insoluble materials and acids, which may create materials compatibility issues, filter plugging, corrosion, durability problems and deposit formation. At present, the rate of fuel aging and the factors that affect aging during vehicle storage have not been adequately investigated. Results of this study show that the aging rate is strongly dependent on storage conditions, with large variations between vehicle types. Particularly rapid changes in stability occurred in passenger vehicles compared to light-duty vans. The study raises a number of questions that require further study, including why such large variation occurred between different vehicle designs and also why the rate of biodiesel aging decreased following very rapid change in the first weeks.

II. INTRODUCTION

In the past few years diesel fuel quality has undergone rapid change, driven by the need to reduce exhaust emissions and minimise carbon footprint. In addition, advanced biofuels contribute to long term energy security and provide economic benefits derived from local energy production. For these reasons increasing penetration of FAME in many markets is quite certain.

The EN 590 standard for European On-Road diesel has been updated to allow use of up to 7.0 Vol% FAME (B7), while CEN has started development of a standard for B10. Higher FAME levels (B20-30) are allowed in some countries and in niche-market applications. In the US ASTM D975 may contain up to 5.0 Vol% FAME, while ASTM D7467 allows between 6 and 20 Vol% FAME for fleet applications. These standards contain oxidation stability requirements and these must continue to be evaluated in order to provide consistently high fuel quality in the marketplace.

Care must be taken with FAME containing fuels during vehicle storage and long term parking. Most standards define fuel quality at the point of sale, while those for B100 can only apply before blending with diesel occurs, which may be some time prior to sale. It is likely that aging may occur within the vehicle, particularly in applications that only see occasional use in a warm environment.

Insoluble polymers and products of age-related degradation can lead to filter clogging and deposits which can occur anywhere in the vehicles fuel system. The most likely consequences are filter plugging and deposits around injectors where high temperatures can cause a reaction of the organic material, leading to a build-up of deposit. Formation of acidic products may affect durability and also cause materials compatibility issues in some cases. It is for these reasons that vehicle and equipment manufacturers have issued joint statements limiting components exposure to FAME.(1, 2) To combat these effects, it is essential that a sufficient level of fuel stability be mandated during the ongoing standards development processes around the world.

A number of previous studies (3, 4) have measured the changes that occur in the fuel during extended storage. In general, these studies evaluated conditions simulating a well controlled commercial storage environment in bulk tanks or drums. Such conditions do not reflect those in a practical application, in which the fuel will be exposed to high temperatures, circulation, aeration and potentially exposure to moisture and catalytic materials. Nonetheless, the results (3, 4) did note a significant effect on stability caused by “simple and occasional agitation of the product in the presence of air”.

DGMK Reports 639 (B5 fuels) and 639-2 (B10 fuels) provide some data for fuel stored in real world conditions.(5) The results also indicated that certain B5 fuels can lead to some increased formation of injector deposits. The oxidation stability of B10 fuel removed from the tank of one slow driving mode vehicle decreased significantly following only 21 days in vehicle storage.

In vehicle storage is likely to affect aging during real world use, along with initial fuel quality and FAME feed type. The present study measures the change in fuel quality that occurs during warm climate storage conditions experienced in vehicles that are only occasionally operated with three B10 fuels having different initial fuel qualities. These comparatively severe storage conditions could be experienced by a vehicle parked at an airport or on a dealer forecourt for an extended period. A number of measurements have also been performed on fuel samples that were stored in sealed plastic and metal containers to serve as a baseline for comparison.

III. TEST METHODOLOGY

TEST VEHICLES - The vehicle test matrix is summarized in Table 1. It consists of three light commercial vehicles (denoted as A, C and E) from a single manufacturer (X) and three passenger cars (denoted as B, D and F) from two different manufacturers (Y and Z). Each of the vehicles is fitted with high density polyethylene (HDPE) fuel tanks and a common rail fuel injection system.

The vehicles were parked under identical ambient conditions in direct sunlight in Martigues, France. The ambient temperature surrounding the vehicles as well as the temperature in the air space above the fuel in the tank of each vehicle type was recorded continuously and the averaged trend is plotted in Figure 1. No significant differences were observed between the in tank and ambient conditions measured between the vehicles. A more detailed temperature profile taken from vehicle B is provided in Figure A-1 in the Appendix.

Prior to testing, all residual fuel was removed from the vehicle tanks, which were then rinsed twice with the appropriate test fuel. The fuel filters replaced to avoid cross contamination and removal of semi soluble materials by FAME. A total volume of 45 L of the appropriate fuel was placed in each vehicle tank, as detailed in Table 1. Fuel samples were drawn from the fuel line after the fuel filter (in vehicles A, C, D and E) or directly from the tanks (in vehicles B and F) at regular intervals and placed in HDPE sample bottles. The samples were stored the dark in an air conditioned laboratory until analyzed. The analysis was scheduled to occur with minimum time delay following sampling from the vehicle.

Table 1: Summary of Test Vehicles

Fuel Type	Vehicle	Manuf.	Type	Tank Size, L	Tank Material	Lift Pump	Tank Breather opening pressure
High Stability	A	X	Light Com.	80	HDPE	None	Normally Open
	B	Y	Pass. Car	62	HDPE	None	35 mBar
Medium Stability	C	X	Light Comm.	80	HDPE	None	Normally Open
	D	Z	Pass. Car	60	HDPE Monolayer	In Tank	10 mBar
Low Stability	E	X	Light Comm.	80	HDPE	None	Normally Open
	F	Y	Pass. Car	62	HDPE	None	35 mBar

The vehicles were operated intermittently to simulate the occasional use that is likely to occur in real world operation. Prior to aging, but after taking the week 0 fuel sample, each vehicle was idled for a total of 6 hours. The engine was revved to 3000RPM for 1 minute every hour during the idling period, to minimize potential for deposit build up. This extended idling cycle only occurred at week 0. No fuel was added after the idling cycle was completed and the tanks were not opened until the end of the 27 week period. Following this, the vehicles were operated at idle for 15 minutes twice per week during the 27 week aging period. The vehicles successfully started and idled throughout the 27-week testing period.

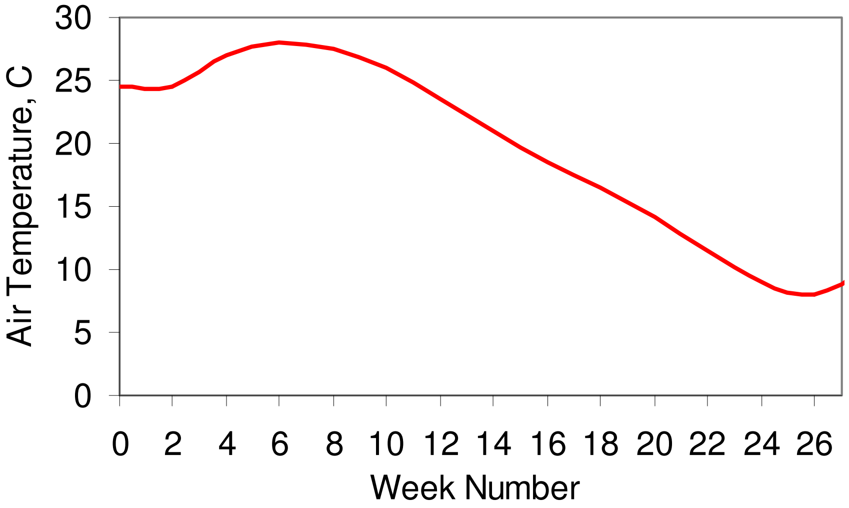

Figure 1: Average Ambient Temperature During Fuel Aging by Calendar Week

AGING IN DRUMS – A sample of each of the three fuels was placed into sealed epoxy lined steel drums, which were then stored in an indoor air conditioned environment. Samples were taken for analysis according to the Rancimat method (EN 15751) at weeks 0, 15 and 27. This was considered as a best case storage environment. A sample of each fuel was also placed in sealed polyethylene plastic drums, which were stored at ambient outdoor temperatures, but shaded from light. Samples were taken at weeks minus 2 (as blended), 18 and 27. The analysis of the fuel in the polyethylene drums was performed by the fuel blender.

TEST FUELS – The principle characteristics of the three test fuels are summarized in Table 2, while a detailed analysis may be found in Table A1 in the appendix. The fuels consist of a single diesel base meeting EN 590, blended with 10% vol of three different FAME's having different oxidation stabilities meeting EN 14214. Analysis of the three pure FAME fuels used to make each blend is provided in Table A2 in the Appendix.

The FAME feed stocks for the High and Medium stability fuel blends were treated with 1000 and 100 mg/kg of butylated hydroxy toluene (BHT) antioxidant additive respectively. Each of the three fuel blends was being treated with 500 mg/kg 2-EHN cetane improver additive. The low stability fuel was intentionally blended below the EN 590 specifications.

Table 2: Summary of B10 Test Fuels

Parameter	High Stability	Medium Stability	Low Stability
Rancimat Stab (EN 15751), h	50.3	28.9	16.1
FAME, % v/v	10	10	10
BHT Conc. mg/kg	100	10	0
Cetane Improver, mg/kg	500	500	500
FAME Type	RME*	0.5RME/0.5SME	SME**

*RME = Rape Methyl Ester **SME = Soy Methyl Ester

FUEL CHARACTERIZATION - Laboratory analysis was used to monitor the quality of the aged samples taken at regular intervals from the vehicle, as summarized in Table 3.

Table 3: Summary of Laboratory Test Methods

Test	Method	Units
Rancimat Stability	EN 15751	hours
Petroxy Stability test	prEN 16091	minutes
ΔTAN at 115°C	CEN Draft	mgKOH/g
Insolubles at 115°C	CEN Draft	g/m^3
Appearance	ASTM D-4176	-
Viscosity	EN ISO 3104	mm^2/s
Acid value	EN 14104	mg KOH/g

RANCIMAT - Is the most widely accepted stability test and is defined in EN 15751. In this test air is passed through a sample heated to 110°C. The induction period before the onset of rapid oxidation is taken as a measure of stability. The EN 590 specification for European road diesel (<7.0 %vol FAME) contains a Rancimat stability requirement of 20 hours minimum. In the US the ASTM D975 standard for road diesel (<5.0 vol% FAME) contains no minimum stability requirement, while ASTM D7467 (6-20%vol FAME in closed fleets) has a stability requirement of only 6 hours minimum.

PETROXY – The Petroxy test is an alternative, potentially more rapid test currently undergoing industry evaluation. In the present study, the tests were performed according to the draft CEN test method prEN 16091 at 140ºC. The procedure measures the time required for the onset of severe oxidation as reflected by a decrease in oxygen pressure over the sample. The Petroxy test requires a small sample volume and so was scheduled with samples drawn at regular intervals during the 27 week storage duration.

DELTA TAN AND INSOLUBLES - No standard method exists and the evaluation was performed according to the draft methodology being evaluated by CEN in mid 2009. In this procedure, the fuel is aged at 115ºC for 16 hours while oxygen is bubbled through the sample at a rate of 3 L/hour. Following completion of the test, the change in acid number is measured (Delta TAN) and the total insoluble content is measured according to the procedure defined in EN 12205. This test measures the volume of insoluble formed once considerable aging has occurred. The Delta TAN and insolubles test is scheduled infrequently due to the high volume requirements, which would quickly deplete the available fuel in each tank.

IV. RESULTS

RANCIMAT - The results obtained using the Rancimat test are plotted in Figures 2, 3 and 4 for high, medium and low stability fuels respectively. In each instance, the results are plotted as a function of storage duration, with week zero being the time the fuel was placed in the vehicle. Data are also available from the blending process (week minus 2) and also as delivered to the test site (week minus 1). The overall trends are similar between each of the three fuels.

Comparatively slow rates of aging were observed for fuel samples taken from sealed metal drums, most likely due to lack of oxygen availability. Most of this decrease occurred immediately after the fuel was placed in the drum and so was probably due to residual oxygen in the tank and dissolved within the fuel. More rapid loss of stability occurred for fuel stored within the plastic drums. This may be due in part to the fact that the plastic drums were stored at ambient outdoor conditions. However, it was unexpected given the fact that oxygen is needed for oxidation to occur and the drums are sealed and so have limited oxygen availability.

Relatively slow aging was observed in fuel samples taken from the commercial vehicles containing each of the three fuels. The rate of aging was normally lower than for sealed plastic drums and in some instances was similar to that observed in the sealed epoxy lined steel drums. By comparison, rapid fuel aging was observed in passenger cars, with the Rancimat induction period decreasing by approximately 75% during the first 10 weeks. The rate of aging decreases after week 10, which may be due in part to falling ambient temperatures.

A fluctuation in the stability of several fuel samples was observed around week zero. This does not appear to be an anomaly, as it is present in both the Rancimat and Petroxy test data. The fluctuation could potentially be an artifact of the sampling technique used to remove the fuel from the vehicle, although no evidence exists to support this.

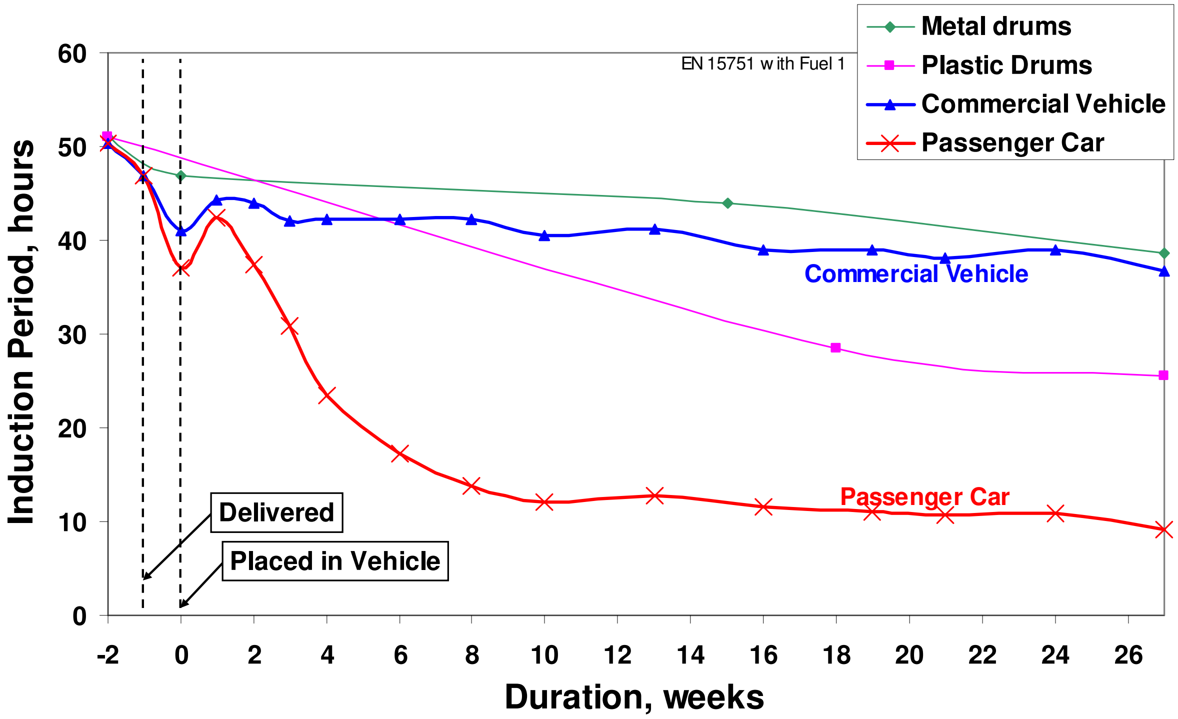

Figure 2: Aging of High Stability B10 Blend Measured Using the Rancimat (Similar trends seen with Petroxy Test)

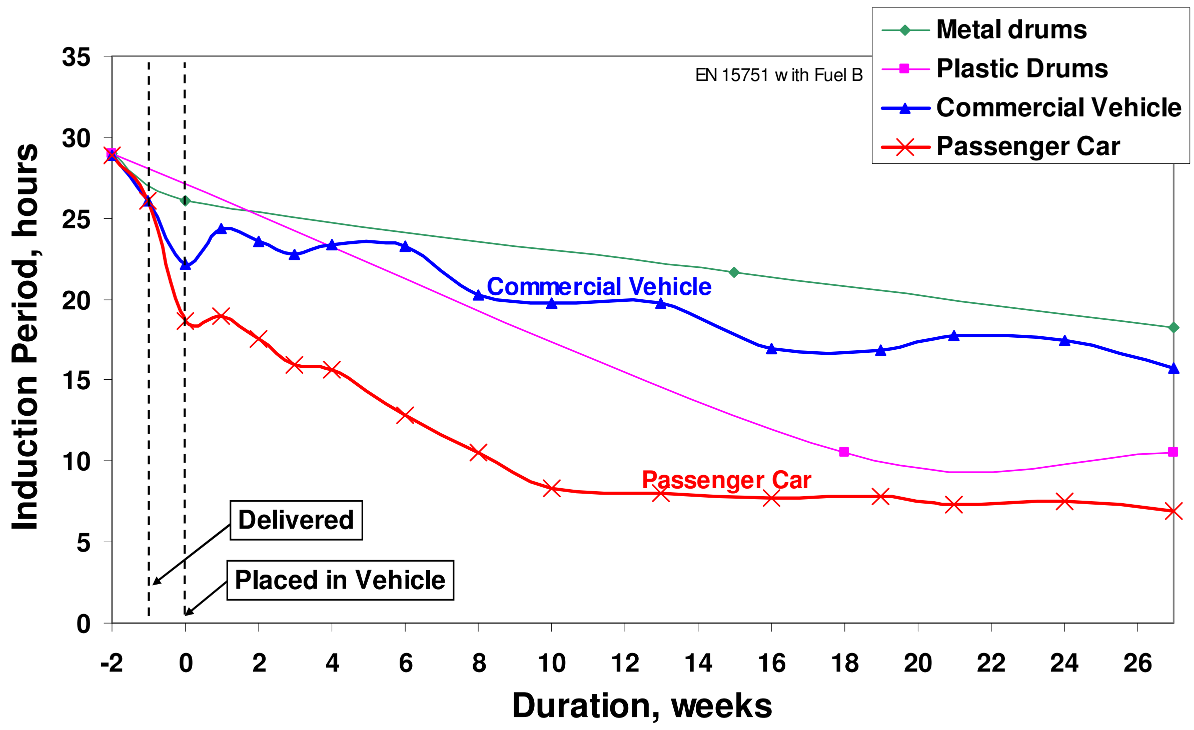

Figure 3: Aging of Medium Stability Fuel B10 Blend Measured Using the Rancimat (Similar trends seen with Petroxy Test)

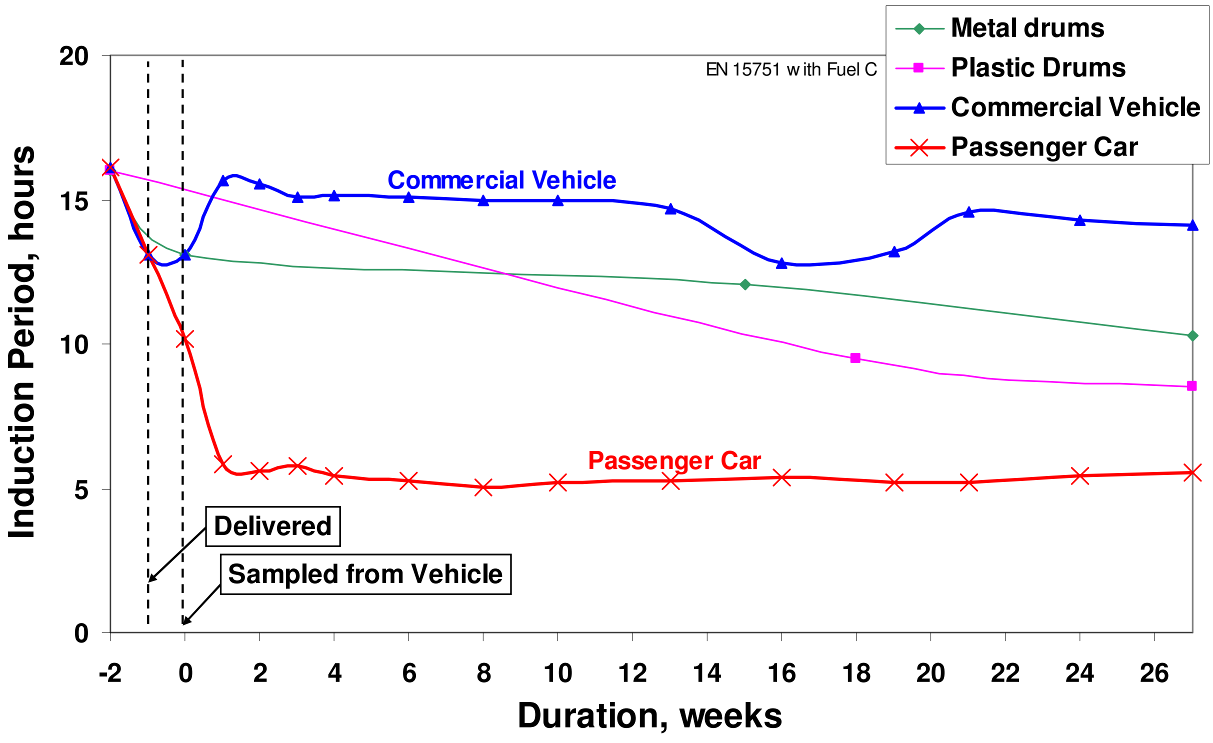

Figure 4: Aging of Low Stability Fuel B10 Blend Measured Using the Rancimat (Similar trends seen with Petroxy Test)

ELEMENTAL ANALYSIS – Inductively Coupled Plasma (ICP) analysis was performed on the new and aged fuel samples. The results indicate that elements normally associated with fuel aging (i.e. copper, zinc) are not present in any of the samples at concentrations down to the detection limit of 1 mg/kg.

More accurate elemental analysis was performed using Atomic Absorption Spectroscopy (AAS) on samples taken from each of the vehicles approximately 11 weeks after conclusion of the study, with the results shown in Table 4. The quantification limit on the AAS analysis is 10 ppb. Similar analysis of samples taken from the sealed metal drums is provided in Table 5. The concentration of the elements analyzed was relatively low. In particular copper which is an effective catalyst for fuel oxidation was present at concentrations below 10 PPB in all samples. The concentrations of the remaining elements including iron, calcium, zinc and sodium, were somewhat higher in the fuel samples taken from the vehicles than those from the drums. In general, elemental contamination does not serve as a basis to explain the variation in aging rate between the fuels and vehicles.

Table 4: Results of Elemental Analysis on Fuel Samples Drawn From Test Vehicles Following Conclusion of the Study Using AAS

	High Stability Fuel Blend		**Medium Stability Fuel Blend**		**Low Stability Fuel Blend**	
	Commercial Vehicle	**Passenger Car**	**Commercial Vehicle**	**Passenger Car**	**Commercial Vehicle**	**Passenger Car**
Copper, PPB	<10	<10	<10	<10	<10	<10
Iron, PPB	60	26	28	61	62	40
Zinc, PPB	132	<10	106	147	50	186
Phosphorous, PPB	<10	<5	<10	<10	<10	<10
Calcium, PPB	296	88	50	214	102	124
Silicon, PPB	13	65	<10	34	24	50
Sodium, PPB	20	83	47	28	103	78

Table 5: Results of Elemental Analysis on Fuel Samples Drawn From metal Storage Drums Following Conclusion of the Study Using AAS

	High Stability Fuel Blend	**Medium Stability Fuel Blend**	**Low Stability Fuel Blend**
Copper, PPB	**<10**	**<10**	**<10**
Iron, PPB	**24**	**26**	**26**
Zinc, PPB	**21**	**16**	**10**
Phosphorous, PPB	**<10**	**<10**	**<10**
Calcium, PPB	**42**	**32**	**37**
Silicon, PPB	**<10**	**14**	**17**
Sodium, PPB	**18**	**28**	**11**

PETROXY - Very similar trends were observed between analysis performed using the Rancimat and Petroxy tests. Because of this the results obtained using the Petroxy tests are not plotted in full. The correlation between the two methods is plotted in Figure 5.

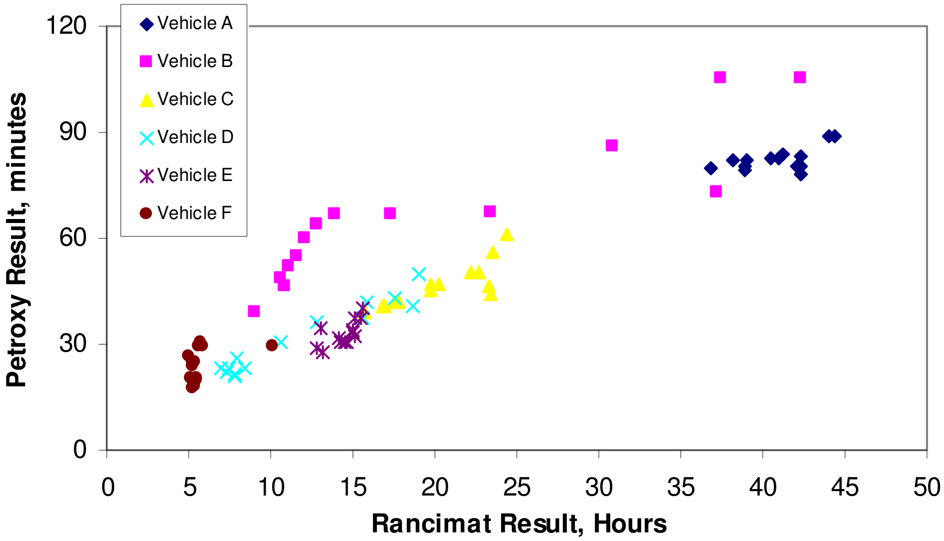

Figure 5: Correlation Between Rancimat and Petroxy Methods

EN ISO 12205 - The fuel standard EN 590 specifies a maximum oxidation stability (insolubles concentration) of 25g/m^3 when measured according to this procedure. Work carried out by the CEN JWG indicates that the applicability of this method is questionable for fuels containing FAME. The results of analysis at weeks 0 and 27 are plotted using a logarithmic scale in Figure 6. An increase in insolubles concentration is observed for each of the fuels during aging. The increase is larger in the passenger vehicles than in the light commercial vehicles, which reflects the previously described changes in Rancimat and Petroxy stability. After 27 weeks of aging in the commercial vehicles, the oxidation stability requirements in EN 590 were still met.

The medium stability fuel would no longer meet the oxidation stability requirements in the EN 590 specification following 27 weeks of aging in a passenger vehicle. (Note the EN 590 specification only applies to fuel at point of sale and is no longer technically applicable when the fuel is in the vehicle). This is despite the fact that the medium stability fuel had an initial Rancimat stability of almost 30 hours, which is more than the 20 hours currently required by EN 590 for B7 and considerably more than the 6 hours required in ASTM D7467 for fuel containing 6 to 20%vol FAME.

The insolubles content for the low stability fuel showed a very large increase. It was 64 time higher than the EN 590 limit following 27 weeks of aging in one passenger vehicle.

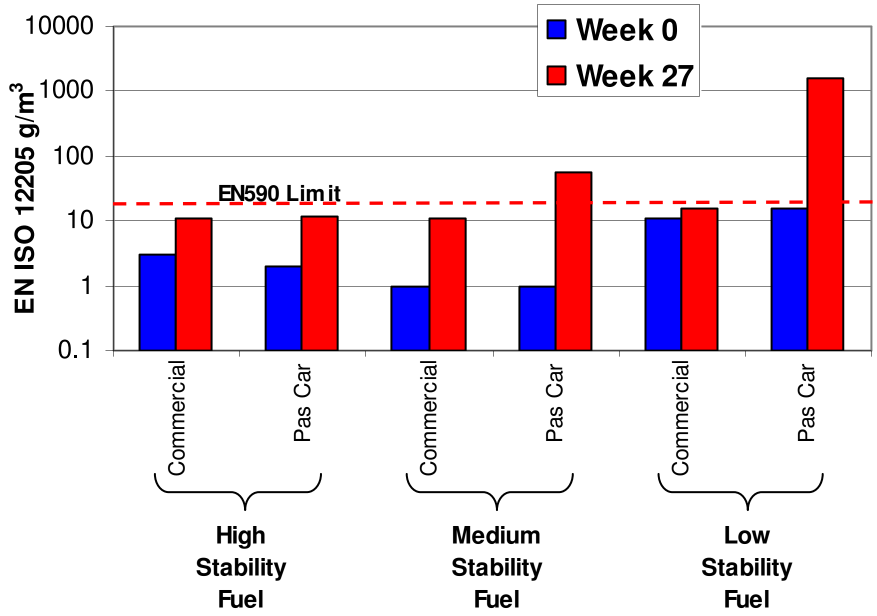

Figure 6: Effect of Vehicle Aging on Insolubles Measured Using EN ISO 12205

DELTA TAN AND INSOLUBLES - Analysis according to the draft method for Delta TAN and insolubles was performed at weeks 0, 4, 13 and 27. The results of Delta TAN testing at 115°C for 16 hours on fuel samples from passenger vehicles are plotted using a logarithmic scale in Figure 7.

There is no requirement or limit for this test in the European Diesel specification, EN 590. However, Japan and France have national limits of 0.13 and 0.3 mg KOH/g respectively. The low stability fuel exceeds both these limits in a matter of weeks when stored in passenger vehicles. The medium stability fuel exceeds the Japanese limit following approximately 8 weeks of aging. In the present study, all of the fuels gave a Delta TAN result of 0.02 or below at week zero. This was unexpected as the FAMEs used have different feeds and concentrations of stability additives and so might be expected to have different stabilities.

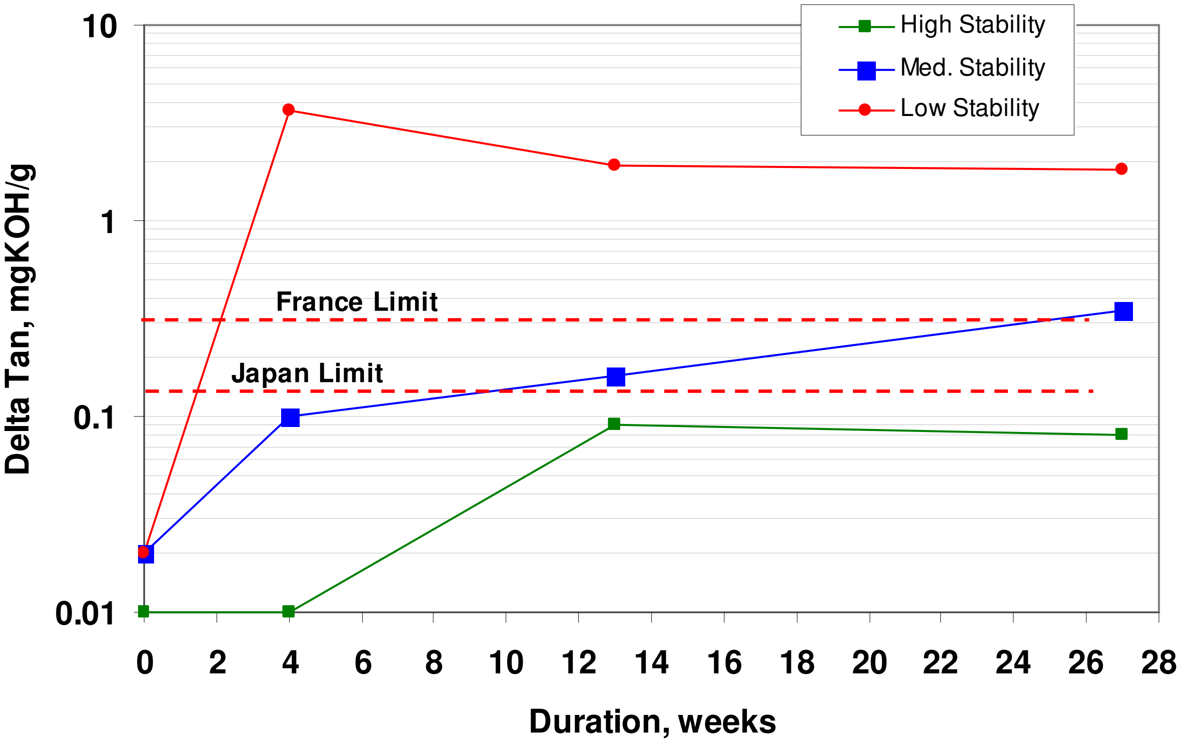

Figure 7: Effect of Aging in Passenger Cars on Delta TAN

The results of insolubles measure following aging at 115°C are plotted in Figure 8 for the three passenger cars. A rapid change was observed for the low stability fuel, resulting in an insolubles concentration of 6500 g/m^3 after 4 weeks of aging. No limit exists for insolubles content.

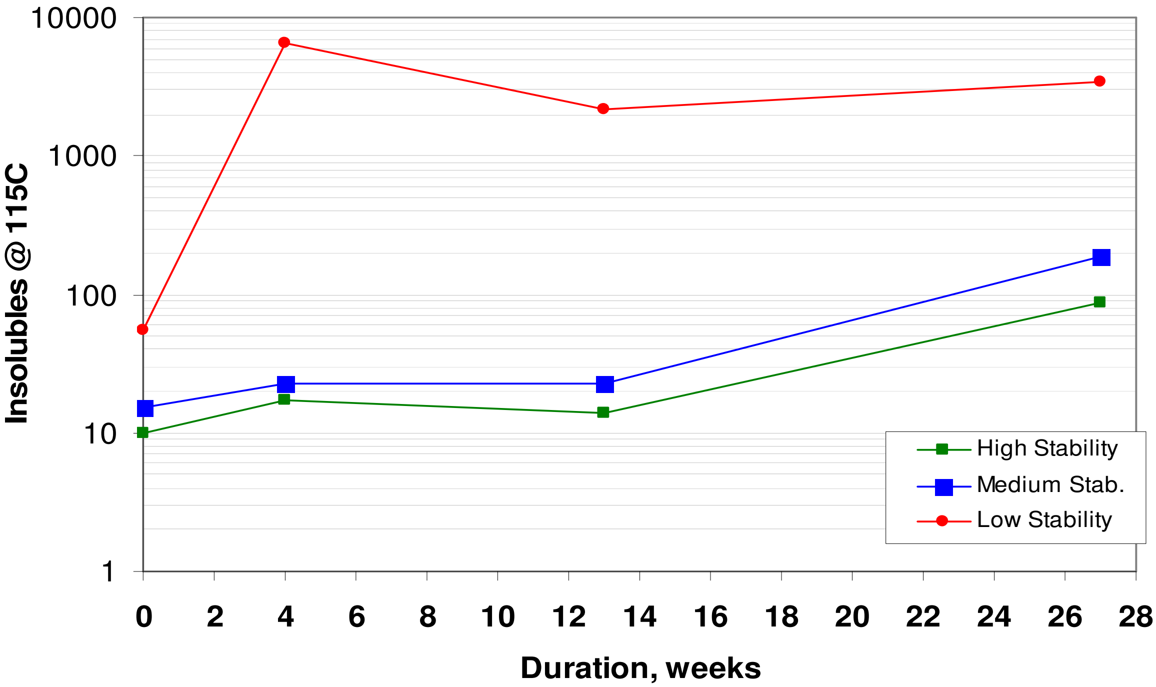

Figure 8: Effect of Aging in Passenger cars on Insolubles, Measured at 115°C

GENERAL FUEL QUALITY – Photographs of the fuel samples taken at weeks 0 and 27 are provided in Figures A-2 and A-3 in the Appendix. The results of analysis according to the parameters defined in the EN 590 specification for road diesel are provided in Table A1 in the appendix. As previously discussed, deterioration in oxidation stability was observed, particularly for low stability fuel in passenger cars. The oxidation stability of all fuels did not deteriorate as significantly in the commercial vehicles as in the passenger cars. Cetane number was increased by aging for all of the fuels, probably due to the increased peroxides content. This change approximates the repeatability of the method, but was observed for all samples. A slight but repeatable increase in sulfur content was observed for several fuels. It is thought that this may be due to leeching from the low pressure system. A slight decrease in lubricity was observed for most of the fuels. However, the resulting wear scar diameter remains well within the 460 µm limit required by EN 590.

The results of peroxides analysis performed according to ASTM D3703 are plotted in Figure 9 for fuel samples drawn from each of the vehicles following conclusion of the study. In general peroxides content increases as fuel stability decreases.

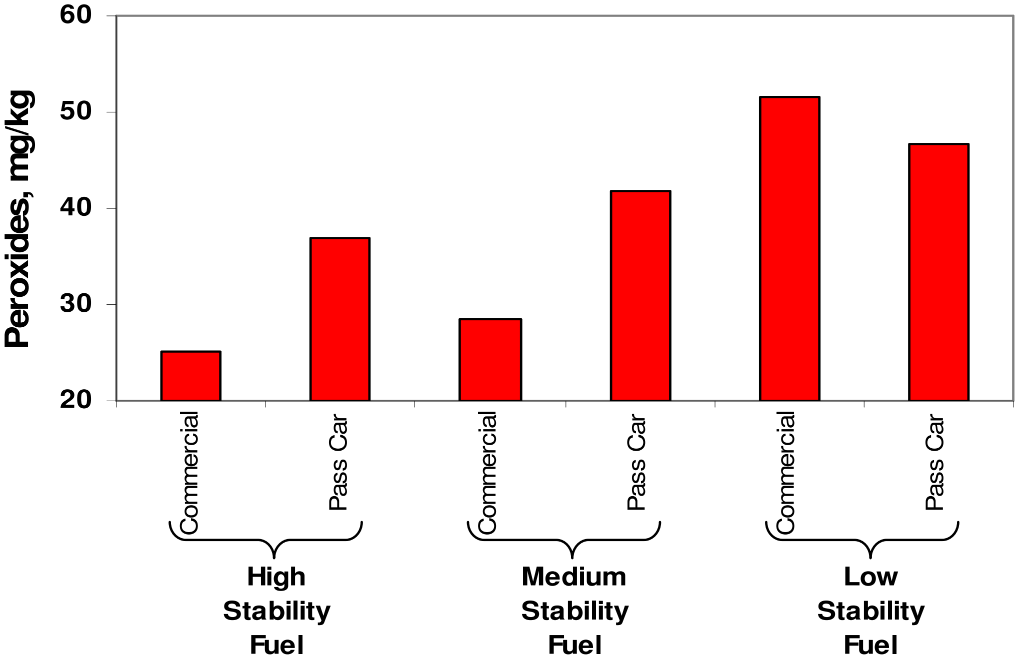

Figure 9: Peroxide Concentration on Fuel Samples Following Conclusion of the Study Measured Using ASTM D3703

V. DISCUSSION

A study was performed to evaluate the effect of in vehicle storage on diesel fuel that contains 10 %vol FAME. Three different FAME blends were used, with oxidation stabilities of 50, 29 and 16 hours, as measured by the Rancimat method (EN 15751). The effects of aging were measured using laboratory scale test methods. The vehicles were started but not driven and so no attempt was made to define the effects of changing fuel quality on system performance. The results indicate aging began immediately following production of the fuel and continued through delivery and placement in the vehicle. In spite of the observed changes in fuel quality with aging, the vehicles started and idled successfully throughout the 27-week testing period.

Aging rate is strongly dependent on storage conditions and vehicle type, with particularly rapid aging occurring in the three passenger cars. This variation was apparent from several of the laboratory test methods, including the Rancimat, Petroxy and EN 12205 methods and so is very unlikely to be an artefact of a single laboratory scale test.

The cause of the repeatable variation between the passenger cars and light commercial vehicles needs to be defined, as the fuel systems in each vehicle are broadly similar with a HDPE fuel tank feeding a high pressure common rail fuel injection system. Similarly, elemental analysis does not indicate the presence of materials known to act as a catalyst to fuel aging in any of the fuels. FAME aging will normally be affected by oxygen availability. However, the tank breathing system on the passenger vehicles in which the fuel aged quickly is normally closed, only opening when the internal tank pressure reaches a preset value. By comparison the light commercial vehicles in which the fuels did not age quickly had a normally open vent. As a result, it is unlikely that variation in oxygen availability is the cause of the variation observed.

Both the Rancimat and Petroxy test methods indicate that the stability of the fuel stabilized following some weeks of storage. This could be due to decreasing ambient temperatures towards the end of the storage period. It is also possible that oxygen availability within the closed tanks is reduced. No conclusive explanation is available as to why the aging rate decreased and it is contrary to normal experience with FAME.

The study observed similar overall trends with each of the three test fuels evaluated. As expected, the most significant effects were observed with the low stability fuel, which had an as blended Induction Period of 16 hours as measured by EN 15751. It should be noted that this still represents a relatively high quality fuel, particularly when compared with US standard D7467 for B6 to B20, which has a minimum stability requirement of only 6 hours.

The vehicles were chosen at random and parked in a warm European climate. Similarly, the fuels were blended to achieve an initial stability target. It is likely that other fuel and additive blends will have different aging characteristics. It is possible that different aging rates could be observed in other applications but additional work would be needed to evaluate this. For example, many commercial and off road vehicles have storage tanks that are exposed to direct sunlight, which could increase oxidation rates.

VI. CONCLUSIONS

The following conclusions may be drawn from the study. It should be noted that the study was not aimed at assessing the impact of the aged fuel on the vehicles. As a result, it is not possible to make an assessment of whether the final condition of the most aged fuels provides enough protection for the vehicles.

a) Storage of B10 in vehicles caused a rapid decrease in stability as measured by both the EN 15751 (Rancimat) and prEN 16091(Petroxy) methods.

b) The rate of aging was strongly dependent on storage conditions.
   - Most rapid aging was observed in passenger vehicles, which lost up to 7 hours of Rancimat IP per week.
   - Reduced aging occurred in commercial vehicles.
   - Slow aging occurred in sealed metal drums, probably due to restricted oxygen availability
   - Relatively rapid aging occurred with fuel stored in polyethylene drums.

c) Storage of B10 caused an increase in insoluble material measured using EN ISO 12205. This increase was particularly significant for the low stability fuel stored in passenger vehicles. One passenger vehicle gave a result of 1589 g/m^3 following 27 weeks of storage. This increase was larger in passenger vehicles than in commercial vehicles.

d) Storage of B10 caused a rapid decrease in stability, as measured by the Delta TAN method. Low and medium stability fuels exceeded the Japanese limits for this test following only 2 and 8 weeks of storage respectively.

e) Good directional correlation was observed between the Rancimat and Petroxy tests applied to both fresh and aged fuels. Both test methods predicted the quality of the test fuel prior to aging and storage.

f) In the present study, the Delta TAN method indicated that the quality of the different test fuels was similar at week zero prior to aging.

g) The results of several different test methods indicate that aging of fuel that contains FAME is strongly affected by storage condition and vehicle design. Further study is required to understand the results, as no correlation was observed with the parameters expected to affect stability, particularly elemental contamination (at current AAS accuracy), fuel tank breather design, or low pressure fuel system design. The concentration of all elements was low, less than 1 ppm in fresh and aged samples.

h) The rate of aging decreased with time, which has not been previously observed. This decrease in aging rate was observed with both the Rancimat and Petroxy measures.

VII. ACKNOWLEDGMENTS

The authors wish to acknowledge the ACEA, CONCAWE, Delphi Diesel Systems, Bosch, Continental, and Denso Corporation for funding this study, ACEA and the manufacturers for providing the test vehicles, and CONCAWE for providing the test fuels. Innospec, Afton and Lubrizol Corporations provided funding to allow additional fuels analysis following conclusion of the study.

VIII. REFERENCES

1) Puetz, W., "Biodiesel for Passenger Cars and Light Trucks in the USA", Joint Statement of the German Passenger Car manufacturers, Diesel Technology Forum Webinar, 15 December 2009.

2) "Fuel Requirements for Diesel Fuel Injection Systems", Joint Statement of the Diesel Fuel Injection Equipment Manufacturers, September 2009. http://delphi.com/manufacturers/auto/powertrain/diesel/

3) Bondioli, P., Gasparoli, A., et al, "Biodiesel Stability Under Commercial Storage Conditions Over One Year," Eur. J. Lipid Sci. Technol. 105, pp. 735-741, 2003.

4) Lacoste, F., Bondioli, P., "Stability of Biodiesel Used as a Fuel for Diesel Engines and Heating Systems," Summary report, Federal Institute of Agricultural Engineering, Wieselburg, Austria, August 2003.

5) DGMK Projects 639-1: "Investigations on FAME in Diesel Fuels (B5)" and 639-2, "Investigations on FAME in Diesel Fuels (B10)".

IX. DEFINITIONS, ACRONYMS, ABBREVIATIONS

AAS: Atomic Absorption Spectroscopy
ASTM: American Society for Testing and Materials
BXX: FAME blend in hydrocarbon diesel at XX Vol%
BHT: butylated hydroxy toluene
CEN: Comite Europeen de Normalisation
FAME: Fatty Acid Methyl Ester
HDPE: High Density Polyethylene
HFRR: High Frequency Reciprocating Rig
ICP: Inductively Coupled Plasma
ppb: Parts per Billion
ppm: Parts per Million
TAN: Total Acid Number

APPENDIX

Table A-1: Laboratory Analysis of Blended Test Fuels According to EN 590

			High Stability Fuel Blend				**Medium Stability Fuel Blend**				**Low Stability Fuel Blend**			
			Commercial Vehicle A		**Passenger Car Vehicle B**		**Commercial Vehicle C**		**Passenger Car Vehicle D**		**Commercial Vehicle E**		**Passenger Car Vehicle F**	
Test	Unit	Method	Wk 0	Wk 27	Wk 0	Wk 27	Wk 0	Wk 27	Wk 0	Wk 27	Wk 0	Wk 27	Wk 0	Wk 27
Density,	kg/m^3	EN 12185	831.6	831.8	831.6	831.8	831.6	831.9	831.6	831.9	831.7	832.0	831.7	831.9
Distillation Rec. @ 250°C	% v/v	EN 3405	28.2	27.1	28.4	27.0	29.4	28.7	28.2	29.2	28.5	27.5	28.9	28.5
Distillation Rec@350°C	% v/v	EN 3405	93.6	94.3	95.0	94.4	94.9	94.8	93.9	95.5	94.9	95.3	94.9	96.1
Distillation 95% Rec.	°C	EN 3405	356.5	352.7	350.1	353.1	350.4	351.1	355.0	347.7	350.6	349.1	350.8	346.8
Sulphur	mg/kg	EN 20846	8.7	9.8	8.7	11.2	8.2	8.5	8.2	8.6	8.0	9.5	8.6	11.0
Water content	mg/kg	EN 12937	63	75	80	91	84	70	96	65	74	65	83	70
Total Cont.	mg/kg	EN 12662	2	3	5	4	5	3	1	2	6	4	6	3
Ash	%m/m	EN 6245	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Cetane Number	N/A	EN 5165	57.6	60.8	57.1	58.9	56.7	60.5	56.8	61.1	57.3	58.4	56.9	59.0
Cetane index	N/A	EN 4264	57.2	57.7	57.0	58.1	56.9	57.1	57.1	56.8	56.9	57.2	56.5	56.8
MCCR on 10%	%m/m	EN 10370	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Cu corr.	N/A	EN 2160	1a	1a	1a	1a	1a	1a	1a	1a	1a	1a	1a	1a
Oxidation Stability	g/m^3	EN 12205	3	11	2	12	1	11	1	54	11	16	15	1,589
	Hr	EN 15751	50.3	36.7	50.3	9.08	28.9	15.77	28.9	6.93	16.1	14.11	16.1	5.53
	minutes	prEN 16091	82.43	79.9	73.07	39.01	50.48	39.33	40.87	23.25	34.63	31.49	29.65	19.6
Flash Point	°C	EN 2719	62.0	65.0	63.0	64.5	63.0	65.5	63.0	64.5	64.0	67.0	64.0	66.5
Lubricity	µm	EN 12156	188	195	193	239	181	230	206	206	201	218	200	248
CFPP	°C	EN 116	-6	-6	-6	-4	-7	-7	-7	-7	-8	-7	-8	-8
Polycyclic Aromatics	%m/m	EN 12916	2.6	2.8	2.7	3.0	3.1	3.0	3.0	3.1	3.0	2.7	3.1	3.2
FAME	%v/v	EN 14078	9.7	9.6	9.5	9.5	9.5	9.8	9.6	9.5	9.7	9.4	9.5	9.6
Peroxides	Mg/kg	D 3703	N/A	25.1	N/A	37.0	N/A	28.5	N/A	41.7	N/A	51.6	N/A	46.7

Table A-2: Laboratory Analysis of FAME Feeds Used to B10 Blend Fuels

			FAME to be used in		
Parameter	**Unit**	**Test Method**	**High Stability Blend**	**Medium Stability Blend**	**Low Stability Blend**
Composition by GC: Methyl ester of:		In house			
myristic acid	% vol		0.1	0.1	0.1
palmitic acid	% vol		4.7	8.1	11.3
palmitoleic acid	% vol		0.2	0.2	0.1
stearic acid	% vol		1.8	3.6	5.1
Oleic acid	% vol		60.7	42.4	23.3
linoleic acid	% vol		20.0	36.2	52.1
linolenic acid	% vol		9.7	7.1	6.8
eicosanoic acid	% vol		0.6	0.5	0.4
eicosenoic acid	% vol		1.2	0.7	0.2
docosanoic acid	% vol		0.3	0.4	0.3
docosenoic acid	% vol		0.1	0.2	<0.1
unidentified acid	% vol		0.7	0.6	0.3
Iodine value (calc. from the methyl ester comp.):	g iodine/100 g		113.5	118.6	128.3
Ester Content	% mass	EN 14103	97.7	98.4	98.3
Linolenic Acid Methyl Ester Content	% mass	EN 14103	9.7	7.1	6.8
Density at 15 °C	kg/m^3	EN 12185	884.0	884.1	886.0
Viscosity at 40 °C	mm^2/s	EN 3104	4.471	4.297	4.175
Flashpoint	°C	ISO 3679	>140	>140	<140
Sulphur content	mg/kg	EN 20846	<5.0	<5.0	<5.0
Cetane Number	ASTM D 6890	ASTM D 6890	59	56.6	no data
Sulphated ash content	% (m/m)	ISO 3987	<0.005	<0.005	<0.005
Water content	mg/kg	EN 12937	280	350	440
Copper corrosion, 3 hrs at 50°C	EN ISO 2160	EN 2160	1a	1a	1a
Oxidation Stability at 110 °C	EN 14112	EN 14112	9.5	5.7	3.1
Acid Value	mg KOH/g	EN 14104	0.31	0.24	0.33
Iodine value	g I/100g	EN 14111	113	120	118
Polyunsat. methyl esters (≥4 double bonds)	% (m/m)	EN 14103	0.25	0.13	0.13
Methanol content	% (m/m)	EN 14110	<0.01	0.05	0.01
Monoglyceride content	% (m/m)	EN 14105	0.56	0.37	0.4
Diglyceride content	% (m/m)	EN 14105	0.15	0.09	0.1
Triglyceride content	% (m/m)	EN 14105	0.11	0.05	0.04
Free glycerol	% (m/m)	EN 14105/6	<0.01	<0.01	<0.01
Total glycerol	% (m/m)	EN 14105	0.18	0.12	0.12
Group I metals (Na + K)	mg/kg	EN 14108/9	<2	<2	<2
Group II (Ca + Mg)	mg/kg	EN 14538	<2	<2	<2
Phosphorus content	mg/kg	EN 14107	<4	<4	<4
CFPP	°C	EN 116	-14	-5	no data

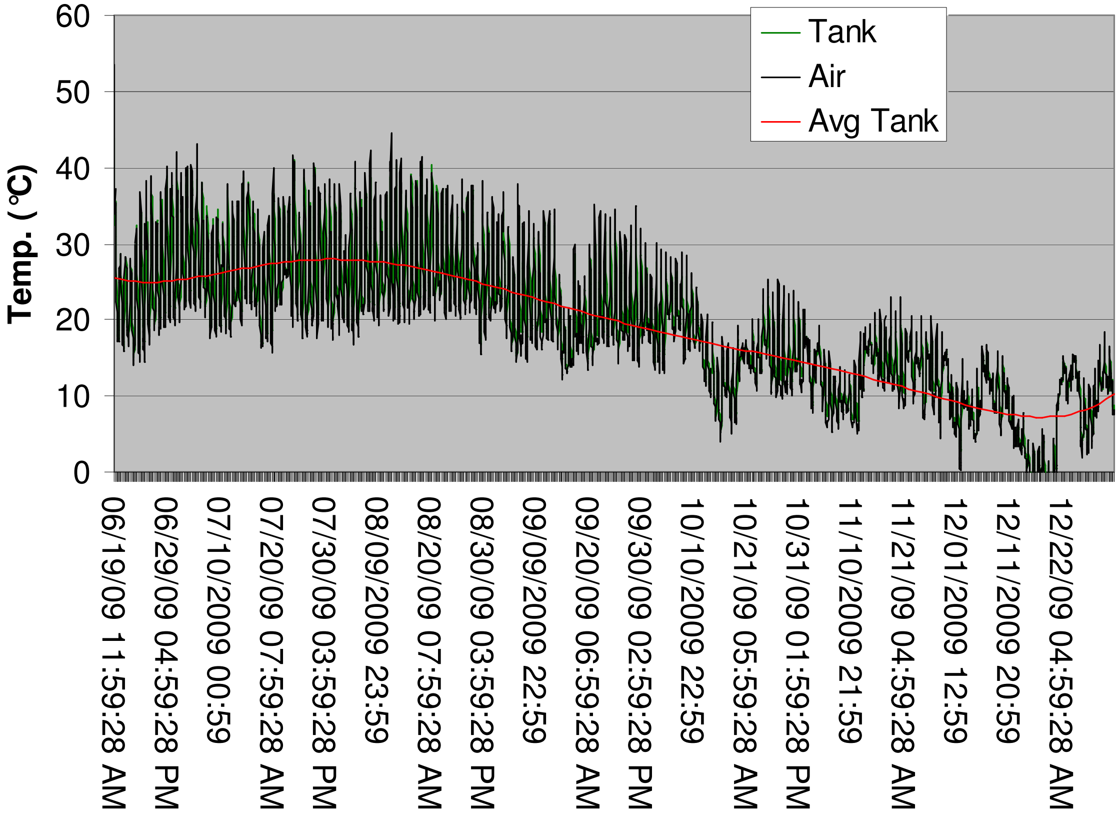

Figure A-1: Temperature Profiles Measured in Vehicle B

Figure A-2: Photograph of Fuel Samples at Week 0 (Fuels from Vehicles A-F, left-to-right)

Figure A-3: Photograph of Fuel Samples at Week 27 (Fuels from Vehicles A-F, left to right)

ISBN 978-2-87567-009-0

9 782875 670090 >