

EBB

European Biodiesel Board

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EBB EUROPEAN BIODIESEL QUALITY REPORT (EBBQR)

Summer 2010 Results Results of the eighth round of tests



Background

The need to ensure that a high quality biodiesel fuel is produced and made available in the market by EBB Member companies, specifically in the perspective of increasing to 10% the maximum FAME content in EN590 diesel fuel, is of significance to the industry. This report is one in a series of bi-annual samplings of all the operational production sites of EBB member companies.

One sampling is operated at the beginning of the winter and one sampling at the beginning of the summer. The samples are taken and sent to the selected laboratory by EBB member companies. There are no unexpected samplings but full co-operation with companies takes place, however EBB Member Companies are bound to gather and send all samples within the specified deadline to the selected laboratory.

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Ester Content

EBBQR – Summer 2010

FAME conforming to the standard must have an ester content over 96.5%. Capillary gas chromatography is used to determine the ester content of a FAME sample.

Summer 2010

EN 14214	> 96,5	% m/m
Min.	89.9	% m/m
Max.	>99	% m/m
Average	98.6	% m/m
Std. Deviation	1.4	% m/m
Range 95% max	101.4	% m/m
Range 95% min	95.7	% m/m
Out of spec	1	-

All values > 99% rounded off 99.1%

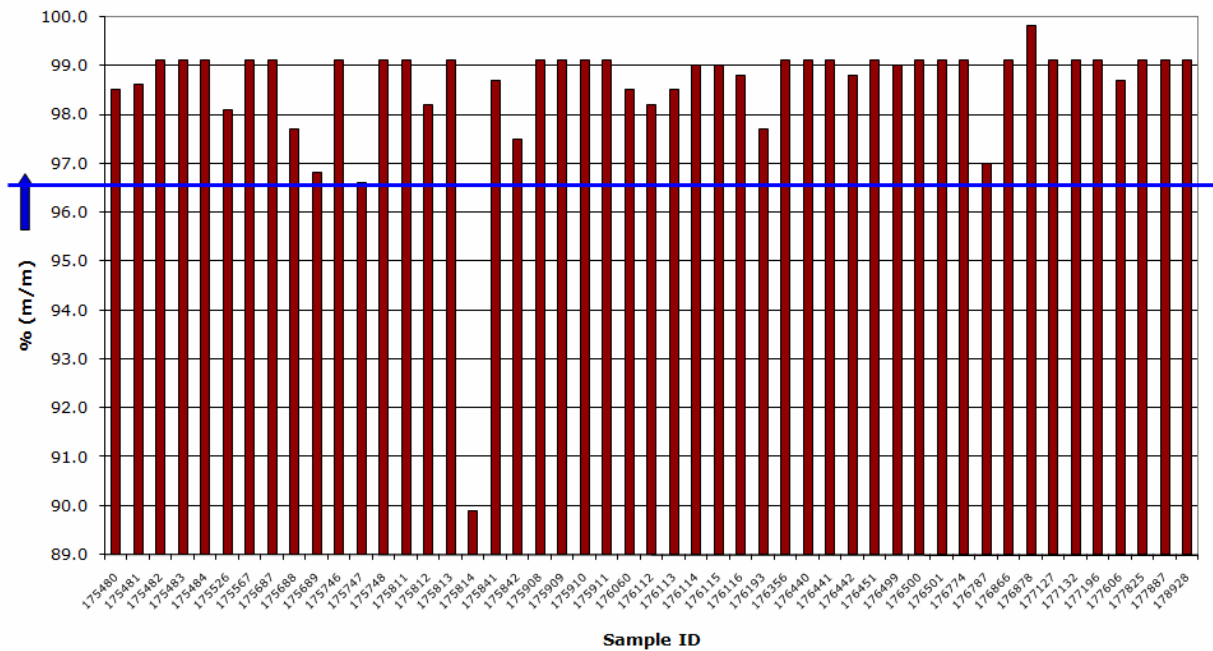
Results

The average value seen in the Summer 2010 EBBQR was 98.6%, with minimum and maximum figures of 89.9% and > 99% respectively. One result was below the 96.5% limit.

The ester content test method (EN14103) can underestimate C17 content, resulting in an incorrectly low overall ester figure for FAMES using animal fat feedstocks.

Summer 2010

Ester Content



Density at 15°C

EBBQR – Summer 2010

Density was measured at a temperature of 15°C. Density may be measured over a range of temperatures from 20°C to 60°C, and in that event a temperature correction formula is used (as supplied within the EN14214 appendix).

Summer 2010

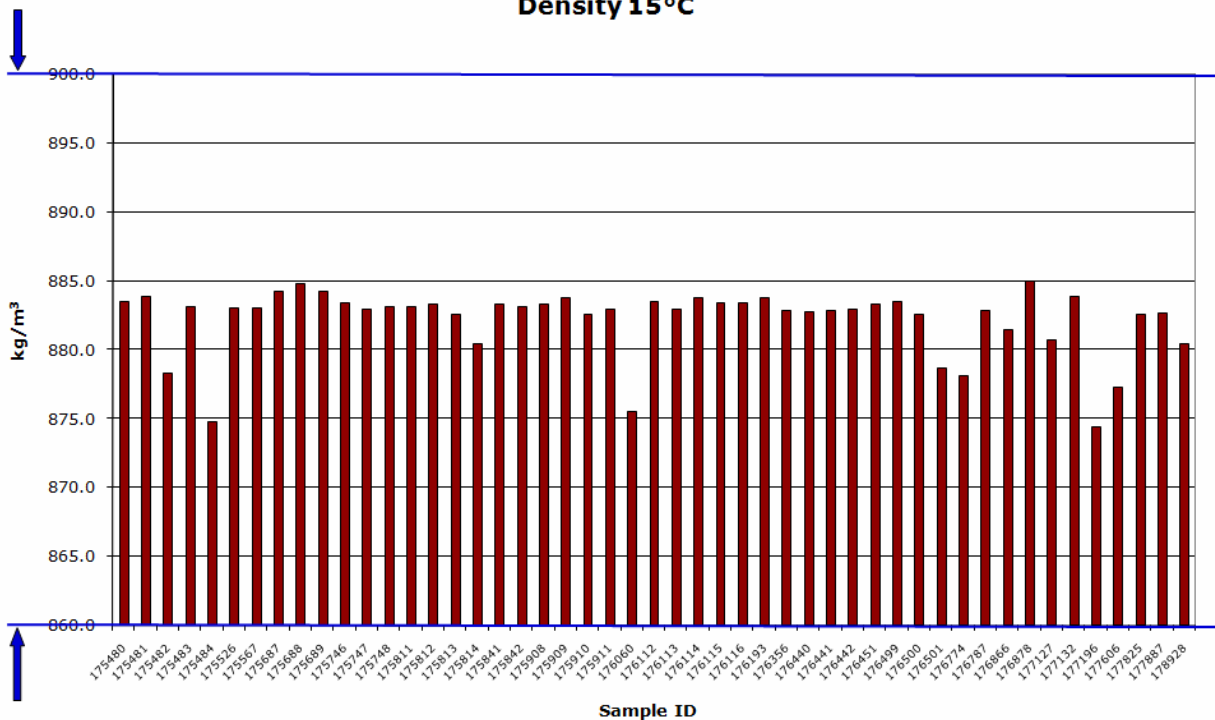
EN 14214	860 - 900	kg/m ³
Min.	874	kg/m ³
Max.	885	kg/m ³
Average	882	kg/m ³
Std. Deviation	2	kg/m ³
Range 95% max	887	kg/m ³
Range 95% min	877	kg/m ³
Out of spec	0	-

Results

The results show all the density figures were within the range of the specification. Biodiesel fuels generally display higher densities than mineral diesel fuel.

Summer 2010

Density 15°C



Kinematic Viscosity at 40°C

EBBQR – Summer 2010

The viscosity of a sample is measured at 40°C. However, if the CFPP of the FAME is -20°C, the viscosity is to be measured at -20°C.

Summer 2010

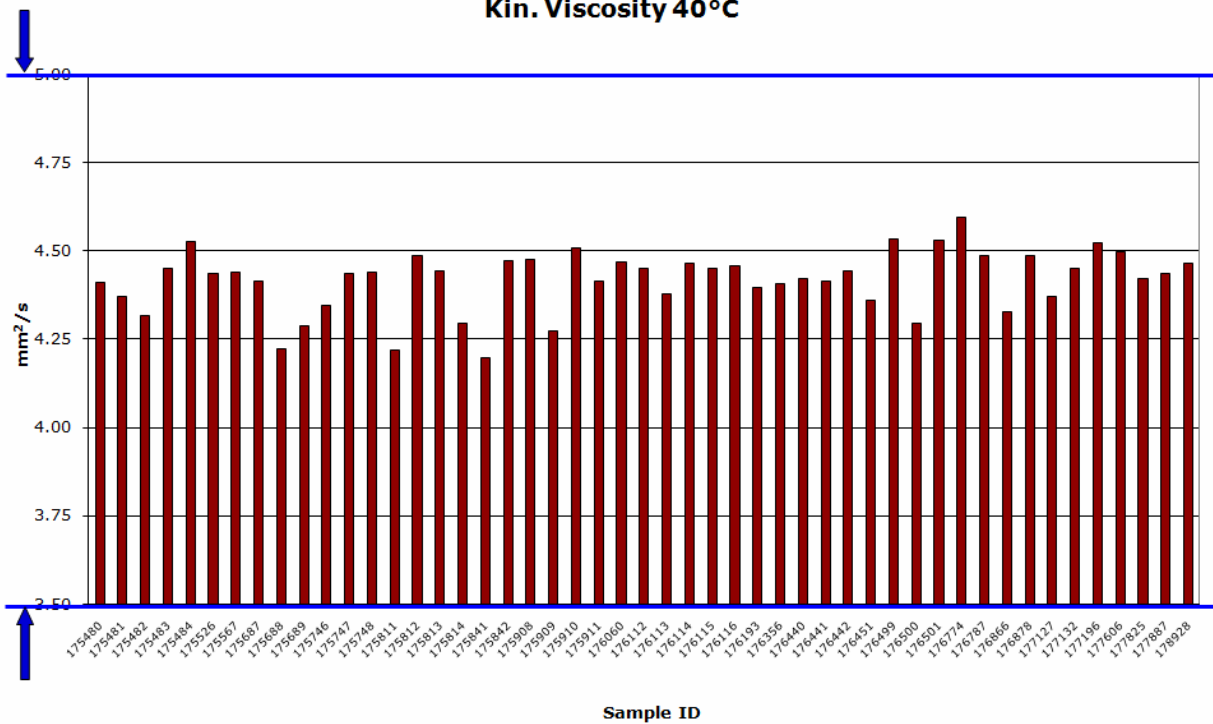
EN 14214	3,50 - 5,00	mm ² /s
Min.	4.20	mm ² /s
Max.	4.60	mm ² /s
Average	4.42	mm ² /s
Std. Deviation	0.09	mm ² /s
Range 95% max	4.59	mm ² /s
Range 95% min	4.25	mm ² /s
Out of spec	0	-

Results

All samples were within range for viscosity. Both the viscosity and density of diesel fuel and biodiesel blends increases with higher concentrations of biodiesel in the fuel blend.

Summer 2010

Kin. Viscosity 40°C



Flash Point

EBBQR – Summer 2010

The flash point is a measure of a fuel's flammability and is considered important for assessing hazards during storage. EN14214 states that flash point can be measured using either EN ISO 2719, or EN ISO 3679.

Summer 2010

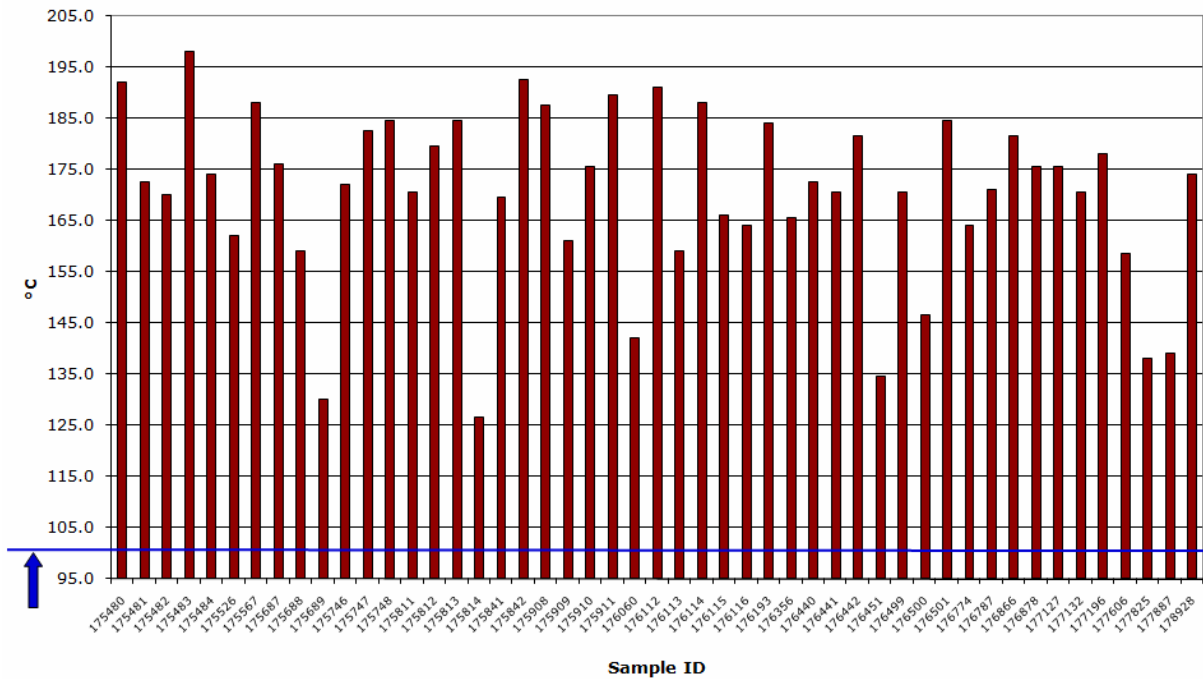
EN 14214	> 101	°C
Min.	127	°C
Max.	198	°C
Average	170	°C
Std. Deviation	17	°C
Range 95% max	204	°C
Range 95% min	137	°C
Out of spec	0	-

Results

All samples adhered to the specification, by exhibiting flash points higher than 101°C. Biodiesels exhibit higher flash points when compared with mineral diesel and this can be seen to be a safety advantage for biodiesel over fossil diesel.

Summer 2010

Flash Point



Cold Filter Plugging Point (CFPP)

EBBQR – Summer 2010

The designated test used for measuring the cold filter plugging point (CFPP) of a fuel is EN116. The CFPP limits required for the final gasoil are dependent upon national regulations; different climates have different cold flow requirements on their fuels.

Summer 2010

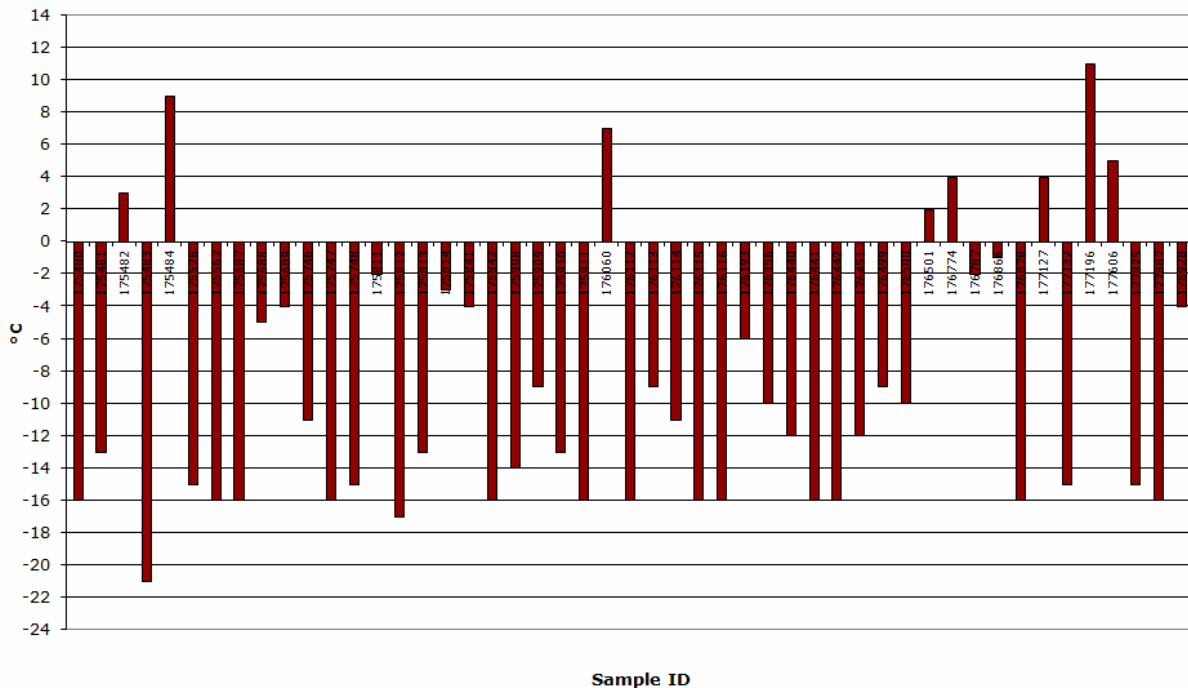
EN 14214	national regulations	°C
Min.	-21	°C
Max.	11	°C
Average	-9	°C
Std. Deviation	8.0	°C
Range 95% max	7	°C
Range 95% min	-25	°C
Out of spec	8 above 0 °C	-

Results

The results of the CFPP tests show EBB member companies exhibited good cold flow properties (obtained with or without cold flow improvers depending on the national regulations). Improvements in the precision of the EN116 test method are currently being investigated as it was originally designed to measure fossil fuels.

Summer 2010

CFPP



Sulphur Content

EBBQR – Summer 2010

The sulphur limit of FAME as defined by EN14214 is 10mg/kg. Up until the end of 2008 EN590 diesel fuel was allowed to have a sulphur content of 50mg/kg; this was subsequently lowered to 10mg/kg.

Summer 2010

EN 14214	< 10	mg/kg
Min.	<1	mg/kg
Max.	12.1	mg/kg
Average	4.2	mg/kg
Std. Deviation	2.9	mg/kg
Range 95% max	9.9	mg/kg
Range 95% min	-1.6	mg/kg
Out of spec	2	-

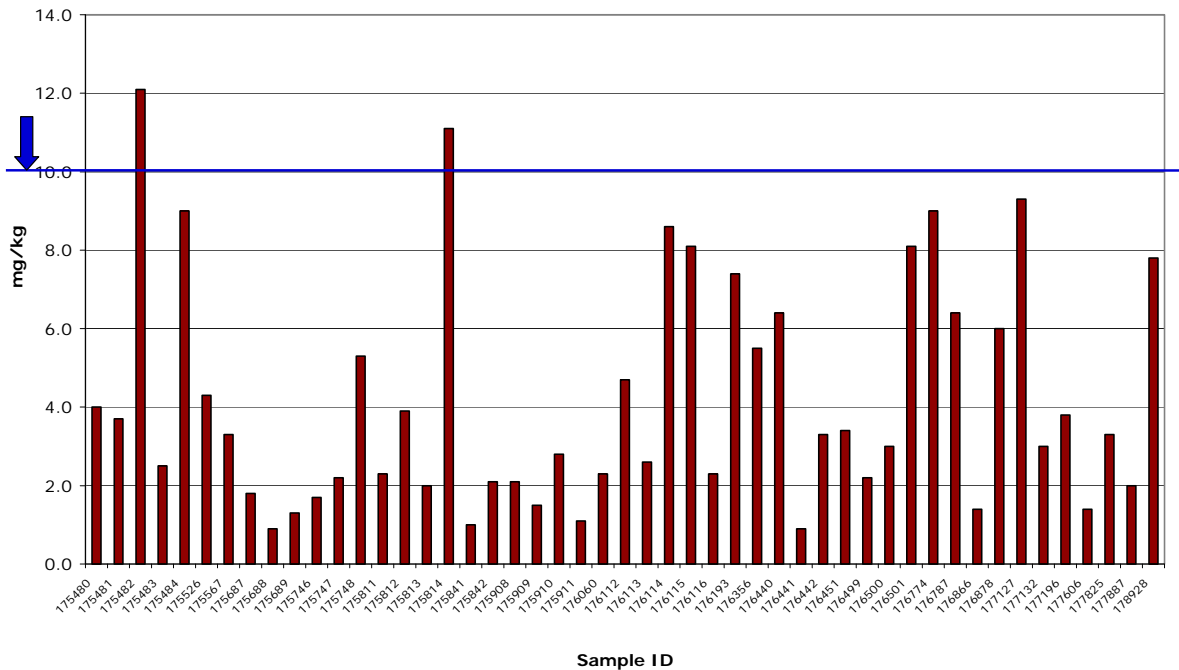
All values < 1 mg/kg rounded off 0,9 mg/kg

Results

The results of the samples were within the limits except for two results which were within the reproducibility limit of the test method.

Summer 2010

Sulfur Content



Carbon Residue (10%)

EBBQR – Summer 2010

The carbon residue of the sample is determined on the 10% distillation residue of the sample, and has a 0.30% limit by mass. For FAME fuels, carbon residue correlates with the amounts of glycerides, free fatty acids, soaps and catalyst residues.

Summer 2010

EN 14214	< 0,30	% m/m
Min.	0.01	% m/m
Max.	0.22	% m/m
Average	0.10	% m/m
Std. Deviation	0.05	% m/m
Range 95% max	0.20	% m/m
Range 95% min	-0.01	% m/m
Out of spec	0	-

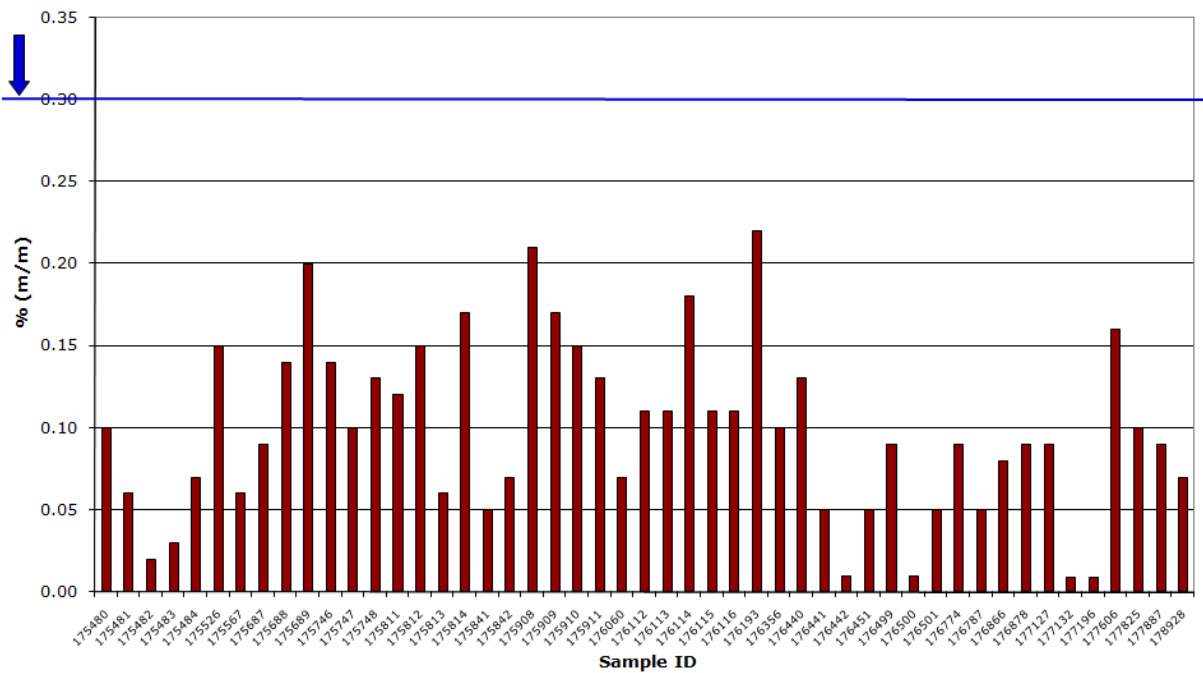
Results

All samples complied with the specification requirements. This test is under consideration by CEN and may not be required within future versions of EN14214.

All values < 0.01 % (m/m) rounded off 0.009% (m/m)

Summer 2010

Carbon Residue (10% D.)



Cetane Number

EBBQR – Summer 2010

The cetane number gives an indication of the ignition quality of fuels. A lower limit of the cetane number parameter of 51 is required in both EN590 and EN14214.

Summer 2010

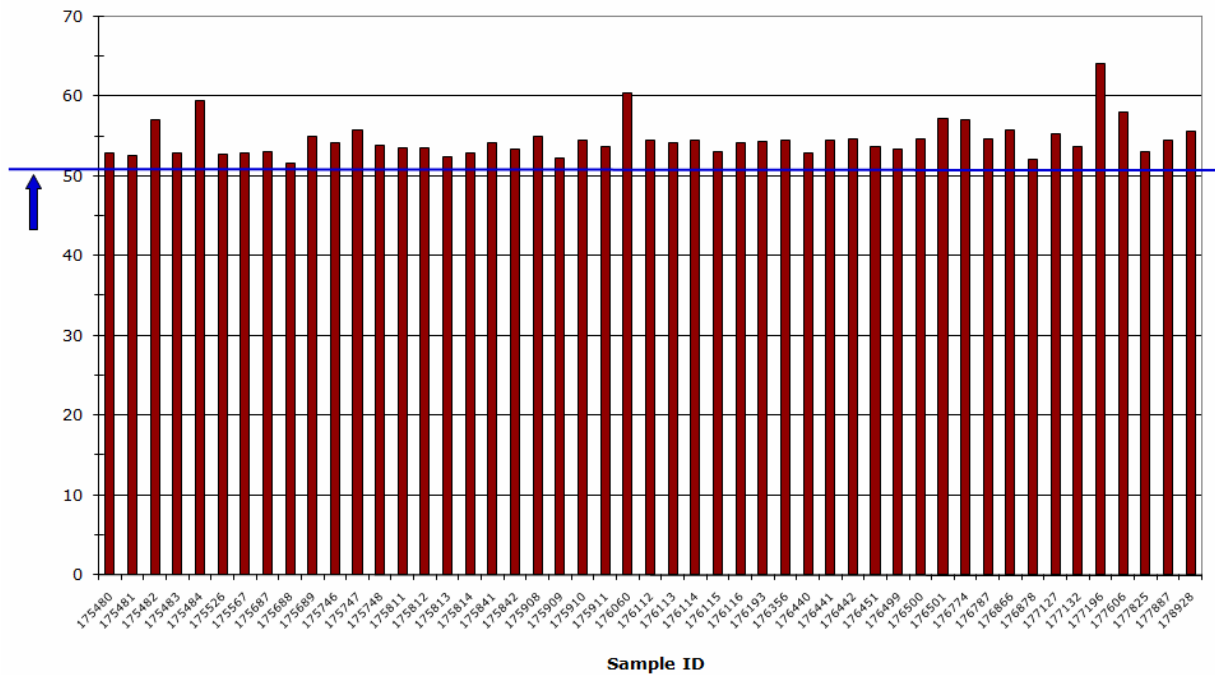
EN 14214	> 51	-
Min.	51.6	-
Max.	64.0	-
Average	54.5	-
Std. Deviation	2.2	-
Range 95% max	59.0	-
Range 95% min	50.0	-
Out of spec	0	-

Results

All samples exhibited cetane numbers above the specification limit of 51. Higher cetane number fuels have shorter ignition delays, which promote smooth engine running and cold temperature starts.

Summer 2010

Cetane Number



Sulphated Ash

EBBQR – Summer 2010

The corresponding value within the EN590 diesel fuel standard cannot be directly compared to the limit for FAME, as in diesel fuel the relevant contaminants are determined as oxides and not in the form of sulphates¹.

Summer 2010

EN 14214	<0,02	% m/m
Min.	<0,01	% m/m
Max.	0.009	% m/m
Average	0.009	% m/m
Std. Deviation	0.000	% m/m
Range 95% max	0.009	% m/m
Range 95% min	0.009	% m/m
Out of spec	0	-

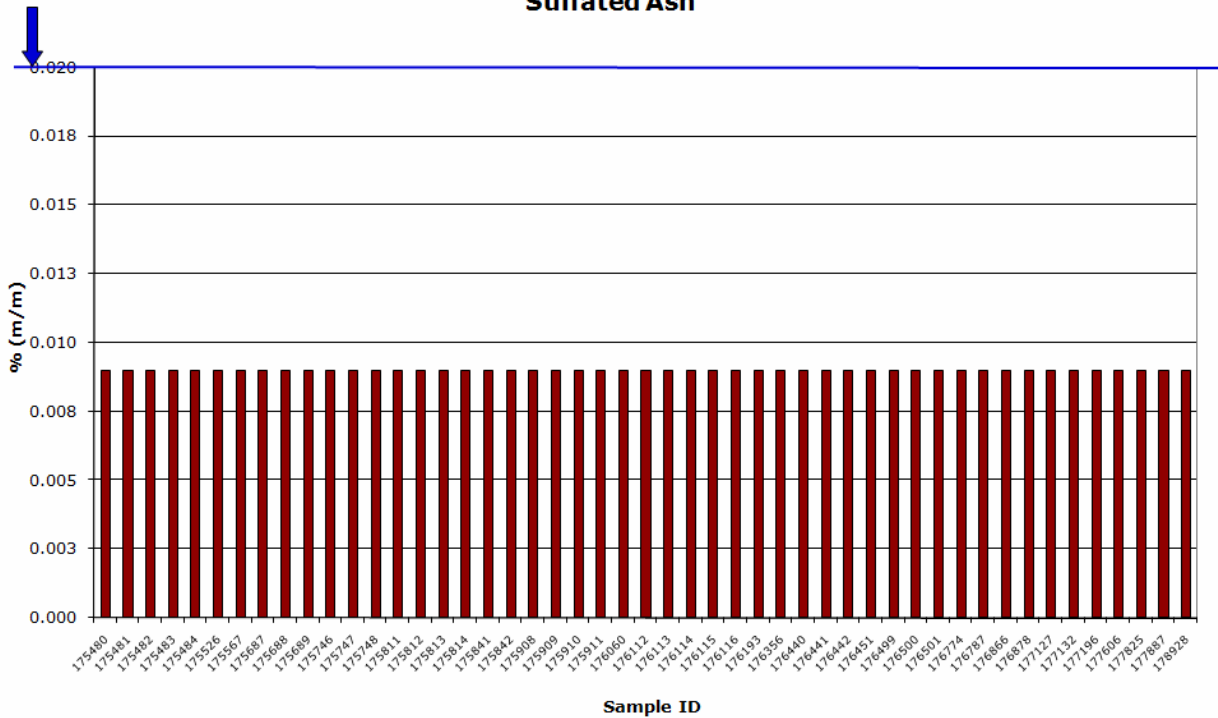
Results

All the samples were within the required limits of the test.

All values < 0,001% (m/m) rounded off 0,0009% (m/m)

Summer 2010

Sulfated Ash



Water Content

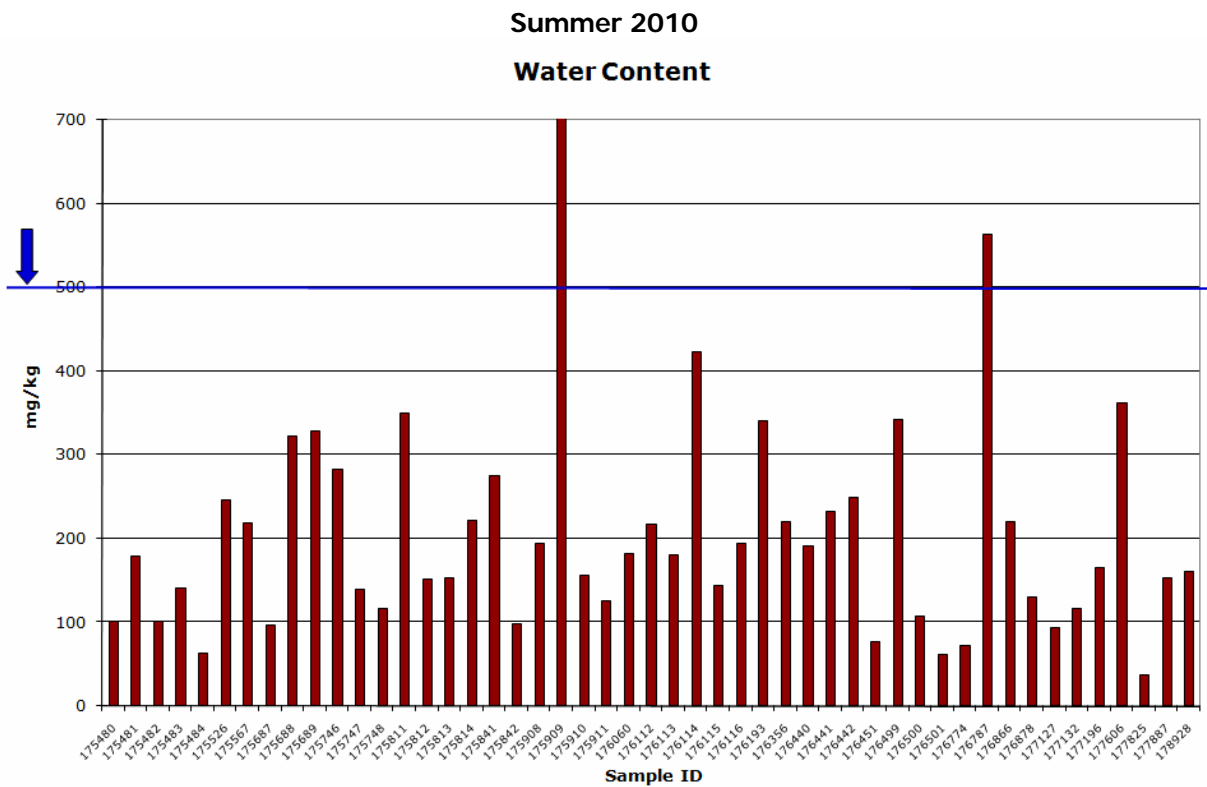
EBBQR – Summer 2010

The EN14214 limit for water content in FAME is 500mg/kg. Water is introduced into the production process during the ester washing step.

Summer 2010		
EN 14214	< 500	mg/kg
Min.	38	mg/kg
Max.	25000	mg/kg
Average	700	mg/kg
Std. Deviation	3509	mg/kg
Range 95% max	7718	mg/kg
Range 95% min	-6317	mg/kg
Out of spec	2	-

Results

The results of the Summer 2010 EBBQR show that all except two samples adhered to the 500mg/kg limit. One high sample was believed to be linked to water contamination of the sample containers. It is worth noting that the samples were taken at the production stage in the FAME process, and higher values are always seen after storage and logistical operations. The FAME must be compliant with EN14214 at the delivery stage.



Total Contamination

EBBQR – Summer 2010

The total contamination limit is 24mg/kg. While an important parameter from the point of view of assessing a fuel's filterability, the method (EN12662) is under review so that the measurement accuracy can be improved.

Summer 2010

EN 14214	< 24	mg/kg
Min.	<1	mg/kg
Max.	105	mg/kg
Average	12	mg/kg
Std. Deviation	16	mg/kg
Range 95% max	44	mg/kg
Range 95% min	-20	mg/kg
Out of spec	4	-

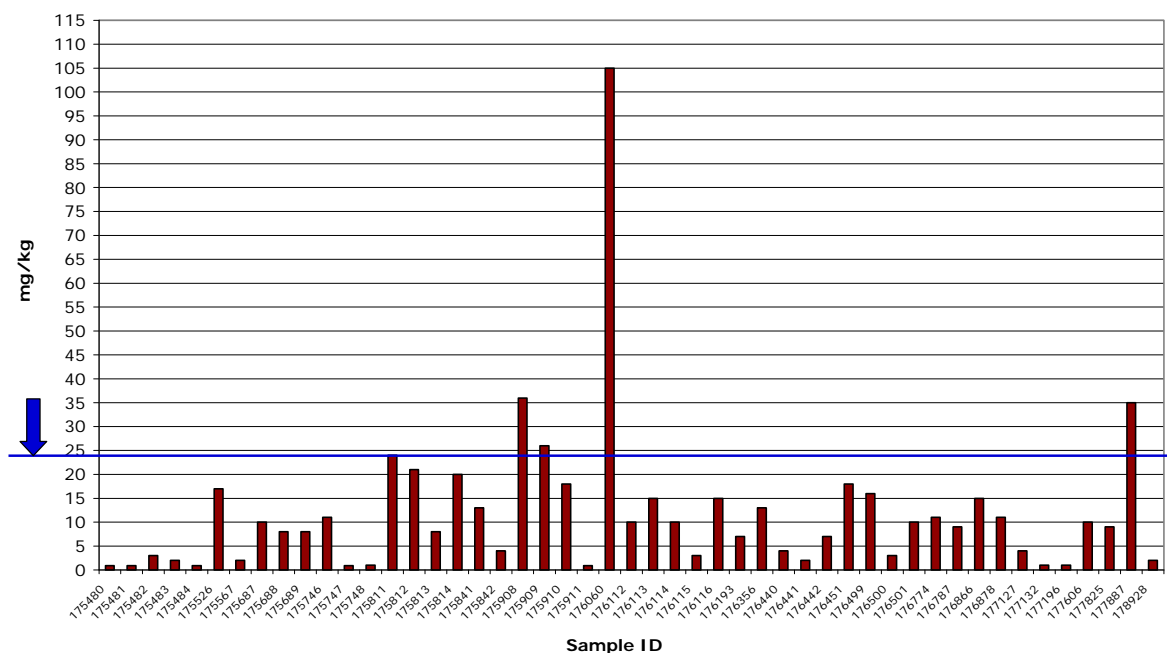
All values < 1 mg/kg rounded off 0.9 mg/kg

Results

One sample was above the test limit value of 24mg/kg and out of the reproducibility range. Three other samples produced results above the specification but remained within the method reproducibility.

Summer 2010

Total Contamination



Copper Strip Corrosion

EBBQR – Summer 2010

This test assesses the tendency of a fuel to corrode the copper, zinc and bronze parts of a car engine and tank, and is the same for both diesel fuel and FAMES.

Summer 2010

EN 14214	Class 1	-
Min.	Class 1	-
Max.	Class 1	-
Average	Class 1	-
Std. Deviation	-	-
Range 95% max	-	-
Range 95% min	-	-
Out of spec	0	-

Oxidation Stability, 110°C

EBBQR – Summer 2010

The oxidation stability limit is described as a minimum of a 6 hour Rancimat induction period, and is said to be the period of time after which FAME samples, aged under a constant air stream at 110°C begin to form volatile acids.

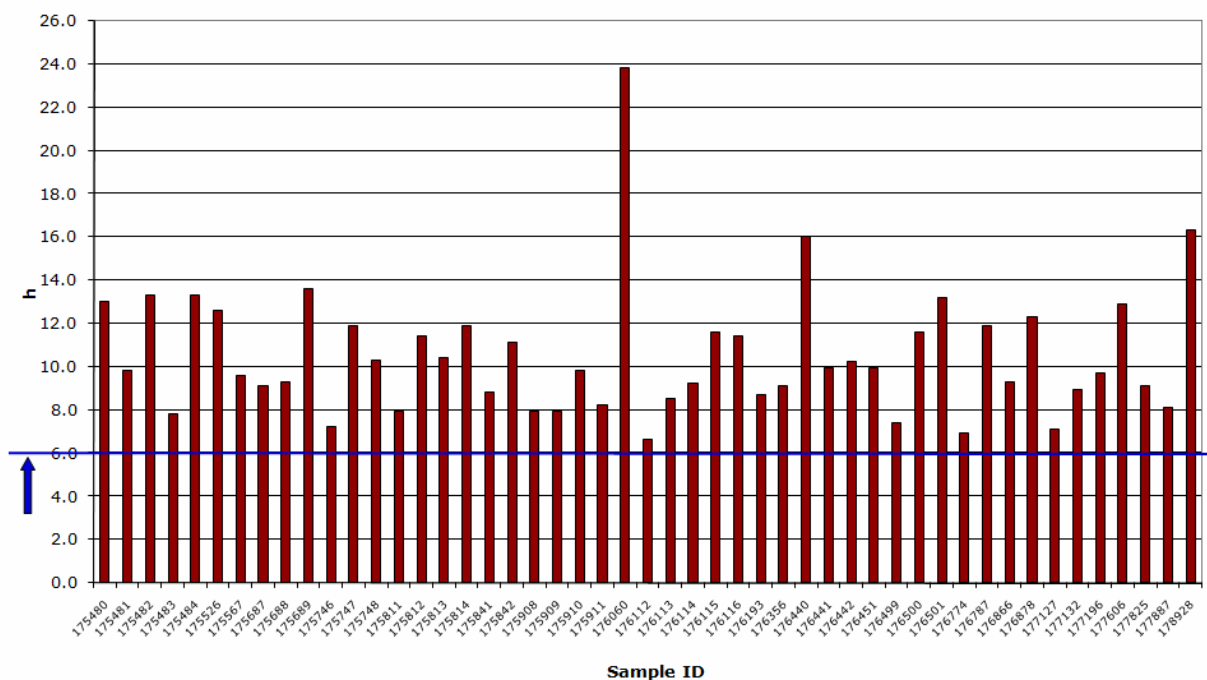
Summer 2010

EN 14214	> 6,0	h
Min.	6.6	h
Max.	23.8	h
Average	10.5	h
Std. Deviation	3.0	h
Range 95% max	16.4	h
Range 95% min	4.6	h
Out of spec	0	-

Results

All samples were above the 6 hour oxidation stability limit. The FAME must be compliant with EN14214 at the delivery stage.

Summer 2010 Oxidation Stability



Acid Value

EBBQR – Summer 2010

The acid value is a measure of the free fatty acids and mineral acids contained in a FAME sample. It is expressed in mg of KOH required to neutralise 1g of FAME.

Summer 2010

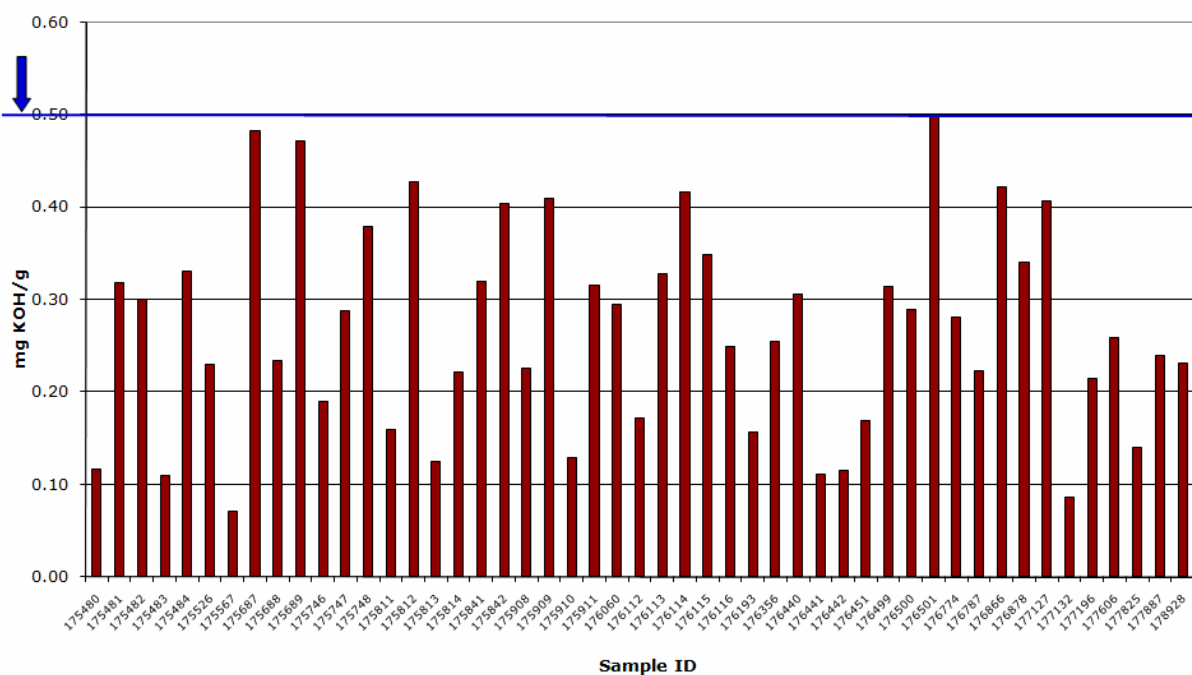
EN 14214	< 0,50	mg KOH/g
Min.	0.07	mg KOH/g
Max.	0.50	mg KOH/g
Average	0.27	mg KOH/g
Std. Deviation	0.11	mg KOH/g
Range 95% max	0.49	mg KOH/g
Range 95% min	0.05	mg KOH/g
Out of spec	0	-

Results

All samples met the limit of 0.50 mg KOH/g. The acid value is influenced by the type of feedstock used and the process parameters, and also gives an indication of fuel aging during storage.

Summer 2010

Acid Number



Iodine Value

EBBQR – Summer 2010

The iodine value is a measure of the total unsaturation within a mixture of fatty materials, and is expressed in grams of iodine which react with 100g of FAME sample.

Summer 2010

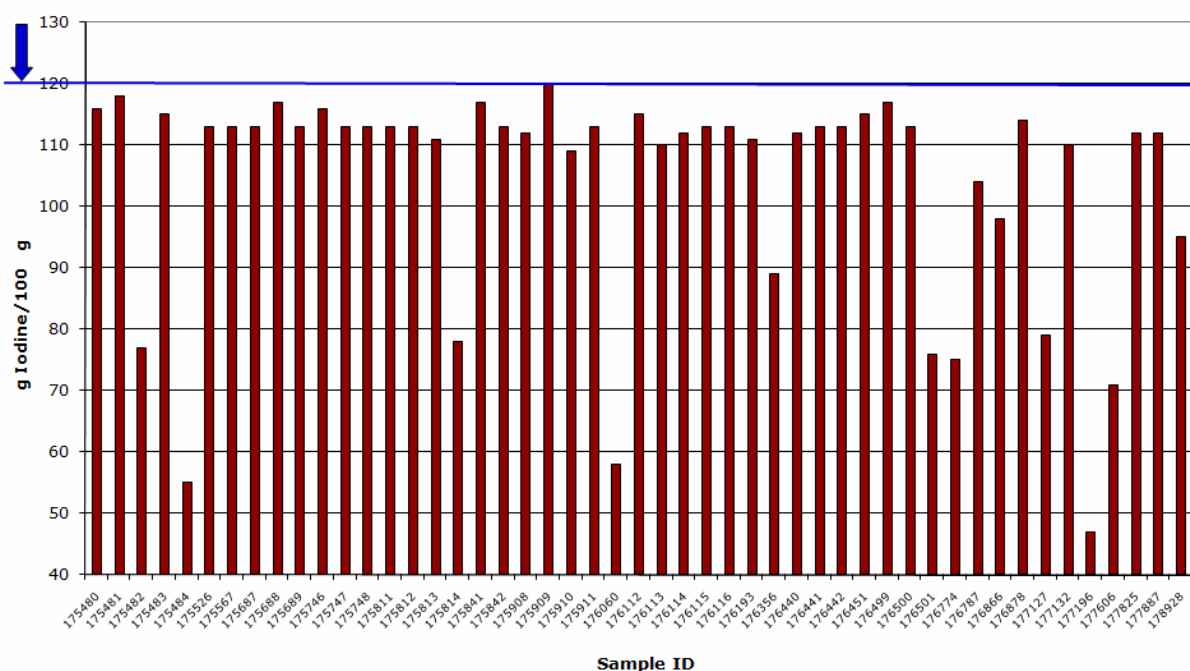
EN 14214	< 120	g Iodine/100 g
Min.	47	g Iodine/100 g
Max.	120	g Iodine/100 g
Average	104	g Iodine/100 g
Std. Deviation	18	g Iodine/100 g
Range 95% max	140	g Iodine/100 g
Range 95% min	67	g Iodine/100 g
Out of spec	0	-

Results

All samples were within the specification limit of 120 g Iod/100g. Iodine value gives an indication of the stability of a FAME sample against oxidation. Highly unsaturated compounds have also been linked with a decreased tendency to oxidise.

Summer 2010

Iodine Value



Linolenic Acid Methyl Ester

EBBQR – Summer 2010

Linolenic acid is a fatty acid which is considered to have a relatively high oxidation rate, the content of which is determined using the EN14103 method.

Summer 2010

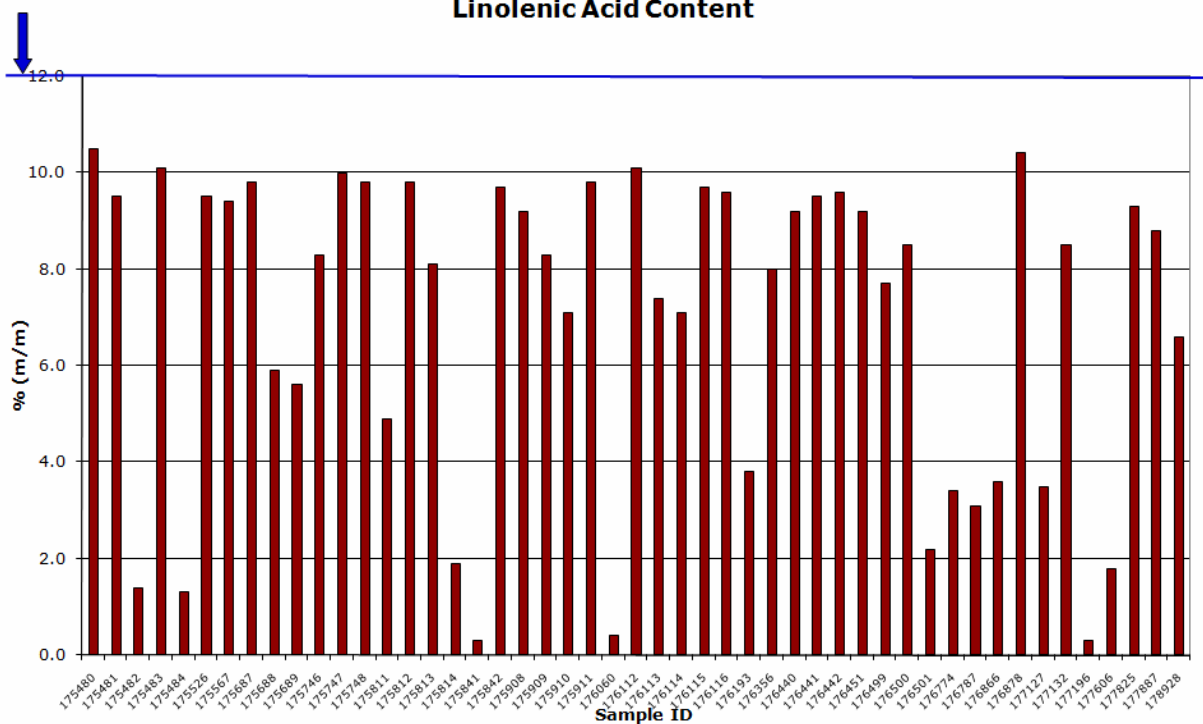
EN 14214	< 12	% m/m
Min.	0.3	% m/m
Max.	10.5	% m/m
Average	7.0	% m/m
Std. Deviation	3.2	% m/m
Range 95% max	13.4	% m/m
Range 95% min	0.5	% m/m
Out of spec	0	-

Results

The 12% (m/m) linolenic acid maximum content limit was respected by all samples.

Summer 2010

Linolenic Acid Content



Methanol Content

EBBQR – Summer 2010

Residual methanol in FAME is removed by washing or a distillation step, and respecting the EN14214 content limit is an important safety factor.

Summer 2010

EN 14214	< 0,20	% m/m
Min.	<0,01	% m/m
Max.	0.11	% m/m
Average	0.03	% m/m
Std. Deviation	0.03	% m/m
Range 95% max	0.09	% m/m
Range 95% min	-0.02	% m/m
Out of spec	0	-

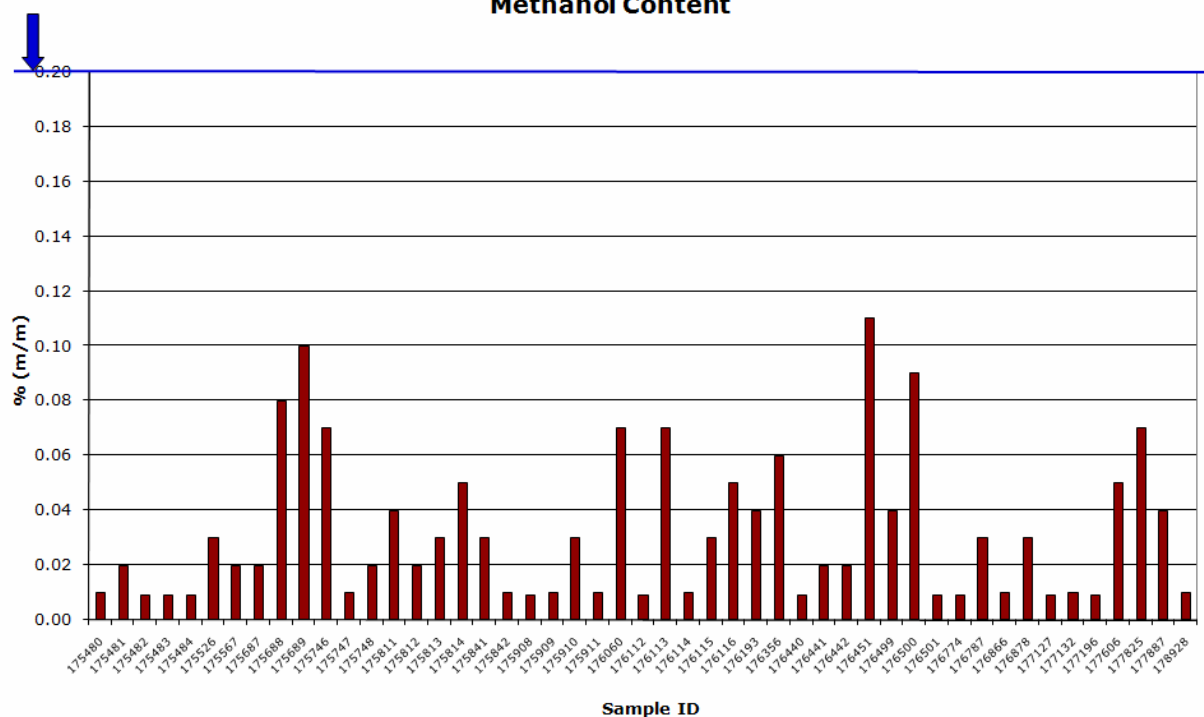
Results

The methanol content limit of 0.2% m/m was respected by all samples. Methanol content is measured using the gas chromatographic EN14110 method.

All values < 0.01% (m/m) rounded off 0.009% (m/m)

Summer 2010

Methanol Content



Free Glycerol

EBBQR – Summer 2010

Free glycerol content depends upon the production process. Free glycerol can collect at the bottom of tanks, and attract other polar compounds such as water.

Summer 2010

EN 14214	< 0,020	% m/m
Min.	<0,01	% m/m
Max.	0.05	% m/m
Average	0.01	% m/m
Std. Deviation	0.01	% m/m
Range 95% max	0.02	% m/m
Range 95% min	0.00	% m/m
Out of spec	2	-

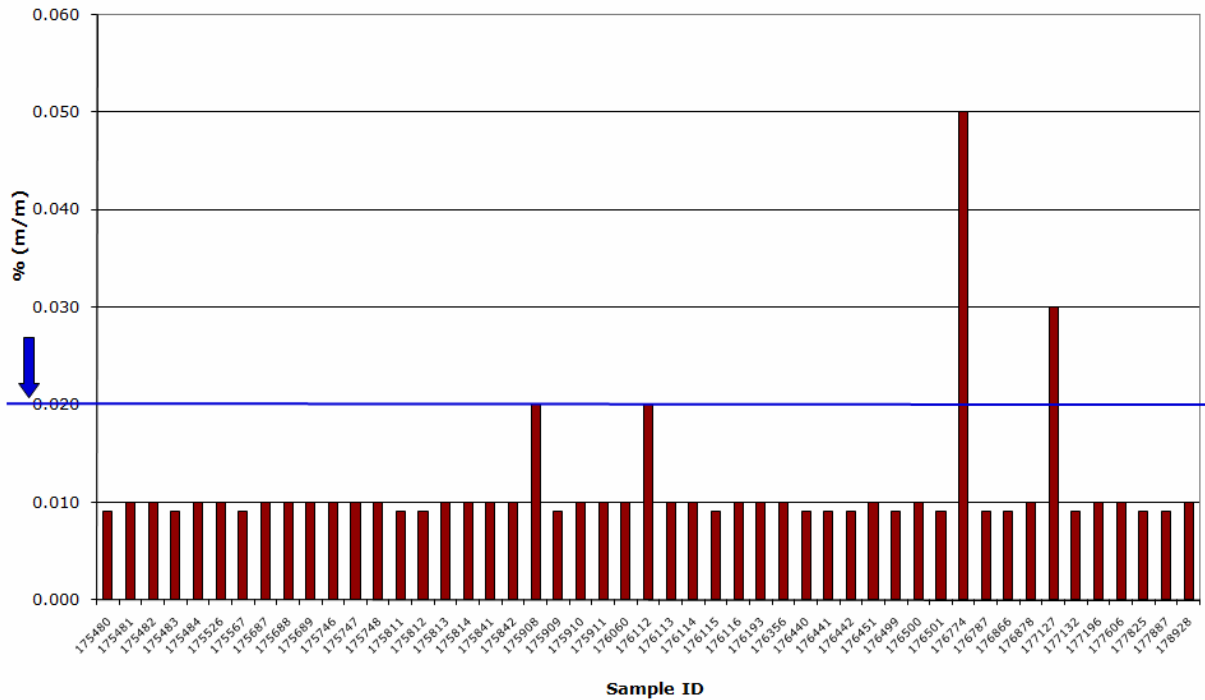
Results

Two samples appear to be above the limit. However during the GC analysis it was found a light ester originating from the animal fat feedstock co-elutes with the glycerol peak giving artificially high results. An alternative glycerol method EN14106:2003 can overcome this issue, however for now EN14105 remains the test method defined in EN14214 and must be used.

All values < 0.01% (m/m) rounded off 0.009% (m/m)

Summer 2010

free Glycerol



Mono, Di and Tri-Glycerides

EBBQR – Summer 2010

The glyceride levels within EN14214 are measured using EN14105 (a gas chromatographic method). The sum of the bound glycerol contained within the glycerides is also used when forming the total glycerol figure.

Summer 2010

Glycerides	Mono	Di	Tri	
EN 14214	< 0,80	< 0,20	< 0,20	% (m/m)
Min.	<0,01	<0,01	<0,01	% (m/m)
Max.	0.82	0.18	0.18	% (m/m)
Average	0.42	0.09	0.03	% (m/m)
Std. Deviation	0.19	0.05	0.03	% (m/m)
Range 95% max	0.80	0.19	0.09	% (m/m)
Range 95% min	0.05	0.00	-0.04	% (m/m)
Out of spec	1	0	0	-

All values < 0,01 %(m/m) rounded off 0,009 %(m/m)

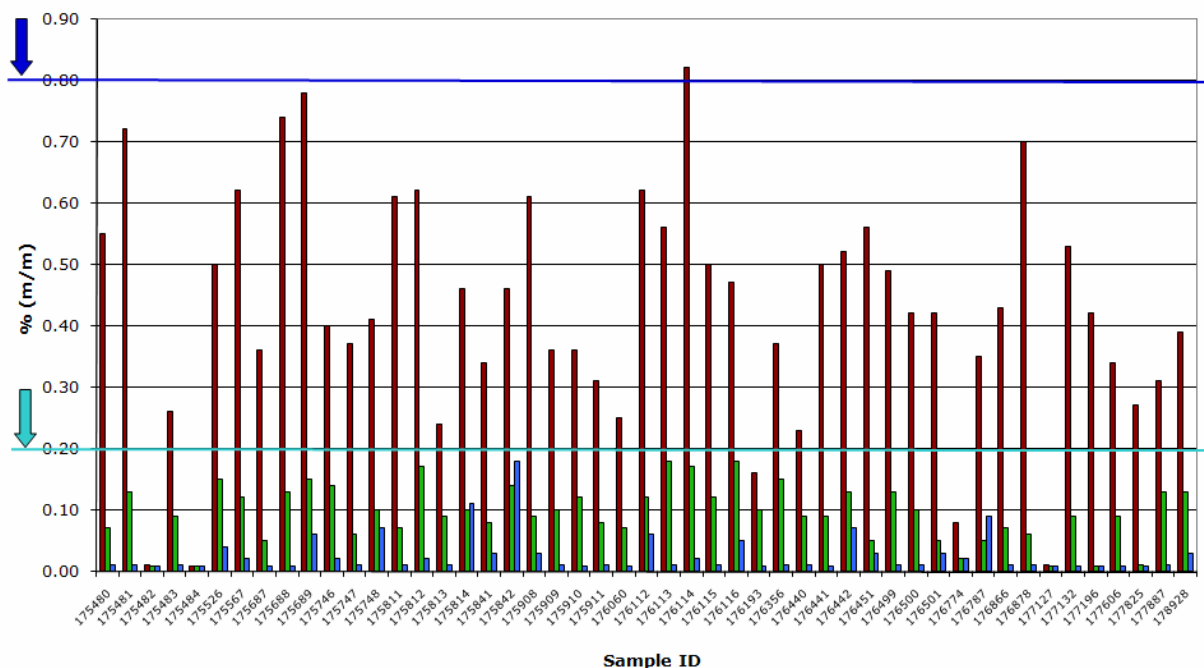
Results

In this testing round all except one monoglyceride result were within the specification limits.

It was noted that almost 80% of EBB members currently produce greater than 0.30% monoglycerides, and over 35% of EBB members produce more than 0.50% monoglycerides.

Summer 2010

Mono - Di - Tri Glyceride Content



Total Glycerol

EBBQR – Summer 2010

Total glycerol levels are measured using EN14105 (a gas chromatographic method).

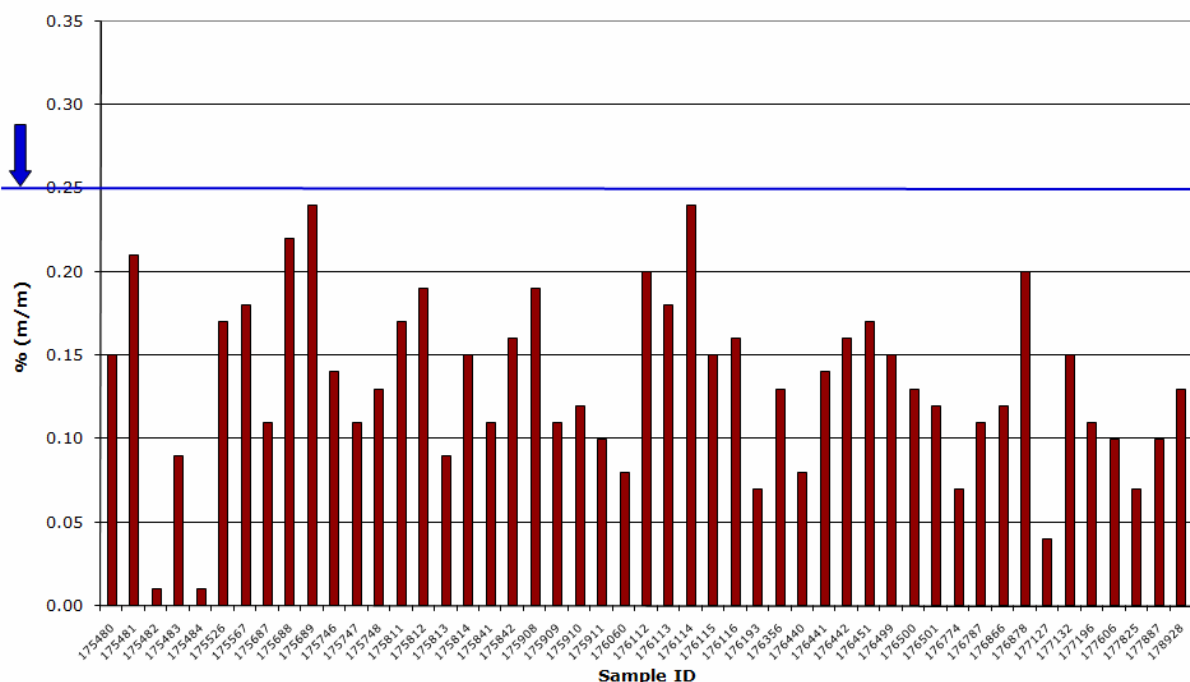
Summer 2010

EN 14214	< 0,25	% m/m
Min.	0.01	% m/m
Max.	0.24	% m/m
Average	0.13	% m/m
Std. Deviation	0.05	% m/m
Range 95% max	0.24	% m/m
Range 95% min	0.03	% m/m
Out of spec	0	-

Results

The results of the Summer 2010 EBBQR showed all samples respected the limit of the specification.

Summer 2010 Total Glycerol Content



Phosphorous Content

EBBQR – Summer 2010

EN14107 is used to measure the phosphorous content of the sample.

Summer 2010

EN 14214	< 4	mg/kg
Min.	<0,5	mg/kg
Max.	1.1	mg/kg
Average	0.4	mg/kg
Std. Deviation	0.1	mg/kg
Range 95% max	0.7	mg/kg
Range 95% min	0.2	mg/kg
Out of spec	0	-

All values <0,5 mg/kg rounded off 0,49 mg/kg

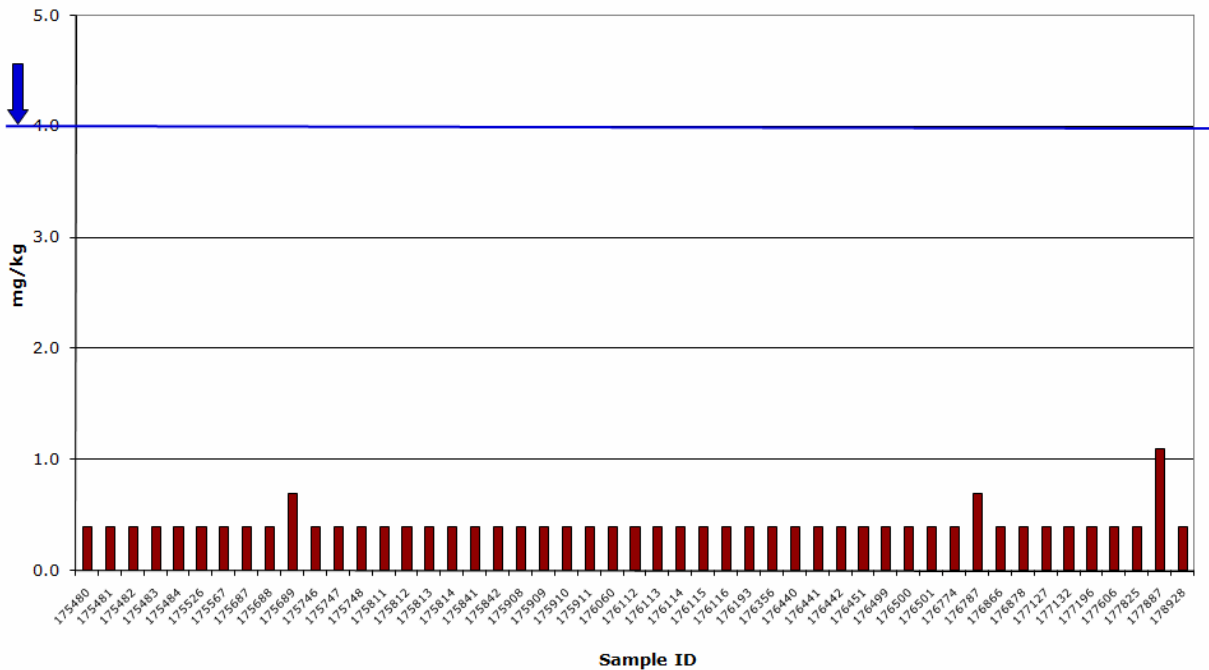
Results

All samples were below the phosphorous content limit of less than 4mg/kg.

It should be noted the test method is valid in the range 4-20 mg/kg, and this must be considered when analysing the results below. There is a new phosphorus test method (with an accuracy of < 1 mg/kg) currently under development by CEN.

Summer 2010

Phosphorous Content



Metals I (Na & K) & Metals II (Ca & Mg)

EBBQR – Summer 2010

EN14108/EN14109 is used to measure the sodium and potassium contents.

Summer 2010

	Metals I (Na + K)	Metals II (Ca + Mg)	
EN 14214	< 5,0	< 5,0	mg/kg
Min.	<0,5	<0,5	mg/kg
Max.	2.7	1.3	mg/kg
Average	0.7	0.4	mg/kg
Std. Deviation	0.6	0.2	mg/kg
Range 95% max	1.9	0.8	mg/kg
Range 95% min	-0.4	0.1	mg/kg
Out of spec	0	0	-

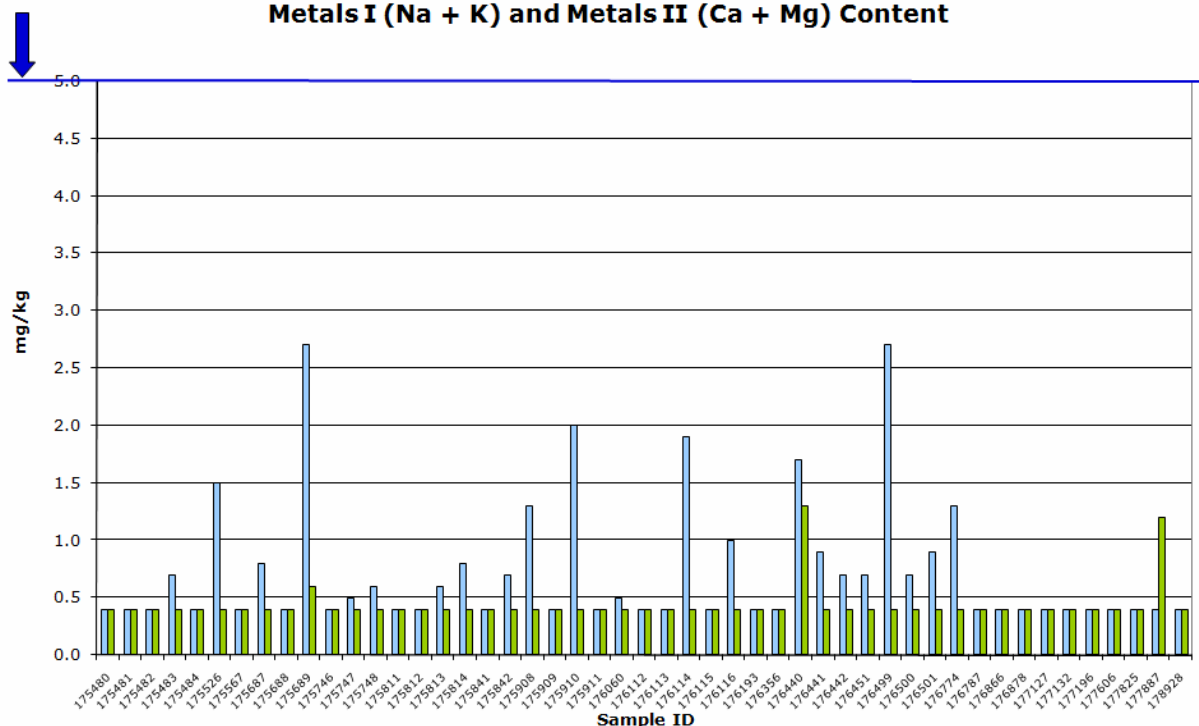
Results

All samples were within the content limits of less than 5mg/kg for both Group I and Group II metals.

All values < 0,5 mg/kg rounded off 0,49 mg/kg

Summer 2010

Metals I (Na + K) and Metals II (Ca + Mg) Content



Summary of Results

Final General Comments

Eighth sampling and analysis Summer 2010 quality

EBB members' production (49 plants monitored) is within the EN14214 limits; borderline samples for sulphur, water, total contamination and total monoglyceride were noted however these fell within the reproducibility (R) of the relevant test method.

The 95% range shows the adherence to EN14214, and also the high quality levels achieved by the European producers.

The results which are not in range with respect to EN14214 are:

Production parameters:

1 result for ester content
2 results for water content
1 result for total contamination

8 results for CFPP above 0°C (certain CFPP results were direct production results, without additives)

ⁱ Mittelbach and Remschmidt, 2004. 'Biodiesel – The Comprehensive Handbook'.