

# **EBB**

## **European Biodiesel Board**

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## **EBB EUROPEAN BIODIESEL QUALITY REPORT (EBBQR)**

### **Winter 2010/2011 Results Results of the ninth 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.

## Table of Contents

Background .....	1
Table of Contents.....	1
Ester Content.....	2
Density at 15°C .....	3
Kinematic Viscosity at 40°C .....	4
Flash Point.....	5
Cold Filter Plugging Point (CFPP) .....	6
Sulphur Content .....	7
Carbon Residue (10%) .....	8
Cetane Number.....	9
Sulphated Ash.....	10
Water Content .....	11
Total Contamination .....	12
Copper Strip Corrosion.....	13
Oxidation Stability, 110°C .....	14
Acid Value .....	15
Iodine Value .....	16
Linolenic Acid Methyl Ester .....	17
Poly Unsaturated Fatty Acid Methyl ester.....	18
Methanol Content.....	19
Free Glycerol.....	20
Mono, Di and Tri-Glycerides .....	21
Total Glycerol.....	22
Phosphorous Content.....	23
Metals I (Na & K) & Metals II (Ca & Mg).....	24
Summary of Results .....	25

## Ester Content

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

EN 14214	> 96,5	% m/m
Min.	95.1	% m/m
Max.	>99	% m/m
Average	98.6	% m/m
Std. Deviation	0.9	% m/m
Range 95% max	100.3	% m/m
Range 95% min	96.9	% m/m
Out of spec	2	-

All values > 99% rounded off 99.1%

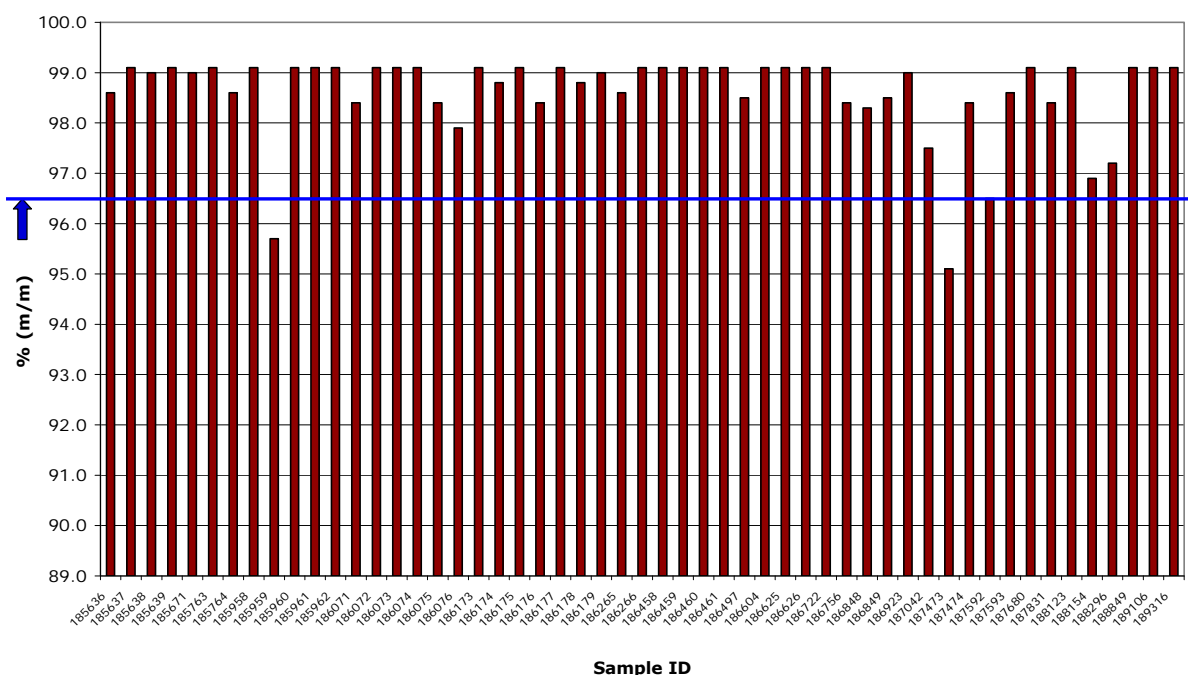
### Results

The average value seen in the Winter 2010/2011 EBBQR was 98.6%, with minimum and maximum figures of 94.5% and > 99% respectively. Two results below the 96.5% limit these were within method reproducibility.

The ester content test method (EN14103) can underestimate C17 content, resulting in an incorrectly low overall ester figure for FAMES using animal fat feedstocks. A new more accurate version, using C19 as a standard is now available.

### Winter 2010/2011

#### Ester Content



## Density at 15°C

EBBQR – Winter 2010/2011

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).

### Winter 2010/2011

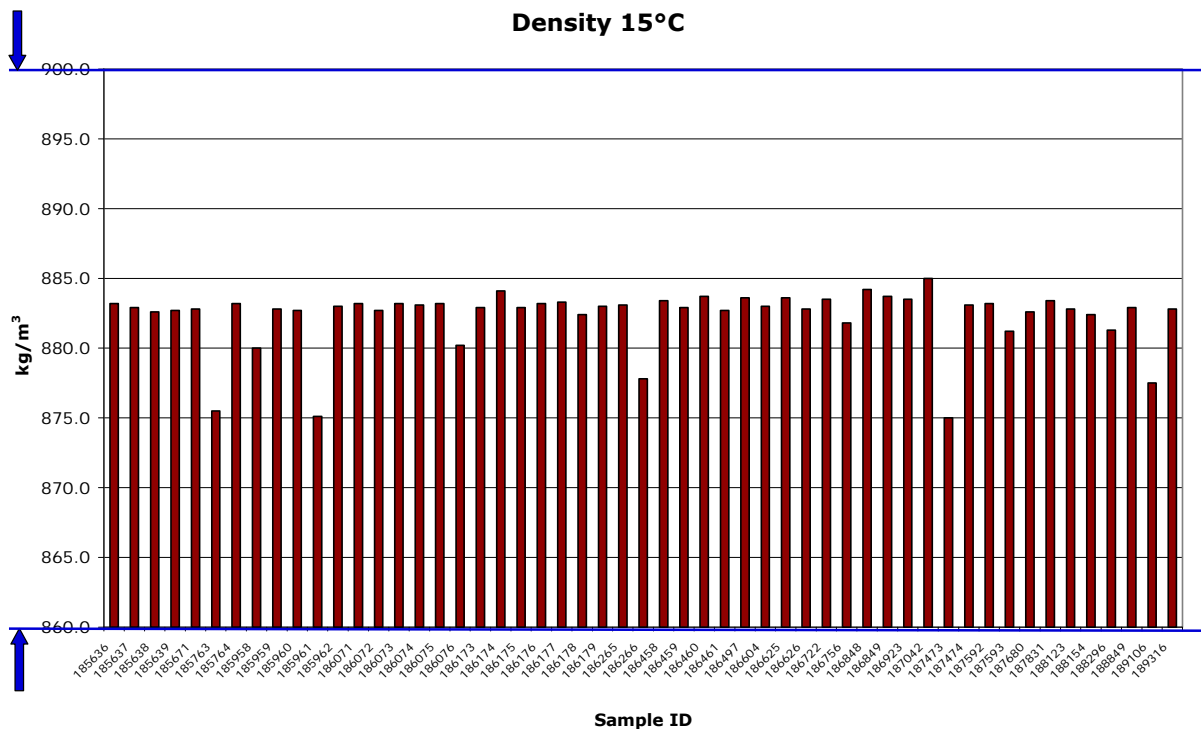
EN 14214	860 - 900	kg/m <sup>3</sup>
Min.	875	kg/m <sup>3</sup>
Max.	885	kg/m <sup>3</sup>
Average	882	kg/m <sup>3</sup>
Std. Deviation	2	kg/m <sup>3</sup>
Range 95% max	887	kg/m <sup>3</sup>
Range 95% min	878	kg/m <sup>3</sup>
Out of spec	0	-

### 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.

### Winter 2010/2011

#### Density 15°C



## Kinematic Viscosity at 40°C

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

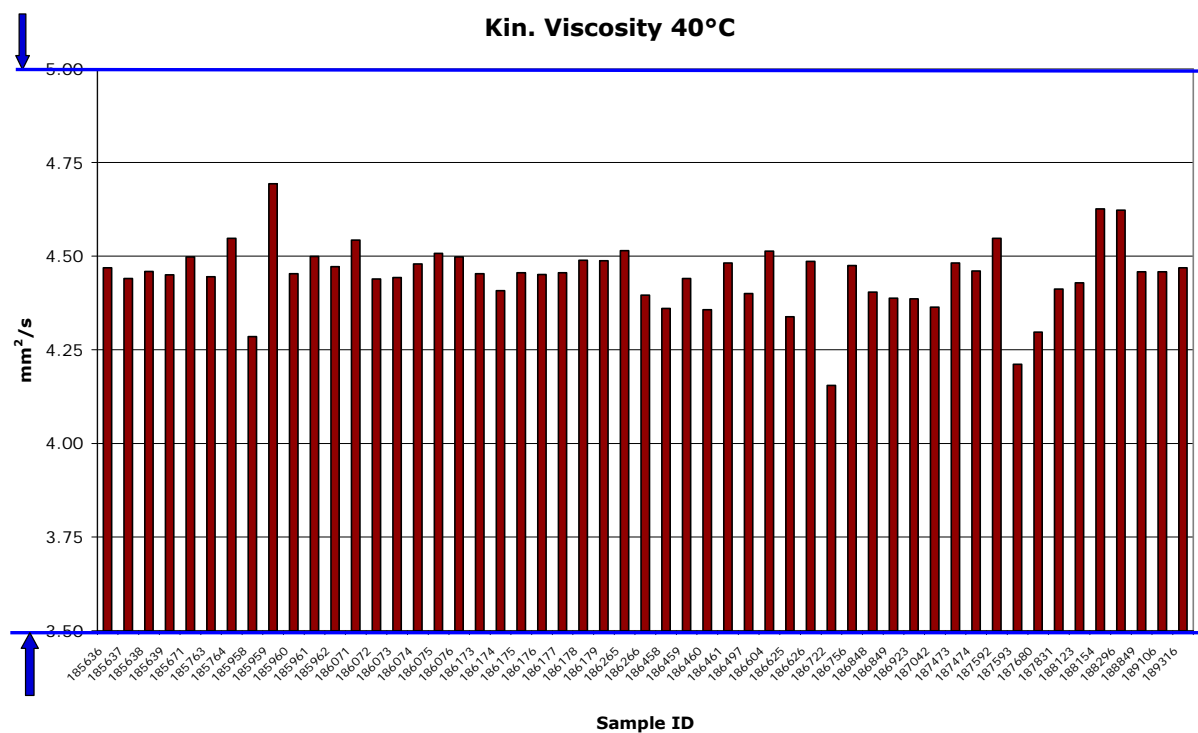
EN 14214	3,50 - 5,00	mm <sup>2</sup> /s
Min.	4.16	mm <sup>2</sup> /s
Max.	4.69	mm <sup>2</sup> /s
Average	4.45	mm <sup>2</sup> /s
Std. Deviation	0.09	mm <sup>2</sup> /s
Range 95% max	4.63	mm <sup>2</sup> /s
Range 95% min	4.27	mm <sup>2</sup> /s
Out of spec	0	-

### 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.

### Winter 2010/2011

#### Kin. Viscosity 40°C



## Flash Point

EBBQR – Winter 2010/2011

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.

### Winter 2010/2011

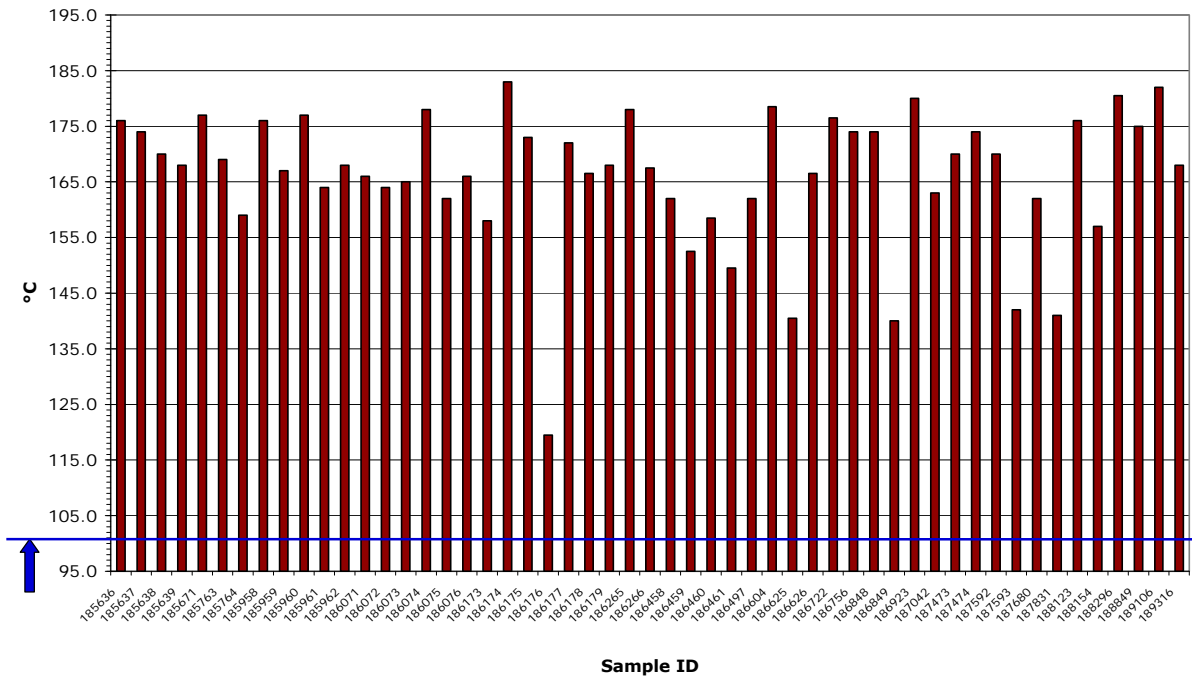
EN 14214	> 101	°C
Min.	120	°C
Max.	183	°C
Average	166	°C
Std. Deviation	12	°C
Range 95% max	191	°C
Range 95% min	142	°C
Out of spec	0	-

### Results

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

### Winter 2010/2011

#### Flash Point



## Cold Filter Plugging Point (CFPP)

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

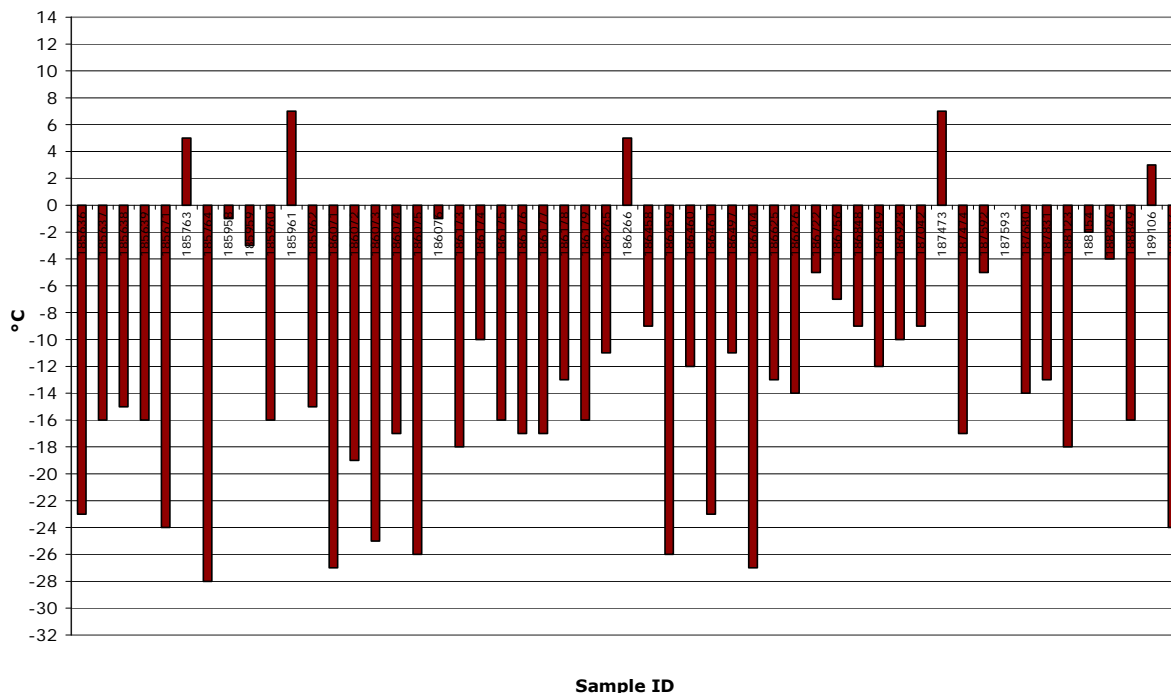
EN 14214	national regulations	°C
Min.	-28	°C
Max.	7	°C
Average	-13	°C
Std. Deviation	9.0	°C
Range 95% max	5	°C
Range 95% min	-31	°C
Above 0°C	5 above 0 °C	-

### 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.

### Winter 2010/2011

#### CFPP



## Sulphur Content

EBBQR – Winter 2010/2011

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.

### Winter 2010/2011

EN 14214	< 10	mg/kg
Min.	<1	mg/kg
Max.	39.9	mg/kg
Average	5.9	mg/kg
Std. Deviation	5.8	mg/kg
Range 95% max	17.4	mg/kg
Range 95% min	-5.6	mg/kg
Out of spec	6	-

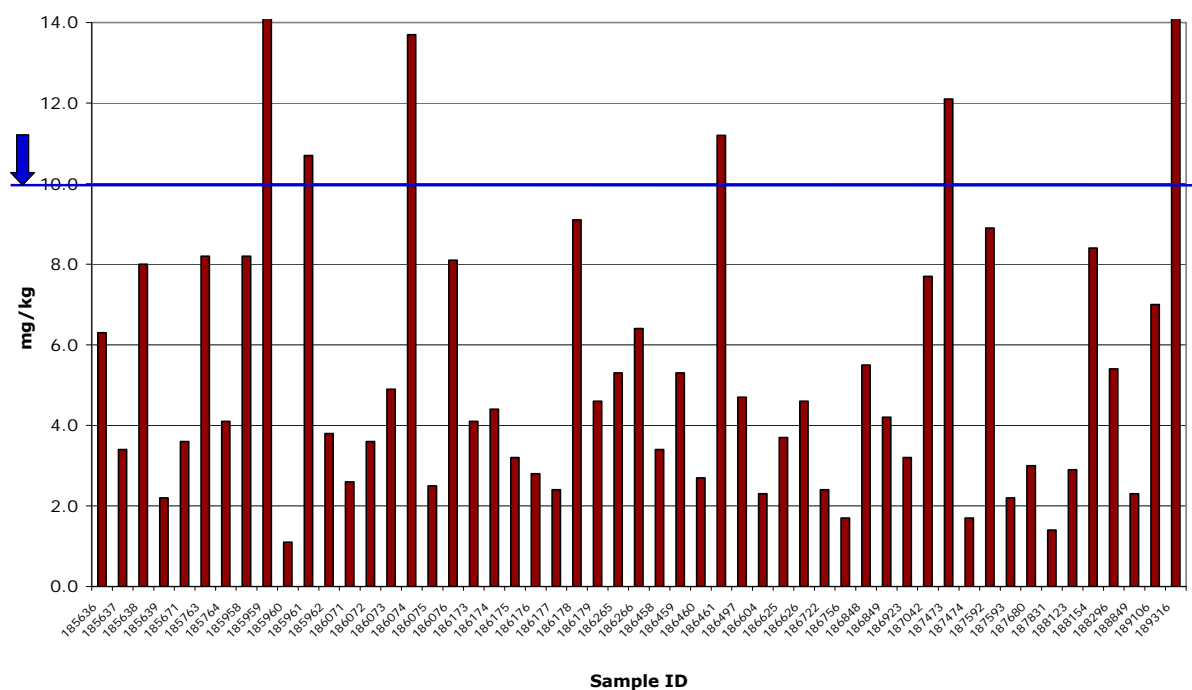
All values < 1 mg/kg rounded off 0,9 mg/kg

### Results

Six results were outside above the limit of the test however three of these results were within the reproducibility limit of the test method.

### Winter 2010/2011

#### Sulfur Content





## Carbon Residue (10%)

*EBBQR – Winter 2010/2011*

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<sup>1</sup>.

### Winter 2010/2011

EN 14214	< 0,30	% m/m
Min.	0.01	% m/m
Max.	0.56	% 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	3	-

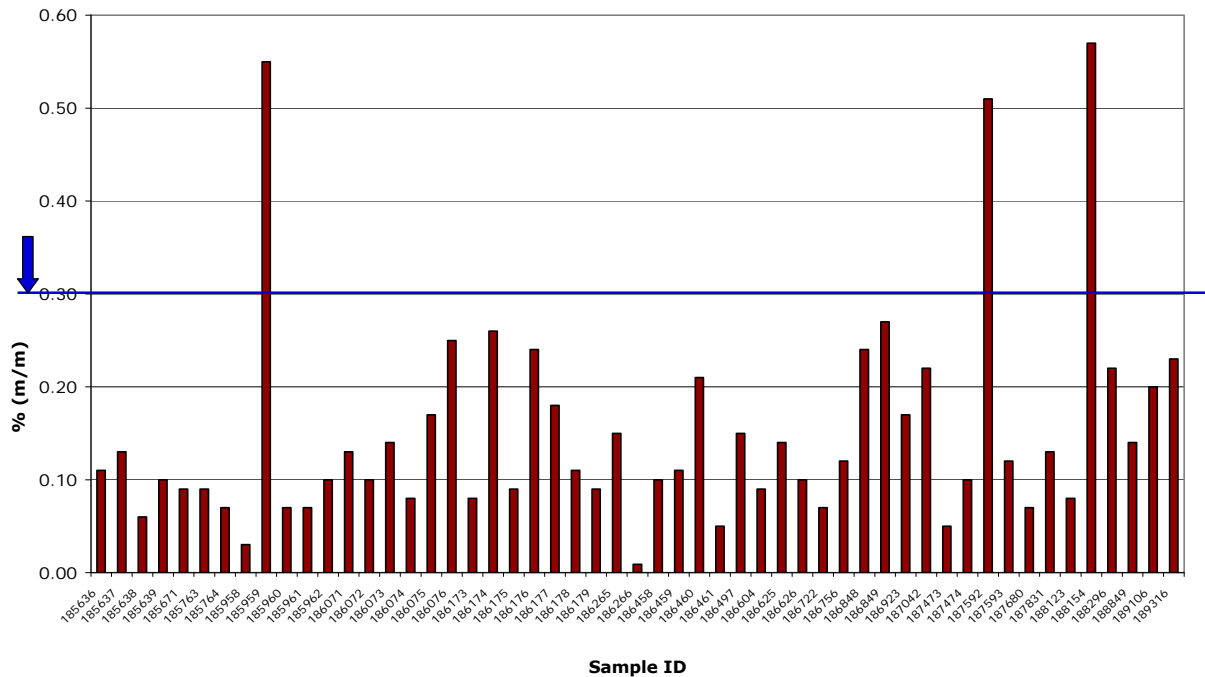
All values < 0.01 % (m/m) rounded off 0.009% (m/m)

### Results

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

### Winter 2010/2011

#### Carbon Residue (10% D.)



## Cetane Number

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

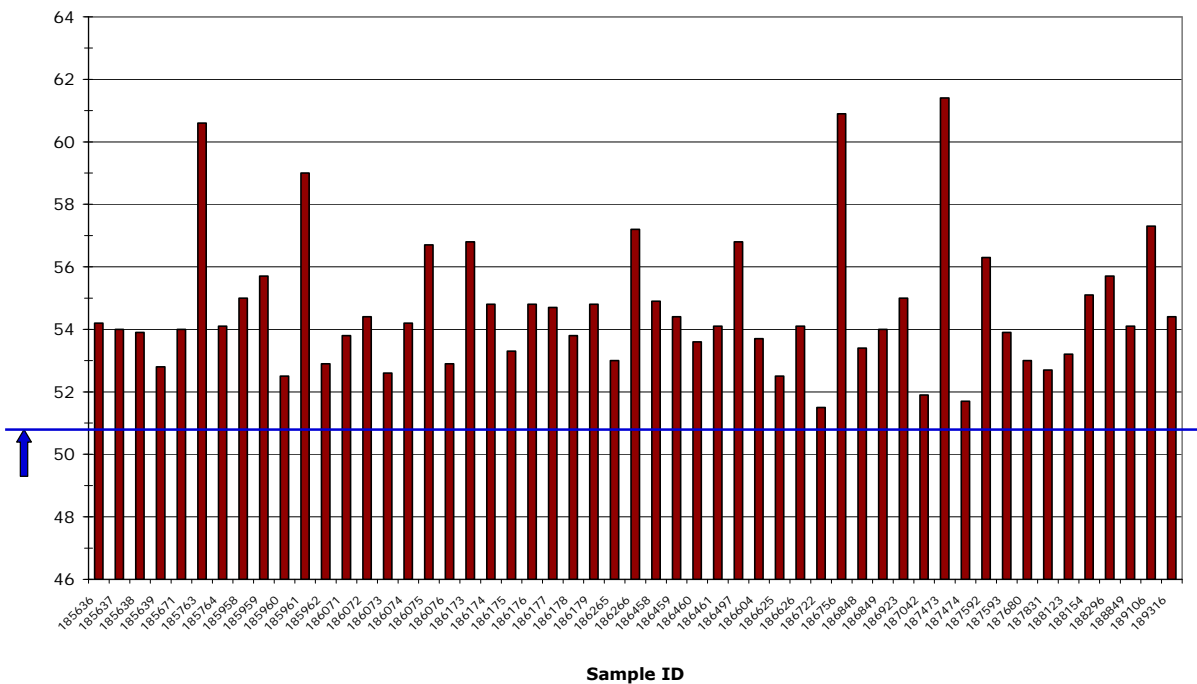
EN 14214	> 51	-
Min.	51.5	-
Max.	61.4	-
Average	54.6	-
Std. Deviation	2.1	-
Range 95% max	58.9	-
Range 95% min	50.4	-
Out of spec	0	-

### 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.

### Winter 2010/2011

#### Cetane Number



## Sulphated Ash

*EBBQR – Winter 2010/2011*

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 sulphates<sup>1</sup>.

### Winter 2010/2011

EN 14214	<0,02	% m/m
Min.	<0,01	% m/m
Max.	0.020	% m/m
Average	0.009	% m/m
Std. Deviation	0.001	% m/m
Range 95% max	0.012	% m/m
Range 95% min	0.006	% m/m
Out of spec	0	-

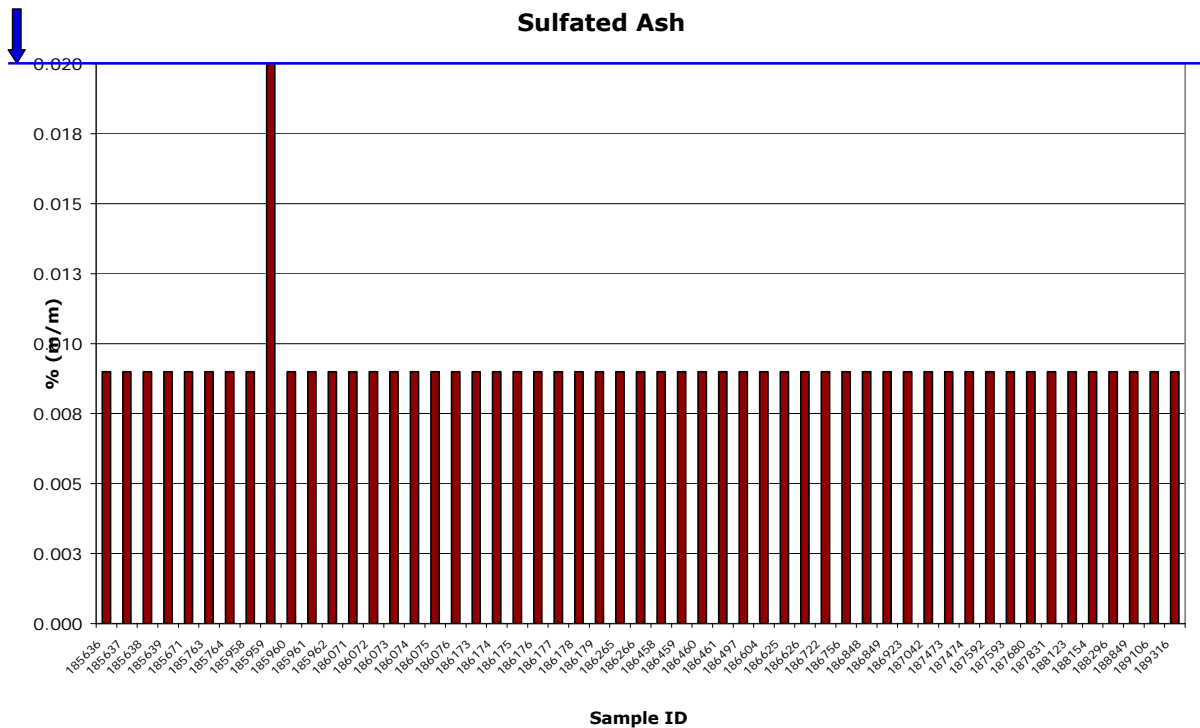
### Results

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

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

### Winter 2010/2011

#### Sulfated Ash





## Total Contamination

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

EN 14214	< 24	mg/kg
Min.	< 1	mg/kg
Max.	51	mg/kg
Average	9	mg/kg
Std. Deviation	8	mg/kg
Range 95% max	26	mg/kg
Range 95% min	-7	mg/kg
Out of spec	1	-

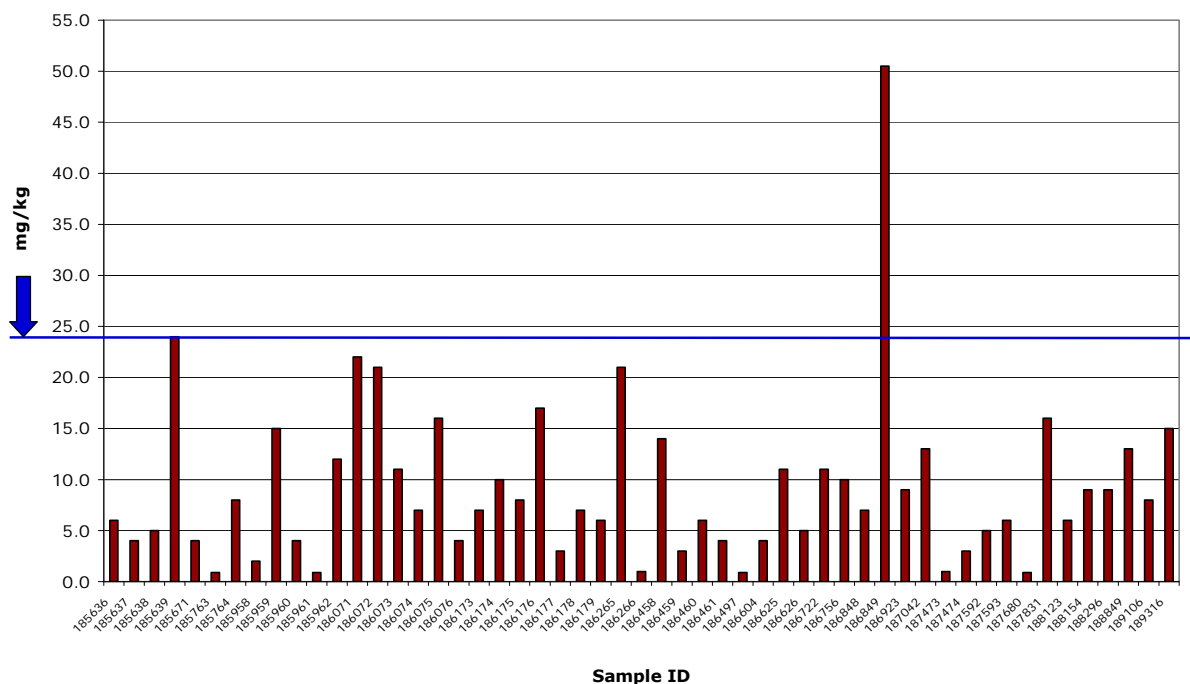
### Results

One sample was above the test limit value of 24mg/kg. All other samples remained within the limits of the specification.

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

### Winter 2010/2011

#### Total Contamination



## Copper Strip Corrosion

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

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 – Winter 2010/2011*

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.

### Winter 2010/2011

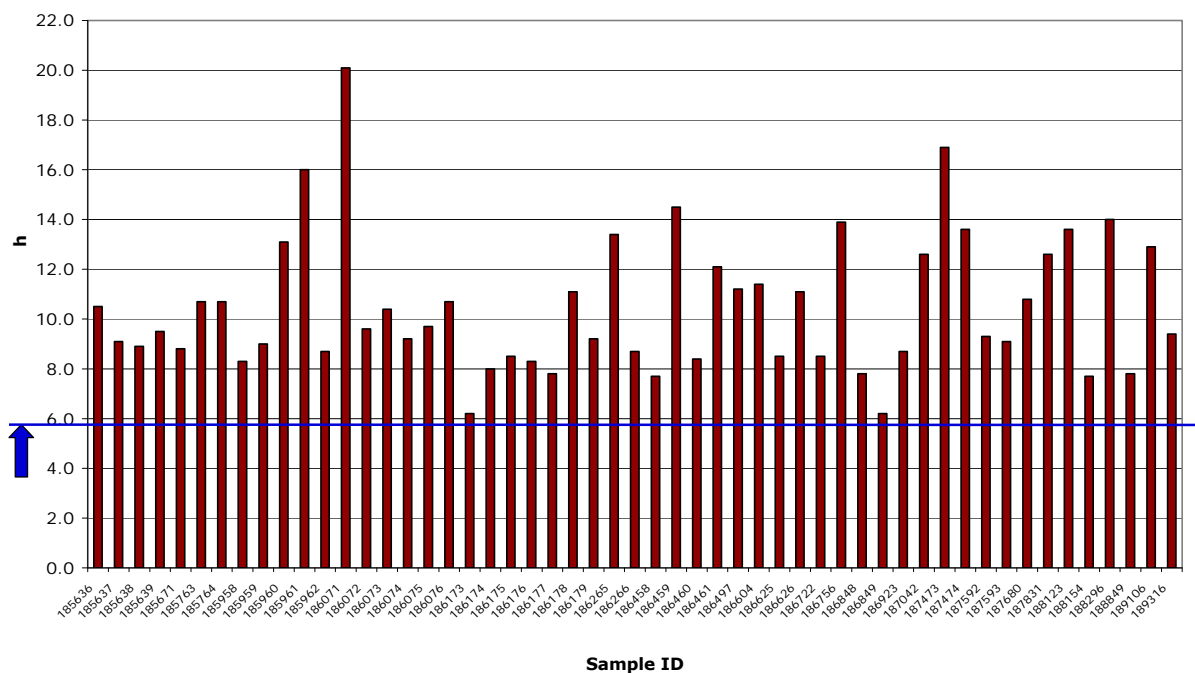
EN 14214	> 6,0	h
Min.	6.2	h
Max.	20.1	h
Average	10.5	h
Std. Deviation	2.7	h
Range 95% max	15.9	h
Range 95% min	5.0	h
Out of spec	0	-

### Results

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

### Winter 2010/2011

#### Oxidation Stability



## Acid Value

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

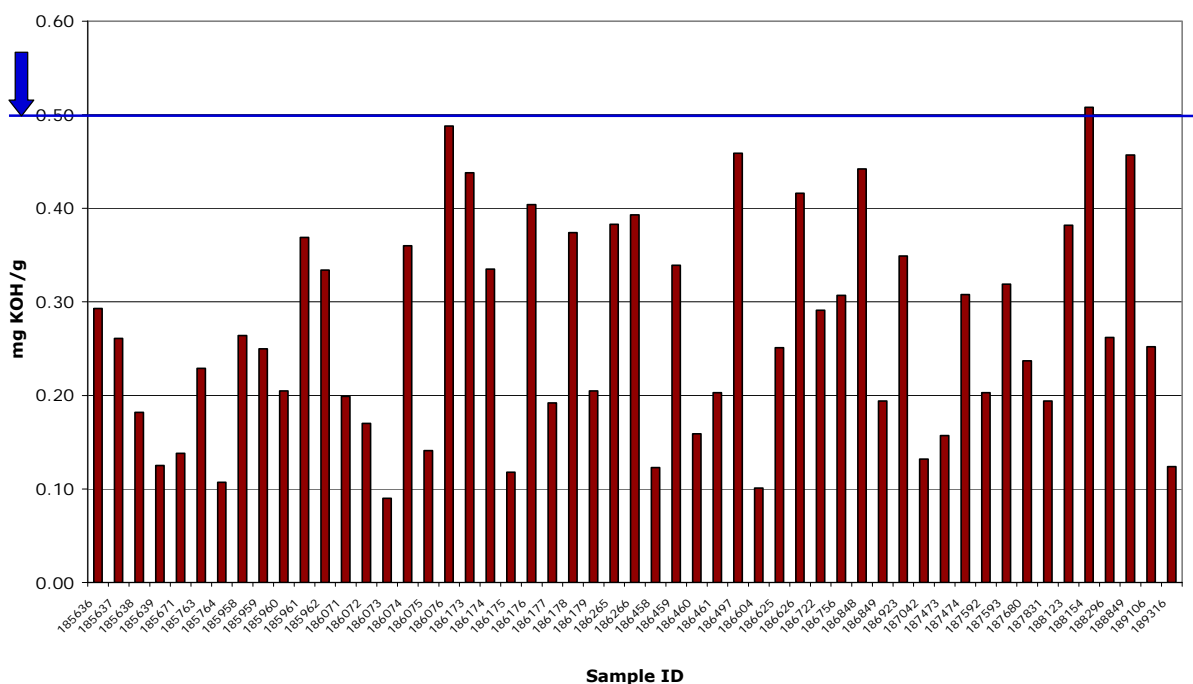
EN 14214	< 0,50	mg KOH/g
Min.	0.09	mg KOH/g
Max.	0.51	mg KOH/g
Average	0.27	mg KOH/g
Std. Deviation	0.11	mg KOH/g
Range 95% max	0.49	mg KOH/g
Range 95% min	0.04	mg KOH/g
Out of spec	1	-

### Results

All samples except one met the limit of 0.50 mg KOH/g and it is also worth noting this was within the method reproducibility. 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.

### Winter 2010/2011

#### Acid Number





## Iodine Value

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

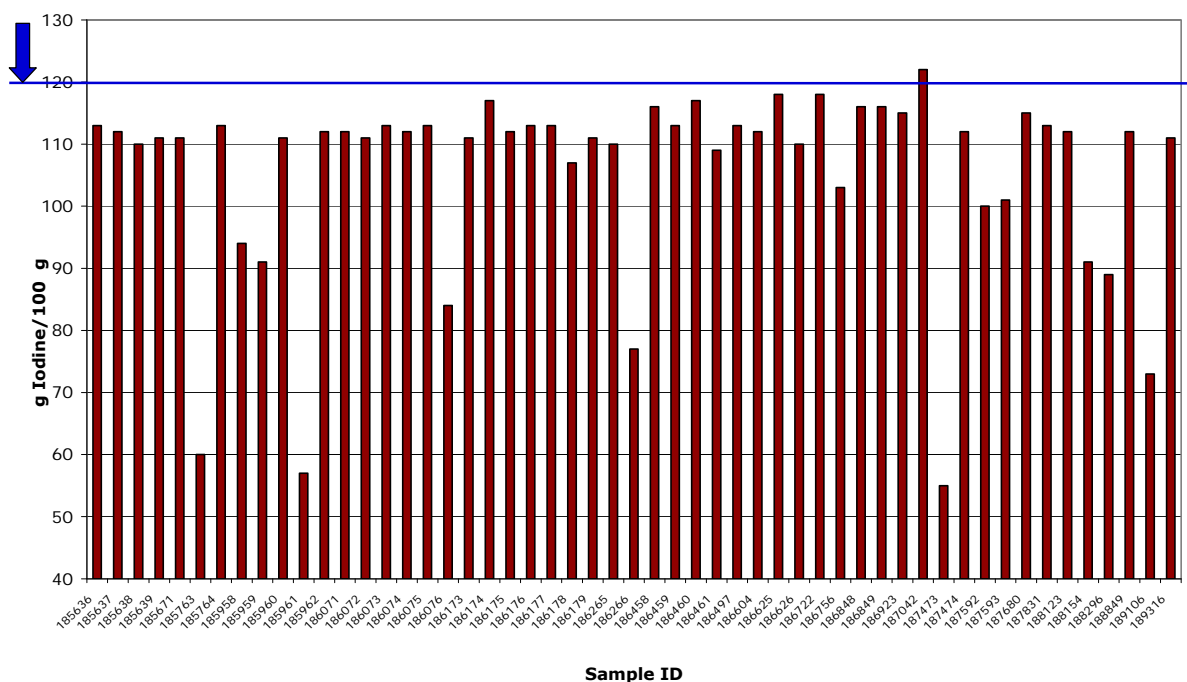
EN 14214	< 120	g Iodine/100 g
Min.	55	g Iodine/100 g
Max.	122	g Iodine/100 g
Average	106	g Iodine/100 g
Std. Deviation	15	g Iodine/100 g
Range 95% max	137	g Iodine/100 g
Range 95% min	75	g Iodine/100 g
Out of spec	1	-

### Results

All samples except one were within the specification limit of 120 g Iod/100g, however this result was within the method reproducibility. 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.

### Winter 2010/2011

#### Iodine Value



## Linolenic Acid Methyl Ester

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

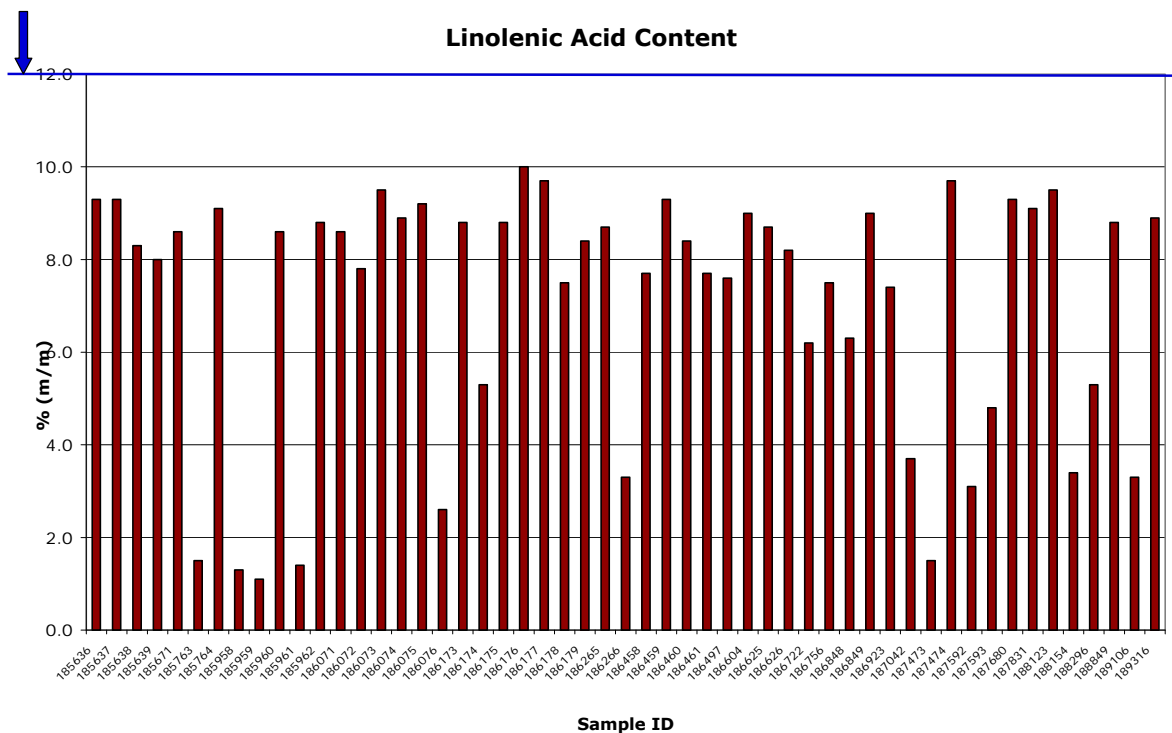
EN 14214	< 12	% m/m
Min.	1.1	% m/m
Max.	10.0	% m/m
Average	7.1	% m/m
Std. Deviation	2.7	% m/m
Range 95% max	12.4	% m/m
Range 95% min	1.7	% m/m
Out of spec	0	-

### Results

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

### Winter 2010/2011

#### Linolenic Acid Content



## Poly Unsaturated Fatty Acid Methyl ester

*EBBQR – Winter 2010/2011*

The EN15779 polyunsaturated ester test has been included in EN14214 in an effort to limit esters which exhibit particularly poor oxidation stabilities, and further increase FAME quality.

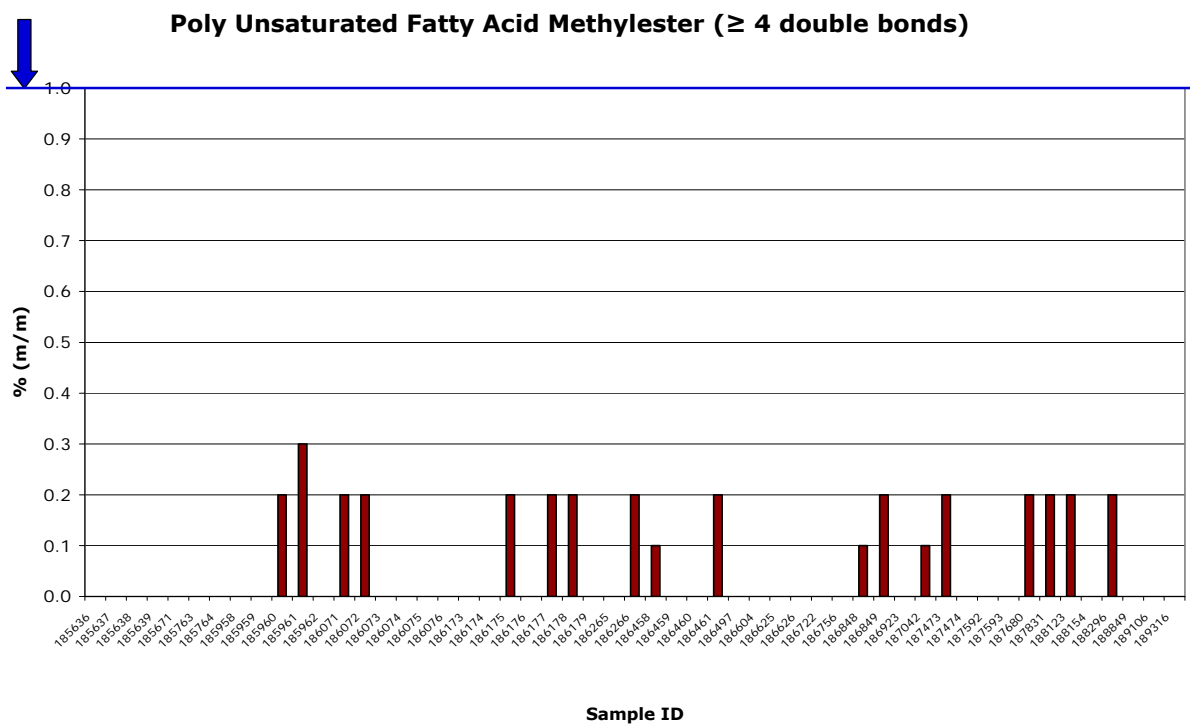
### Winter 2010/2011

EN 14214	< 1	% m/m
Min.	0.1	% m/m
Max.	0.3	% m/m
Average	0.2	% m/m
Std. Deviation	0.0	% m/m
Range 95% max	0.3	% m/m
Range 95% min	0.1	% m/m
Out of spec	0	-

### Results

As this was the first instance of the requirement of this new test, not all EBB members opted for it. However all those that did were within the limit of the poly unsaturated fatty acid methyl ester limit of <1% m/m.

### Winter 2010/2011



## Methanol Content

EBBQR – Winter 2010/2011

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

### Winter 2010/2011

EN 14214	< 0,20	% m/m
Min.	<0,01	% m/m
Max.	0.14	% m/m
Average	0.03	% m/m
Std. Deviation	0.03	% m/m
Range 95% max	0.10	% m/m
Range 95% min	-0.03	% m/m
Out of spec	0	-

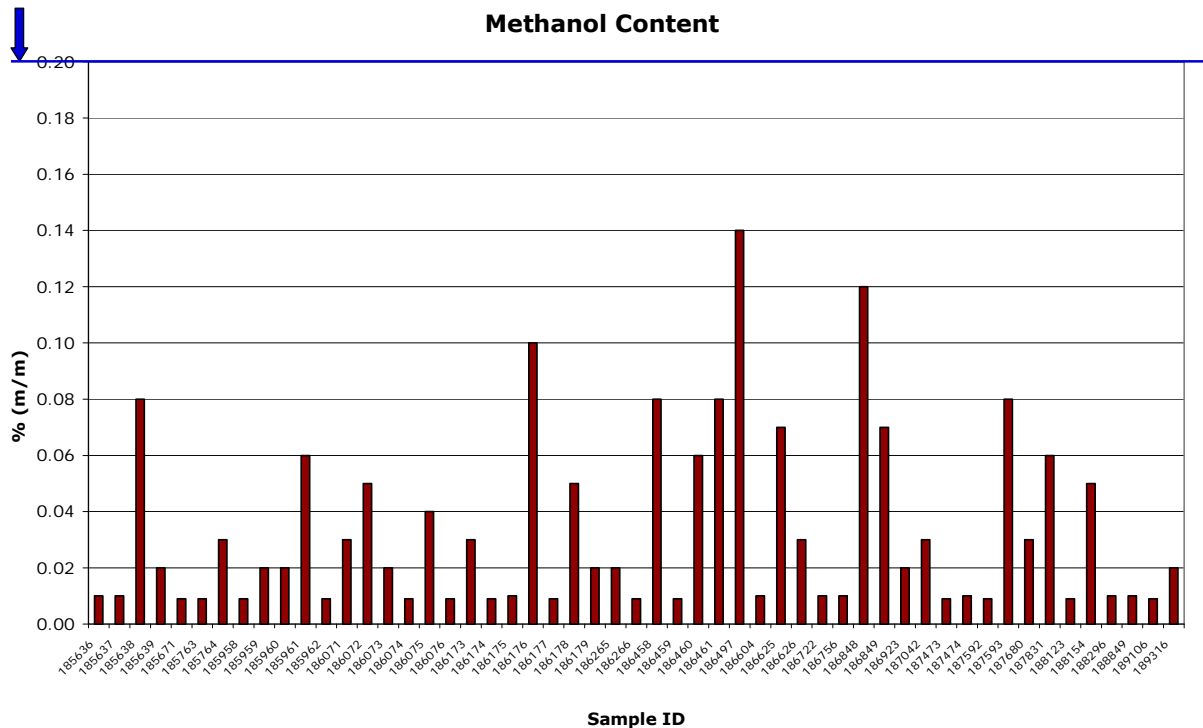
### Results

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)

### Winter 2010/2011

#### Methanol Content



## Free Glycerol

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

EN 14214	< 0,020	% m/m
Min.	<0,01	% m/m
Max.	0.03	% m/m
Average	0.01	% m/m
Std. Deviation	0.00	% m/m
Range 95% max	0.02	% m/m
Range 95% min	0.00	% m/m
Out of spec	1	-

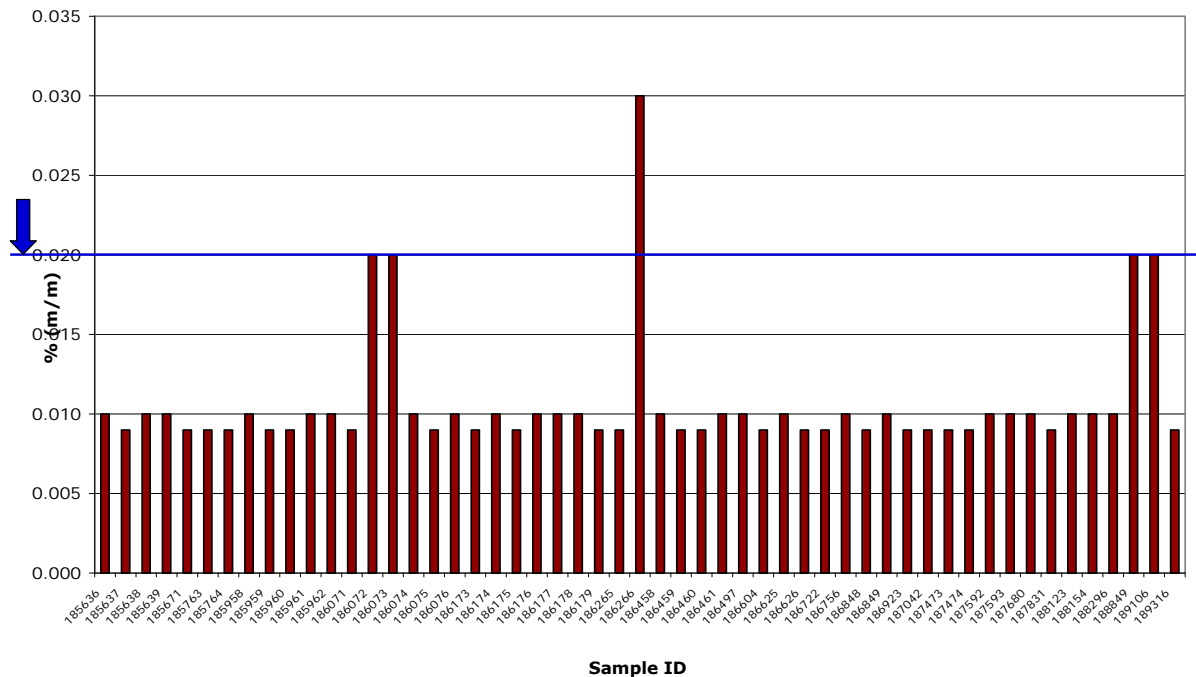
### Results

One sample was above specification but within the method reproducibility. It should be noted that during the GC analysis it is believed other material can co-elute with the glycerol peak giving artificially high results. An alternative glycerol method EN14106:2003 may overcome this issue, further investigations are currently on-going.

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

### Winter 2010/2011

#### Free Glycerol



## Mono, Di and Tri-Glycerides

*EBBQR – Winter 2010/2011*

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.

### Winter 2010/2011

Glycerides	Mono	Di	Tri	
EN 14214	< 0,80	< 0,20	< 0,20	% (m/m)
Min.	0.01	<0,01	<0,01	% (m/m)
Max.	0.73	0.16	0.14	% (m/m)
Average	0.42	0.10	0.04	% (m/m)
Std. Deviation	0.18	0.04	0.03	% (m/m)
Range 95% max	0.79	0.19	0.10	% (m/m)
Range 95% min	0.05	0.01	-0.03	% (m/m)
Out of spec	0	0	0	-

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

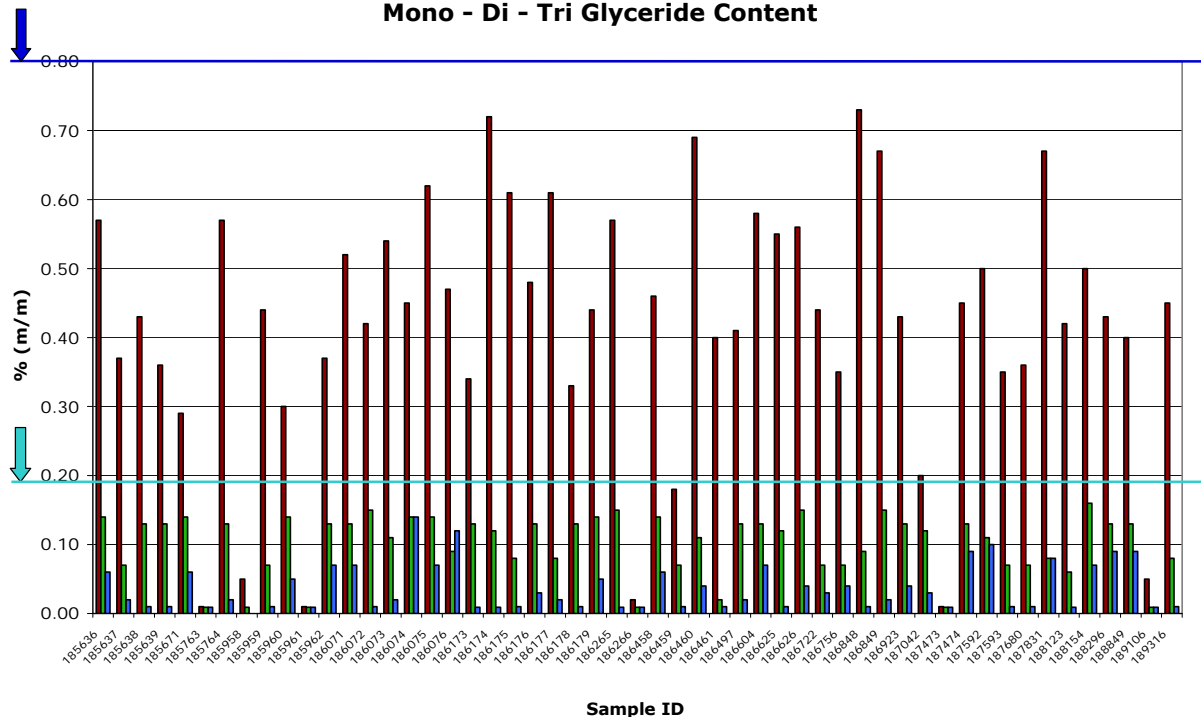
### Results

In this testing round all results 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.

### Winter 2010/2011

#### Mono - Di - Tri Glyceride Content



## Total Glycerol

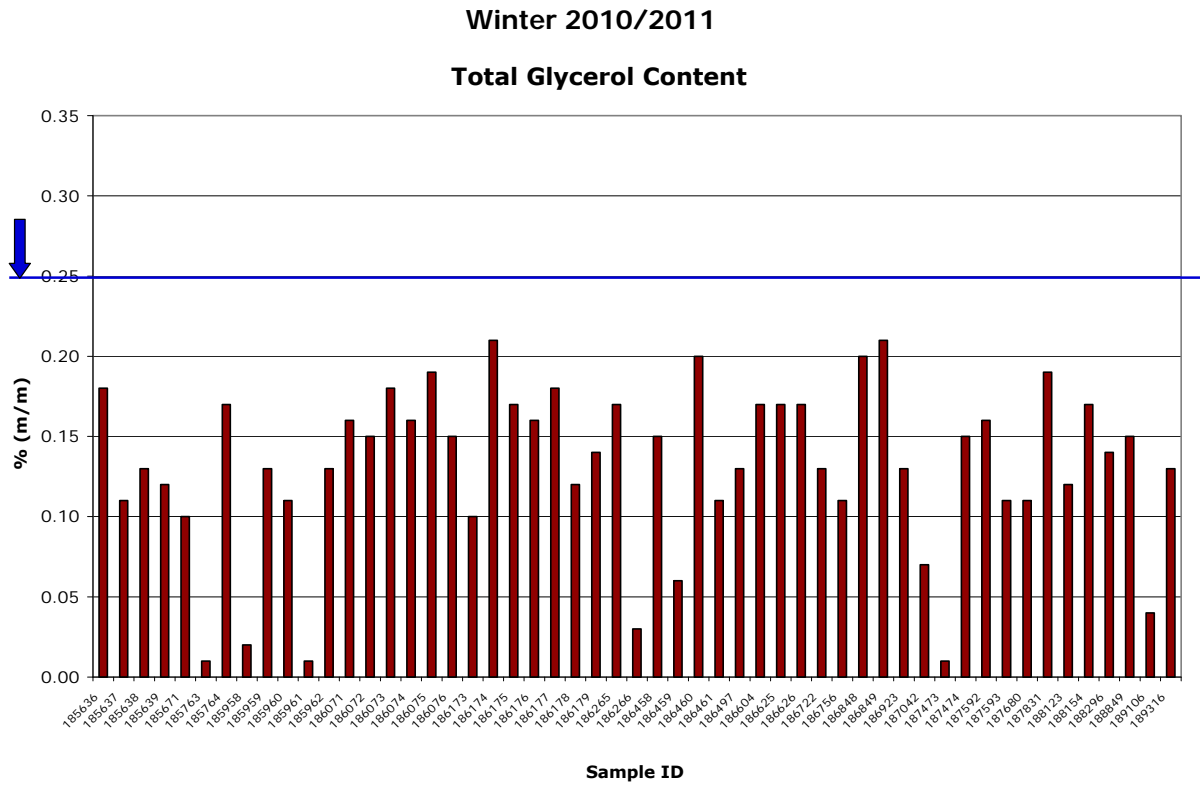
EBBQR – Winter 2010/2011

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

Winter 2010/2011		
EN 14214	< 0,25	% m/m
Min.	0.01	% m/m
Max.	0.21	% m/m
Average	0.13	% m/m
Std. Deviation	0.05	% m/m
Range 95% max	0.23	% m/m
Range 95% min	0.03	% m/m
Out of spec	0	-

### Results

The results of the Winter 2010/2011 EBBQR showed all samples respected the limit of the specification.



## Phosphorous Content

*EBBQR – Winter 2010/2011*

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

### Winter 2010/2011

EN 14214	< 4	mg/kg
Min.	<0,5	mg/kg
Max.	15.6	mg/kg
Average	0.8	mg/kg
Std. Deviation	2.1	mg/kg
Range 95% max	4.9	mg/kg
Range 95% min	-3.4	mg/kg
Out of spec	1	-

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

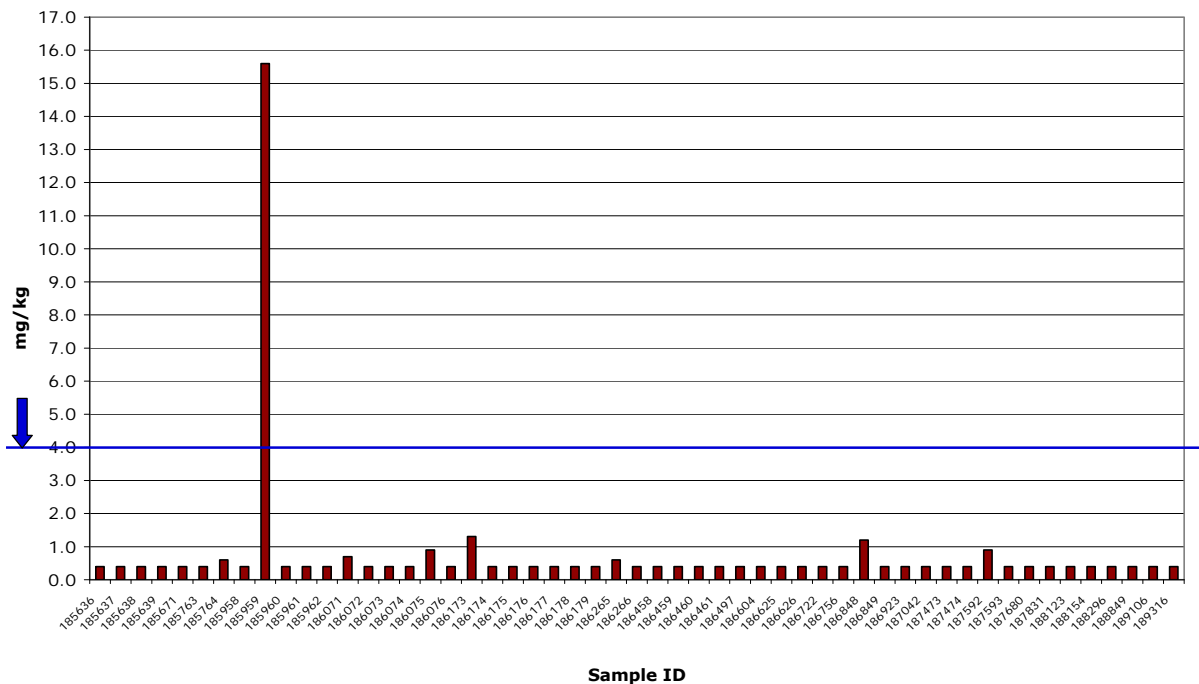
### Results

All samples except one were below the phosphorous content limit of less than 4mg/kg. It should be noted this incorrectly taken sample was however not final product.

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 improved accuracy, and a possible future limit of 2.5mg/kg is under discussion.

### Winter 2010/2011

#### Phosphorous Content





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

EBBQR – Winter 2010/2011

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

### Winter 2010/2011

	Metals I (Na + K)	Metals II (Ca + Mg)	
EN 14214	< 5,0	< 5,0	mg/kg
Min.	<0,5	<0,5	mg/kg
Max.	4.0	1.5	mg/kg
Average	0.9	0.5	mg/kg
Std. Deviation	0.8	0.2	mg/kg
Range 95% max	2.5	0.9	mg/kg
Range 95% min	-0.6	0.0	mg/kg
Out of spec	0	0	-

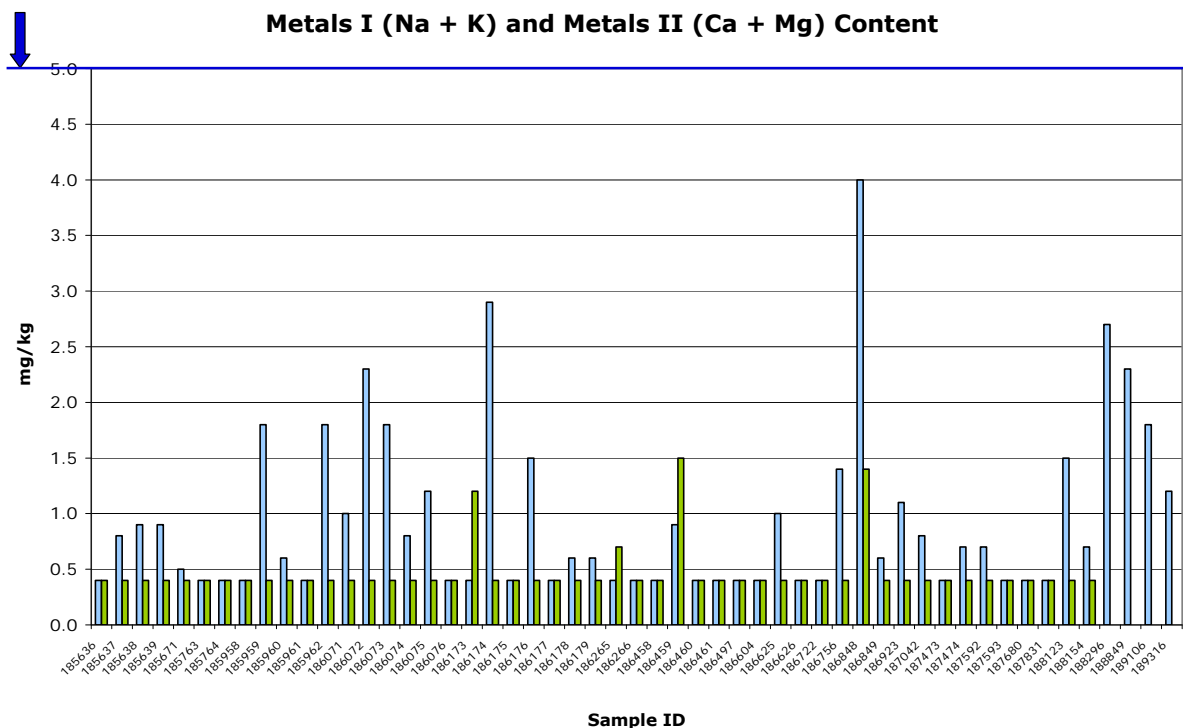
### Results

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

### Winter 2010/2011

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



## Summary of Results

### *Final General Comments*

#### **Eighth sampling and analysis Winter 2010/2011 quality**

EBB members' production (53 plants monitored) is within the EN14214 limits; borderline samples for ester content, free glycerol, iodine value and acid value 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 were not in range with respect to EN14214 are:

#### **Production parameters:**

3 results for sulphur content  
1 result for total contamination  
3 results for carbon residue  
and 1 result for phosphorus content

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

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<sup>i</sup> Mittelbach and Remschmidt, 2004. 'Biodiesel – The Comprehensive Handbook'.