Biodiesel Quality in Germany

Sampling results of biodiesel producers and warehouse operators of

Association Quality Management Biodiesel (AGQM)

2017

Project Management and Report:
Maren Dietrich (AGQM)

Analytics performed by:
ASG Analytik-Service GmbH
Trentiner Ring 30
86356 Neusaess / Germany

Inhaltsverzeichnis

Association Quality Management Biodiesel
Claire-Waldoff-Str. 7
D-10117 Berlin / Germany

1 Abstract

In all EU member states, biodiesel – also known as FAME (Fatty Acid Methyl Ester) – contributes to the reduction of CO_2 emissions and the use of fossil fuels by its use as admixture to Diesel fuel at a rate of 7 % (B7). To guarantee a marketable and suitable product it is essential to reliably meet the high-quality requirements resulting from EU standard EN 14214 and thus to only market products of high quality. To comply with this claim Association Quality Management biodiesel (AGQM) which is the alliance of most German biodiesel producers and traders, was founded in Germany in 1999. In the past years an AGQM quality label was introduced to provide customers and traders on the market with technical reliability.

One of the most important AGQM quality assurance measures is the annual and unannounced member sampling. Unannounced sampling safeguards that the results reflect the real production processes of producers and warehouse operators. This report gives an overview of the samplings carried out in 2017. In addition, the measured quality parameters and their meaning for the biodiesel production process are briefly discussed.

All 15 AGQM biodiesel producers and traders participated in the unannounced sampling in 2017. In the main three sampling campaigns 53 biodiesel samples were taken, analysed, and evaluated. Since 2017 all member companies with detected irregularities during the main three campaigns (violation of a limit or acceptance limit) must participate in an additional campaign. In 2017 all 53 samples tested in the main campaigns met the requirements of DIN EN 14214 and the more stringent AGQM limits within the acceptance limits. However, there were 14 violations of the acceptance limits. In the additional campaigns 13 of 14 analysed samples complied with the requirements.

If limits or acceptance limits are violated during sampling, AGQM always addresses the member company in question, looks into the matter jointly with the company to detect the reasons and supports the implementation of corrective and preventive measures.

The result of this year's report shows, once again, that the quality level of biodiesel produced by AGQM members is very high. Nonetheless AGQM and its members permanently develop the quality management system in order to also be prepared for any future challenges from the market.

2 Description of Sampling

As part of the Quality Management System (QM system) AGQM's Committee for Quality Assurance (QA committee) stipulates the parameters to be tested which comprise all parameters legally required by the 36. BImSchV (German Federal Emission Protection Directive) as proof for biofuel properties. Sampling and analysis are assigned to an independent laboratory accredited for biodiesel analysis. Basis of all analysis is always the current version of DIN EN 14214. In 2017 the demanded standard limits as well as the associated acceptance limits – derived from the precision of the relevant method – complied with DIN EN 14214:2014-06. Table 2 of the annex lists the parameters to be tested with their limits according to DIN EN 14214. For parameters 'Water Content', 'Total Contamination', and 'Cold Filter Plugging Point (CFPP)' AGQM's requirements for its members are more stringent than the legal ones which underlines AGQM's particularly high quality standard. All parameters with AGQM limits are shown in Table 3 of the annex.

In 2017 three sampling campaigns took place with 14 biodiesel producers and 1 warehouse operator. There were three campaigns which were carried out in periods 'winter', 'intermediate', and 'summer'. The reason for that is that in the (German) national attachment NB of DIN EN 14214 different limits for parameters 'Cloud Point' and 'CFPP', which concern the cold properties of the product, were stipulated for winter, intermediate and summer grades. These parameters can be determined individually by EU nations because the climatic conditions of some vary widely.

The sampling schedules are listed below:

C 1:	6 to 17 February	Winter grade
C 2:	19 to 30 June	Summer grade
C 3:	4 to 17 October	Intermediate grade

A total of 53 samples were taken and analysed during the main campaigns and 14 samples in the ensuing additional campaigns.

The implementation of the AGQM QM system by AGQM's members is assessed by means of a point system. Bonus points are awarded for the participation in quality assuring measures; sanction points are received for violations against the QM system. If the necessity of sanction measures must be assessed, the percentage ratio of sanction and bonus points is considered.

AGQM assesses all tested parameters after their analysis the result of which is subsequently communicated to the member company. In case of doubt concerning the result of the analysis, member companies can address AGQM to apply for arbitration proceedings. For that, the member

assigns an independent laboratory accredited for biodiesel analytics. The arbitration sample is one of the retain samples taken during the sampling campaign. The result of the arbitrational analysis is binding for both parties. If any irregularities are confirmed by the arbitrational analysis the member may be given sanction points and must participate in the next unannounced additional campaign.

3 Individual Sampling Results and Evaluation

In the following section the test method used, the limit according to DIN EN 14214, the AGQM limit – if applicable –, the acceptance limit as well as a description can be found for every parameter followed by a graphical illustration of the measured values as well as their evaluation.

The results given in this report were made anonymous and do not reveal the origin of the sample. In the diagrams the measuring values for every sampling campaign are given in ascending order to illustrate the spread. The axis 'Number of Samples' illustrates how many samples were taken in the relevant campaign. In the diagrams the limits are marked with a black line, the acceptance limits – determined considering the precision of the method – with a red line. The acceptance limits are the decisive factor when it comes to Customs matters as well as to the assignment of sanction points according to AGQM'S QM system. In the diagrams of parameters 'total contamination' and 'water content' AGQM's more stringent limit as well as acceptance limit are both given additionally.

One AGQM warehouse operator also participates in the annual sampling campaigns beside all biodiesel producers.

Biodiesel as Blend Component for Biofuels

Alternative raw materials to produce biodiesel, such as used cooking oils and fats as well as fatty acids, are used increasingly to improve the greenhouse gas balance and to support the idea of 'circular economy'. AGQM also supports producers of such products in quality assurance matters. Biodiesel made of such raw materials is primarily used as blend component for biodiesel produced of traditional raw materials (mainly rapeseed oil); it is thus not marketed as pure fuel. Since admixing such blend components to other products results in an overall standard-conform biodiesel and thus specification leeway is given.

In 2013 the AGQM QM system was amended with a special regulation for blend components for biodiesel. At first such fuels were exempted from the determination of parameters 'sulphur content',

'CFPP', and 'Cloud Point' if the producer had applied for the exception in advance. Those three parameters are strongly determined by the fatty acid profile and/or contamination in the raw material and can hardly be influenced during the production process. Therefore, in case of blend components for biodiesel, limit violations of the parameters mentioned before were not sanctioned during the period of report. All measurements resulting from this special regulation, are not illustrated in the following diagrams.

In autumn 2017 a separate chapter for biodiesel blend components was integrated into AGQM's QM system which comprises special AGQM limits for parameters 'sulphur content', 'CFPP', and 'Cloud Point' which will be checked in future unannounced samplings.

3.1 Fatty Acid Methyl Ester Content

Test method:	*DIN EN 14103:2015*
Limit of DIN EN 14214:2014:	*min. 96.5 % (w/w)*
Acceptance limit:	*min. 94.0 % (w/w)*

Information on the purity of biodiesel can be derived from the content of fatty acid methyl esters, briefly ester content. Dependent on the type and condition of the raw material and the reaction control, the ester content of the biodiesel can be reduced if by-products form or substances ingress the final product.

The ester content is determined by gas chromatography and given as sum of all fatty acid methyl esters from C6:0 to C24:1 in weight by weight [% (w/w)]. DIN EN 14214 demands a minimum fatty acid methyl ester content of 96.5 % (w/w). As a rule, after esterification or transesterification the ester content of a distilled final product is higher, since unwanted substances must thus be separated.

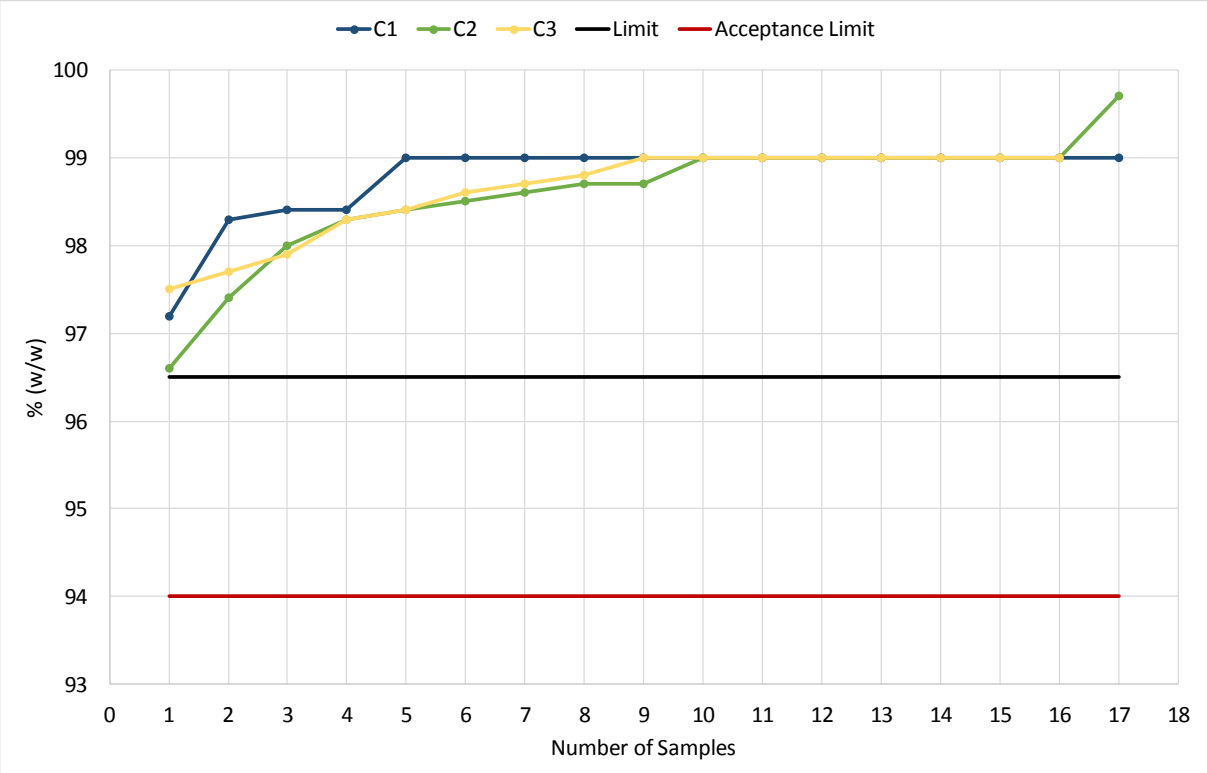

Diagram 1: Fatty Acid Methyl Ester Content according to DIN EN 14103

Diagram 1 shows the values of the ester content of the tested samples. The measurements of all samples are standard-conform.

3.2 Density at 15 °C

Test method:	*DIN EN ISO 12185:1997*
Limit of DIN EN 14214:2014:	*min. 860 und max. 900 kg/m³*
Acceptance limit:	*min. 859.7 kg/m³ und max. 900.3 kg/m³*

The density of a substance is the quotient of its mass and volume at a stipulated temperature. It is determined by means of an oscillating u-tube density meter. According to DIN EN 14214, the density of biodiesel at 15 °C must be between 860-900 kg/m³. Both FAME composition and biodiesel purity have an influence on the density. The density may also be influenced by contamination. For example, a higher methanol content would reduce the density.

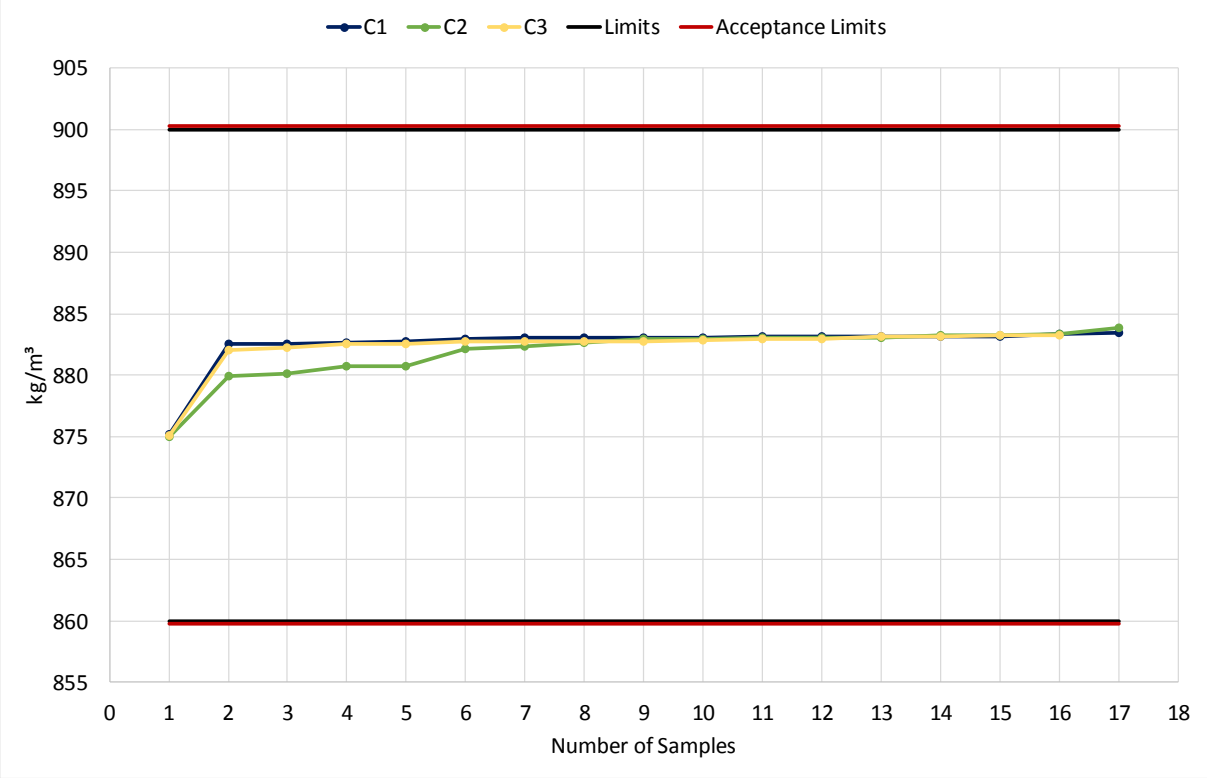

Diagram 2: Density at 15 °C according to DIN EN ISO 12185

As illustrated by diagram 2 all analysed samples stay within the density range required by the standard. Almost all samples are in the tight range between 881 and 883 kg/m³ which can be concluded from the use of rapeseed oil as raw material. However, there are also reduced densities of about 875 kg/m³. In those cases, it can be assumed that other raw material was used.

3.3 Sulphur Content

Test method: *DIN EN ISO 20846:2011*
Limit of DIN EN 14214:2014: *max. 10 mg/kg*
Acceptance limit: *max. 11.3 mg/kg*

Sulphur can already be contained in raw materials used for the biodiesel production. Plants can absorb sulphur compounds during growth, which usually results in a sulphur content of between 2 and 7 mg/kg. Animal and used cooking fats and oils can contain sulphur in the form of protein compounds which may result in a sulphur content of up to 30 mg/kg. Dependent on the kind of sulphur compound, its content in biodiesel can be reduced by washing processes or distillation.

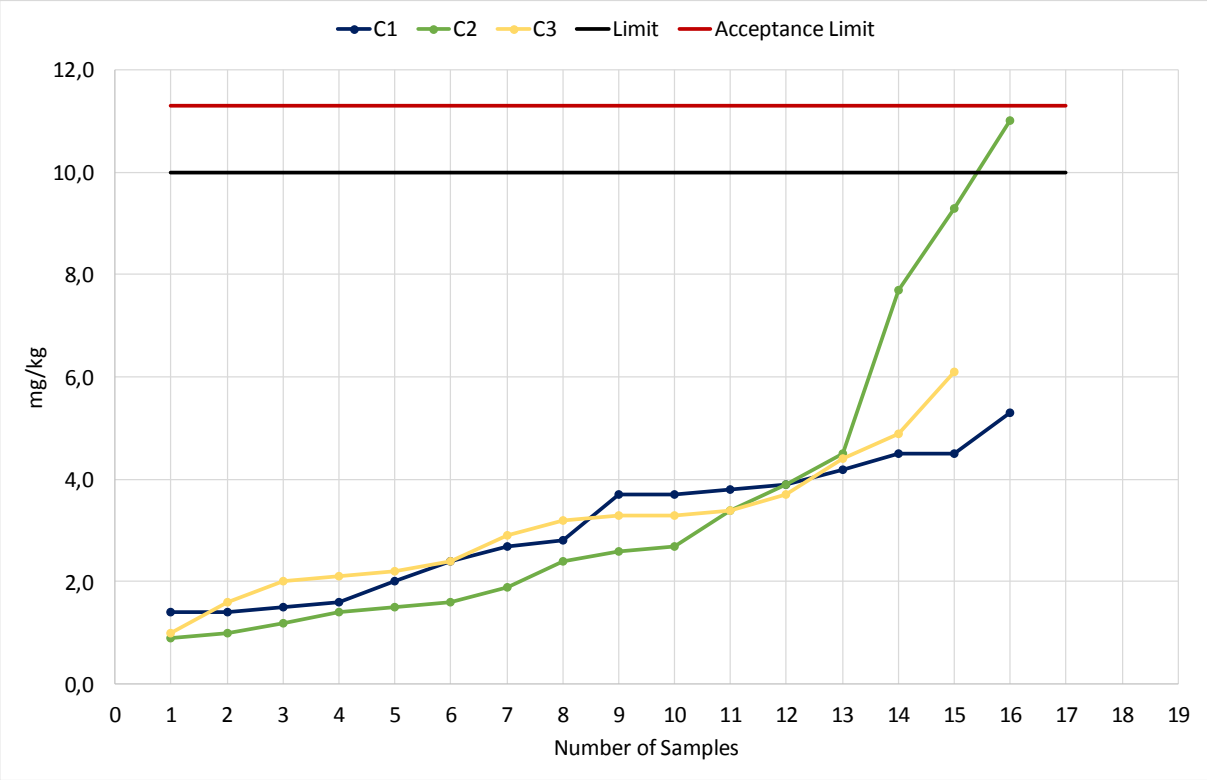

Diagram 3: Sulphur Content according to DIN EN ISO 20846

As can be seen from diagram 3 one sample of C2 exceeds the limit (10 mg/kg) with a value of 11mg/kg within the acceptance limit (11.3 mg/kg). Most samples have a sulphur content of below 7 mg/kg and thus significantly under the limit.

3.4 Water Content

Test method:	*DIN EN ISO 12937:2002*
Limit of DIN EN 14214:2014:	*max. 500 mg/kg*
Acceptance limit:	*max. 591 mg/kg*
AGQM limit:	*max. 220 mg/kg for producers*
Acceptance limit:	*max. 280 mg/kg*
AGQM limit:	*max. 300 mg/kg for warehouse operators*
Acceptance limit:	*max. 370 mg/kg*

Unlike hydrocarbon-based fuels, biodiesel can physically dissolve larger amounts of water due to its higher polar properties (up to 1500 mg of water/kg biodiesel). Since water wash is part of almost all production processes, at the end of the biodiesel production process the product must be dried. Subsequently the storage conditions must be selected accordingly to prevent further contamination of the biodiesel due to air humidity.

Fossil Diesel fuels can only absorb very small amounts of water which means, when mixed with biodiesel, the water dissolved therein may precipitate. Then in winter piping systems may be blocked due to its freezing. In summer corrosion can be caused and microbial growth promoted. DIN EN 14214 demands a maximum water content of 500 mg/kg. Due to the difficulties described above, AGQM's quality standards are stricter and require a maximum water content of 220 mg/kg ex works for member companies.

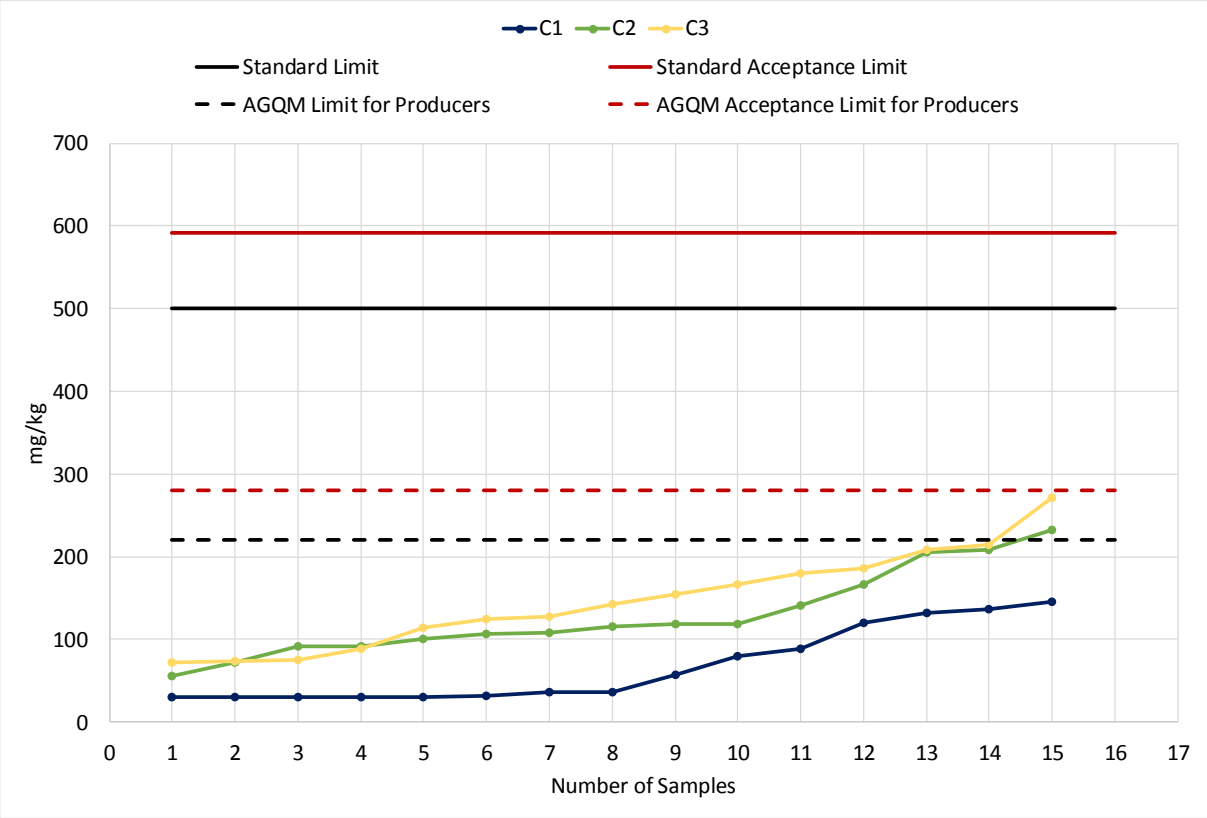

Diagram 4: Water Content according to DIN EN ISO 12937

Diagram 4 shows the measurements of the water content. It is obvious that all tested samples are clearly below the standard limit. In campaigns 2 and 3 one sample each exceeds the AGQM limit for producers with 233 and 271 mg/kg. However, both samples range within AGQM's acceptance limit (280 mg/kg).

The results of the samples of the single warehouse operator are not give here to protect the identity of the member. The samples meet the required AGQM limit for warehouse operators (300 mg/kg) without any difficulties.

3.5 Total Contamination

Test method: *DIN EN 12662:1998*

Due to the fact that the current version of DIN EN 12662:2014 is unsuitable for FAME concerning the determination of parameter 'Total Contamination', DIN EN 12662:1998 applies for AGQM's checks. This procedure is based on a recommendation by CEN TC19 – JWG 1 of 13 July 2014.

Limit of DIN EN 14214:2014: *max. 24 mg/kg*
Acceptance limit: *max. 32 mg/kg*
AGQM limit: *max. 20 mg/kg*

AGQM's limit for parameter 'total contamination' is also AGQM's acceptance limit.

'Total contamination' is a measure for the content of insoluble particles ('rust and dust') in the product. It is determined gravimetrically after filtration of a heated sample by weighing the filters. Biodiesel is usually not distilled which is why 'total contamination' is an important quality feature. High proportions of insoluble particles can cause filter blocking and wear of the injector system. AGQM set its own more stringent acceptance limit of 20 mg/kg to account for the imprecision of the method.

Diagram 5 shows that all samples can meet the tightened AGQM limit for 'total contamination'. In additional campaign 2 (AC2, not illustrated) one member exceeded the AGQM limit for 'total contamination' (20 mg/kg) with 22 mg/kg. The arbitration sample revealed a value of 29 mg/kg which led to the assignment of one sanction point. However, the acceptance limit of DIN EN 14214 (32 mg/kg) was not exceeded.

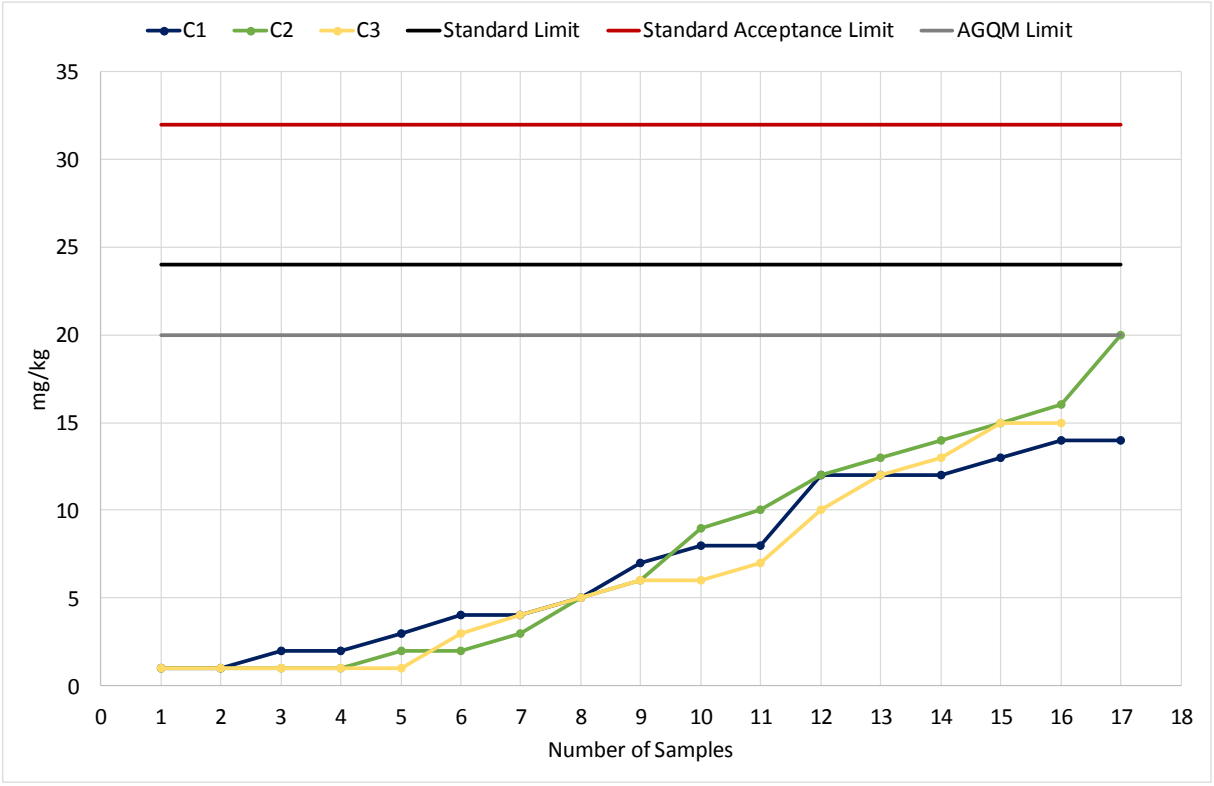

Diagram 5: Total Contamination according to DIN EN 12662

3.6 Oxidation Stability

Test method:	*DIN EN 14112:2014*
Limit of DIN EN 14214:2014:	*min. 8 h*
Acceptance limit:	*min. 6.6 h*

The oxidation stability of biodiesel is the measure for the resilience against oxidative processes. Test method for biodiesel is the so-called 'Rancimat test'. At 110 °C a constant air stream is passed through the test sample. Upon degrading the oxidation reserve (natural reserve plus additives) of the sample, large amounts of volatile oxidation products form which – together with air – are passed through the test liquid of the measuring vessel where they increase the conductivity. The time until such oxidation products are detected is called induction period. DIN EN 14214 stipulates a minimum oxidation stability of 8 hours.

In addition to natural antioxidants (e.g. tocopherols) which are contained in vegetable oils and slow down the ageing process, synthetic stabilizers are used. Upon request of interested additive producers

AGQM annually tests products which can be used to enhance the biodiesel oxidation stability. Additives passing the test are published in the so-called 'No-Harm List' on AGQM's homepage.

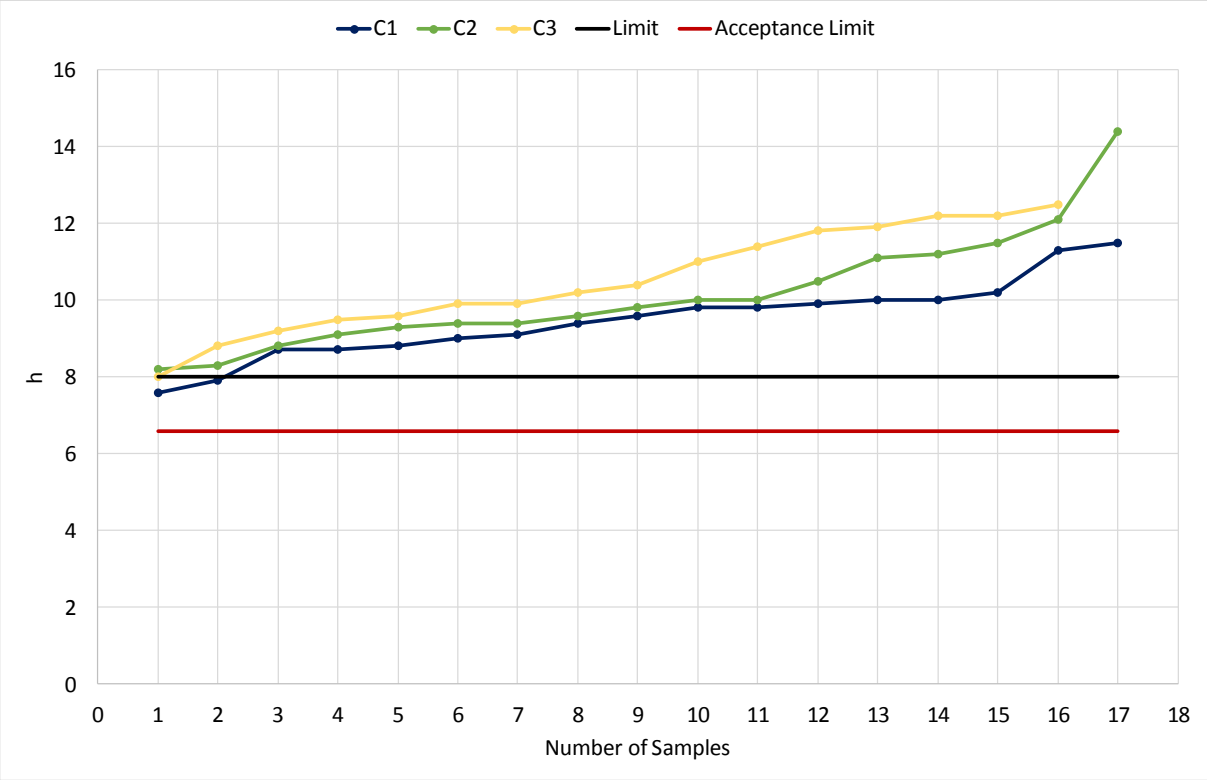

Diagram 6: Oxidation Stability according to DIN EN 14112

Diagram 6 illustrates that AGQM member companies have no issues to meet the requirements for oxidation stability. Two samples of C1 fall below the limit with 7.9 h and 7.6 h. However, those values are within the acceptance limit (6.6 h).

3.7 Acid Number

Test method:	*DIN EN 14104:2003*
Limit of DIN EN 14214:2014:	*max. 0.5 mg KOH/g*
Acceptance limit:	*max. 0.54 mg KOH/g*

The acid number is the measure for free acids (especially fatty acids) in biodiesel. Fatty acids are weak acids and therefore only little corrosive. If washed with inorganic acids small soap residues are broken and the free fatty acids thus forming can remain in the biodiesel. The acid number may also rise during the storage of FAME when ageing processes (primarily oxidation) cause ester cleavage and the formation of short-chain carbon acids. However, this effect can hardly be observed under regular storage conditions. DIN EN 14214 stipulates a maximum acid number of 0.5 mg KOH/g.

Diagram 7 shows the measurements for the acid number. All samples comply with the requirements of the standard easily.

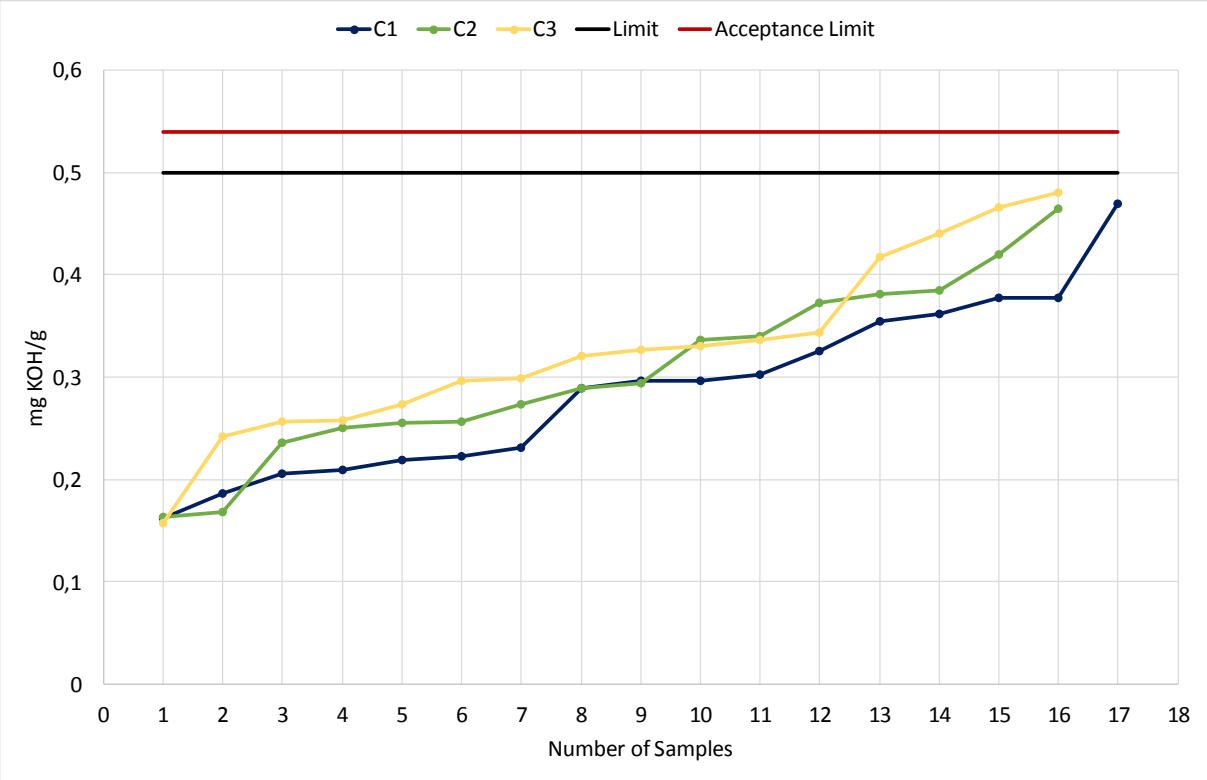

Diagram 7: Acid Number according to DIN EN 14104

3.8 Iodine Number

Test method: *DIN EN 16300:2012*
Limit of DIN EN 14214:2014: *max. 120 g iodine/100g*
Acceptance limit: *max. 124 g iodine/100g*

The iodine number is a measure for the proportion of double bonds in fats and oils as well as fatty acid methyl esters. It varies dependent on the raw material used. Since unsaturated fatty acids are more prone to oxidative reactions, the oxidation stability of biodiesel decreases with the rising number of double bonds which means rising iodine number. Therefore, apart from the oxidation stability, the iodine number is an indicator for the stability of biodiesel.

The standard stipulates two different methods for its determination. For the AGQM sampling the iodine number is determined arithmetically based on the fatty acid profile measured by gas chromatography according to DIN EN 16300. The result is given in g of iodine/100 g biodiesel.

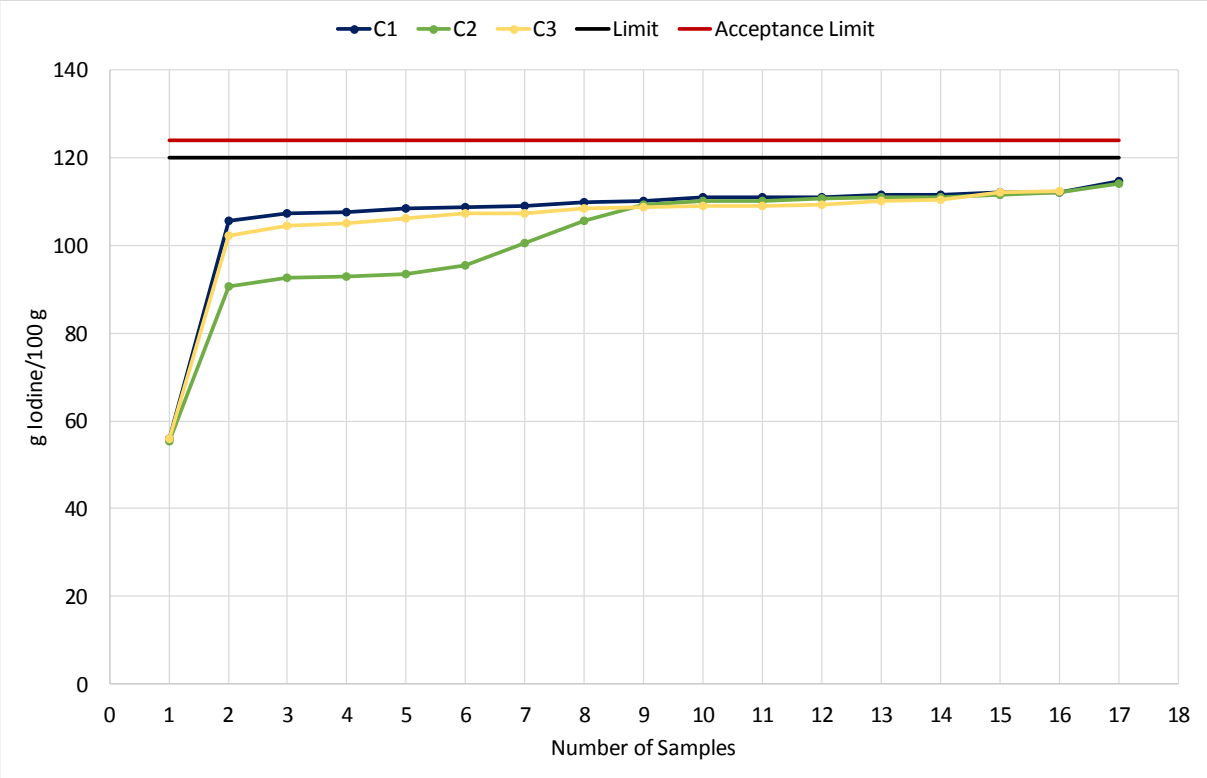

Diagram 8: Iodine Number according to DIN EN 16300 (determined from the methyl ester profile)

The results for the iodine number are given in diagram 8. All tested samples stay below the standard limit. In the second campaign (C2) it is noticeable that some measurements of the iodine number are lower which can be explained with the use of raw materials with a higher saturation level. A high saturation level causes worse cold properties (CFPP and Cloud Point) which is less important during the summer months. Some samples have an iodine number of less than 60 g of iodine/100 g biodiesel all year round which can also be attributed to the raw material used.

3.9 Mono-, Di-, and Triglycerides; free Glycerol

Test method:	*DIN EN 14105:2011*
Monoglycerides	
Limit of DIN EN 14214:2014:	*max. 0.70 % (w/w)*
Acceptance limit:	*max. 0.82 % (w/w)*
Diglycerides	
Limit of DIN EN 14214:2014:	*max. 0.20 % (w/w)*
Acceptance limit:	*max. 0.24 % (w/w)*
Triglycerides	
Limit of DIN EN 14214:2014:	*max. 0.20 % (w/w)*
Acceptance limit:	*max. 0.27 % (w/w)*
Free Glycerol	
Limit of DIN EN 14214:2014:	*max. 0.020 % (w/w)*
Acceptance limit:	*max. 0.026 % (w/w)*

During the transesterification of vegetable oils with methanol, varying contents of intermediate products (mono- and diglycerides; free glycerol) and unprocessed vegetable oil (triglycerides) can be found next to the main product ‘fatty acid methyl ester’. Since glycerol is practically insoluble in biodiesel, it can be almost completely removed by distillation and subsequent water wash. The contents ratio of mono-, di-, and triglycerides is a measure for the completeness of the transesterification reaction because in general, the concentration increases in the order ‘triglycerides < diglycerides < monoglycerides’. The cleavage of the last fatty acid residue is the slowest step of the reaction which is why the limit of 0.70 % (w/w) for monoglycerides required by the standard is slightly higher than the one for both di- and triglycerides with 0.20 % (w/w). The content of mono-, di-, and triglycerides can only be reduced to a certain degree, since chemical equilibrium between products and educts adjusts in any case. Glycerides can only be completely removed by distillation.

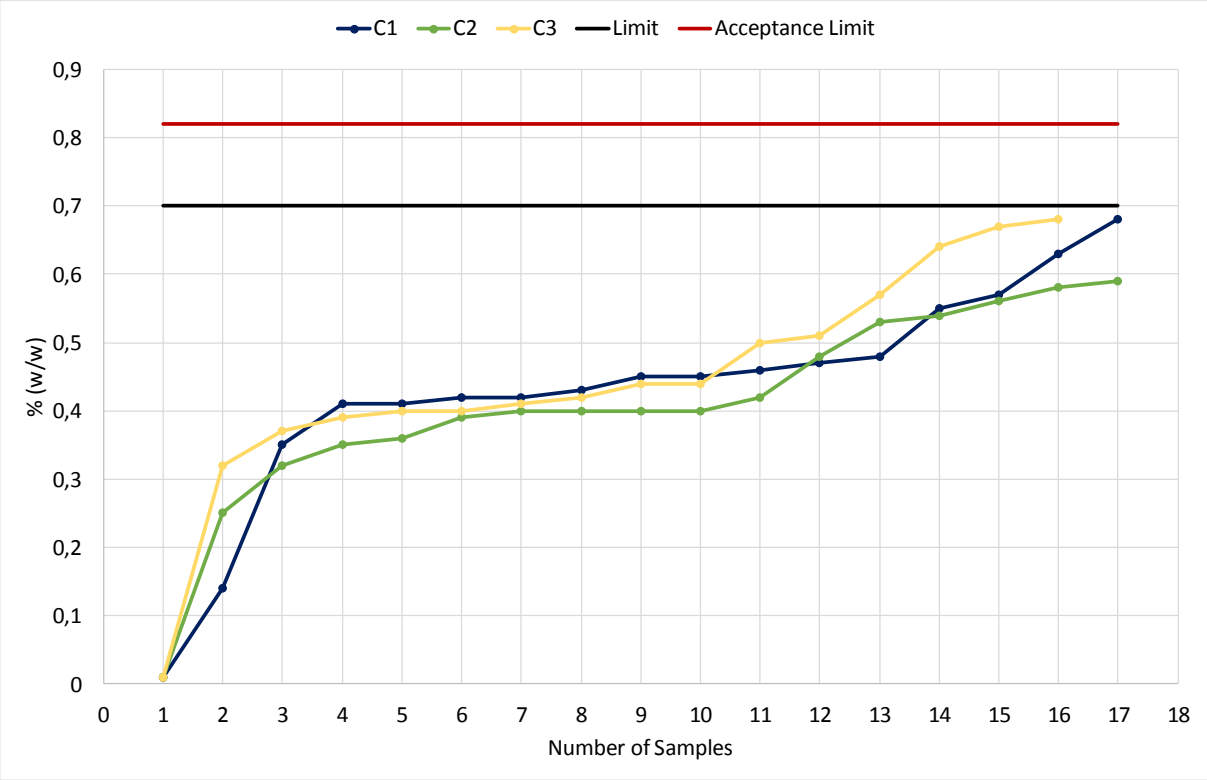

Diagram 9: Monoglycerides according to DIN EN 14105

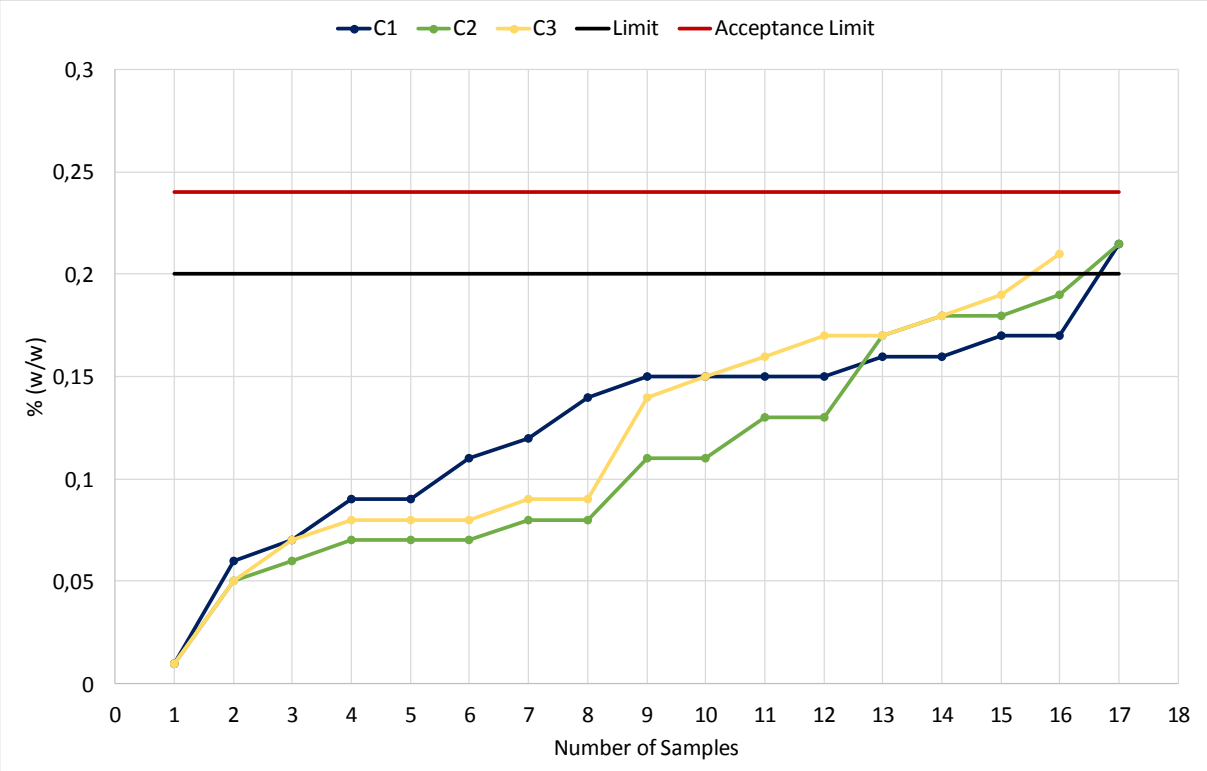

Diagram 10: Diglycerides according to DIN EN 14105

The measurements for monoglycerides are shown in diagram 9. All tested samples fall below the limit of 0.70 % (w/w). The value of some samples is almost 0 % (w/w) which suggests that the production process included a distillation step.

Diagram 10 shows the values for the content of diglycerides. In all three campaigns one sample each slightly exceeds the standard limit of 0.20 % (w/w). However, all violations (0.215 % (w/w) each in C1 and C2 and 0.21 % (w/w) in C3) range within the acceptance limit of 0.24 % (w/w) and are thus not sanctioned.

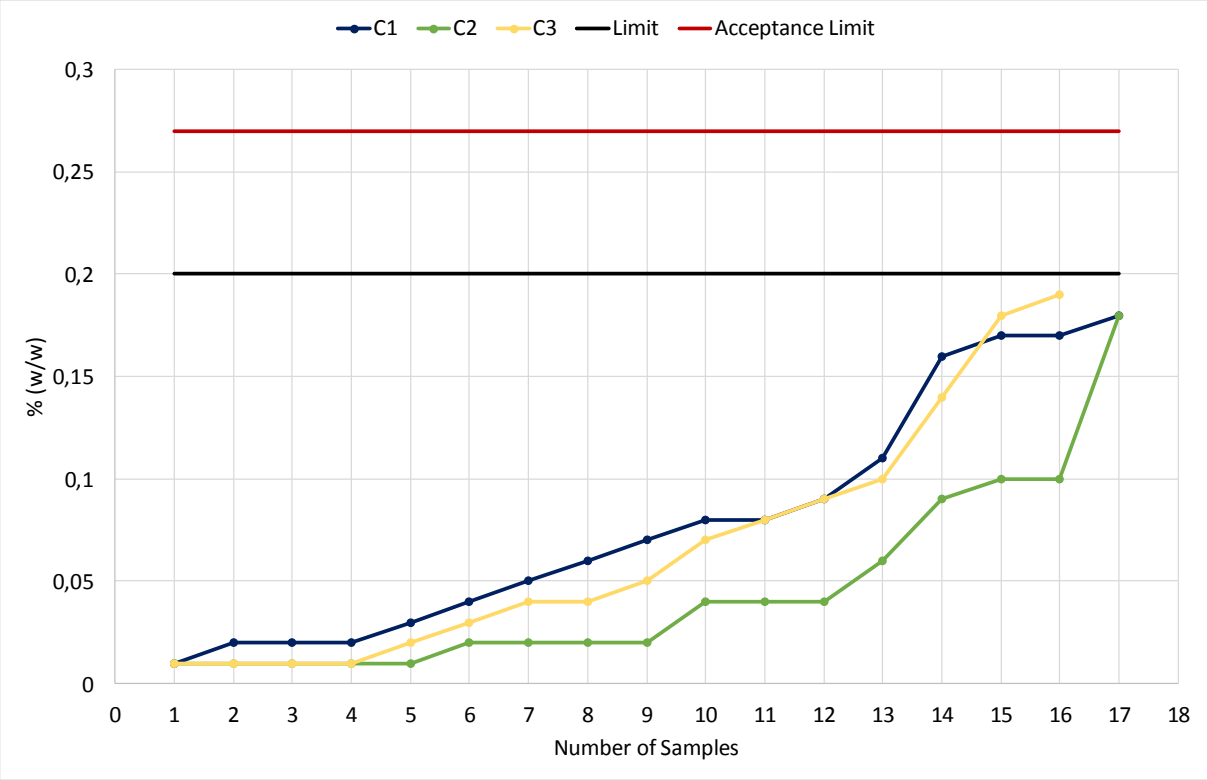

Diagram 11: Triglycerides according to DIN EN 14105

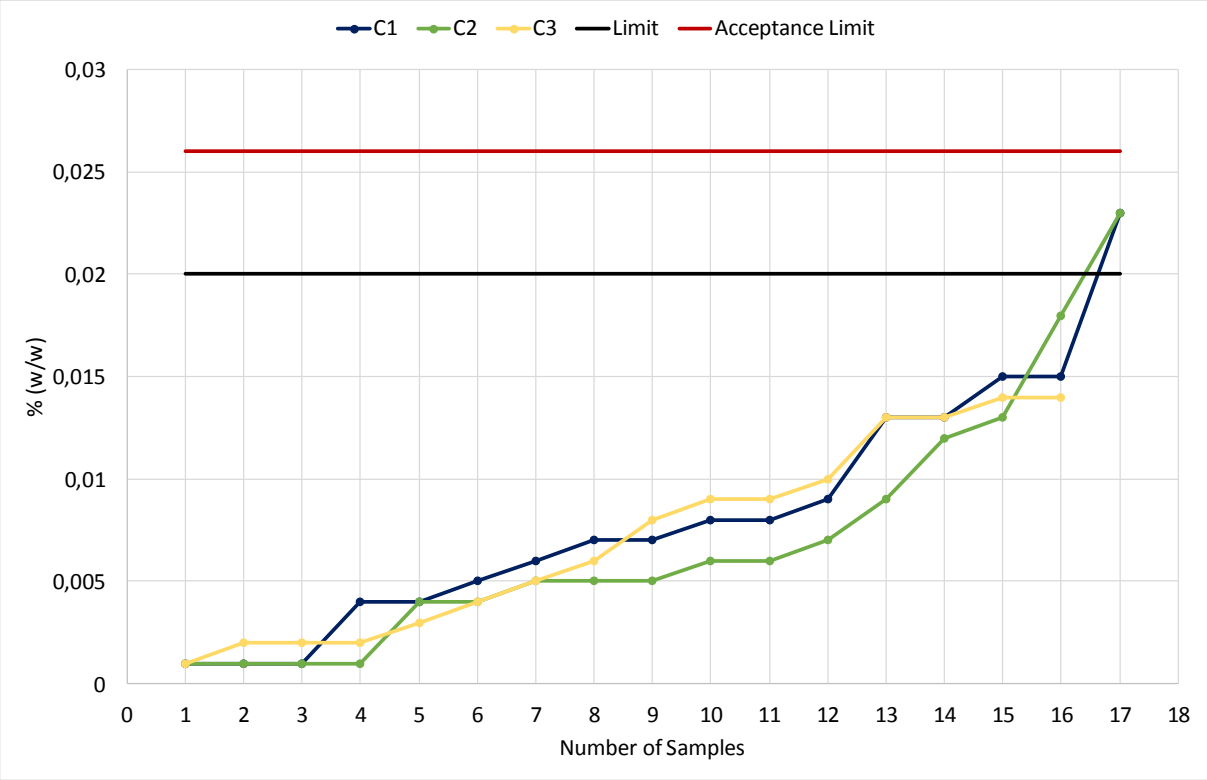

Diagram 12: Free Glycerol nach DIN EN 14105

In diagram 11 the measuring results for the content of triglycerides are shown. All tested samples fall below the limit.

The content of free glycerol of all samples but two falls below the standard limit (see diagram 12). However, the two samples with higher contents of free glycerol in C1 and C2 still range within the acceptance limit (0.026 % (w/w).

3.10 Alkali (Sodium and Potassium) and Earth Alkali Metals (Calcium + Magnesium)

Test method:	*DIN EN 14538:2006*
Limit of DIN EN 14214:2014:	*max. 5 mg/kg*
Acceptance limit:	*max. 6.1 mg/kg*

Sodium and potassium hydroxides or methylates are normally used as catalysts for the production of biodiesel. Residues thereof are often present in biodiesel as soaps which were not completely removed during washing. Soaps can cause filter plugging and clogging of injector pumps and nozzle needles. Alkali metals are also associated with formation of ash. Sodium as well as potassium deposit on the surface of particle filters and oxidising catalytic converters and thus reduce the effectivity and service life of the systems.

Earth alkali metals calcium and magnesium are either introduced into the process by raw material or they can get into the final product during the production process when tab water is used for washing. Calcium and magnesium soaps, which are more voluminous than alkali metal soaps, form by reacting with free fatty acids. The use of softened water for the washing process can reduce the ingress of earth alkali metals into biodiesel.

In this respect AGQM biodiesel producers attribute major importance to quality assurance and to very low contents of alkali and earth alkali metals in the final product.

Diagrams 13 and 14 clearly demonstrate that all efforts have been successful. The measurements for alkali metals sodium and potassium are clearly below 3 mg/kg (limit max. 5 mg/kg), and for earth alkali metals magnesium and calcium the measurements are even significantly below the determination limit of 1 mg/kg.

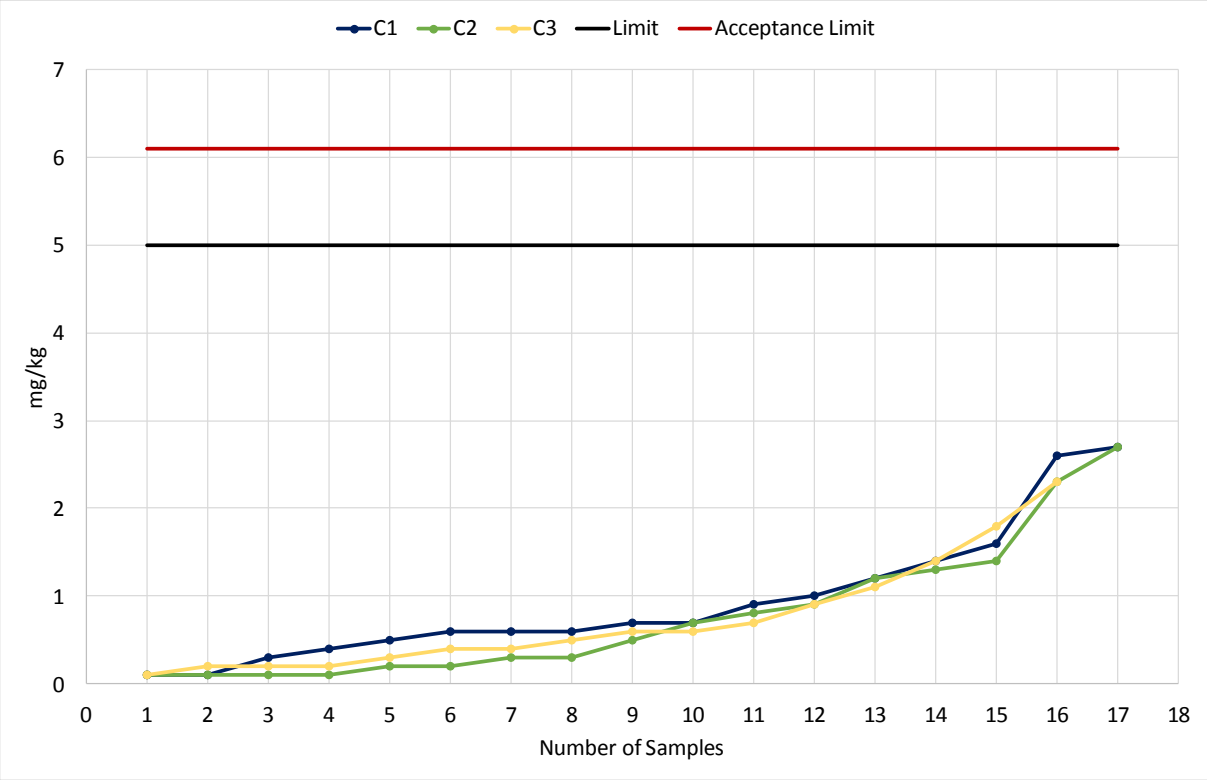

Diagram 13: Sum of Alkali Metals Sodium and Potassium according to DIN EN 14538

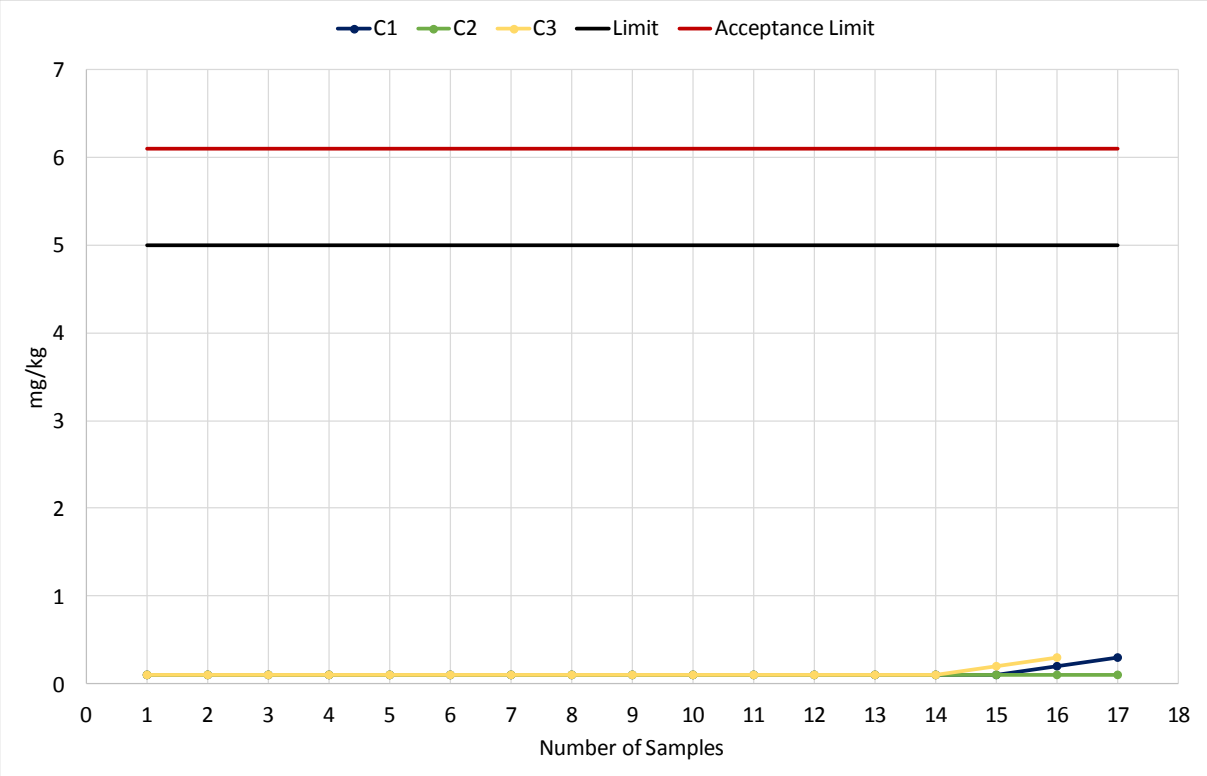

Diagram 14: Sum of Earth Alkali Metals Calcium and Magnesium according to DIN EN 14538

3.11 Phosphorus Content

Test method:	*DIN EN 14107:2003*
Limit of DIN EN 14214:2014:	*max. 4 mg/kg*
Acceptance limit:	*max. 4.5 mg/kg*

Vegetable oils and animal fats contain phosphorus in the form of phospholipids which influence the transesterification process because they act as emulsifiers and thus interfere with the separation of the phases. Furthermore, phosphorus is a catalyst poison which impairs the effectivity of exhaust gas after treatment systems. The maximum limit is 4 mg/kg; currently an additional tightening of the limit is not possible because of the precision of the method. The phosphorus content must already be considered when the raw material is selected, or it must be reduced by a refining process before transesterification. Phosphorus can also be introduced into biodiesel during the production process when phosphoric acid is used to separate soaps. In general, however, it can be well removed with water. The measurements for the phosphorus content are illustrated in diagram 15. All values fall below the limit; all measurements but two even fall below 0.5 mg/kg.

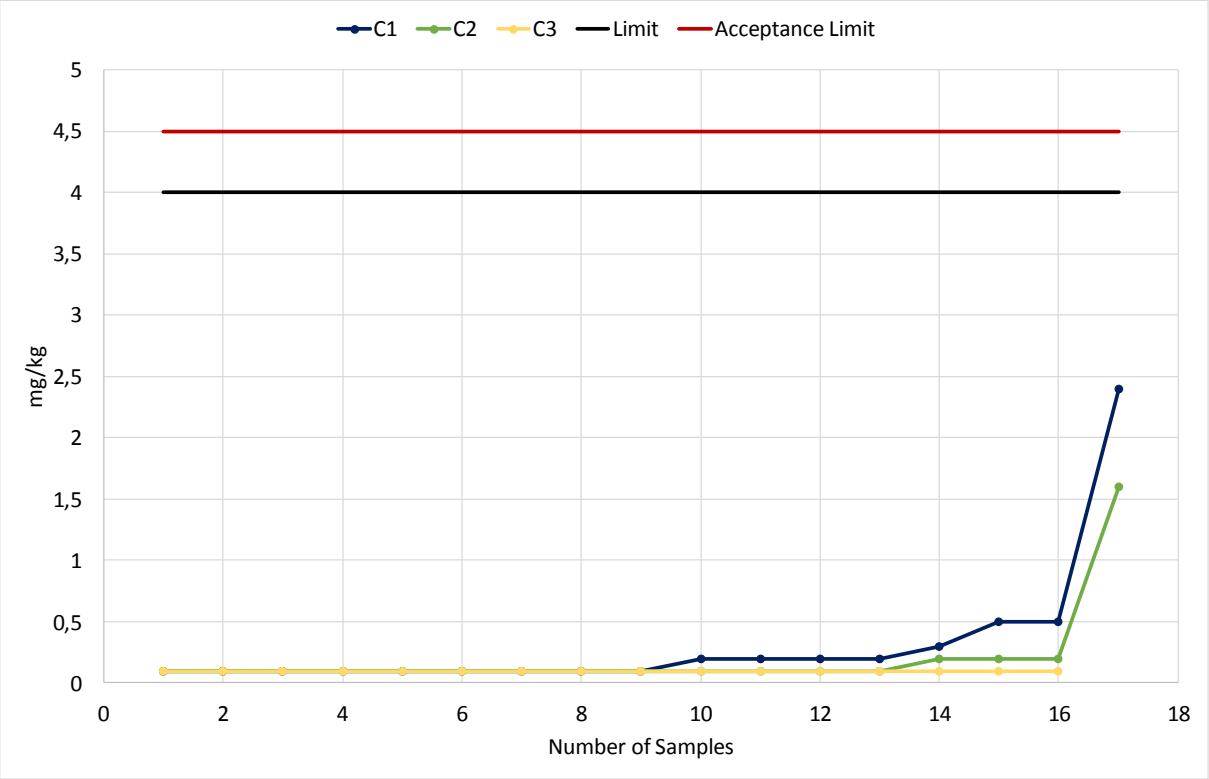

Diagram 15: Phosphorus Content according to DIN EN 14107 / Campaigns 1 to 4

3.12 Content of Linolenic Acid Methyl Ester

Test method: *DIN EN 14103:2015*
Limit of DIN EN 14214:2014: *max. 12.0 % (w/w)*
Acceptance limit: *max. 14.9 % (w/w)*

The content of linolenic acid methyl ester is determined by gas chromatography from the fatty acid profile. Linolenic acid is a triple unsaturated fatty acid with 18 carbon atoms (C18:3). Due to its chemical structure, it is extremely prone to oxidative attacks which is why the content of linolenic acid in biodiesel is limited to 12 % (w/w).

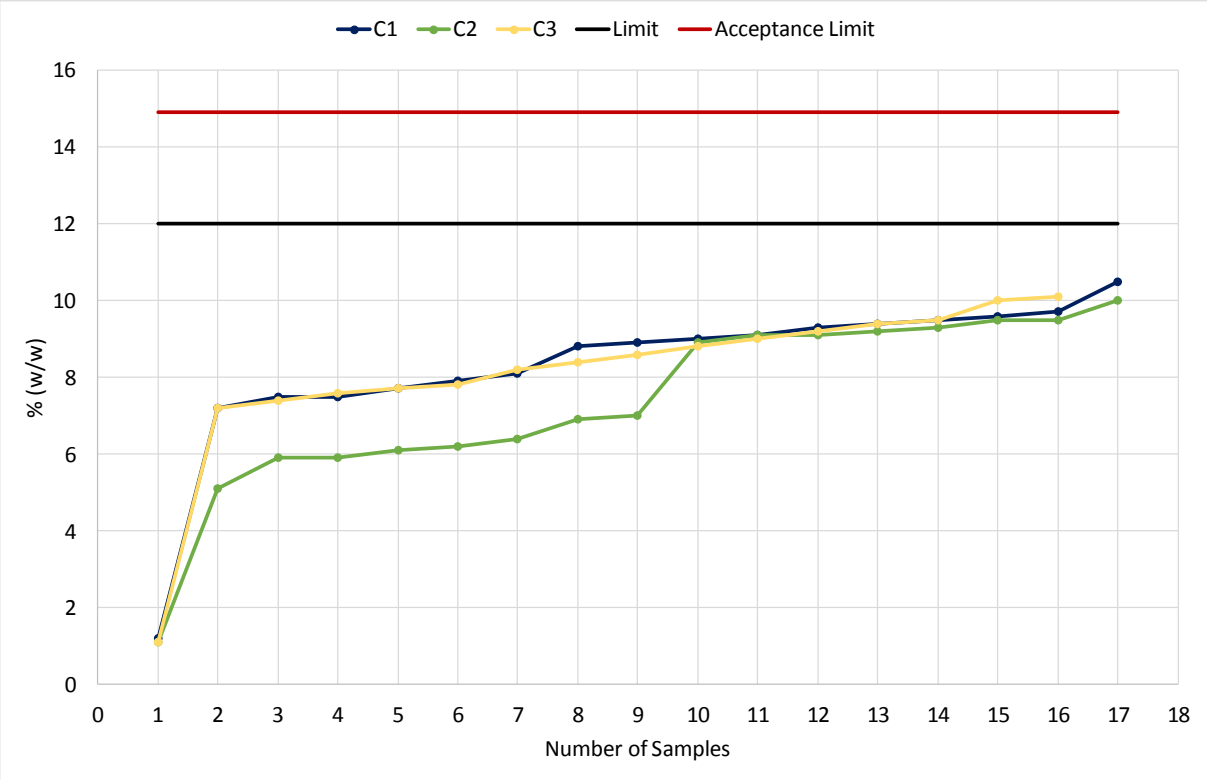

Diagram 16: Content of Linolenic Acid Methyl Ester according to DIN EN 14103

As can be seen from diagram 16 the content of linolenic acid methyl ester ranges within the requirements of the standard. The content of linolenic acid of pure rapeseed oil is usually between 8 and 10 %. The lower contents of linolenic acid methyl ester of many samples of the summer campaign (C2) suggest that raw material 'rapeseed oil' which is normally used to produce biodiesel, was – at least partly – substituted by other oils.

3.13 Cold Filter Plugging Point (CFPP)

Test method: *DIN EN 116:2015*

Limits according to DIN EN 14214:2014 for biodiesel as Blend Component in Diesel fuel

Period	*Limit*	*Acceptance limit*	
from 15 April to 30 September	*0 °C*	*+1.5 °C*	*Summer Grade*
from 1 October to 15 November	*-5 °C*	*-3.2 °C*	*Intermediate Grade*
from 16 November to 28/29 February	*-10 °C*	*-7.9 °C*	*Winter Grade*
from 1 March to 14 April	*-5 °C*	*-3.2 °C*	*Intermediate Grade*

The CFPP is the measure for the filterability of biodiesel at low temperatures. The requirements for 'resistance to cold' are regulated nationally according to the prevailing climatic conditions. As applicable for Diesel fuel, there are differing requirements for summer, intermediate and winter grades.

According to German legal regulations applicable for cold properties, biodiesel as blend component for Diesel fuel must adhere to the CFPP value of -10 °C between 16 November and 28/29 February if the value of -20 °C demanded by DIN EN 14214 can be achieved by additivation which is done at the refineries of the mineral oil companies when Diesel fuel is blended with biodiesel. Due to German legal regulations concerning mineral oil tax, marketing pure biodiesel (B100) came to a standstill which means that biodiesel is marketed almost exclusively as blend component.

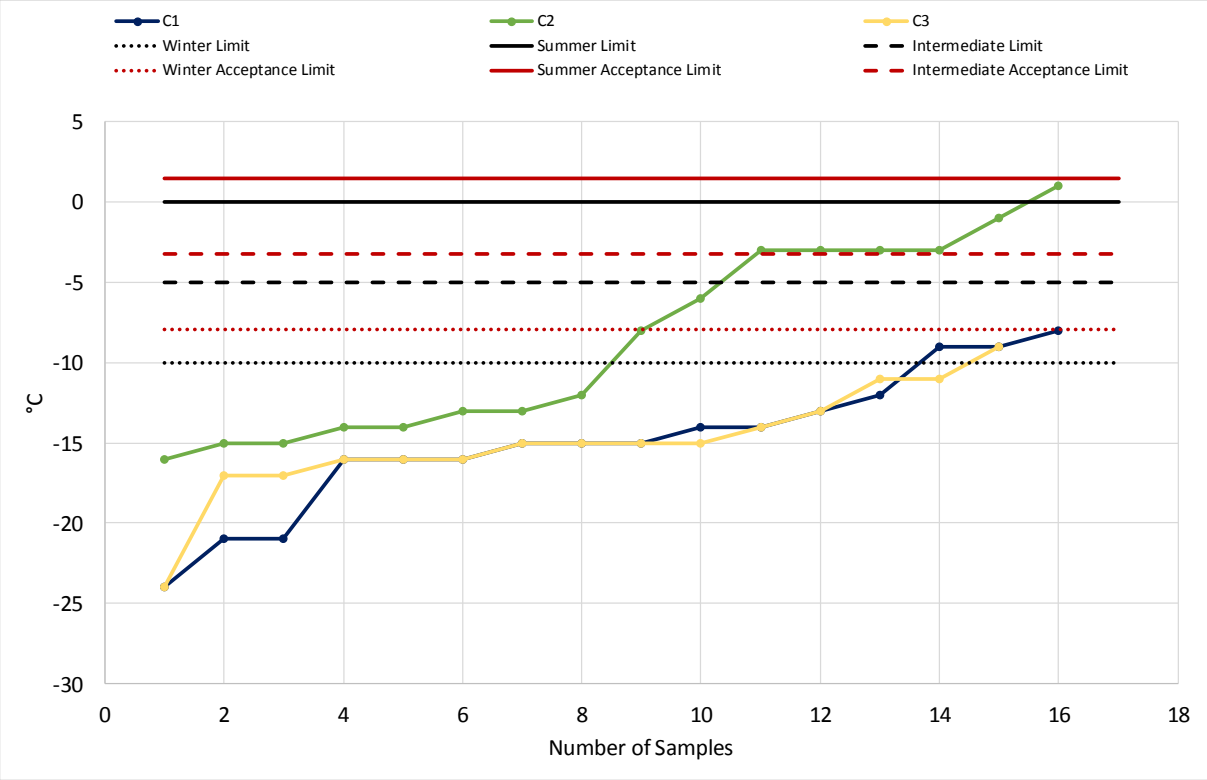

Diagram 17: CFPP according to DIN EN 116

Diagram 17 shows the measurements as well as different limits for the CFPP. The limit for the winter period is illustrated by a dotted line; the limit for intermediate grade is given by a dashed line; and the uninterrupted line represents the summer limit.

In C1 three samples exceed the winter limit (-10 °C) with -9 °C and -8 °C but they range within the acceptance limit (-7.9 °C). The summer limit (0 °C) is exceeded by one sample with 1 °C in C2 which is within the acceptance limit of 1.5 °C. No limit violations were found in C3.

3.14 Cloud Point (CP)

Test method: *DIN EN 23015:2013*

Limits according to DIN EN 14214:2014 for biodiesel as Blend Component in Diesel fuel

Period	*Limit*	*Acceptance limit*	
from 15 April to 30 September	*5 °C*	*7.4 °C*	*Summer Grade*
from 1. October to 15 November	*0 °C*	*2.4 °C*	*Intermediate Grade*
from 16 November to 28/29 February	*-3 °C*	*-0.6 °C*	*Winter Grade*
from 1 March to 14 April	*0 °C*	*2.4 °C*	*Intermediate Grade*

The Cloud Point is the temperature at which temperature-induced clouding sets in when a clear liquid product is cooled down under stipulated test conditions. Upon publication of DIN EN 14214:2012 in 2012 the Cloud Point has since been part of the requirements for biodiesel as blend component in Germany.

Diagram 18 shows all Cloud Point measurements. All tested samples can easily comply with the required limit.

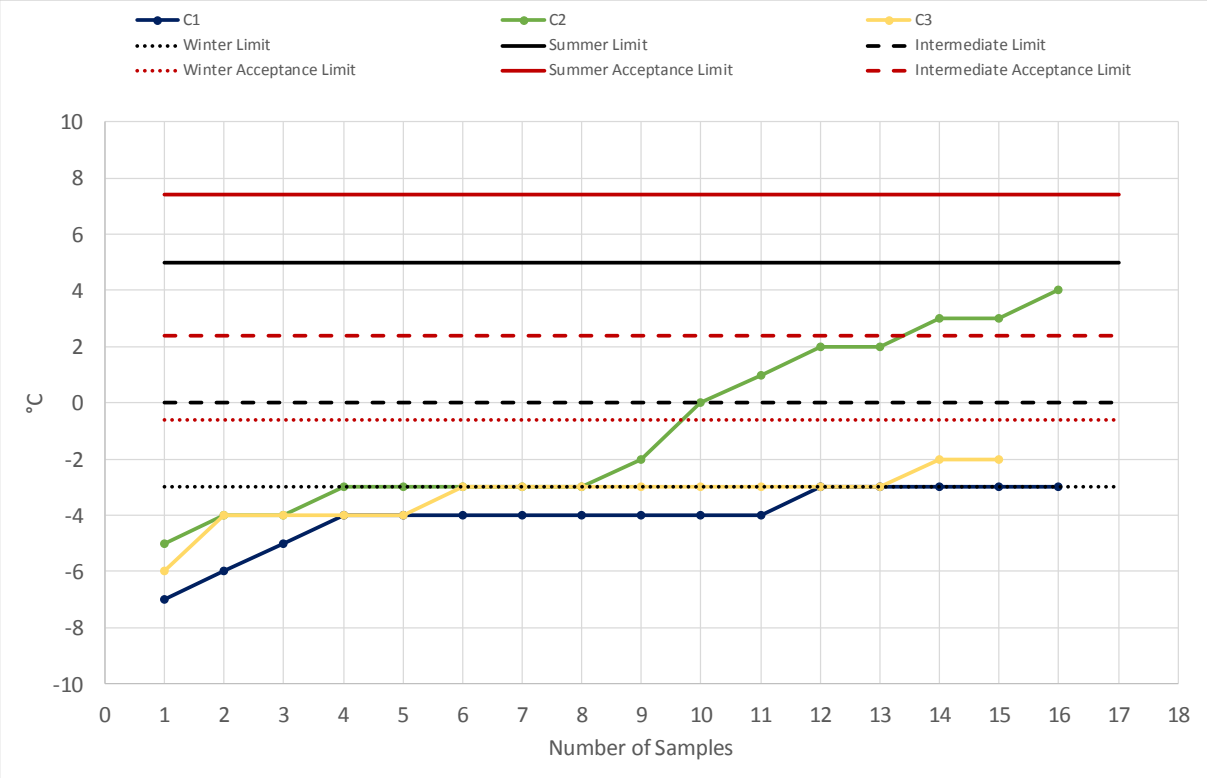

Diagram 18: Cloud Point according to DIN EN 23015

4 Summary

Since 2010 AGQM has published an annual report on the quality of the biodiesel produced and traded by AGQM members. The results of all unannounced samplings of the year are given in this report.

Compared with the year before the biodiesel quality improved again in 2017. In 2016 fifteen violations were determined and an additional 3 acceptance limits were violated. In 2017 there were three main campaigns when 53 samples were taken and 1060 parameters analysed; 14 limit violations – all within the acceptance limits – were determined for oxidation stability and CFPP as well as the contents of sulphur, water, diglycerides, and free glycerol.

Since 2017, member companies are re-sampled in an additional campaign if irregularities (violations of limit or acceptance limit) were found during the main campaigns. Thus 14 samples were taken in three additional campaigns; there were no irregularities in campaigns AC2 and AC3. One member company exceeded the AGQM limit for 'total contamination' in AC1 which resulted in the assignment of one sanction point.

Table 1: List of all samples violating limits

Parameter	Method	Samples per Campaign					
		C1	AC1	C2	AC2	C3	AC3
Sulfur Content	DIN EN ISO 20846			1			
Water Content	DIN EN ISO 12937			1		1	
Total Contamination	DIN EN 12662		1				
Oxidation Stability	DIN EN 14112	2					
Diglyceride Content	DIN EN 14105	1		1		1	
Content of free Glycerol	DIN EN 14105	1		1			
CFPP	DIN EN 116	3		1			

Limit Violations within Acceptance Limit
Violations of the Acceptance Limit of DIN EN 14214
Violations of AGQM's Acceptance Limit

The results provide clear evidence that AGQM-associated biodiesel producers and traders safeguard the compliance with legal regulations at all times and that their quality level is progressing continuously.

5 Annex

5.1 Limits and Test Methods

Table 2: Limits and Test Methods for the Parameter tested according to DIN EN 14214:2014

Test Parameter	Method	Year of Publication	Measuring Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Fatty Acid Methyl Ester Content	DIN EN 14103	2015	% (w/w)	96.5	-	94.0	-
Density at 15 °C	DIN EN ISO 12185	1997	kg/m³	860	900	859.7	900.3
Sulphur Content (UV)	DIN EN ISO 20846	2011	mg/kg	-	10.0	-	11.3
Water Content K.-F.	DIN EN ISO 12937	2000	mg/kg	-	500	-	591
Total Contamination	DIN EN 12662	1998[1]	mg/kg	-	24	-	32
Oxidation Stability (at 110 °C)	DIN EN 14112	2014	h	8.0	-	6.6	-
Acid Number	DIN EN 14104	2003	mg KOH/g	-	0.50	-	0.54
Iodine Number	DIN EN 16300	2012	g iodine/100g	-	120	-	124
Content of Linolenic Acid Methyl Ester	DIN EN 14103	2015	% (w/w)	-	12.0	-	14.9

[1] Due to the fact that the current version of DIN EN 12662 is not suitable for the determination of parameter 'total contamination' in FAME, DIN EN 12662:1998 applies until further notice.

Arbeitsgemeinschaft
Qualitätsmanagement Biodiesel e.V.
Claire-Waldoff-Str. 7
10117 Berlin

Test Parameter	Method	Year of Publication	Measuring Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Content of Free Glycerol	DIN EN 14105	2011	% (w/w)	-	0.02	-	0.026
Content of Monoglycerides		2011	% (w/w)	-	0.70	-	0.82
Content of Diglycerides	DIN EN 14105	2011	% (w/w)	-	0.20	-	0.24
Content of Triglycerides		2011	% (w/w)	-	0.20	-	0.27
Overall Glyceride Content		2011	% (w/w)	-	0.25	-	0.28
Content of Alkalimetals (Na + K)	DIN EN 14538	2006	mg/kg	-	5.0	-	6.1
Sodium Content		2006	mg/kg	-	5.0	-	6.1
Potassium Content		2006	mg/kg	-	5.0	-	
Content of Earth Alkali Metals (Ca + Mg)		2006	mg/kg	-	5.0	-	6.1
Calcium Content		2006	mg/kg	-	5.0	-	6.1
Magnesium Content		2006	mg/kg	-	5.0	-	
Phosphor Content	DIN EN 14107	2003	mg/kg	-	4.0	-	4.5
CFPP (if used as blend component for Diesel fuel)	DIN EN 116	2015	°C	15 April to 30 September 1 October to 15 November 16 November to 28/29 Feb 1 March to 14 April	0 -5 -10 -5	- - - -	1.5 -3.2 -7.9 -3.2
Cloud Point (if used as blend component for Diesel fuel)	DIN EN 23015	2013	°C	15 April to 30 September 1 October to 15 November 16 November to 28/29 Feb 1 March to 14 April	5 0 -3 0	- - - -	7.4 2.4 -0.6 2.4

Table 3: Limits and Test Methods for the Parameter tested according to AGQM's Quality Management System

Test Parameter	Method	Year of Publication	Measuring Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Water Content (for producers)	DIN EN ISO 12937	2000	mg/kg	-	220	-	280
Water Content (for warehouse operators)	DIN EN ISO 12937	2000	mg/kg	-	300	-	370
Total Contamination	DIN EN 12662	1998[2]	mg/kg	-	20	-	20
CFPP (if used as blend component for Diesel fuel)	DIN EN 116	2015	°C	from 19 Oct to 28/29 Feb	-10	-	-17.3

[2] Due to the fact that the current version of DIN EN 12662 is not suitable for the determination of parameter 'total contamination' in FAME, DIN EN 12662:1998 applies until further notice.

5.2 Abbreviations

AC 1	Additional Campaign 1
AC 2	Additional Campaign 2
AC 3	Additional Campaign 3
AGQM	Association Quality Management Biodiesel
BImSchV	Bundes-Immissionsschutzverordnung (German Federal Emission Protection Directive)
C 1	Campaign 1
C 2	Campaign 2
C 3	Campaign 3
CEN	Comité Européen de Normalisation (European Standardisation Committee)
CFPP	Cold Filter Plugging Point
DIN	Deutsches Institut für Normung (German Institute for Standardisation)
DIN EN 14214:2014	German Version EN 14214:2012+A1:2014
EN	European Standard
FAME	Fatty Acid Methyl Ester
QM System	Quality Management System
QA Committee	Committee for Quality Assurance
TC	Technical Committee