Biodiesel Quality in Germany

Sampling results of biodiesel producers and Warehouse operators of Association Quality Management Biodiesel (AGQM)

2020

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1 Introduction

In view of the current discussions on climate change and the European Commission's Green Deal that aims to make Europe the first “climate-neutral” continent by 2050, the importance of biofuels for the decarbonization of the transport sector continues to increase.

With the use of biofuels, an average greenhouse gas reduction of approx. 83% could be achieved for the year 2019 (BLE Report 2019).

Biodiesel or FAME (fatty acid methyl ester), with an average greenhouse gas reduction of 81%, is the most important biofuel in the member states of the European Union and is used via an admixture with diesel fuel to reduce CO_2 emissions and the use of fossil fuels. In Germany, admixtures of up to 7% (B7) are available at public filling stations. The European Standard EN 14214 describes the qualitative requirements a biodiesel has to meet in order to market it as a ready-to-use product.

German biodiesel producers and warehouse operators founded the Association Quality Management Biodiesel (AGQM) in 1999 to meet the quality requirements of the resulting German standard DIN EN 14214. AGQM has become one of the most important institutions in the field of promoting and monitoring biodiesel quality in Germany and increasingly in other European countries over the past 20 years.

This report summarizes the results of the annual unannounced samplings that are carried out by AGQM at its members as a quality assurance measure several times a year. Sampling at member companies is carried out without prior notice. This ensures that the results are consistent with the actual operations of the producers and warehouse operators. The report relates to the campaigns carried out in 2020.

2 Sampling

The Quality Management System (QM system) of the AGQM stipulates that unannounced sampling is carried out on the members at least three times a year. Since 2017, all members who have been found to have an irregularity (violation of a limit or acceptance limit) in one of the three main campaigns have to participate in an additional campaign that follows the respective main campaign and is also unannounced.

Sampling and analysis are performed by an independent laboratory accredited for biodiesel analysis.

In 2020, 17 production sites and two warehouses were sampled. One winter, one intermediate and one summer campaign were carried out, as the National Annex NB of DIN EN 14214 sets different limits for the respective season for the parameters cloud point and CFPP. Each country can set these limits individually since the climatic conditions sometimes differ greatly.

The periods of the sampling were:

Campaign 1:	27. January to 07. February	Winter grade
Campaign 2:	15. June to 26. June	Summer grade
Campaign 3:	05. October to 16. October	Intermediate grade

In the main campaigns 56 samples were taken and analysed and in the resulting additional campaigns 15 samples. Three representative samples are taken during each sampling. One sample is used for analysis, the other two serves as potential arbitration samples.

The analysis results are evaluated by the AGQM office and the member companies are subsequently informed about the result. If there are doubts about the results of the sampling, members can address AGQM to apply arbitration proceedings. The member assigns an independent laboratory accredited for biodiesel analytics. As arbitration sample one of the two samples taken during the sampling is used. The result of the arbitral analysis is binding for both sides. If a deviation is confirmed in the arbitral analysis, the member has to participate in the next unannounced additional campaign and further sanction measures may be initiated.

The implementation of the QM system of the AGQM is evaluated for each member based on a points system. Bonus points are awarded for participation in quality assurance measures and sanction points for violations of the QM system. The percentage ratio of sanction points to bonus points is used to assess the need for sanctions.

3 Quality requirements

It is anchored in the QM system of AGQM that all quality parameters listed in the legal requirements of the 36th BImSchV (Federal Immission Control Ordinance) for the verification of the biofuel properties are examined while sampling. The analyses are always based on the valid version of DIN EN 14214. In 2020, the required standard limit values and the associated acceptance limit values resulting from the precision of the respective method corresponded to DIN EN 14214:2012 + A2:2019.

For the parameters water content, total contamination and cold filter plugging point (CFPP), AGQM sets higher demands on the biodiesel quality of its members than required by legislative authority. Separate limit values for warehouse operators were also defined in the AGQM guidelines for the parameter water content.

In the annex, Table 1 lists the parameters to be tested with their limit values according to DIN EN 14214 and Table 2 lists the AGQM limit values for the corresponding parameters.

In addition, the market development of recent years shows that to improve the greenhouse gas balance and support the circular economy concept, alternative raw materials for the production of biodiesel, e.g., used cooking oils and fats as well as fatty acids are used. Due to the nature of the raw materials, some of these products show quality differences compared to DIN EN 14214. Biodiesel from these alternative raw materials is usually not used as a pure fuel, but exclusively as a blending component for biodiesel from classic raw materials (primarily rapeseed oil). Since manufacturers of biodiesel from alternative raw materials are also members of the AGQM, a separate chapter for blending components for biodiesel was implemented in the QM system in autumn 2017. By blending such components with other goods, an overall standard-compliant biodiesel can be produced, which is why the specification can be extended within a narrow scope. For example, special limit values for blend components for biodiesel were included in the QM system for the parameters sulphur content, CFPP and cloud point. These three parameters are strongly determined by the fatty acid composition or impurities in the raw material and can hardly be influenced in the production process. If a producer applies to the AGQM for a corresponding exemption, the specific limit values (see annex Table 3) for blend components for biodiesel are used as the limit values to be complied with for the parameters sulphur content, CFPP and cloud point, rather than the values of DIN EN 14214. The samples that are blend components for biodiesel are shown in separate diagrams for the above-mentioned parameters.

4 Results of the sampling and evaluation

The results of the unannounced sampling at AGQM member companies are shown graphically in the following section. The applicable limit values and acceptance limit values are listed for each parameter and the parameter is classified regarding the influence on the product quality.

The results presented in this report are anonymous and do not indicate the origin of the sample. The values in the charts are shown in ascending order for each campaign to show the distribution. The "Sample Number" axis shows how many samples were taken in each campaign.

The limits are indicated in the diagrams by an orange line, the acceptance limits, which are calculated considering the precision of the method, by a red line. Under customs law, but also regarding the award of sanction points under the QM system, these acceptance limits are decisive.

For the parameters sulphur content, CFPP and cloud point, in addition to the diagrams with the standard limit values, an additional diagram is also shown in each case with the specific limit and acceptance limit values for blend components for biodiesel.

4.1 Fatty Acid Methyl Ester Content

Test method:	*DIN EN 14103:2011*
Limit of DIN EN 14214:	*min. 96.5% (m/m)*
Acceptance limit:	*min. 94.0% (m/m)*

The fatty acid methyl ester content, or short ester content, provides information on the purity of the biodiesel. Depending on the nature of the raw material and the reaction conditions, by-products may be present in the final product which reduce the ester content. It is determined by gas chromatography and expressed as the sum of all fatty acid methyl esters of C6:0 to C24:1 in mass per cent [% (m/m)]. EN 14214 requires an ester content of at least 96.5% (m/m). A distilled product after the transesterification usually has a higher ester content since undesirable substances can be separated in this way. According to DIN EN 14103, determined ester contents that are greater than 99.0% (m/m) are indicated with > 99.0% (m/m) as the result.

Figure 1 shows that in campaigns 2 and 3, one sample each was found to be below the standard limit (96.5% (m/m)). In general, the ester content is highest on average in the winter campaign. This is possibly due to the increased use of rapeseed oil as a feedstock for biodiesel in winter because of its good cold properties.

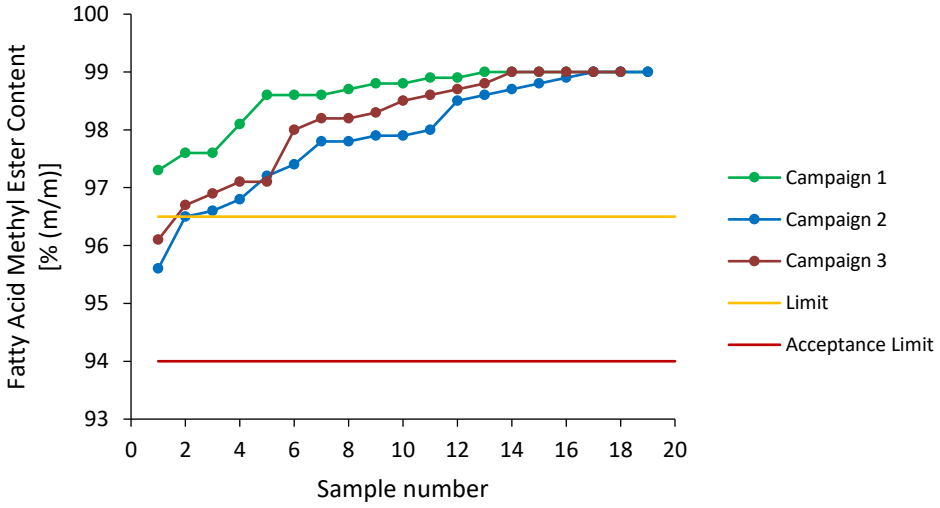

Figure 1: Fatty Acid Methyl Ester Content acc. to DIN EN 14103.

4.2 Density at 15 °C

Test method:	*DIN EN ISO 12185:1996*
Limit of DIN EN 14214:	*min. 860 kg/m³ and max. 900 kg/m³*
Acceptance limit:	*min. 859.7 kg/m³ and max. 900.3 kg/m³*

The density of a substance is the quotient of its mass and its volume at a specified temperature. It is determined by 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 the FAME composition and the purity of the biodiesel have an influence on the density. It can also be influenced by impurities. A higher methanol content for example reduces the density.

Figure 2 shows the density of the samples analysed. All samples comply with the density range required by the standard. Almost all samples are in a very narrow range between 880 kg/m³ and 883 kg/m³, which suggests the use of rapeseed oil as raw material. But there are also lower densities of about 875 kg/m³, suggesting the use of other raw materials.

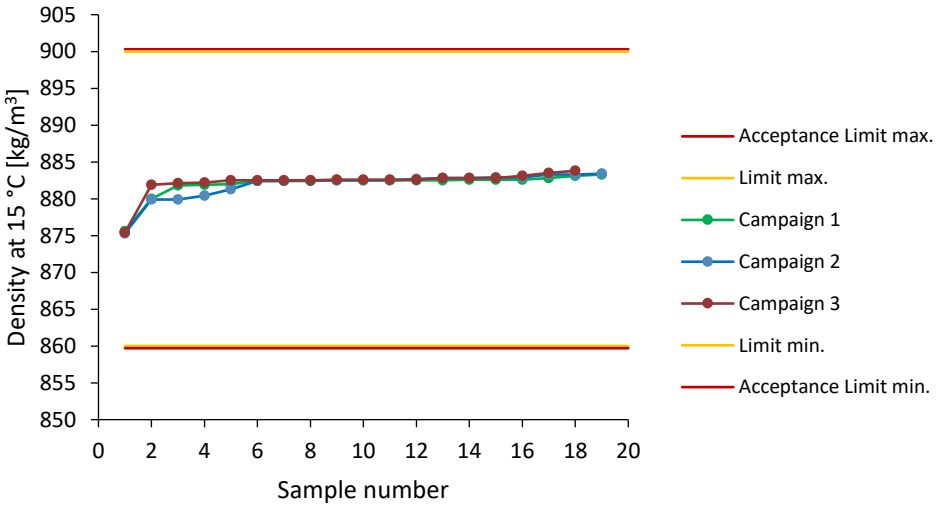

Figure 2: Density at 15 °C acc. to DIN EN ISO 12185.

4.3 Sulphur Content

Test method:	*DIN EN ISO 20846:2011*
Limit of DIN EN 14214:	*max. 10 mg/kg*
Acceptance limit:	*max. 11.3 mg/kg*
AGQM limit for blend components for biodiesel:	*max. 13 mg/kg*
AGQM acceptance limit for blend components for biodiesel:	*max. 14.5 mg/kg*

Sulphur can already be contained in the raw materials used for biodiesel production. In plants that can take up sulphur compounds during growth, the sulphur content is usually between 2 mg/kg and 7 mg/kg. Animal fats and used cooking oils can contain sulphur in the form of protein compounds, resulting in a sulphur content of up to 30 mg/kg. Depending on the type of sulphur compound, the content in the biodiesel can be reduced by washing processes or distillation of the biodiesel.

As can be seen in Figure 3, the required limits are met by all samples. The 95% quantile is 7.3 mg/kg. The 95% quantile describes the value below or above wich 95% of all results are laying.

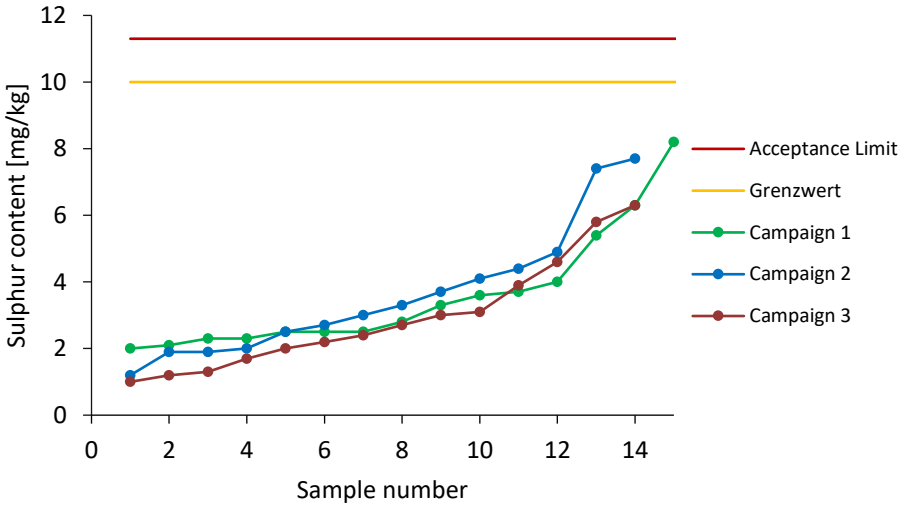

Figure 3: Sulphur content acc. to DIN EN ISO 20846.

Figure 4 shows those samples that are used as blend components for biodiesel. According to AGQM QM system section 2.1.1, deviating limit values apply to these samples (see Appendix, Table 3). All samples of blend components for biodiesel were below the specific limit of 13 mg/kg.

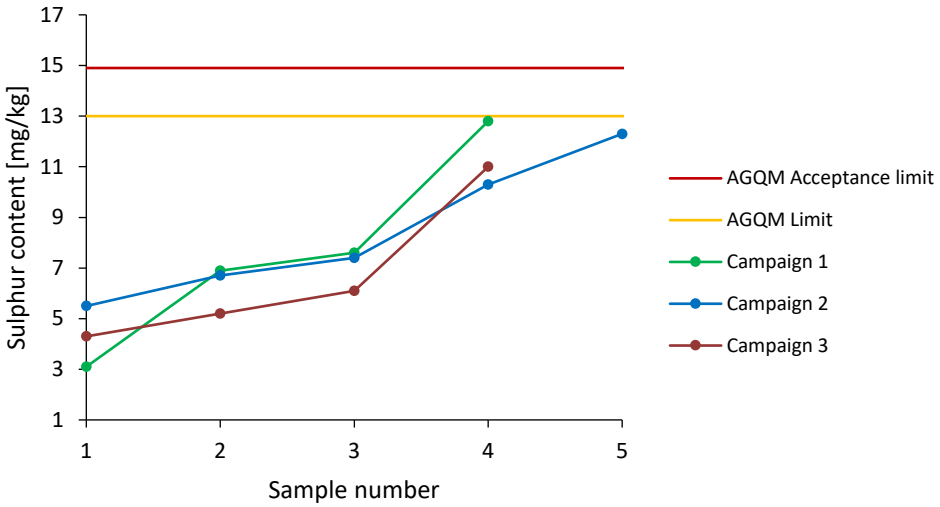

Figure 4: Sulphur content of blend components for biodiesel acc. to DIN EN ISO 20846.

4.4 Water Content

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

Biodiesel can physically dissolve up to 1500 mg of water/kg biodiesel because it has a higher polarity than hydrocarbon-based fuels. Since almost all production processes include a water wash, the product needs to be dried at the end of the biodiesel production. Subsequently, the storage conditions must be selected accordingly to avoid a renewed contamination of the biodiesel by atmospheric moisture.

Fossil diesel fuels can dissolve only very small amounts of water, so when mixing with biodiesel with a very high water content, the dissolved water can precipitate. Freezing of potential free water can block filters or piping and it can cause corrosion or promote microbial growth. DIN EN 14214 requires a maximum water content of 500 mg/kg. Due to the problems described above, AGQM has set stricter requirements with a maximum water content of 220 mg/kg for producers and 300 mg/kg for warehouse operators.

Figure 5 shows the values for the water content. All samples tested are well below the standard limit.

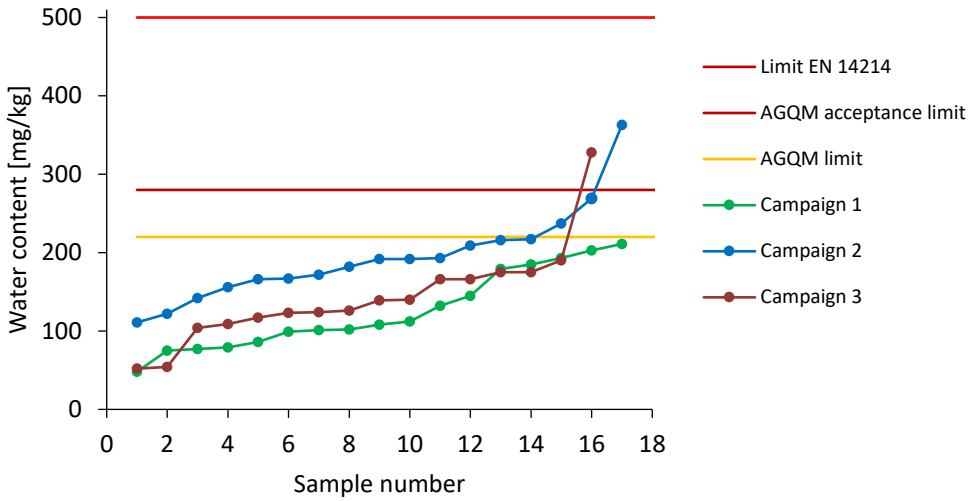

Figure 5: Water content acc. DIN EN ISO 12937.

In campaign 2 and 3, the AGQM limit for manufacturers and the corresponding AGQM acceptance limit are exceeded twice by one company with 363 mg/kg and 328 mg/kg. The company had itself identified a technical fault in a heat exchanger. Corresponding sanction points were awarded for exceeding the limit. There were also two further exceedances of the AGQM limit value by two companies in campaign 2, but within the corresponding acceptance limit value.

To preserve the anonymity of the only sampled warehouse operator, the results of these samples are not presented here.

4.5 Total Contamination

Test method: *DIN EN 12662:1998*
Limit of DIN EN 14214: *max. 24 mg/kg*
AGQM limit: *max. 20 mg/kg*

AGQM 's limit for parameter Total Contamination is also AGQM's acceptance limit.

The total contamination is a measure of the content of non-soluble particles ("rust and dust") in the product. The determination is carried out after filtration of a heated sample gravimetrically by weighing the filter. Biodiesel is normally not distilled, which is why total contamination is an important quality feature. High levels of insoluble particles can lead to filter blockages and wear on the injection system. The AGQM has set its own stricter limit of 20 mg/kg as the acceptance limit to address this issue.

Figure 6 shows that all samples can meet the stricter AGQM limit for total contamination.

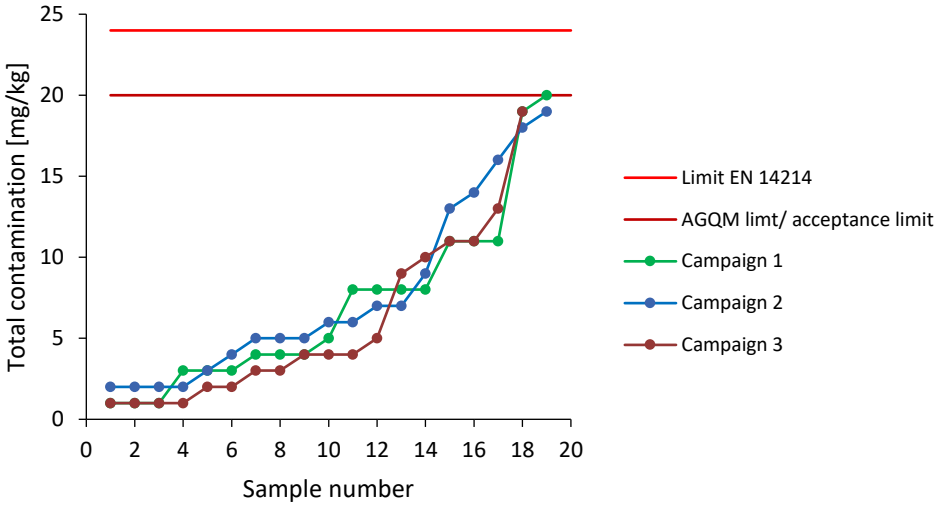

Figure 6: Total contamination acc. to DIN EN 12662.

4.6 Oxidation Stability

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

Vegetable oils and biodiesel derived therefrom contain natural antioxidants (e.g., tocopherols) that slow down the aging process. In addition, synthetic stabilizers are also used. Once a year, AGQM tests products from interested additive producers that can be used to increase the oxidation stability of biodiesel. Additives that pass the test are published in the so-called "No-Harm List" on the AGQM website.

As test method for the oxidation stability of biodiesel, the so-called Rancimat test is performed. At 110 °C, a constant stream of air is passed through the sample. After the oxidation reserve (natural reserve and additives) of the sample has been degraded, volatile oxidation products are formed, which together with the air are transferred into the test liquid of the measuring cell, where they increase the conductivity. The time until these oxidation products are detected is referred to as induction time or oxidation stability. DIN EN 14214 requires a minimum oxidation stability of 8 hours.

Figure 7 shows the oxidation stabilities of the tested samples. In campaigns 1 and 2, a total of five samples falls below the standard limit of 8.0 h, but all within the acceptance limit (6.6 h). In campaign 3 there are no abnormalities.

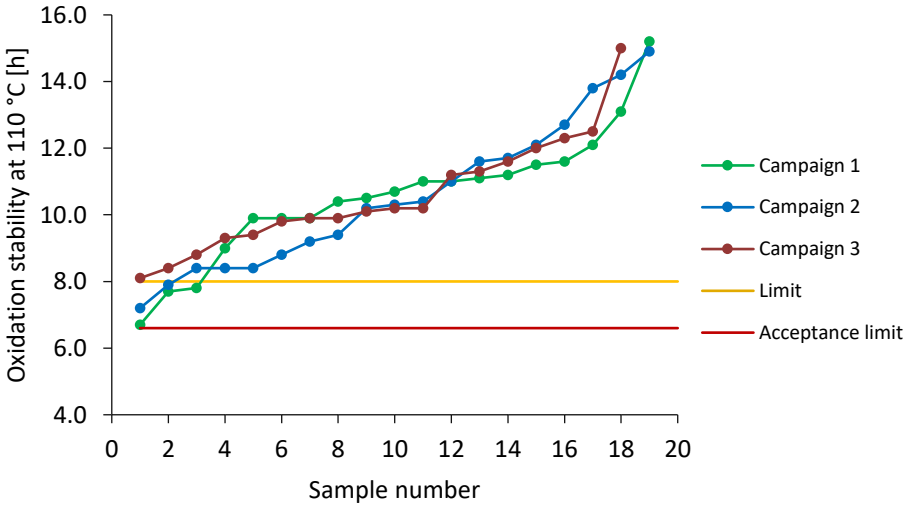

Figure 7: Oxidation Stability acc. to DIN EN 14112.

4.7 Acid Number

Test method:	*DIN EN 14104:2003*
Limit of DIN EN 14214:	*max. 0.50 mg KOH/g*
Acceptance limit:	*max. 0.54 mg KOH/g*

The acid number is a measure of the free acids (especially fatty acids) in biodiesel. Fatty acids are weak acids and therefore only slightly corrosive. In the production process, small residues of alkaline metal soaps are cleaved by washing with inorganic acids. The resulting free fatty acids can remain in the biodiesel. The acid number can also increase during storage of FAME, as aging processes (especially oxidation) lead to ester cleavage or the formation of short-chain carboxylic acids. Under typical storage conditions, however, this effect is hardly observed. DIN EN 14214 requires an acid value of no more than 0.50 mg KOH/g.

Figure 8 shows the measured values for the acid value. Only one sample exceeds the standard limit of 0.50 mg KOH/g in campaign 3 with a content of 0.52 mg KOH/g within the acceptance limit.

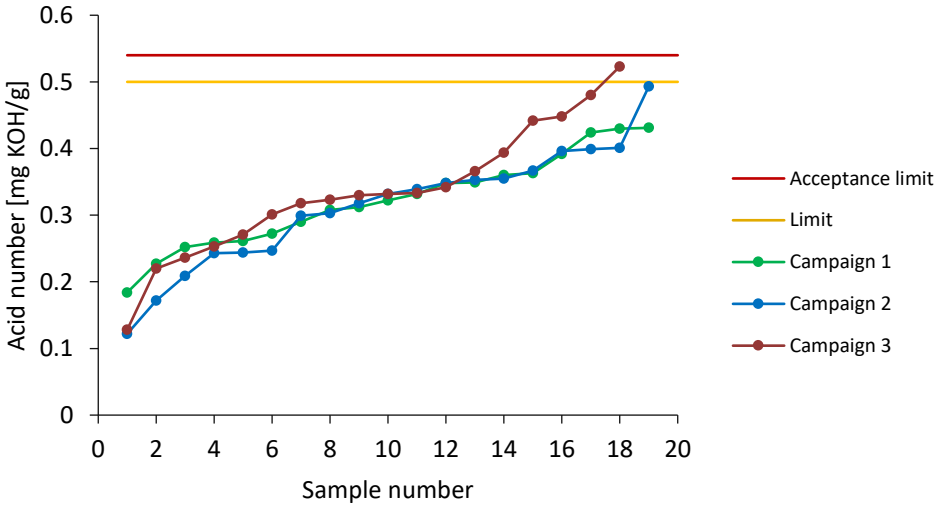

Figure 8: Acid Number acc. to DIN EN 14104.

4.8 Iodine Number

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

The iodine number is a measure of the proportion of double bonds which is present in fatty acid methyl ester. It varies with the type of raw material used. Since unsaturated fatty acids are more prone to oxidation reactions, the stability of biodiesel decreases with increasing number of double bonds, i.e., increasing iodine number. Therefore, the iodine number, in addition to the oxidation stability, is an indicator of the stability of biodiesel.

For determination, two different methods are specified in DIN EN 14214. In the AGQM sampling campaigns, the iodine number is calculated from the fatty acid profile measured by gas chromatography in accordance with DIN EN 16300. The result is given in g iodine/100 g biodiesel.

Figure 9 shows the results for the iodine number. All examined samples are below the standard limit value. The products of one company show iodine numbers below 60 g iodine/100 g biodiesel all year round, which is also due to the raw material used.

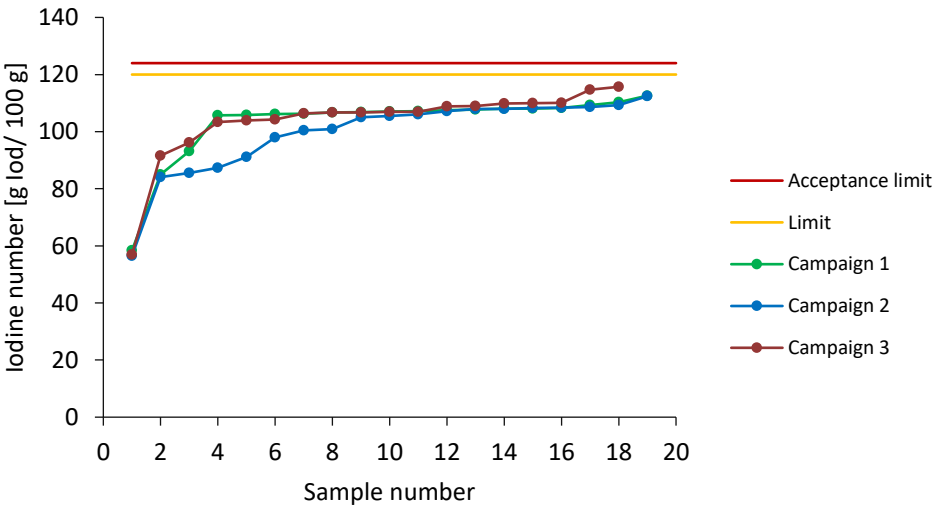

Figure 9: Iodine number acc. to DIN EN 16300.

4.9 Mono-, di- and triglycerides, total glycerol and free glycerol

Test method:	*DIN EN 14105:2011*
Monoglycerides	
Limit of DIN EN 14214:	*max. 0.70% (m/m)*
Acceptance limit:	*max. 0.82% (m/m)*
Diglycerides	
Limit of DIN EN 14214:	*max. 0.20% (m/m)*
Acceptance limit:	*max. 0.24% (m/m)*
Triglycerides	
Limit of DIN EN 14214:	*max. 0.20% (m/m)*
Acceptance limit:	*max. 0.27% (m/m)*
Total glycerol	
Limit of DIN EN 14214:	*max. 0.25% (m/m)*
Acceptance limit:	*max. 0.28% (m/m)*
Free glycerol	
Limit of DIN EN 14214:	*max. 0.020% (m/m)*
Acceptance limit:	*max. 0.026% (m/m)*

In the transesterification of vegetable oils with methanol, in addition to the main product (fatty acid methyl ester) also different levels of by-products like mono- and diglycerides, free glycerol occur. Furthermore, unreacted vegetable oil (triglycerides) is found in the reaction mixture. Since glycerol is virtually insoluble in biodiesel, it can be separated almost completely by decanting and subsequent water washing. The ratio of the content of mono-, di- and triglycerides is a measure of the completeness of the transesterification reaction since the concentration usually increases in the order triglycerides < diglycerides < monoglycerides. The cleavage of the last fatty acid residue is the slowest step of the reaction, therefore the standard limit for monoglycerides is slightly higher at 0.70% (m/m) than that for di- and triglycerides at 0.20% (m/m). The content of mono-, di- and triglycerides can only be reduced to a certain degree, since in each case a chemical equilibrium between products and educts is established. The almost complete removal of the glycerides is possible only by distillation.

Figure 10 to Figure 12 show the results for mono-, di- and triglycerides. In campaign 3, the standard limit of 0.02% (m/m) for the content of diglycerides was exceeded by one member within the corresponding acceptance limit. All other samples did not show any abnormalities. For the content of monoglycerides, some samples even show values < 0.01% (m/m), which suggests that the production process includes a distillation step.

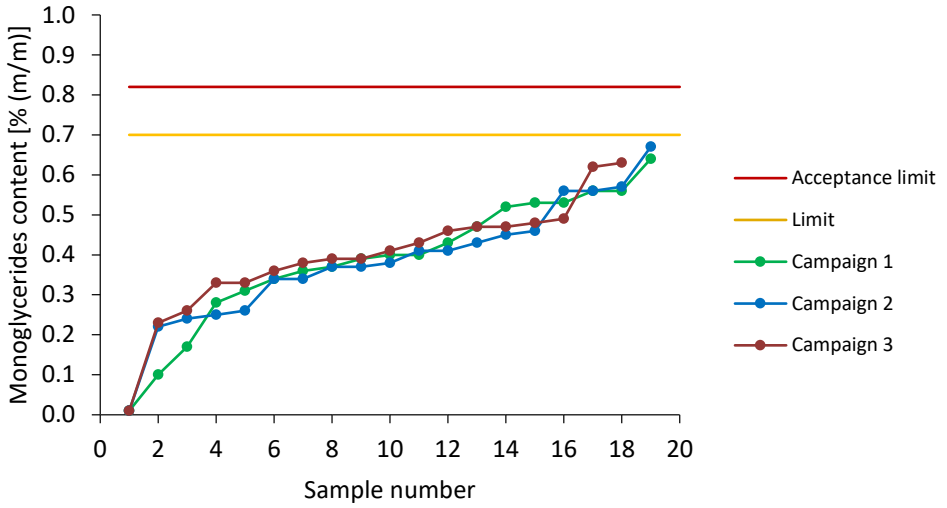

Figure 10: Monoglycerides acc. to DIN EN 14105.

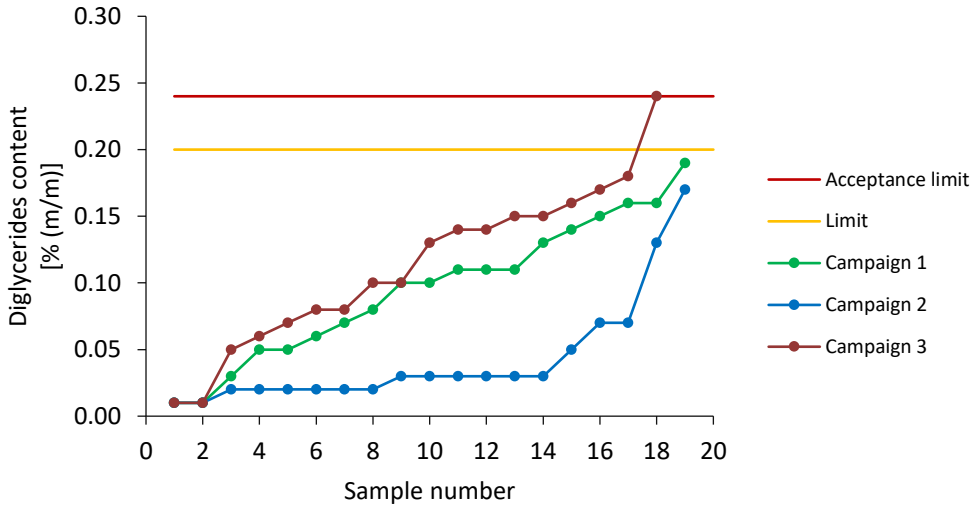

Figure 11: Diglycerides acc. to DIN EN 14105.

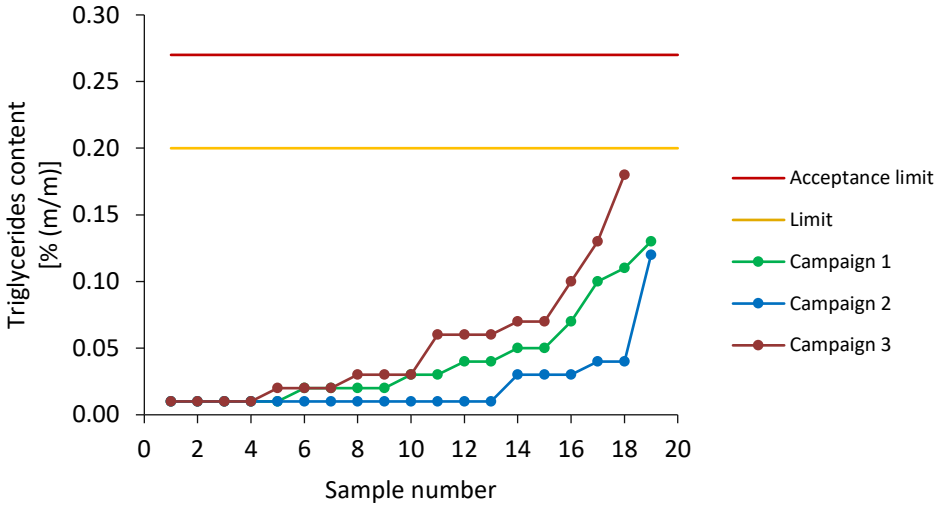

Figure 12: Triglycerides acc. to DIN EN 14105.

The total glycerol content is shown in Figure 13. All samples analysed comply with the standard limit of 0.25% (m/m).

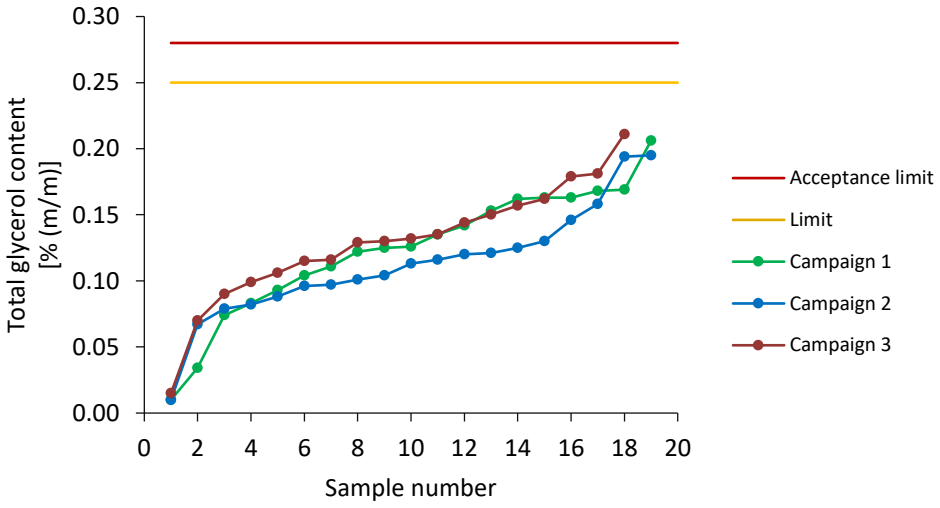

Figure 13: Total glycerol acc. to DIN EN 14105.

Figure 14 shows the free glycerol content. One sample in campaign 3 exceeds the standard limit of 0.020% (m/m) with a content of 0.024% (m/m) within the acceptance limit. In the subsequent additional sampling, the same member was found to have a high content of free glycerol outside the acceptance limit of 0.026% (m/m). The analysis showed a content of 0.027% (m/m). The member received one sanction point for exceeding the acceptance limit. Problems in the washing process could be identified as the cause, which could then be remedied. All other samples comply with the standard limit.

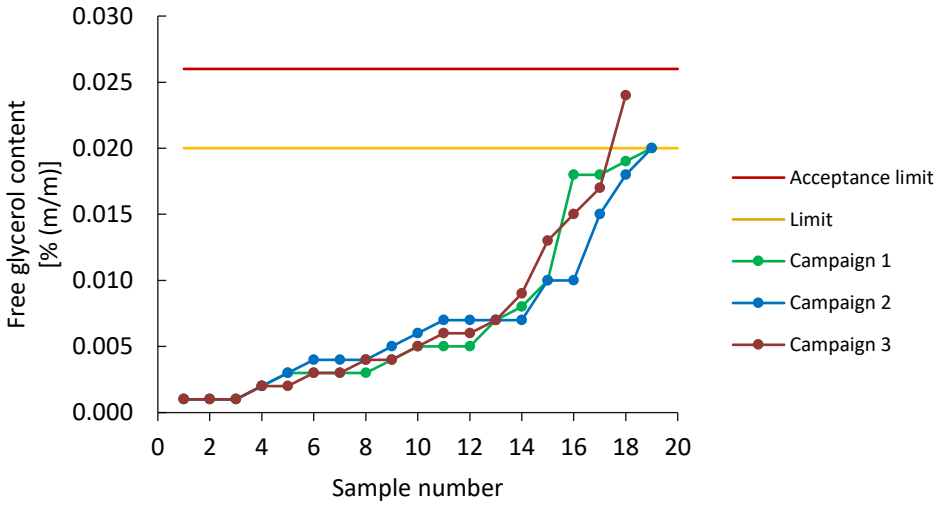

Figure 14: Free glycerol acc. to DIN EN 14105.

4.10 Alkaline Metals (Sodium + Potassium) and Alkaline Earth Metals (Calcium + Magnesium)

Test method:	*DIN EN 14538:2006*
Limit of DIN EN 14214:	*max. 5.0 mg/kg (sum parameter)*
Acceptance limit:	*max. 6.1 mg/kg (sum parameter)*

For biodiesel production, sodium and potassium hydroxides or methylates are usually used as catalysts. If residues of it could not be completely removed in the wash process, these are usually present in biodiesel in form of soaps. Soaps can lead to filter plugging and clogging of injection pumps and nozzle needles and are also associated with ash formation.

The alkaline earth metals calcium and magnesium are either added to the raw material in the process or can reach the product through the use of tap water for water washing during the production

process. The reaction with free fatty acids produces calcium and magnesium soaps that are more voluminous than alkaline metal soaps.

Figure 15 and Figure 16 clearly show that biodiesel producers attach great importance to low levels of alkaline and alkaline earth metals. The contents of the alkaline metals sodium and potassium are, except for three samples, all below 2 mg/kg, 95% of the samples have alkaline metal contents below 1.5 mg/kg. The contents of the alkaline earth metals magnesium and calcium are even well below the limit of quantification of 0.1 mg/kg.

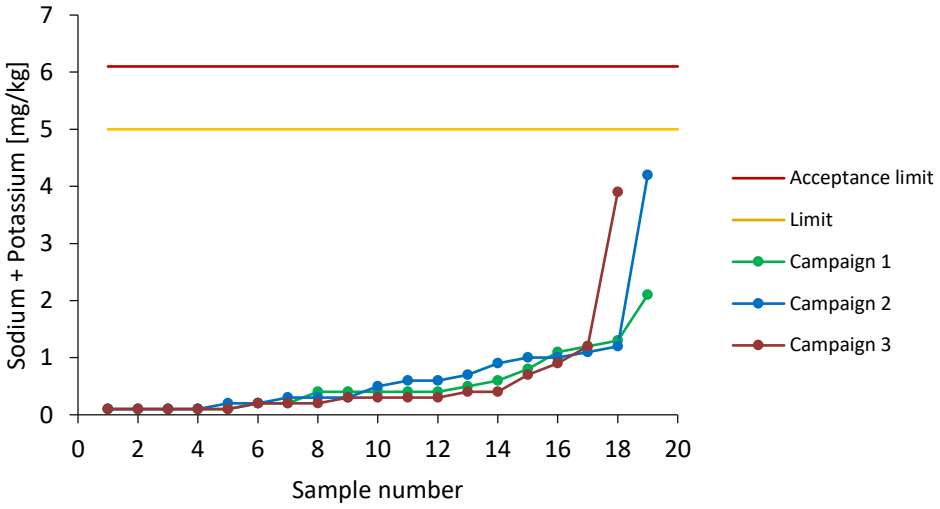

Figure 15: Sum of alkali metals Sodium and Potassium acc. to DIN EN 14538.

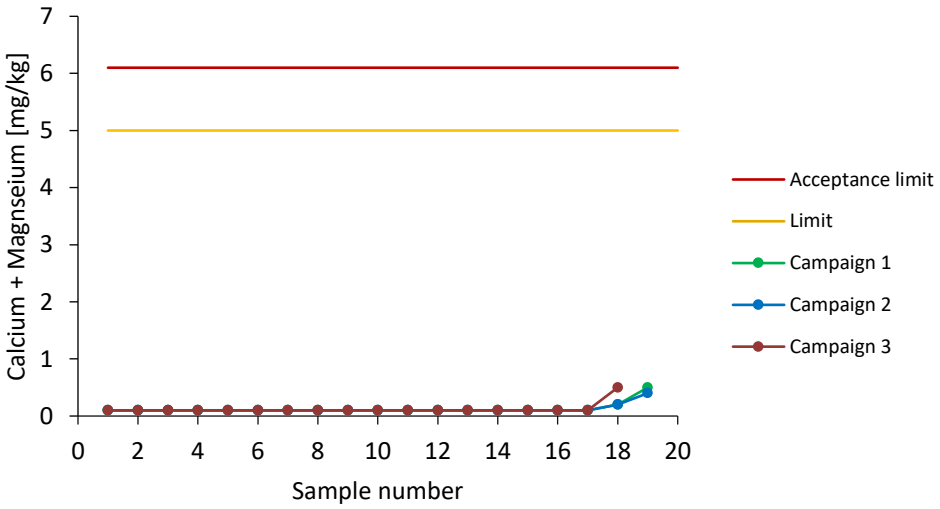

Figure 16: Sum of alkaline earth metals Calcium and Magnesium acc. to DIN EN 14538.

4.11 Phosphorous Content

Test method:	*DIN EN 14107:2003*
Limit of DIN EN 14214:	*max. 4.0 mg/kg*
Acceptance limit:	*max. 4.5 mg/kg*

The phosphorus content must already be considered during the selection of raw materials or reduced by a refining process before transesterification. Vegetable oils and animal fats contain phosphorous in form of phospholipids. These can hinder the transesterification process since they act as emulsifiers and thus disrupt the phase separation. Phosphorus can also enter the biodiesel during production if phosphoric acid is used to break down the soaps, but it is usually easy to remove with water. Since phosphorus is a catalyst poison, it can affect the effect of exhaust gas aftertreatment systems. Currently, work is in progress to include the parameter in EN 14538.

Figure 17 shows the values for the phosphorus content. Except for one sample in campaign 3, the contents are significantly lower than 2 mg/kg and are thus well below the standard limit of 4.0 mg/kg. The 95% quantile of the values is lower than 1.5 mg/kg.

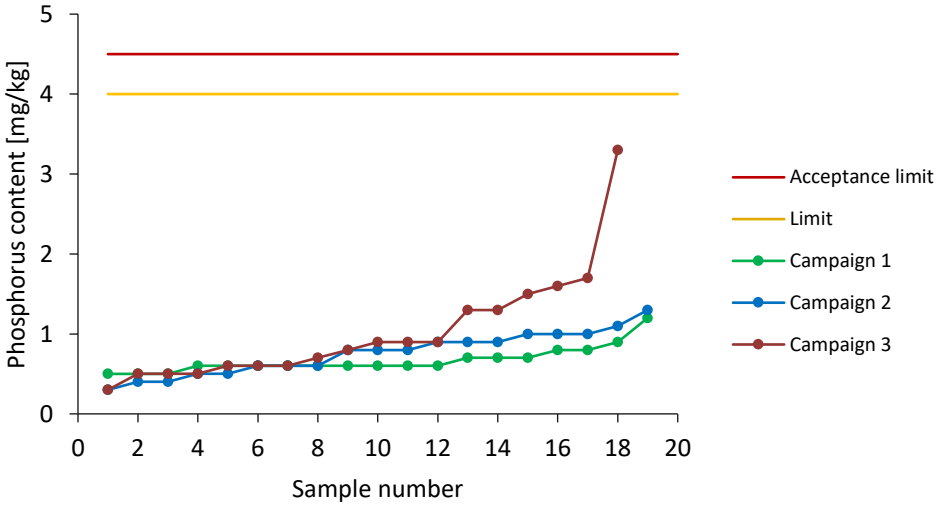

Figure 17: Phosphorus content acc. to DIN EN 14107.

4.12 Content of Linolenic Acid Methyl Ester

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

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 methyl ester in biodiesel is limited to 12% (m/m). It is determined from the fatty acid profile by gas chromatography.

As shown in Figure 18, all analysed samples have a content of linolenic acid methyl ester within the requirements of the standard. The linolenic acid content of pure rapeseed oil is usually between 7% and 10%. The lower contents in a large part of the samples show that the raw material rapeseed oil usually used in biodiesel production in winter was at least partially replaced by other oils in the summer campaign (campaign 2).

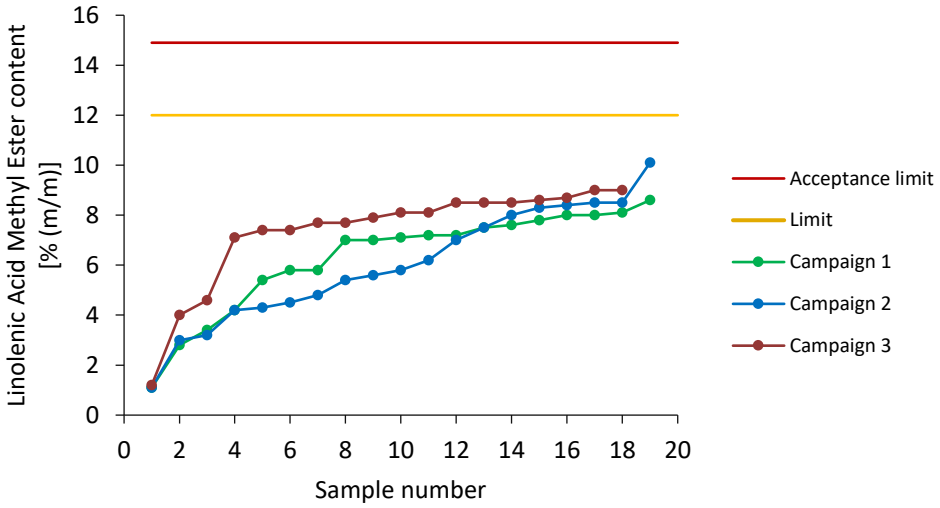

Figure 18: Content of Linolenic Acid Methyl Ester acc. to DIN EN 14103.

4.13 Cold Filter Plugging Point (CFPP)

Test method: *DIN EN 116:2015*

Limits according to DIN EN 14214 for biodiesel as blend component in diesel fuel:

Period	*Limit*	*Acceptance limit*	
from 15.04. to 30.09.	*0 °C*	*+1.8 °C*	*Summer period*
from 01.10. to 15.11.	*-5 °C*	*-3.2 °C*	*Intermediate period*
from 16.11. to 28./29.02.	*-10 °C*	*-7.9 °C*	*Winter period*
from 01.03. to 14.04.	*-5 °C*	*-3.2 °C*	*Intermediate period*
AGQM limit for blend components for biodiesel	*+10 °C*	*+11.4 °C*	*All year*

The CFPP is a measure of the filterability of biodiesel at low temperatures. The requirements for "cold resistance" are regulated nationally depending on the prevailing climatic conditions resulting in different requirements for summer, intermediate and winter quality.

In Germany, regarding the cold properties, the legal regulation applies that biodiesel as a blend component for diesel fuel must comply with a CFPP of -10 °C between 16.11. and the 28./29.02. if the -20 °C required in DIN EN 14214 can be achieved by additivation. The additivation of the mixture of diesel fuels and biodiesel then takes place in the refineries.

Figure 19 shows the measured values and various limit values for the CFPP. The winter limits and campaign 1 are shown in green, the summer limits and campaign 2 in blue and the transition period with campaign 3 in red.

In campaign 1, the winter acceptance limit of -7.9 °C is exceeded by a member with a CFPP of +2 °C. The member was already producing biodiesel as a blending component for biodiesel during this period but did not make use of the exemption until campaign 2. Consequently, there were no further anomalies in the subsequent campaigns. The specific limit values are complied with for all other samples.

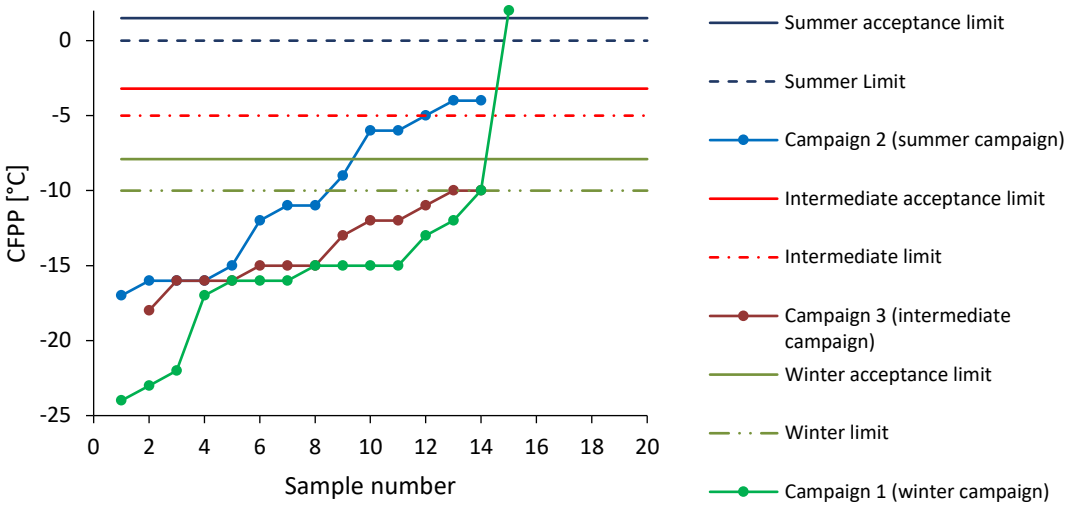

Figure 19: CFPP acc. to DIN EN 116.

Figure 20 shows the measured values of the members for the CFPP that make use of the exemption for blend components for biodiesel according to QM system section 2.1.1. For this case, an AGQM limit value of +10 °C, which applies all year round, was set. All samples comply with the corresponding limit value.

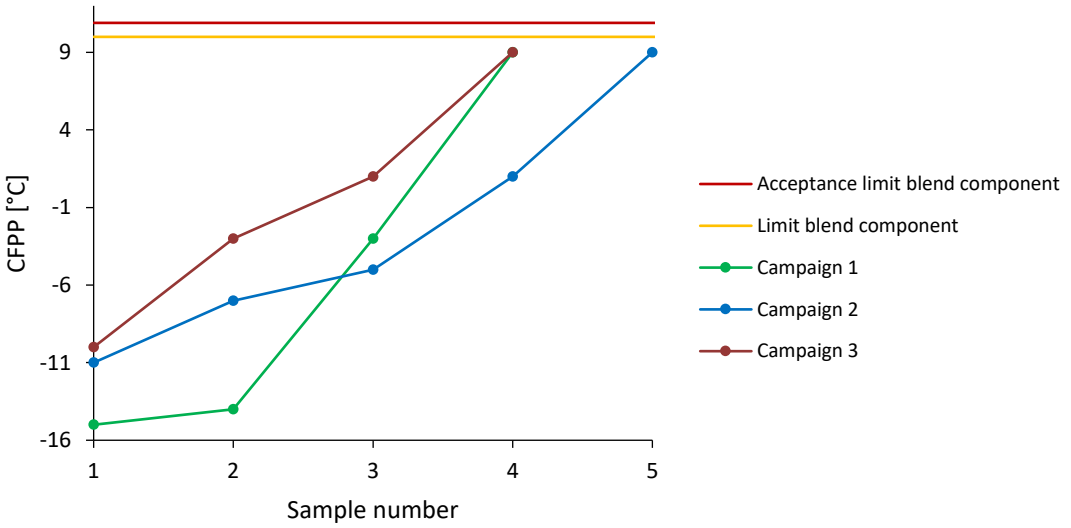

Figure 20: CFPP of blend components for biodiesel acc. to DIN EN 116.

4.14 Cloud point (CP)

Test method: *DIN EN 23015:2013*

Limit according to DIN EN 14214 for biodiesel as blend component in diesel fuel:

Period	*Limit*	*Acceptance limit*	
from 15.04. to 30.09.	*5 °C*	*7.4 °C*	*Summer period*
from 01.10. to 15.11.	*0 °C*	*2.4 °C*	*Intermediate period*
from 16.11. to 28./29.02.	*-3 °C*	*-0.6 °C*	*Winter period*
from 01.03. to 14.04.	*0 °C*	*2.4 °C*	*Intermediate period*
AGQM limit for blend components for biodiesel	*+15 °C*	*+17.4 °C*	*All year*

The cloud point is the temperature at which the first temperature-related turbidities ("clouds") form in a clear, liquid product on cooling under specified test conditions. Since 2012 with the publication of DIN EN 14214:2012, the cloud point in Germany is part of the requirement for biodiesel as blend component for diesel fuel.

Figure 21 shows the measured values for the cloud point. The winter limits and campaign 1 are shown in green, the summer limits and campaign 2 in blue and the transition period with campaign 3 in red. In campaign 1 there are a total of four exceedances of the winter limit value of -3 °C, one of which is outside the corresponding acceptance limit value. This anomaly is also due to the member that only claimed the exemption for blend components for biodiesel from campaign 2 onwards (cf. section 4.13). A sanction point was also awarded for this exceedance. For all other samples, the corresponding limit values are complied with.

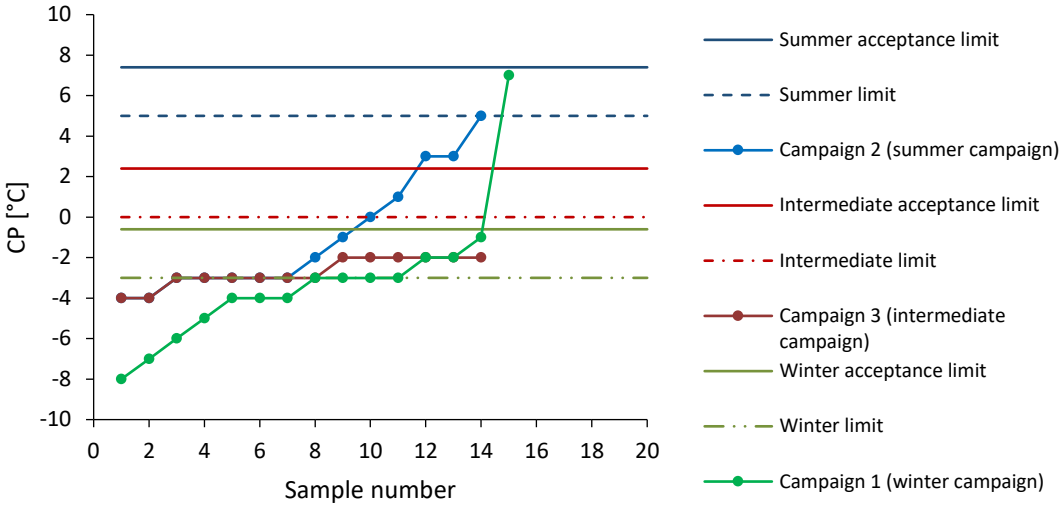

Figure 21: Cloud point acc. to DIN EN 23015.

As with the CFPP, separate limits apply to the cloud point for manufacturers of blend components for biodiesel.

In Figure 22, the measured cloud points are shown for the products of those members which make use of the exemption for blend components for biodiesel. The analysed samples of the blend components for biodiesel are all below the year-round specific limit of +15 °C.

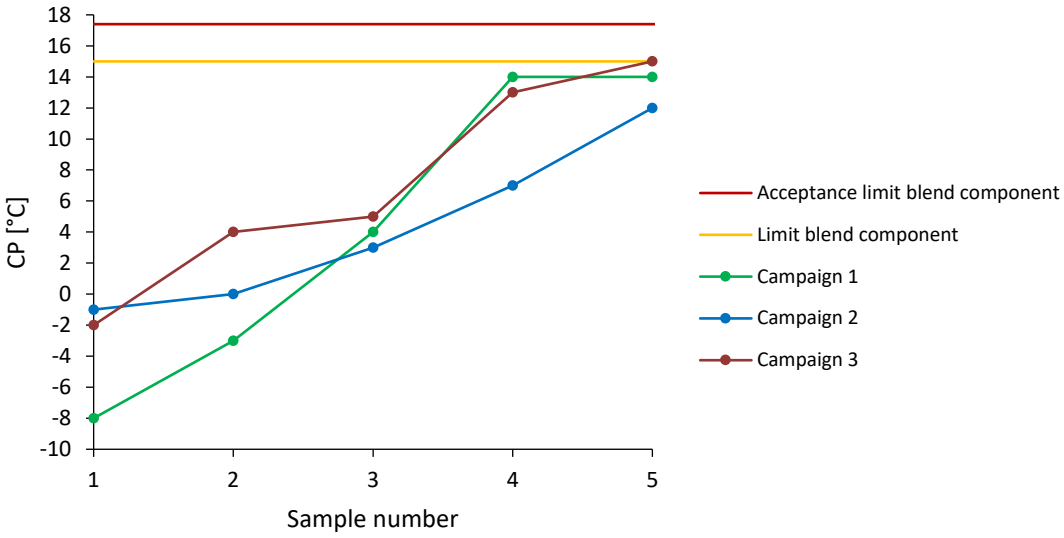

Figure 22: Cloud point of blend components for biodiesel acc. to DIN EN 23015.

Additional Campaigns

Since 2017, members who have been identified as having a deviation (violation of limit or acceptance limit) in a main campaign will have to attend an additional unannounced sampling campaign. In 2020, a total of three additional campaigns had to be carried out in which a total of 15 samples were taken.

In the additional campaign 1, a sum of five companies were sampled and a violation of the limit value outside the corresponding acceptance limit value was detected. A sanction point was awarded to the company. Upon request, the company stated that appropriate measures had already been taken to remedy the problem.

Six companies participated in the additional campaign 2. In the additional campaign, there were five violations of the limit value within the acceptance limit value by a total of three companies.

In the additional campaign 3, four companies were sampled. Three violations of the corresponding limit value were detected by two companies within the acceptance limit value. One company additionally exceeded a limit value outside the rejection limit value. A sanction point had to be awarded to the company in question. The company stated that it had already detected the problem itself and corrected it accordingly.

5 Summary

Since 2010, AGQM has published an annual report on the quality of the biodiesel produced and traded by its members. In this report, the results of the unannounced sampling of the year 2020 are presented.

A comparison of the sample numbers for 2019 and 2020 shows that the total number of samples tested in 2020 (71) was significantly higher than the number of samples tested in 2019 (59). Likewise, a larger number of limit violations were recorded in the respective main campaigns in 2020 than in 2019, so that more companies had to participate in the additional campaigns. However, the number of violations of the acceptance limit values remained roughly constant compared to the previous year, so that the quality of the products in terms of compliance with the standard remained the same.

The companies where abnormalities were found while sampling also detected the deviations within the scope of self-monitoring, so that it was possible to effectively prevent the goods from being placed on the market. In addition, with the support of the AGQM office, measures were taken to optimize the production process to prevent further limit violations.

The results show that unannounced sampling is an effective measure of detecting abnormalities and taking countermeasures as quickly as possible. In this way, the samples provide intensive support for the company's own quality management as an independent control instrument. The AGQM supports all members with various support measures (e.g., audits or coaching) in researching the causes and remedying problems and promotes access to and the exchange of know-how with various expert committees. The worldwide unique FAME round robin test, which is organized by AGQM in cooperation with the DIN FAM, promotes continuous improvement and further development of the operating and analysis laboratories located at the member companies.

In this way, AGQM and its members make an important contribution to the stable and high-quality supply of the European fuel market with biodiesel as a sustainable and greenhouse gas reduced biofuel. The AGQM label is therefore a reliable quality feature for customers and dealers in the market.

6 Annex

6.1 Limits and Test Methods

Table 1: Limits and test methods for the parameters tested according to DIN EN 14214:2019.

Test Parameter	Method	Year of Publication	Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Fatty Acid Methyl Ester Content	DIN EN 14103	2011	% (m/m)	96.5	-	94.0	-
Density at 15 °C	DIN EN ISO 12185	1996	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	2002	mg/kg	-	500	-	654
Total Contamination	DIN EN 12662	1998[1]	mg/kg	-	24	-	31
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/ 100 g	-	120	-	124
Content of Linolenic Acid Methyl Ester	DIN EN 14103	2011	% (m/m)	-	12.0	-	12.4
Content of Free Glycerol	DIN EN 14105	2011	% (m/m)	-	0.02	-	0.026
Content of Monoglycerides			% (m/m)	-	0.70	-	0.82

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

Test Parameter	Method	Year of Publication	Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Content of Diglycerides	DIN EN 14105	2011	% (m/m)	-	0.20	-	0.24
Content of Triglycerides			% (m/m)	-	0.20	-	0.27
Content of total Glycerol			% (m/m)	-	0.25	-	0.28
Content of Alkali Metals (Na+K)	DIN EN 14538	2006	mg/kg	-	5.0	-	6.1
Sodium Content			mg/kg	-	5.0	-	6.1
Potassium Content			mg/kg	-	5.0	-	
Content of Earth Alkali Metals (Ca+Mg)			mg/kg	-	5.0	-	6.1
Calcium Content			mg/kg	-	5.0	-	6.1
Magnesium Content			mg/kg	-	5.0	-	
Phosphorous 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	from 15.04. to 30.09. from 01.10. to 15.11. from 16.11. to 28/29.02 from 01.03. to 14.04	0 -5 -10 -5	- - - -	1.8 -3.1 -7.9 -3.1
Cloud point (if used as blend component for diesel fuel)	DIN EN 23015	2013	°C	from 15.04. to 30.09. from 01.10. to 15.11 from 16.11. to 28/29.02 from 01.03. to 14.04	5 0 -3 0	- - - -	7.4 2.4 -0.6 2.4

Table 2: Limits and test methods for the parameters tested according to the QM system of AGQM.

Test Parameter	Method	Year of Publication	Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Water Content (for Producers)	DIN EN ISO 12937	2002	mg/kg	-	220	-	322
Water Content (for Traders)	DIN EN ISO 12937	2002	mg/kg	-	300	-	419
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 19.10. to 28/29.02	-10	-	-7.9

Table 3: Limits and test methods for the tested parameters for blend component for biodiesel according to the QM system of AGQM.

Test Parameter	Method	Year of Publication	Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Sulphur Content	DIN EN ISO 20846	2011	mg/kg	-	13.0	-	14.9
Cloud point	DIN EN 23015	2013	°C	-	15	-	17.4
CFPP	DIN EN 116	2015	°C	-	10	-	10.9

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