Biodiesel Quality in Germany

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

2021

Project Management and Report:
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1 Introduction

In view of the Fit for 55 packages of measures and the European Commission's Green Deal, according to which the EU is to become climate-neutral by 2050, the importance of biofuels for the decarbonisation of the transport sector continues to increase.

This is also shown by the data published in the 2020 BLE report on the total greenhouse gas emission savings of all biofuels, which, as in the previous year, were again around 83%. The largest share is attributable to the use of biodiesel (fatty acid methyl esters - FAME).

Thus, biodiesel is considered the most important biofuel in the member states of the European Union and is used there as an admixture to diesel fuel and pure fuel to reduce CO_2 emissions and the consumption of fossil fuels. In Germany, blends of up to 7% (B7) are available at public filling stations. The European standard EN 14214 describes the qualitative requirements that a biodiesel must meet to be marketed as a neat fuel or blend component.

The Association Quality Management Biodiesel (AGQM) was founded in 1999 by German biodiesel producers and traders in order to ensure that the quality requirements of the German biodiesel fuel market are met and has since developed into one of the most important institutions in the field of promoting and monitoring biodiesel quality in Germany and other European countries.

As a quality assurance measure, AGQM carries out sampling at its members' sites several times a year. Sampling at the member companies is carried out without prior notice, thus ensuring that the results correspond to the real operations of the producers and storage operators. This quality report summarises the results of these demanding sampling campaigns from the year 2021.

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 are 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 2021, samples were taken at 16 production sites and two warehouses. A campaign was carried out in each of the winter, intermediate and summer periods to take into account the regulations of the

national Annex NB of DIN EN 14214 for the limit values of the parameters cloud point and CFPP. Each member state can set these limit values individually, as climatic conditions differ greatly in some cases.

Campaign 1:	01. February to 12. February	Winter grade
Campaign 2:	31. Mai to 11. June	Summer grade
Campaign 3:	18. October to 29. October	Intermediate grade

A total of 54 samples were taken and analysed in the main campaigns and 9 samples in the necessary additional campaigns. Three representative samples are taken in each sampling. One sample is used for analysis, one sample is stored at the analysis laboratory as a potential arbitration sample and one sample remains with the producer as a reserve sample.

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 further measures.

3 Quality requirements

According to the AGQM QM system, all quality parameters listed in the legal requirements of the 36th BImSchV (Federal Immission Control Ordinance) are examined during sampling to verify the biofuel properties.

The analyses are always based on the valid version of DIN EN 14214. In 2021, the required standard limit values as well as the associated acceptance limit values 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 and for the parameter total contamination the AGQM limit value also corresponds to the AGQM acceptance limit value.

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 to produce biodiesel, e.g., used cooking oils and fats as well as free 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. Thus, 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 for assessment instead of the values of DIN EN 14214 as the limit values to be complied with for the parameters sulphur content, CFPP and cloud point. 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

In the following section, the results of the sampling at the AGQM member companies in the three main campaigns are presented graphically. 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 by a red line. Under customs law, but also regarding the award of sanction points under the QM system, these acceptance limits are decisive. The acceptance limits can be calculated according to DIN EN ISO 4259 using the following formulas:

$$AL = L\,(min) - (0.59 \cdot R) \qquad bzw. \qquad AL = L\,(max) + (0.59 \cdot R)$$

With AL = acceptance limit; L = limit (from EN 14214); R = comparability (from standard method)

For example:

$$AL\,(ester\ content) = 96.5\%\,(m/m) - (0.59 \cdot 4.14\%\,(m/m)) \;=\; 94.0\%\,(m/m)$$

For some parameters, the 95 quantile is also given, which describes the value below or above which 95% of all results lie.

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 all three campaigns there was no undercutting of the standard limit. Compared to the previous year, the ester content is at a similar level in all three campaigns and there is hardly any difference between winter and summer goods regarding the ester content. The 95% quantile is 97.6% (m/m).

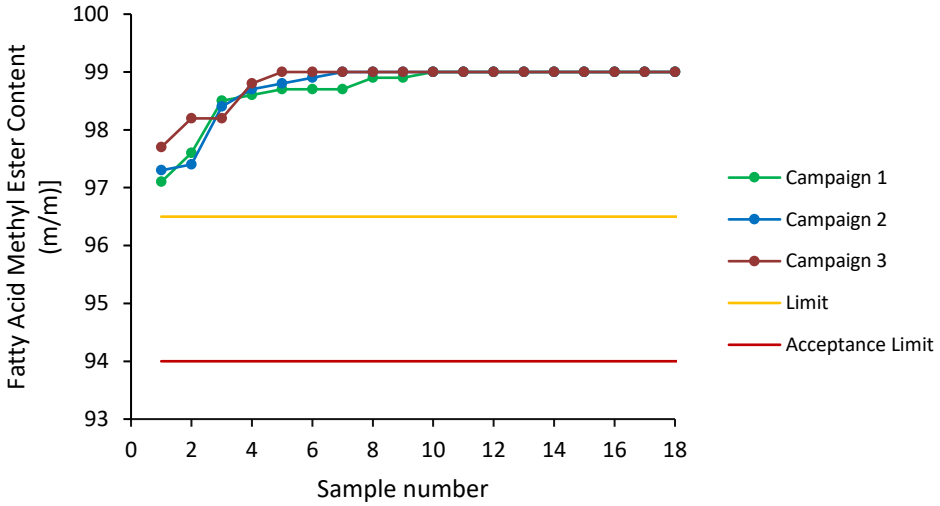

Figure 1: Fatty Acid Methyl Ester Content acc. to DIN EN 14103.

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. An increased methanol content, for example, lowers the density.

Figure 2 shows the density of the analysed samples. 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 indicates the main use of rapeseed oil as feedstock. Lower densities of approx. 875 kg/m³ are due to the use of other raw materials. Also, no differences can be found between the individual campaigns in terms of density.

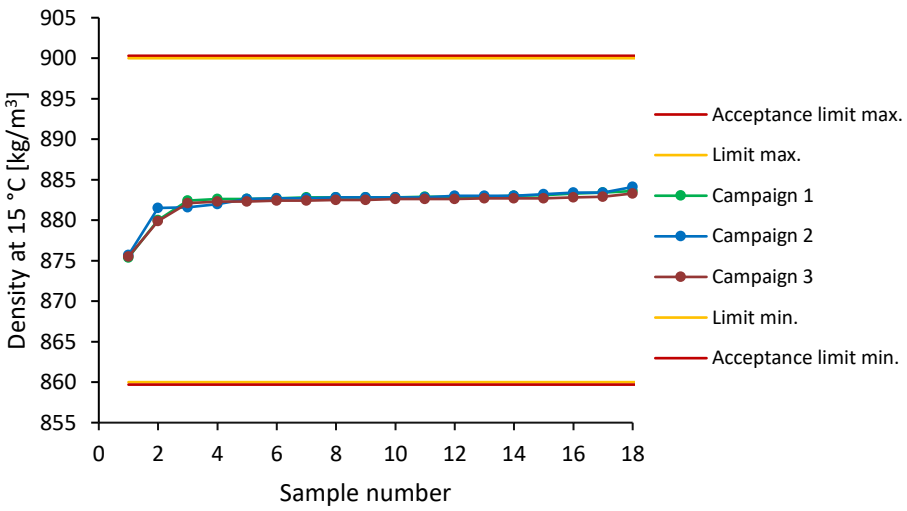

Figure 2: Density at 15 °C acc. to DIN EN ISO 12185.

4.3 Sulphur Content

Test method:	*DIN EN ISO 20846:2011*
Limit of DIN EN 14214:	*max. 10.0 mg/kg*
Acceptance limit:	*max. 11.3 mg/kg*
AGQM limit for blend components for biodiesel:	*max. 13.0 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 7.1 mg/kg.

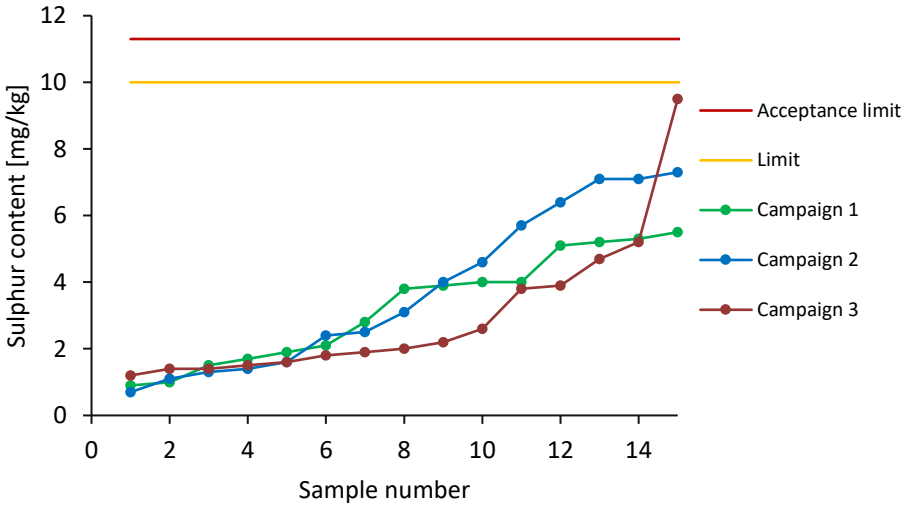

Figure 3: Sulphur content acc. to DIN EN ISO 20846.

Figure 4 shows the analytical results of the samples of those companies that have made use of the exemption for blending components for biodiesel. According to AGQM QM system section 2.1.1, different limit values apply to these samples (see Appendix, Table 3). In campaign 2 there was one limit and acceptance limit violation by a member with a sulphur content of 16.3 mg/kg, which according to the member was due to the use of typical raw materials with an increased sulphur content. One sanction point has been awarded to the member.

In order to take into account current market requirements and raw material offers, the AGQM limit value for the sulphur content for blend components for biodiesel has been raised to a maximum of 20 mg/kg in September 2021. The limit value has also been defined as a acceptance limit value. All samples of blend components for biodiesel in 2021 were below the new, specific limit value of 20 mg/kg. Most of the samples would comply with the DIN EN 14214 limit value of 10 mg/kg.

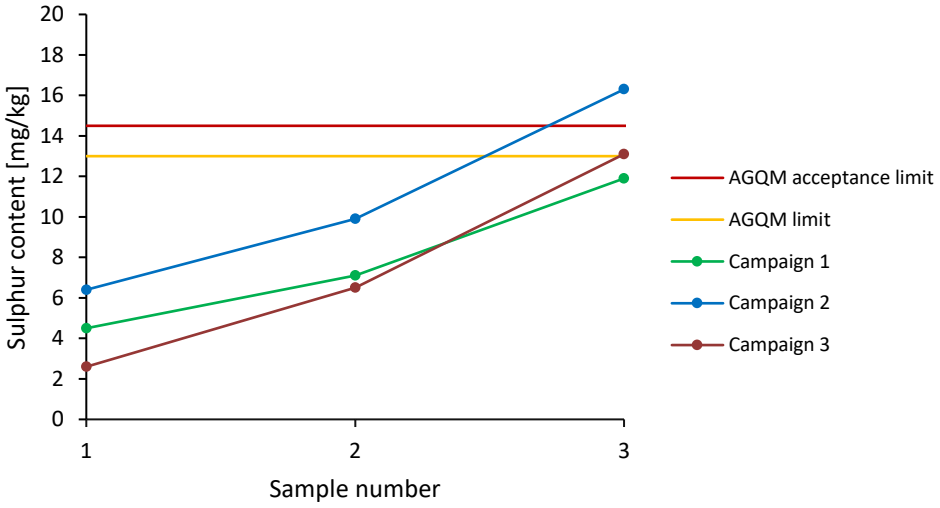

Figure 4: Sulphur content of blend components for biodiesel acc. to DIN EN ISO 20846.

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. To consider current market conditions and requirements from fuel and automotive industries, the AGQM limit value for the water content has been raised to max. 270 mg/kg for producers and max. 320 mg/kg for warehouse operators in September 2021.

Figure 5 shows the values for the water content. All samples tested are well below the standard limit and there are only two exceedances of the AGQM limit.

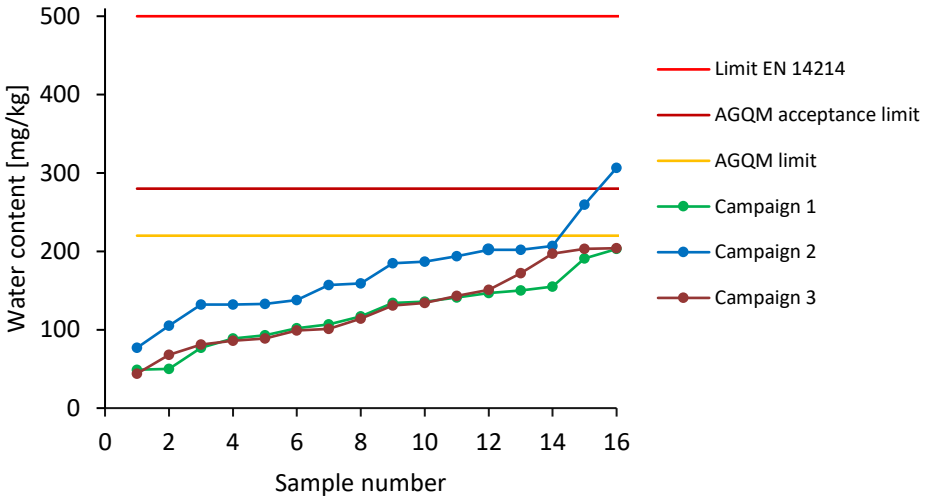

Figure 5: Water content acc. DIN EN ISO 12937.

In campaign 2, the AGQM limit is exceeded by two samples with a content of 260 mg/kg and a content of 307 mg/kg. In the second sample, the AGQM acceptance limit of 280 mg/kg is exceeded. A sanction point was awarded to the company concerned for the violation of the limit value.

To preserve the anonymity of the only warehouse operator sampled in 2021, its results 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 an acceptance limit to account for this issue and the relatively poor precision of the method.

Figure 6 shows that all samples comply with the stricter AGQM limit for total contamination and the 95% quantile is only 13 mg/kg.

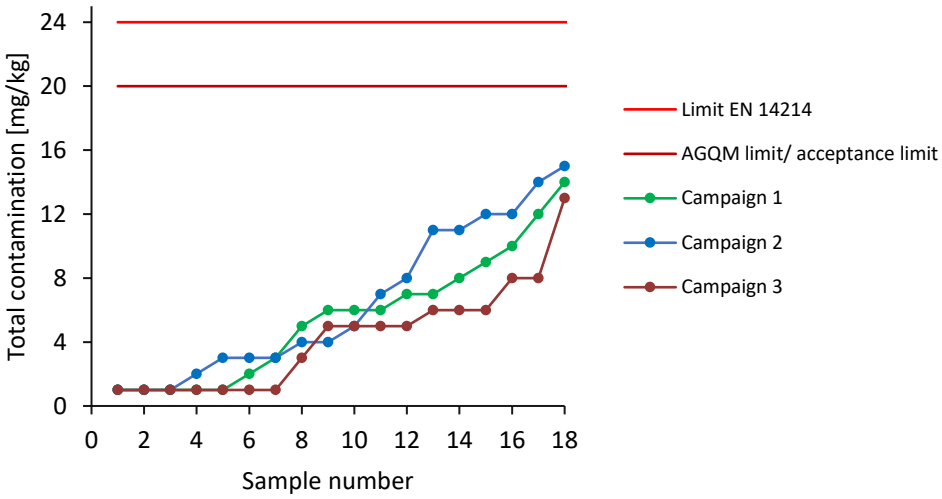

Figure 6: Total contamination acc. to DIN EN 12662.

4.6 Oxidation Stability

Test method:	*DIN EN 14112:2016*
Limit of DIN EN 14214:	*min. 8.0 h*
Acceptance limit:	*min. 6.6 h*

Oxidation stability is a measure of the resistance of a fuel to oxidative processes. Vegetable oils and biodiesel produced from them contain natural antioxidants (e.g., tocopherols) that slow down the ageing process. Synthetic stabilizers are also used. Once a year, AGQM tests products that can be used to increase the oxidation stability of biodiesel at the request of interested additive manufacturers. Additives that pass the test are published in the so-called "No-Harm List" on the AGQM website.

The test method for the oxidation stability of biodiesel is the so-called Rancimat test. At 110 °C, a constant flow of air is passed through the sample to be tested. After the oxidation reserve (natural reserve and additives) of the sample is degraded, volatile oxidation products are formed, which are conducted together with the air into the test liquid of the measuring cell and increase the conductivity there. The time until the detection of these oxidation products is called induction time or oxidation stability. DIN EN 14214 requires a minimum oxidation stability of 8.0 hours.

Figure 7 shows the oxidation stabilities of the samples tested. In the three campaigns that took place in 2021, the limit value was complied with by all samples.

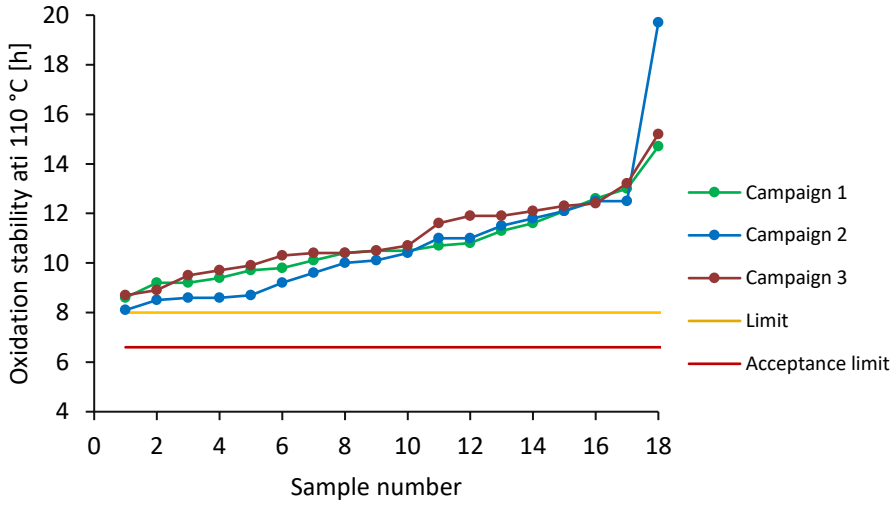

Figure 7: Oxidation Stability acc. to DIN EN 14112.

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 exceeded 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. In the following additional campaign, no further anomalies were found.

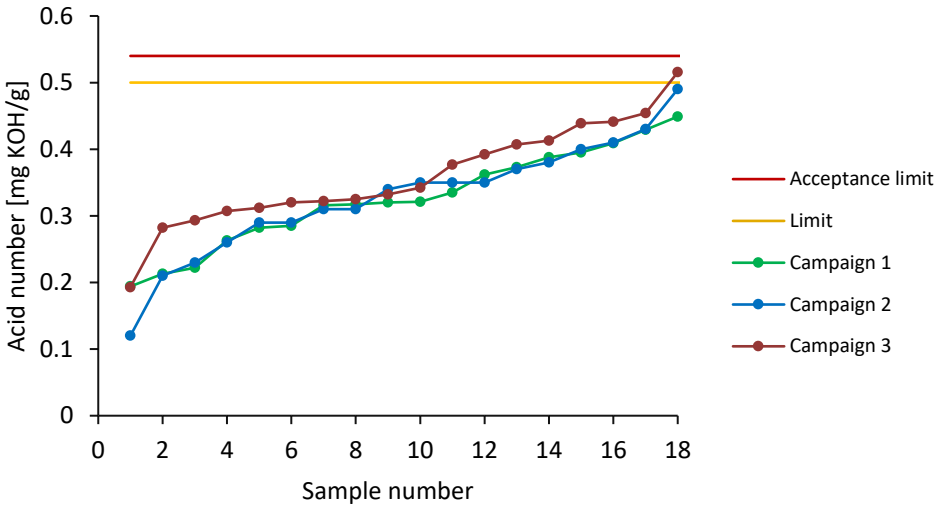

Figure 8: Acid Number acc. to DIN EN 14104.

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 value. All samples analysed are below the standard limit and show an almost identical course within the three campaigns carried out, which overall indicates a constant raw material use over the year.

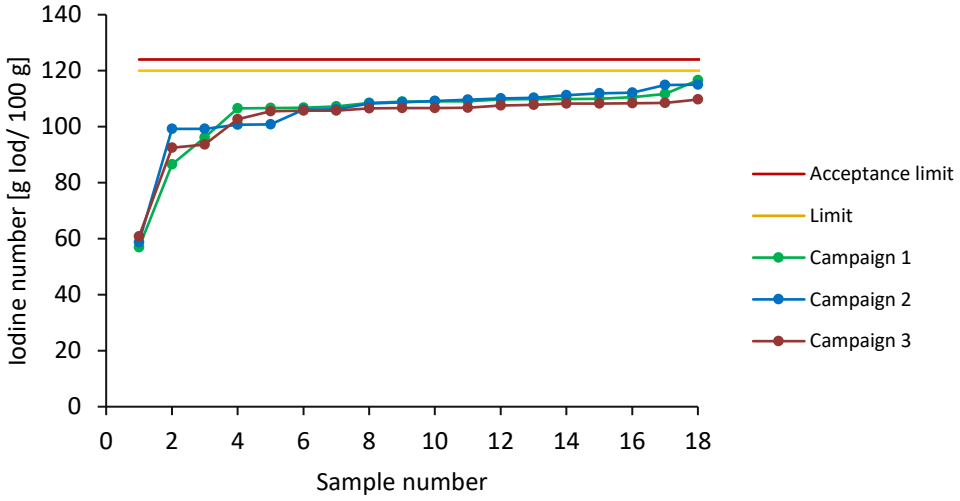

Figure 9: Iodine number acc. to DIN EN 16300.

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 of the analyses for mono-, di- and triglycerides. In campaign 3, the content of diglycerides exceeded the standard limit by 0.02% (m/m) of one member, within the corresponding acceptance limit. All other samples showed no abnormalities about mono-, di- and

triglycerides. For the monoglyceride content, some samples even show values < 0.01% (m/m), which suggests that the production process includes a distillation step.

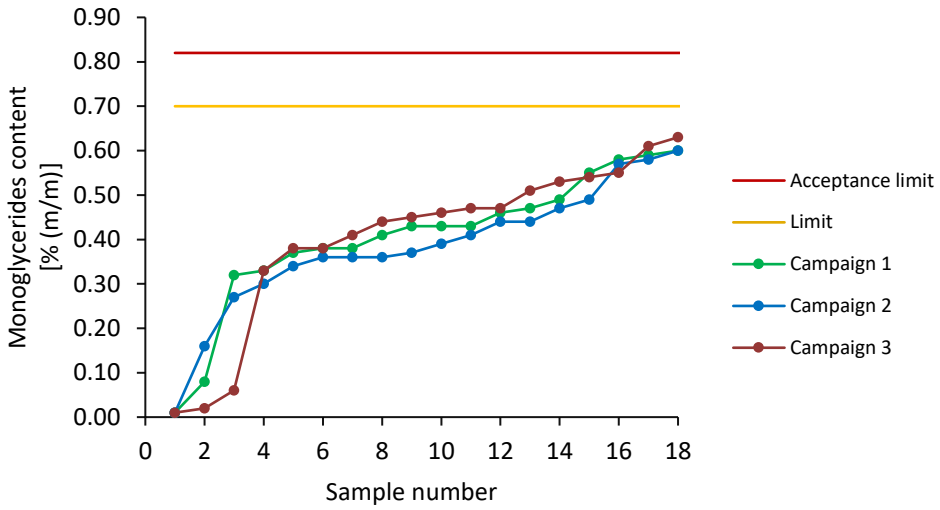

Figure 10: Monoglycerides acc. to DIN EN 14105.

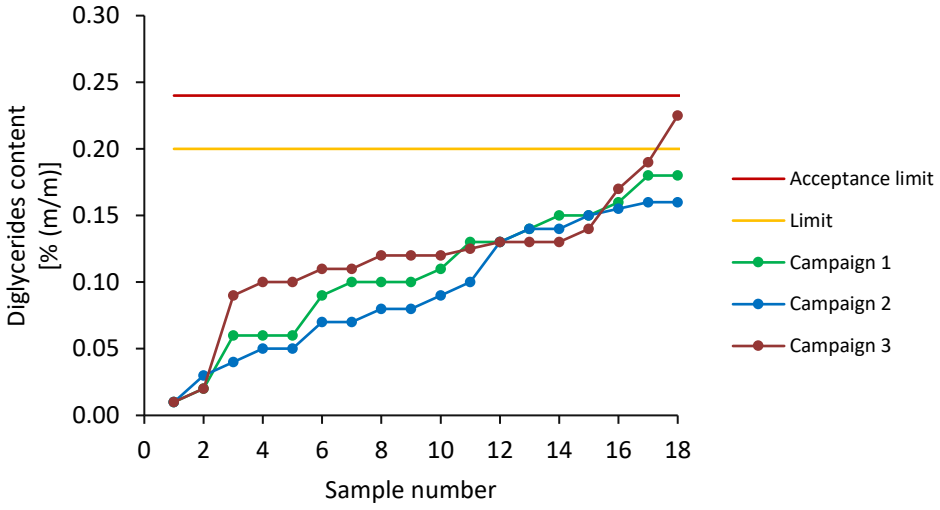

Figure 11: Diglycerides acc. to DIN EN 14105.

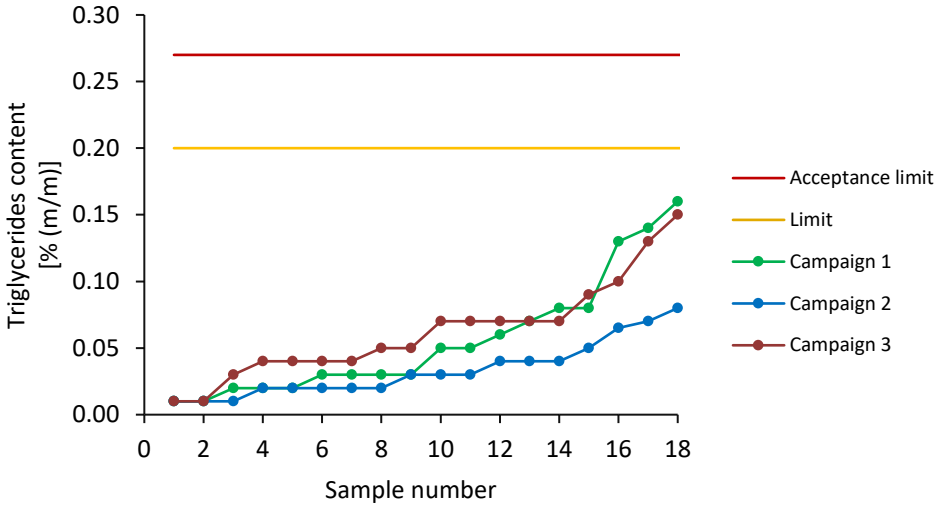

Figure 12: Triglycerides acc. to DIN EN 14105.

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

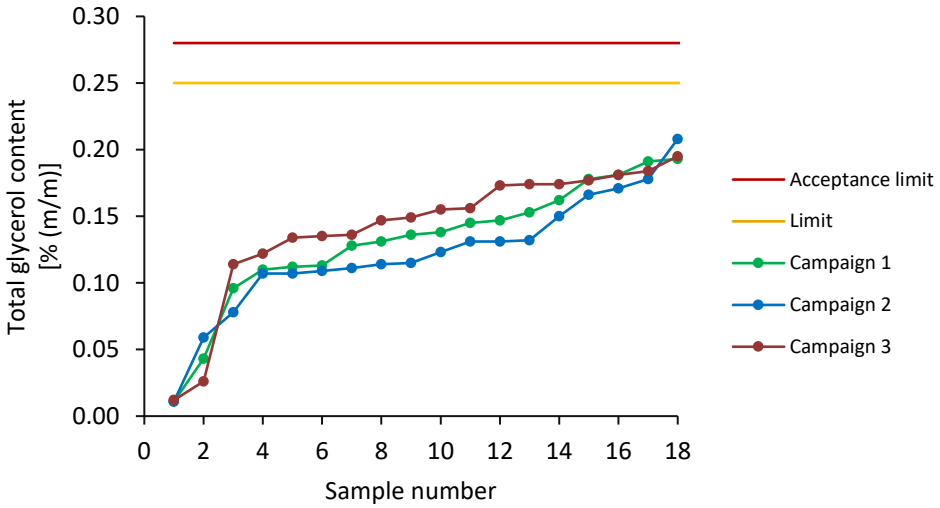

Figure 13: Total glycerol acc. to DIN EN 14105.

Figure 14 shows the free glycerol content. One sample in campaign 2 exceeded the standard and acceptance limit with a free glycerol content of 0.032% (m/m). A sanction point had to be awarded to the member. In the additional sampling that subsequently took place, no abnormalities were found for the same member. In the third campaign, the sample of that member again showed an exceedance of the limit value within the acceptance limit value with a content of 0.021% (m/m) of free glycerol. The measures taken to eliminate the anomalies were implemented, so that no further exceedances occurred in the subsequent samples.

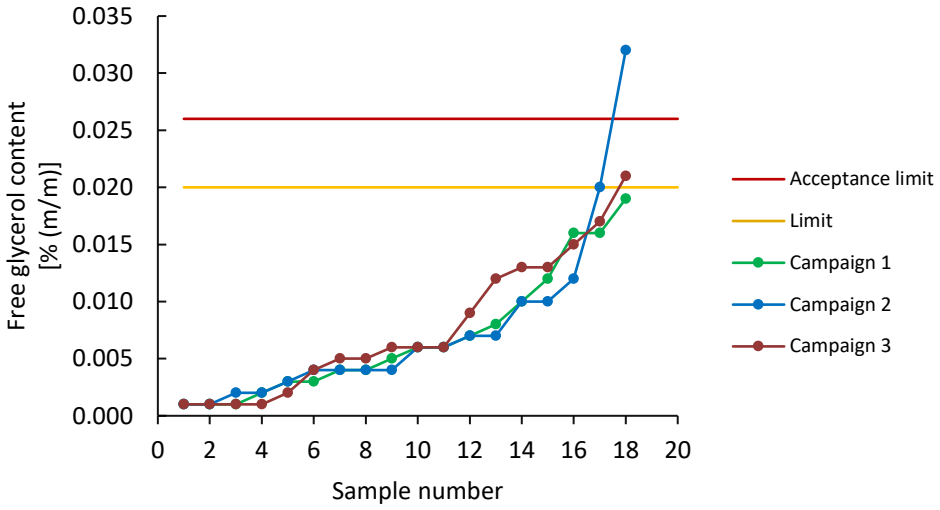

Figure 14: Free glycerol acc. to DIN EN 14105.

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)*

In biodiesel production, sodium and potassium methanolates are usually used as catalysts. If residues of these are not completely removed in the wash, residues of the cations in the biodiesel are usually present in the form of soaps. Soaps can lead to filter blockages, deposits in injection pumps and nozzle needles and 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 six samples, all below 2 mg/kg. The content of the alkaline earth metals magnesium and calcium (Figure 16) is mostly below the determination limit of 0.1 mg/kg. Only one sample in campaign 1 has a slightly higher alkaline earth metal content of 3.5 mg/kg, which is also well below the standard limit of 5 mg/kg.

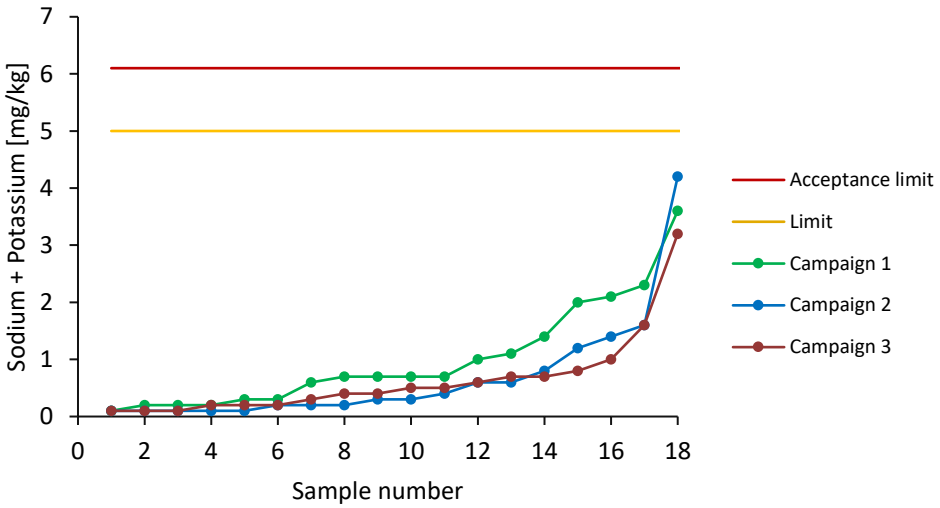

Figure 15: Sum of alkali metals Sodium and Potassium acc. to DIN EN 14538.

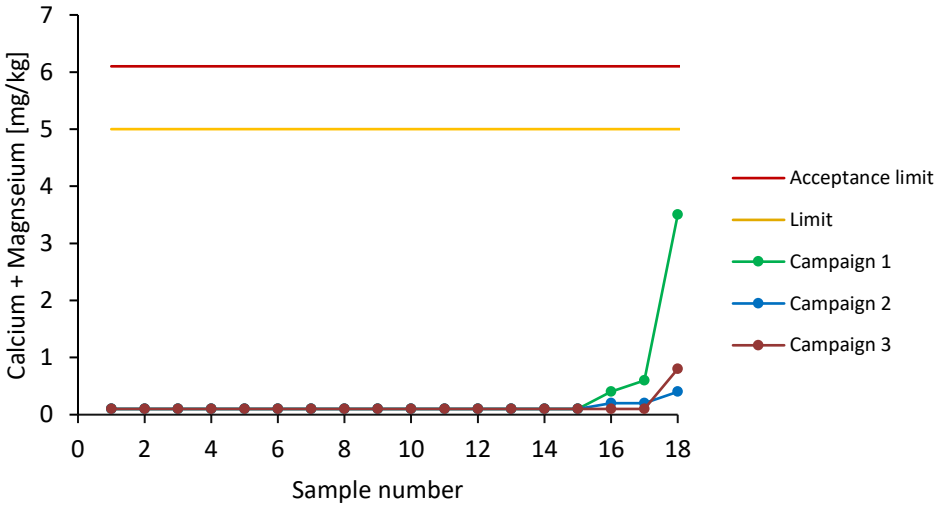

Figure 16: Sum of alkaline earth metals Calcium and Magnesium acc. to DIN EN 14538.

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 1, the contents are significantly lower than 1 mg/kg and are thus well below the standard limit of 4.0 mg/kg. The 95% quantile of the values is 0.8 mg/kg.

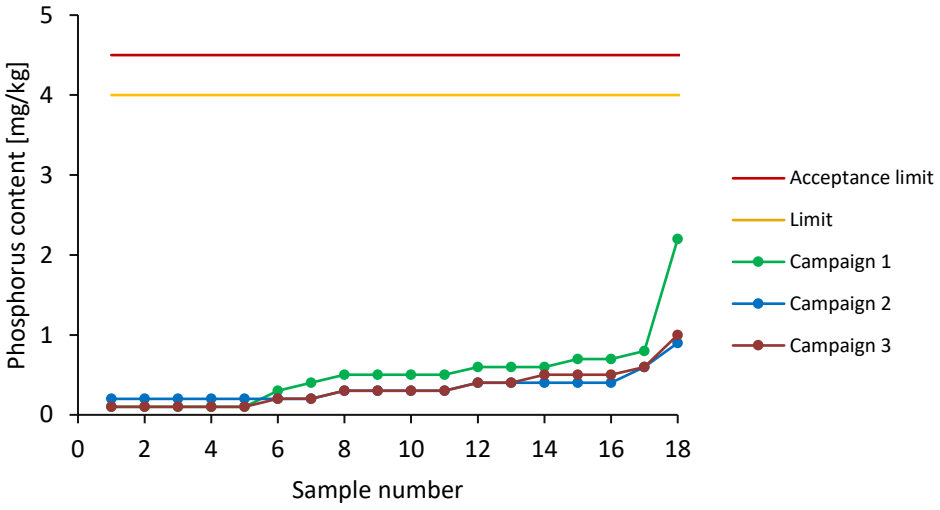

Figure 17: Phosphorus content acc. to DIN EN 14107.

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 can be seen in Figure 18, all samples analysed have a linolenic acid methyl ester content 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). In two samples, the content of linolenic acid methyl ester is even below 2% (m/m) throughout the year.

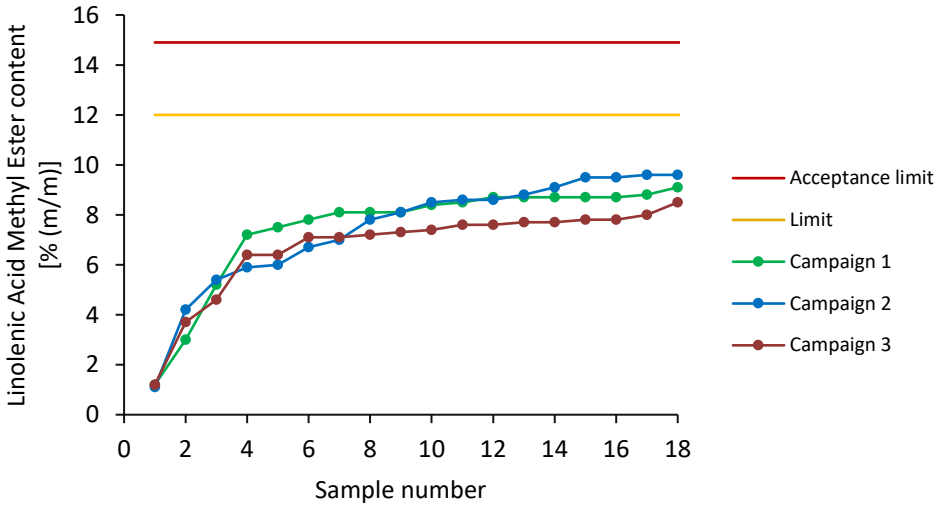

Figure 18: Content of Linolenic Acid Methyl Ester acc. to DIN EN 14103.

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*	*+1.5 °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. In Germany, the additives are then usually used in the refineries of the mineral oil companies for the blends of diesel fuel and biodiesel. Once a year, AGQM tests flow improvers that can be used to lower the CFPP of biodiesel at the request of interested additive manufacturers. Additives that pass the test are published in the so-called "No-Harm List" on the AGQM website.

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.

All samples tested comply with the specific, seasonal limit values.

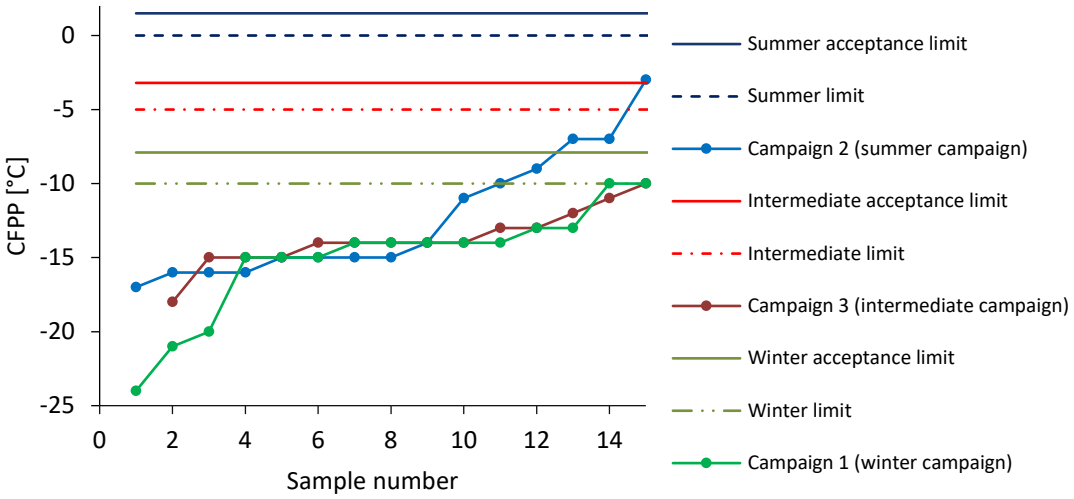

Figure 19: CFPP acc. to DIN EN 116.

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.

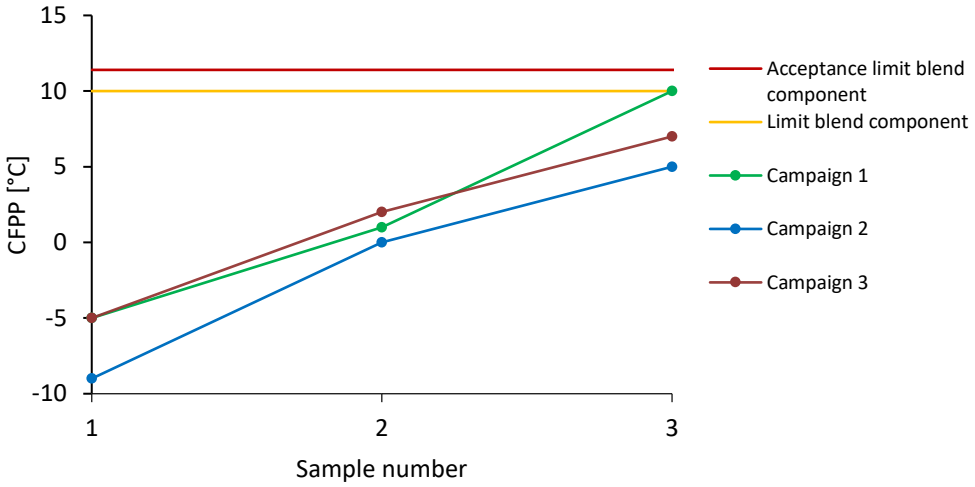

Figure 20: CFPP of blend components for biodiesel acc. to DIN EN 116.

4.14 Cloud point (CP)

Test method: *DIN EN 23015:1994*

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 when it cools under specified test conditions.

All samples investigated comply with the specific, seasonal limit values.

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. All samples investigated comply with the specific, seasonal limit values.

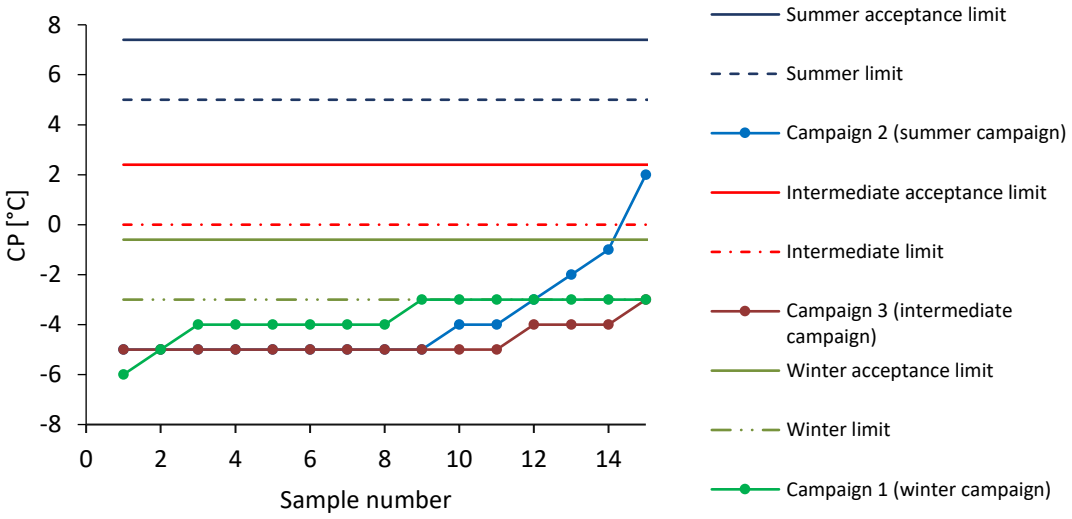

Figure 21: Cloud point acc. to DIN EN 23015.

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.

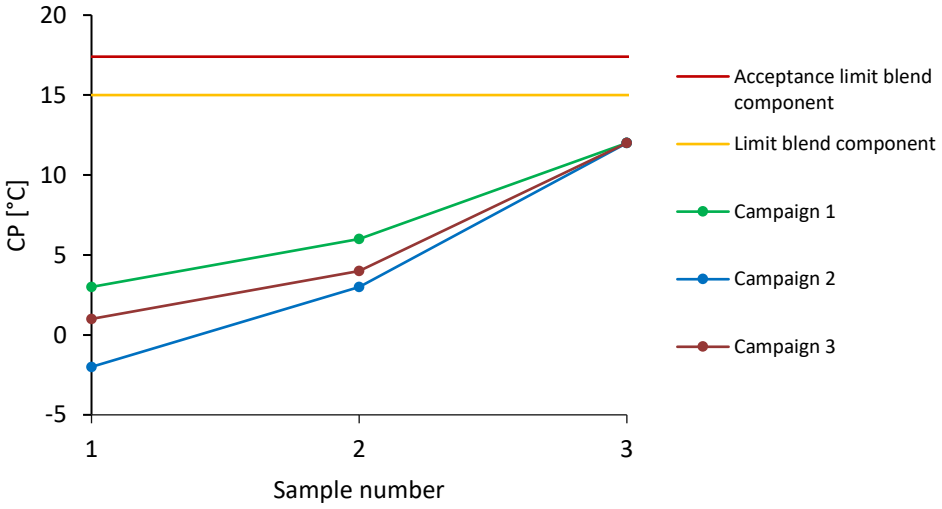

Figure 22: Cloud point of blend components for biodiesel acc. to DIN EN 23015.

Additional Campaigns

Members who are found to be abnormal in a main campaign (violation of limit value or acceptance limit value) must subsequently participate in an additional campaign, which is also unannounced. In 2021, a total of three additional campaigns with nine samples were carried out.

In the additional campaign 1, one company was sampled and a violation of the limit value for the parameter water content, which was already conspicuous in the first campaign, was again detected within the corresponding acceptance limit value. The cause of the anomalies was found and remedied, so that no further limit value violations were found for the member in the subsequent campaigns.

A total of five companies had to participate in the additional campaign 2. Two limit violations outside the AGQM acceptance limit were found for the parameters water content and sulphur content for blend components for biodiesel. A sanction point had to be issued to each of the two members. No further limit value violations were found for the other three companies.

In the final additional campaign 3, which took place in the year, three companies were sampled. One more violation of the corresponding AGQM limit value outside the acceptance limit value for the parameter total contamination was detected by one company. A sanction point was awarded, and the member declared that he had already detected the problem himself and would monitor this parameter more closely in future.

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 2021 are presented.

A comparison of the sample numbers for 2020 and 2021 (Figure 23) shows that the total number of samples analysed in 2021 (63) was significantly lower than the total number of samples analysed in 2020 (71), which is due to fewer limit violations in the main campaigns and thus lower participation of companies in the additional campaigns. Also noteworthy is the more than one-third reduction in limit violations within the acceptance limit compared to 2020. Although only one limit value violation outside an acceptance limit value of DIN EN 14214 was detected, the violations of the specific AGQM acceptance limit values have doubled compared to the year 2020. This shows that the parameters for which stricter limit values are required are precisely those that can be classified as critical and are therefore essential to be able to guarantee a high quality of the end product biodiesel with more than compliance with the standard limit values.

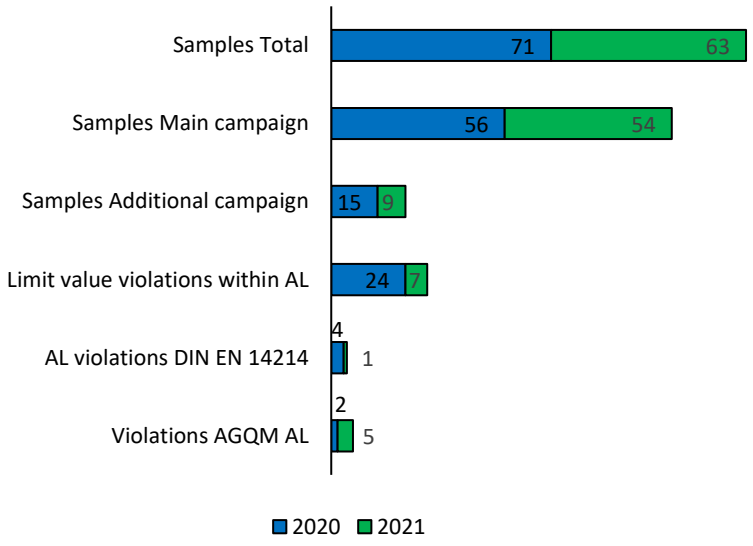

Figure 23: Comparison of the number of samples for main and additional campaigns as well as the number of limit value violations within and outside the acceptance limit values (AL) for the years 2020 and 2021.

The companies where abnormalities were detected during the 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 optimise the production process to avoid the occurrence of further limit value violations in the future.

The result shows that the unannounced sampling is an effective means of detecting anomalies and taking countermeasures as quickly as possible. In this way, the sampling intensively supports the member companies' 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 rectifying problems and promotes access to and the exchange of know-how with various expert committees. The FAME round robin test organised by AGQM in cooperation with DIN FAM, which is unique worldwide, promotes continuous improvement and further development of the operating and analytical 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 biodiesel to the European fuel market. Labelling as AGQM goods thus represents a reliable mark of quality for customers and traders 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	-	28
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
Content of Earth Alkali Metals (Ca+Mg)			mg/kg	-	5.0	-	6.1
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	1994	°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	-	280
Water Content (for Traders)	DIN EN ISO 12937	2002	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 19.10. to 28/29.02	-10	-	-7.9

[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.

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	-	14.5
Cloud point	DIN EN 23015	1998	°C	-	15	-	17.4
CFPP	DIN EN 116	2015	°C	-	10	-	11.4