

Biodiesel Quality in Germany

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

2022



Project Management and Report:

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1 Introduction

In order to achieve the EU's goal of becoming climate neutral by 2050 and to limit the global temperature increase to a maximum of 1.5 degrees Celsius, an immediate switch to climate-friendly fuels and an exhaustion of all available options is imperative. One of these options is the use of biofuels to decarbonise the transport sector. According to the BLE report 2021, the total greenhouse gas emission saving of all biofuels was around 84%. The largest share is attributable to the use of biodiesel (FAME).

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 mainly as an admixture to diesel fuel to reduce CO₂ 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), founded in 1999 by German biodiesel producers and traders, promotes and monitors the biodiesel quality in Germany and Austria.

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 2022.

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 2022, samples were taken at 16 production sites and three warehouse operators. 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:	17. January to 28. January	Winter grade
Campaign 2:	09. May to 20. May	Summer grade
Campaign 3:	04. October to 14. October	Intermediate grade

A total of 57 samples were taken and analysed in the main campaigns and 9 samples in the necessary additional campaigns. For each sampling, one analytical sample, one sample as a potential arbitration sample and one sample that remains with the producer or warehouse operator as a reserve sample are taken.

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.

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. As a rule, biodiesel from these alternative raw materials cannot be used as a pure fuel, but only 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. By blending such components with other goods, an overall standard-compliant biodiesel can be produced. 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 (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.

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:

$$AL = L (min) - (0.59 \cdot R) \quad \text{bzw.} \quad AL = L (max) + (0.59 \cdot R)$$

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 (excluding products with limit value violations and products for which special limit values apply).

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 three of the 57 samples were below the standard limit. In campaign 2, one sample fell below the standard acceptance limit with an ester content of 93.6 % (m/m). The company itself detected the violation and the biodiesel was consequently not placed on the market. A sanction point had to be awarded to the company in question. Eight samples per campaign each had an ester content of 99.0 % (m/m). The 95 % quantile is 97.0 % (m/m).

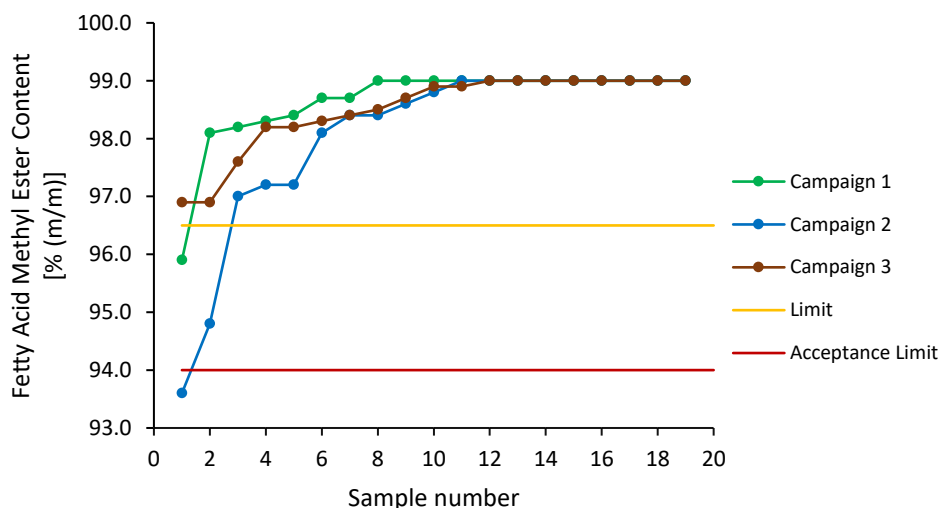


Figure 1: Fatty Acid Methyl Ester Content acc. to DIN EN 14103.

4.2 Density at 15 °C

Test method:	DIN EN ISO 12185:1996
Limit of DIN EN 14214:	min. 860 kg/m ³ and max. 900 kg/m ³
Acceptance limit:	min. 859.7 kg/m ³ and max. 900.3 kg/m ³

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. It can also be stated that the densities of the samples are not subject to seasonal fluctuations and are almost constant within a year.

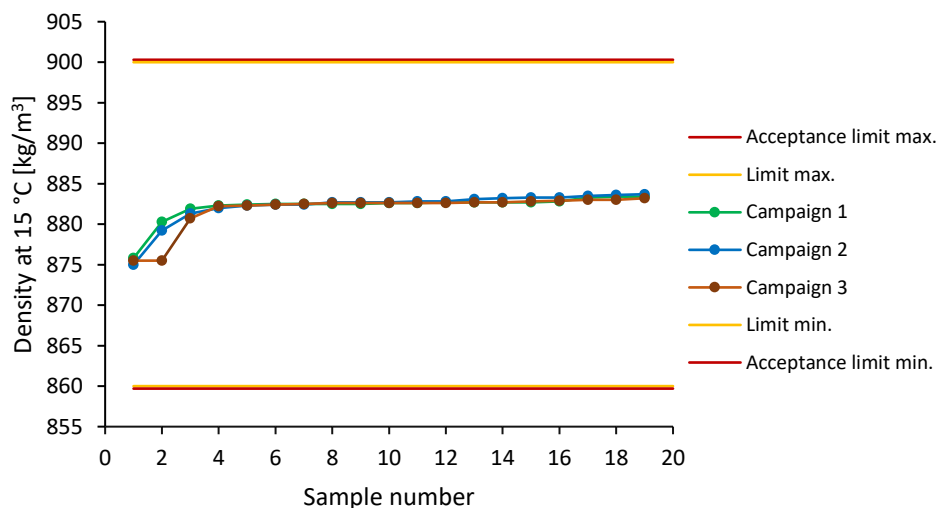


Figure 2: Density at 15 °C acc. to DIN EN ISO 12185.

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. 20.0 mg/kg

The AGQM limit value for sulphur content is equal to the AGQM acceptance limit value.

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. A total of ten of the samples even had a sulphur content below 4 mg/kg in all three campaigns. The 95% quantile is 7.5 mg/kg.

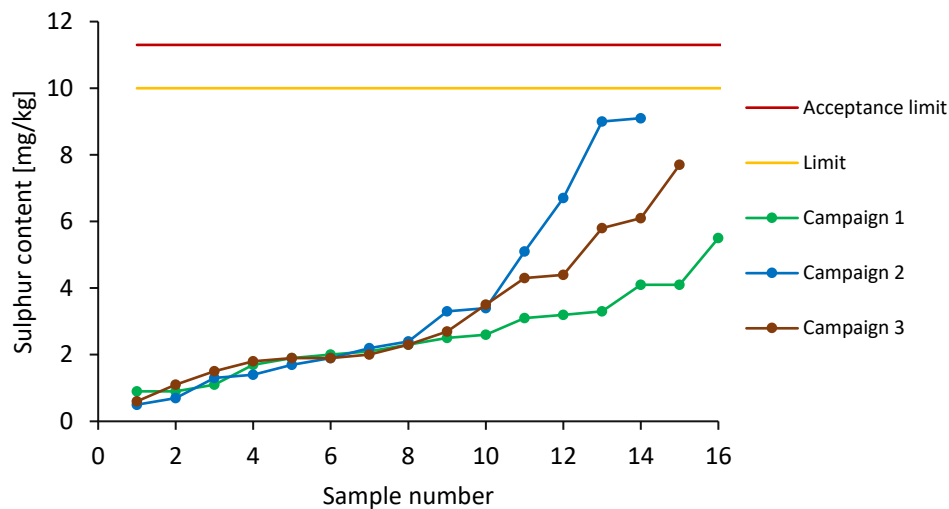


Figure 3: Sulphur content acc. to DIN EN ISO 20846.

Figure 4 shows the analytical results of the samples of those companies that have made use of the exemption for blending components for biodiesel.

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. The limit value has also been defined as an acceptance limit value. All samples of blend components for biodiesel were below the limit value of 20 mg/kg. Most of the samples would comply with the DIN EN 14214 limit value of 10 mg/kg.

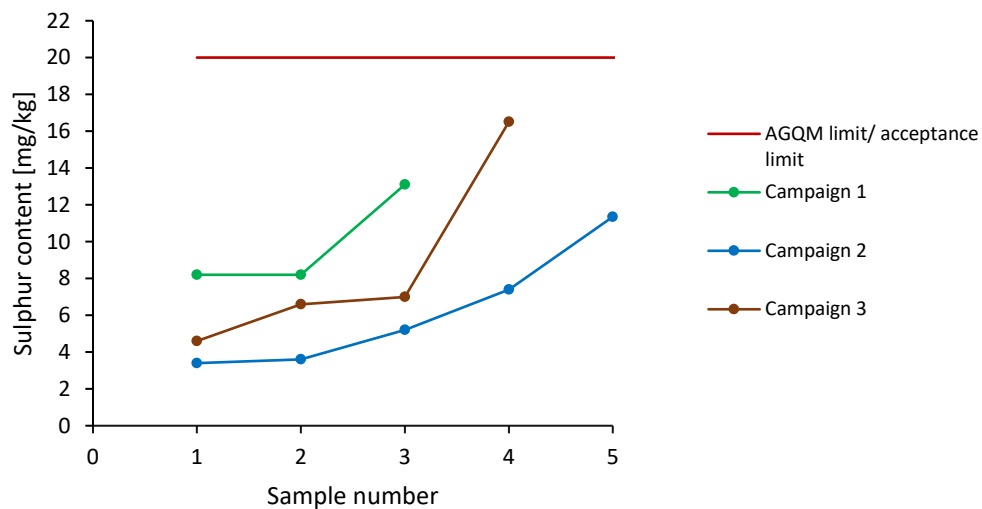


Figure 4: Sulphur content of blend components for biodiesel acc. to DIN EN ISO 20846.

4.4 Water Content

Test method:	DIN EN ISO 12937:2002
Limit of DIN EN 14214:	max. 0.050 % (m/m)
AGQM limit (producers):	max. 0.027 % (m/m)
Acceptance limit (producers):	max. 0.034 % (m/m)
AGQM limit (warehouse operators):	max. 0.032 % (m/m)
Acceptance limit (warehouse operators):	max. 0.039 % (m/m)

Biodiesel can physically dissolve up to 0.0150 % (m/m) because it has a very high polarity. 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 only absorb very small amounts of water, so dissolved water could precipitate when mixed with biodiesel that significantly exceeds the water limit. Free water can block filters or pipes by freezing, cause corrosion or promote microbial growth. DIN EN 14214 requires a maximum water content of 0.050 % (m/m). Due to the problems described above, AGQM has set stricter requirements with a maximum water content of 0.027 % (m/m) for producers and 0.039 % (m/m) for warehouse operators.

Figure 5 shows the values for the water content. All samples tested are well below the standard limit. An exceedance of the AGQM limit value occurred in four cases.

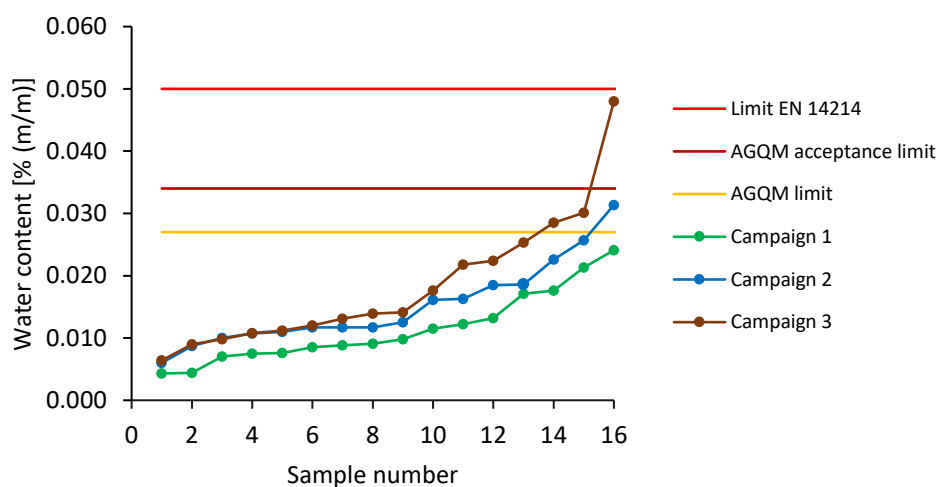


Figure 5: Water content acc. DIN EN ISO 12937.

In campaign 2, the AGQM limit is exceeded by one sample with a content of 0.0314 % (m/m). In the subsequent intermediate campaign 3, the AGQM limit was exceeded by a total of three samples, of which the water content of one sample was 0.048 % (m/m) above the AGQM acceptance limit. For the violation of the acceptance limit, one sanction point was given to the company concerned, which stated that the reason for the violation was an excessive storage time of the product.

Figure 6 plots the sample values for the water content of the warehouse operators. All samples comply with the specific AGQM limit of 0.032 % (m/m).

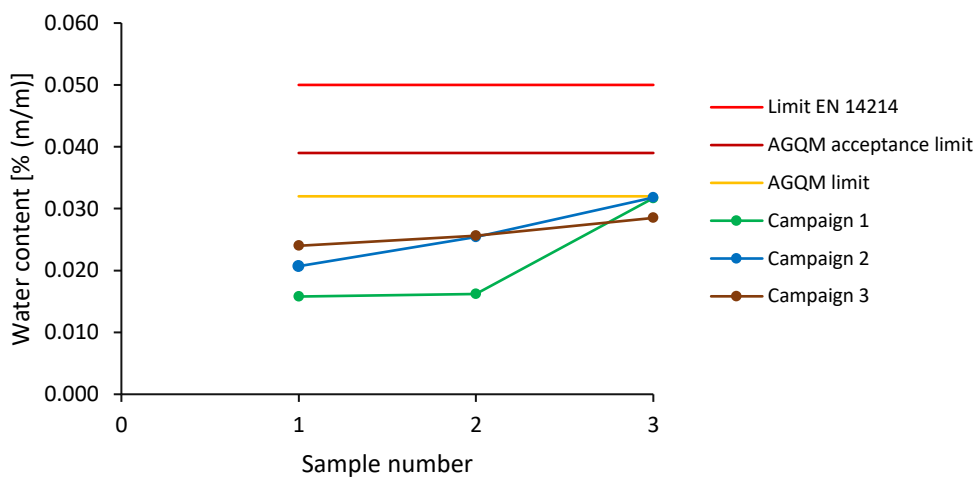


Figure 6: Water content according to DIN EN ISO 12937 of the warehouse operators.

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*

The AGQM limit value for total contamination is equal to the AGQM acceptance limit value.

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 7 shows that, except for one sample, all other samples complied with the stricter AGQM limit for total contamination and the 95% quantile was 14 mg/kg.

The member whose sample in campaign 1 had a total contamination level of 21 mg/kg, exceeding AGQM's own acceptance limit, received one sanction point.

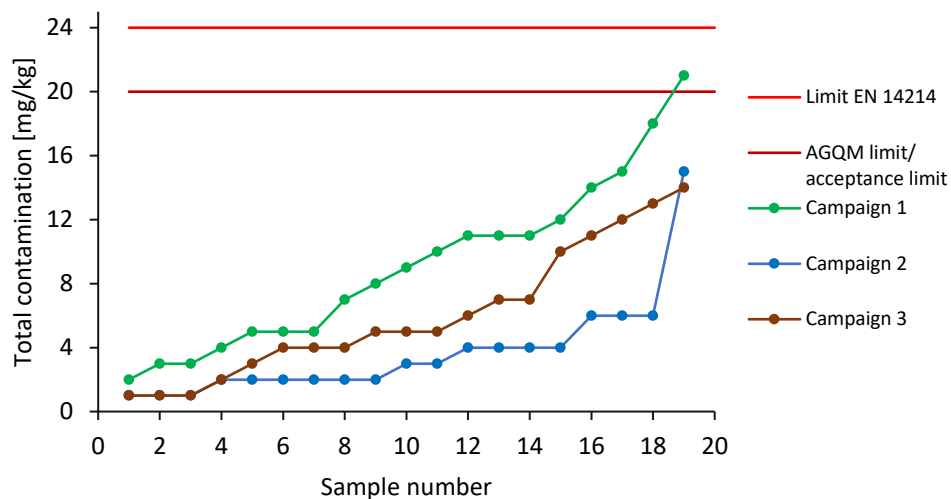


Figure 7: Total contamination acc. to DIN EN 12662.

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 [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 8 shows the oxidation stabilities of the samples tested. In campaign 2, there were underruns of the standard limit value within the acceptance limit value of two samples with oxidation stabilities of 7.7 h and 7.0 h.

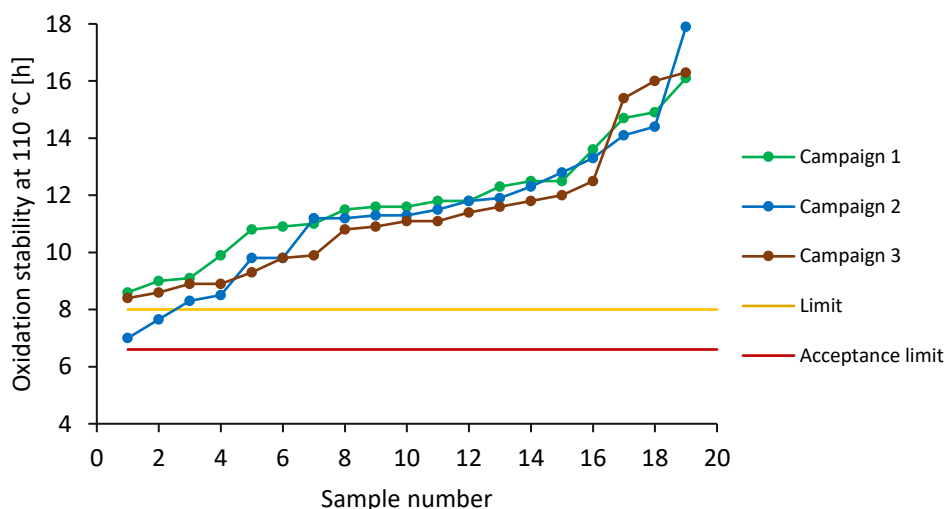


Figure 8: Oxidation Stability acc. to DIN EN 14112.

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 9 shows the measured values for the acid value. All samples comply with the standard limit of 0.50 mg KOH/g.

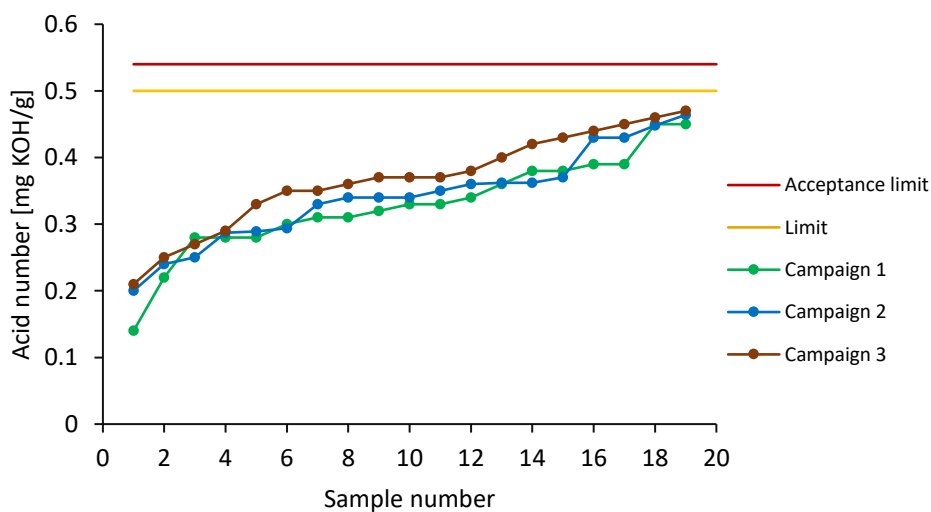


Figure 9: Acid Number acc. to DIN EN 14104.

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 10 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.

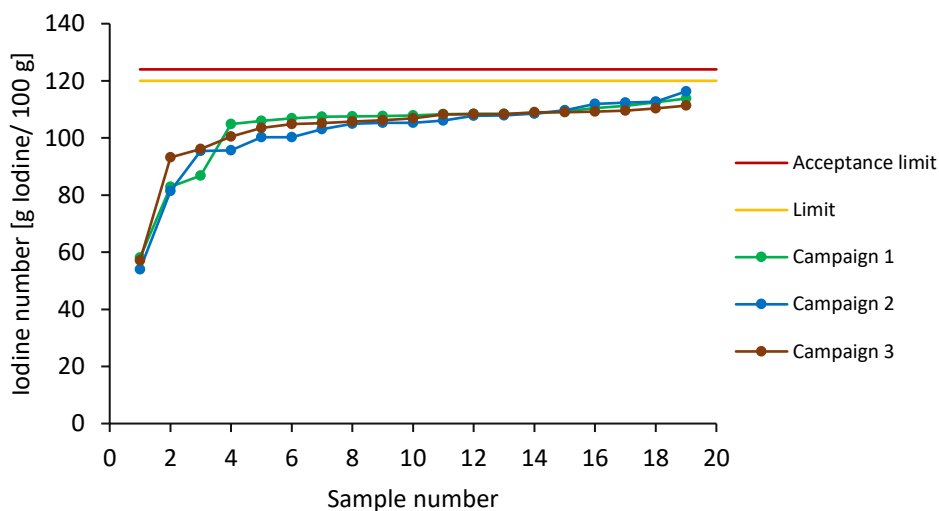


Figure 10: Iodine number acc. to DIN EN 16300.

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 11 to Figure 13 show the results of the analyses for mono-, di- and triglycerides. All samples comply with the corresponding DIN limits of 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.

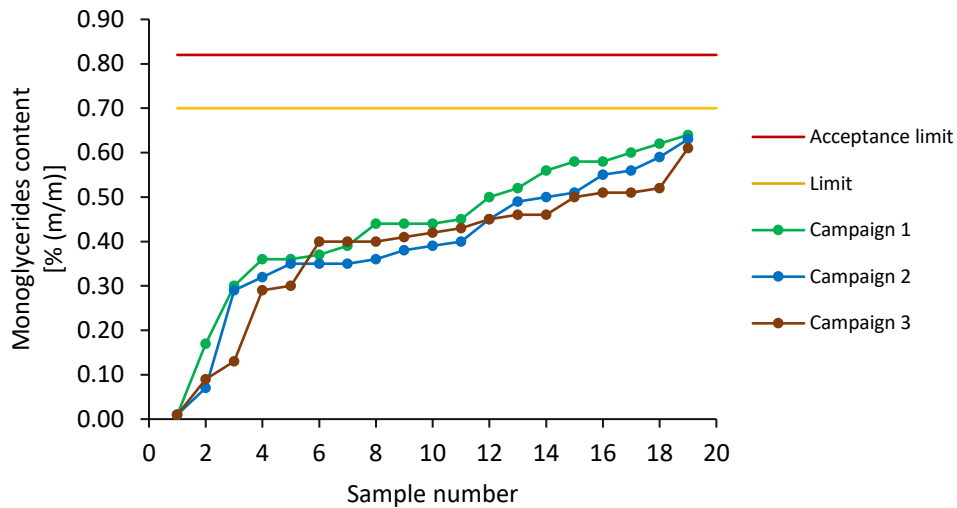


Figure 11: Monoglycerides acc. to DIN EN 14105.

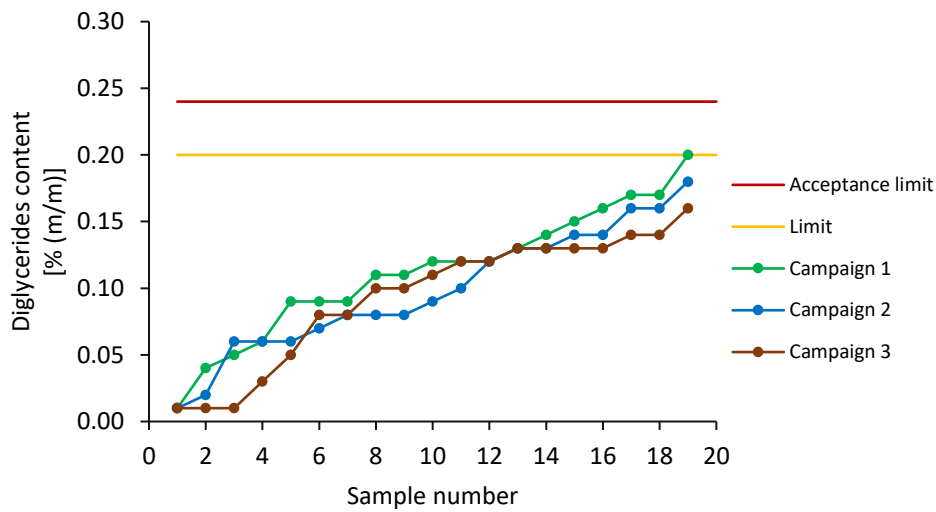


Figure 12: Diglycerides acc. to DIN EN 14105.

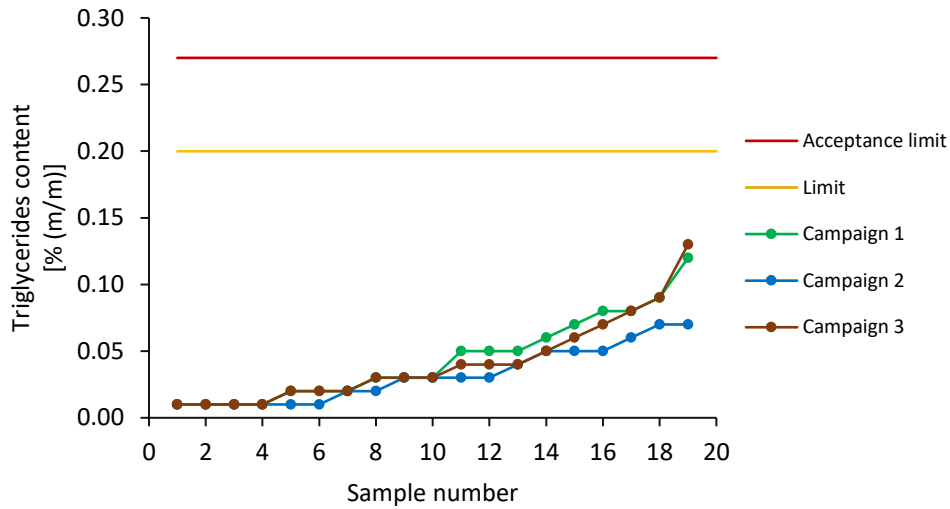


Figure 13: Triglycerides acc. to DIN EN 14105.

The total glycerol content is shown in Figure 14. All samples analysed comply with the standard limit of 0.25% (m/m).

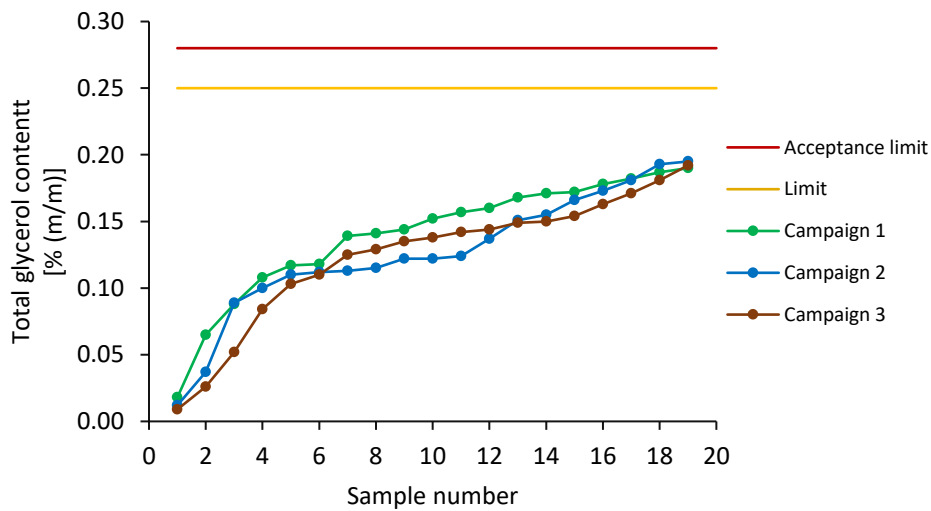


Figure 14: Total glycerol acc. to DIN EN 14105.

Figure 15 shows the free glycerol content. One sample in campaign 2 exceeded the standard limit with a content of 0.024 % (m/m) of free glycerol. All other samples showed no abnormalities.

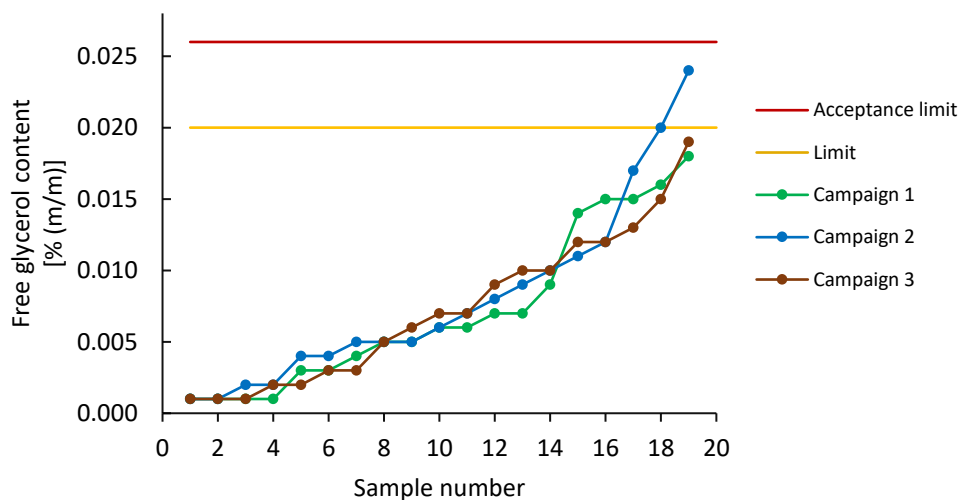


Figure 15: Free glycerol acc. to DIN EN 14105.

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 16 and Figure 17 clearly show that biodiesel producers attach great importance to low levels of alkaline and alkaline earth metals. One sample in campaign 2 was detected to exceed the standard

limit with a content of 5.4 mg/kg of sodium and potassium. The 95% quantile for the sodium and potassium content is 1.7 mg/kg.

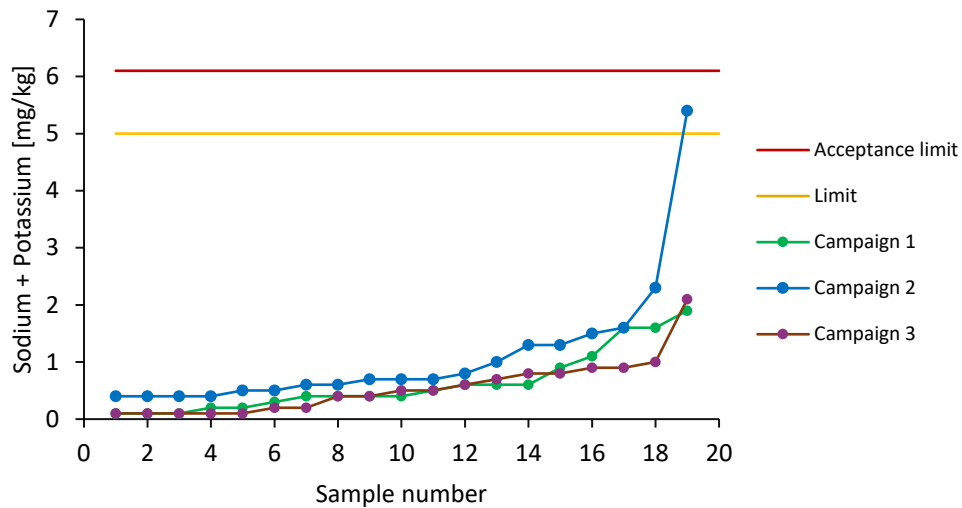


Figure 16: Sum of alkali metals Sodium and Potassium acc. to DIN EN 14538.

The content of the alkaline earth metals magnesium and calcium (Figure 17) 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 2.0 mg/kg. The 95% quantile is only 0.1 mg/kg.

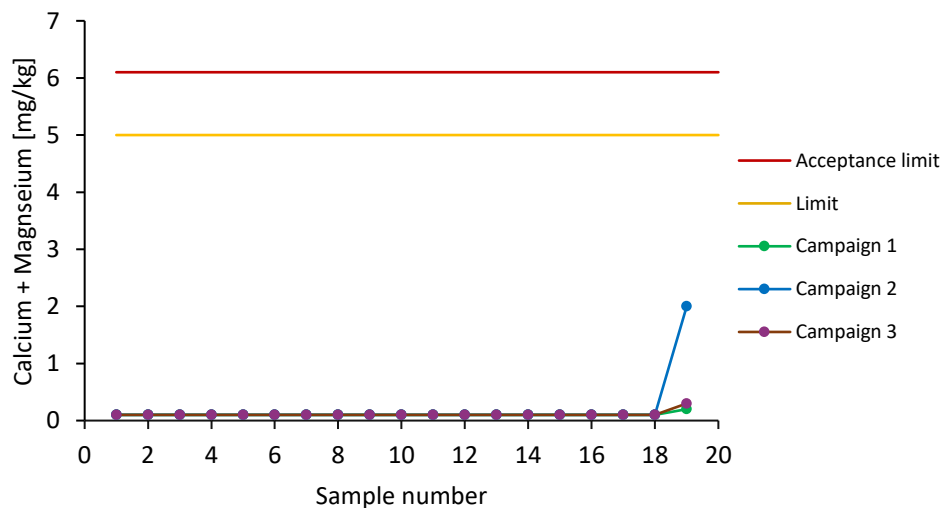


Figure 17: Sum of alkaline earth metals Calcium and Magnesium acc. to DIN EN 14538.

4.11 Phosphorous Content

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

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 phosphorus 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. Work is currently underway to include the parameter in EN 14538.

Figure 18 shows the values for the phosphorus content. All samples are well below the standard limit of 4.0 mg/kg. The 95% quantile of the values is 0.8 mg/kg.

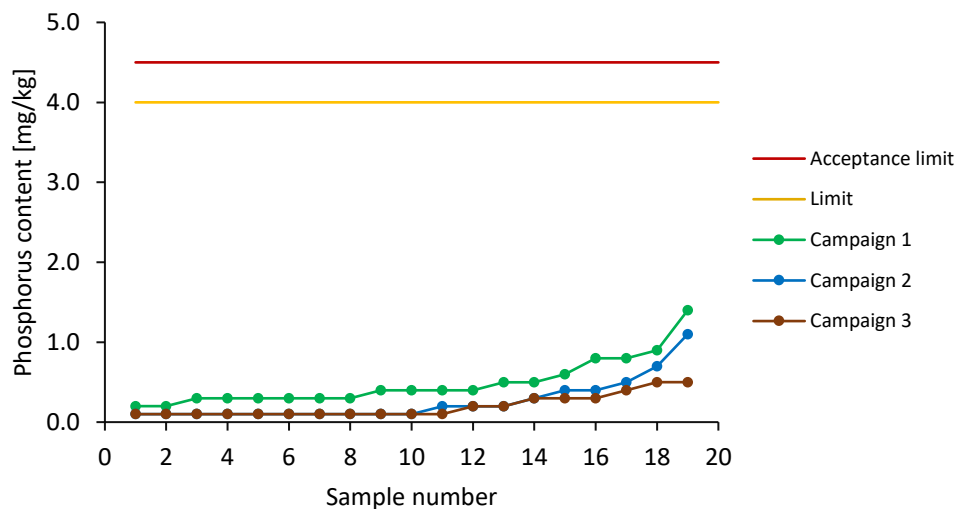


Figure 18: Phosphorus content acc. to DIN EN 14107.

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). This is determined from the fatty acid profile by using gas chromatography.

As can be seen in Figure 19, 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.

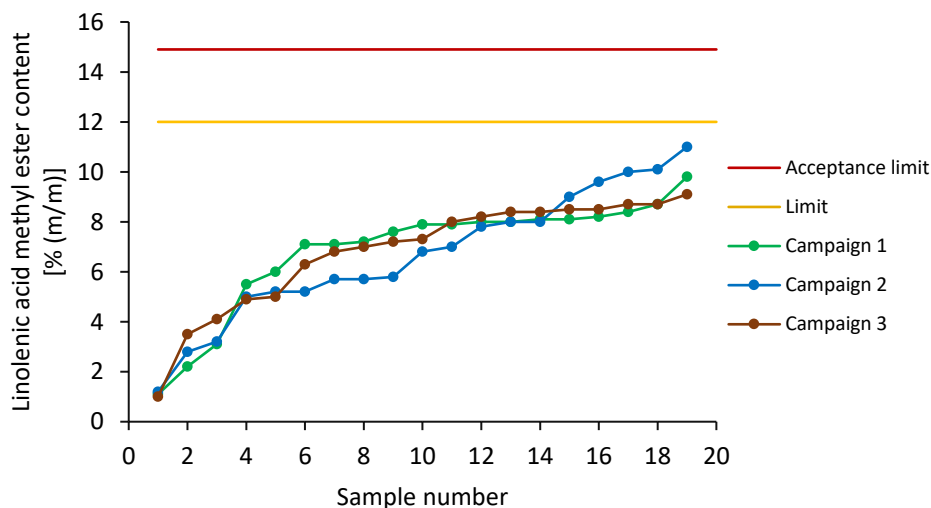


Figure 19: Content of Linolenic Acid Methyl Ester acc. to DIN EN 14103.



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 [No-Harm List BDFI](#) on the AGQM website.

Figure 20 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 intermediate period with campaign 3 in red.

Except for one sample, all samples tested comply with the specific seasonal limits. In campaign 1, one sample exceeded the winter limit with a CFPP of -8 °C within the corresponding acceptance limit.

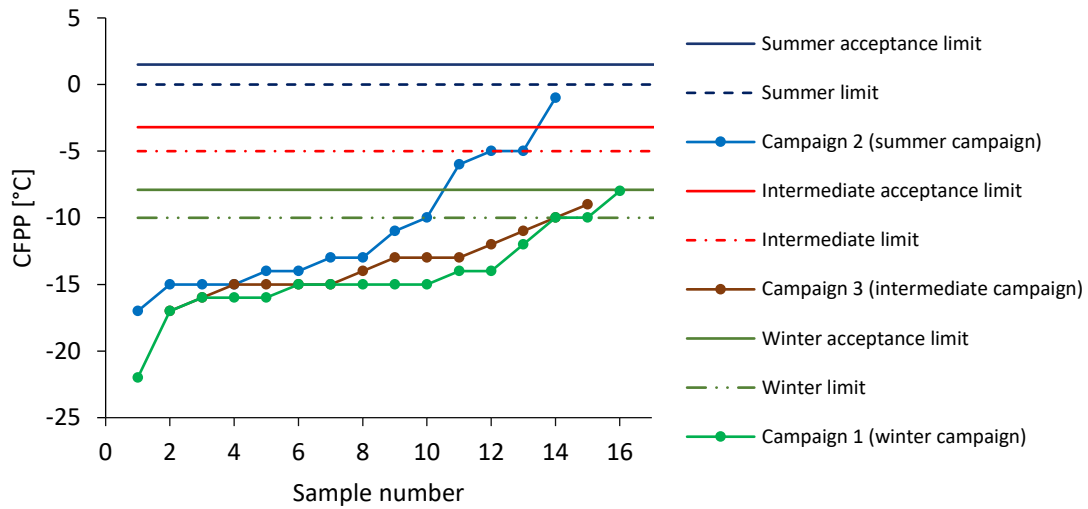


Figure 20: CFPP acc. to DIN EN 116.

Figure 21 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. One of the samples exceeded the AGQM limit with a CFPP of +11 °C within the corresponding acceptance limit in campaign 2.

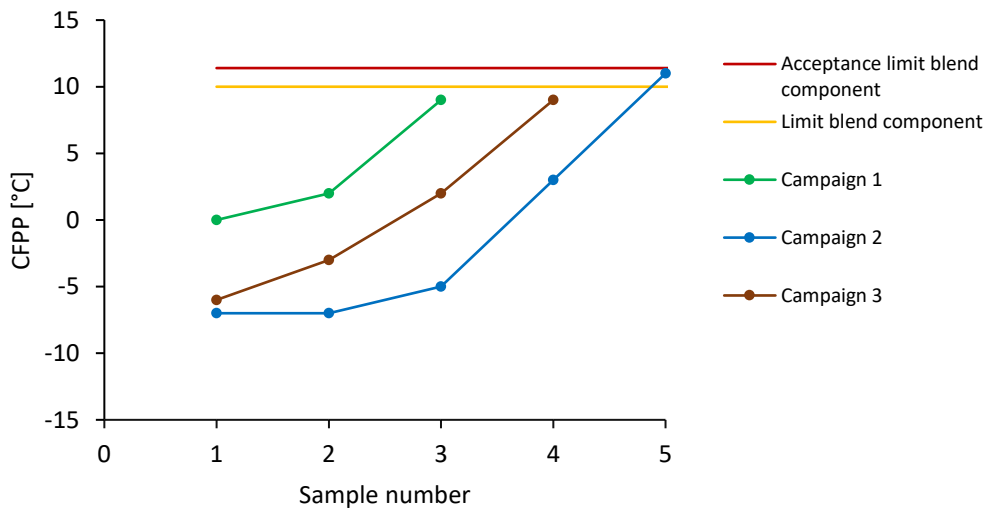


Figure 21: CFPP of blend components for biodiesel acc. to DIN EN 116.



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 22 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 intermediate period with campaign 3 in red.

Except for one sample, all samples tested comply with the specific seasonal limits. In campaign 1, one sample exceeded the winter limit with a CP of -2 °C within the corresponding acceptance limit. The irregularity was also evident in the CFPP (see p. 26).

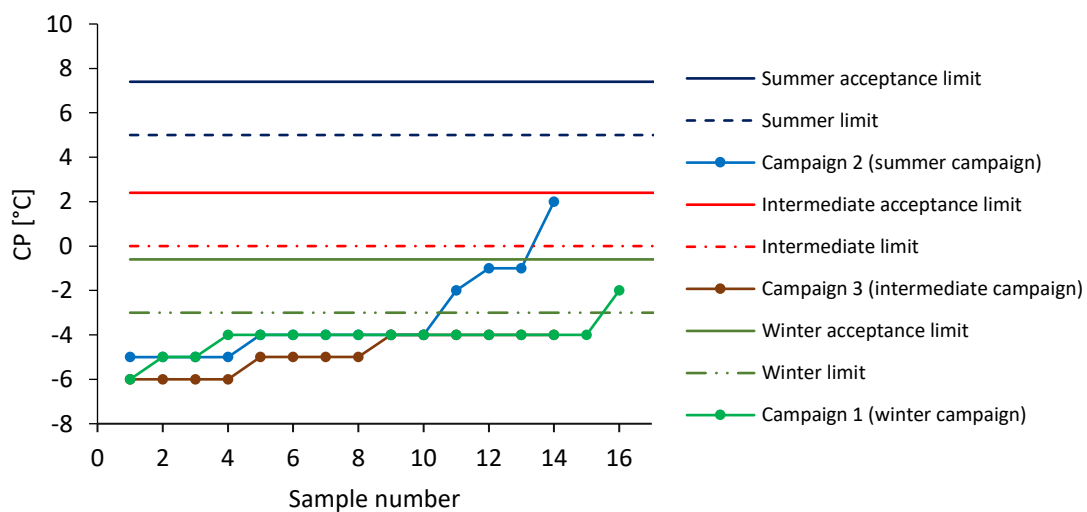


Figure 22: Cloud point acc. to DIN EN 23015.

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

In Figure 23, 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.

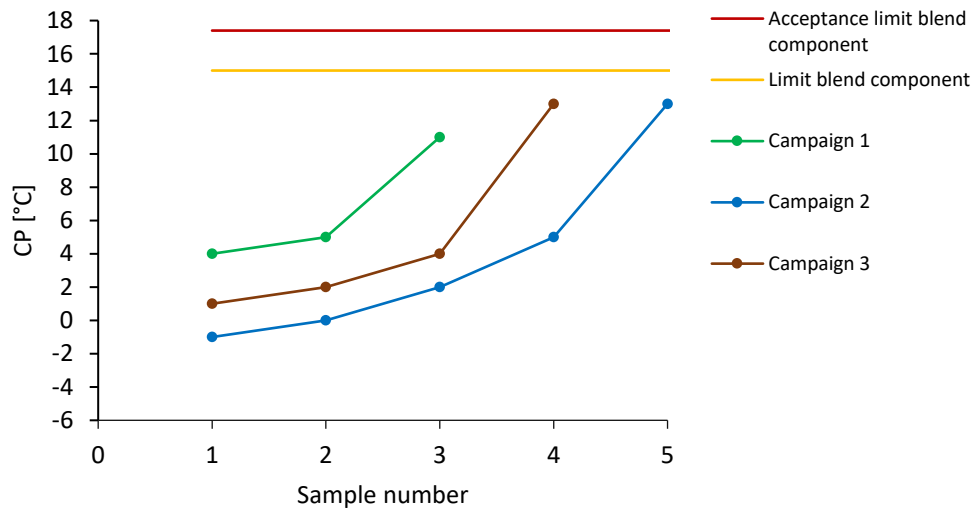


Figure 23: Cloud point of blend components for biodiesel acc. to DIN EN 23015.



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 2022, a total of three additional campaigns with nine samples were carried out.

In the additional campaigns 1 and 2, three companies were sampled again, but they did not show any abnormalities in the additional campaigns and thus complied with all limit values.

In the additional campaign 3, a total of three companies had to participate again. Only one company was found to be below the limit value for the parameter oxidation stability within the acceptance limit value.

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 2022 are presented.

A comparison of the sample numbers for the year 2021 and 2022 (Figure 24) shows that the total number of samples analysed in 2022 was 66, which is three samples higher than in 2021. This is due to the sampling of one additional member and its participation in the three main campaigns. The number of samples in the additional campaigns was kept constant at 9, where on the other hand the number of violations within the acceptance limit increased from 7 to 12 in 2022 compared to 2021. However, at the same time, the total number of violations outside the acceptance limit threshold decreased significantly compared to the previous year. The three acceptance limit violations also involve the specific AGQM acceptance limits for total contamination and water content in two cases. This further shows that the parameters for which stricter limits are required are precisely those that can be classified as critical and are therefore essential in order to be able to guarantee a high quality of the end product biodiesel.

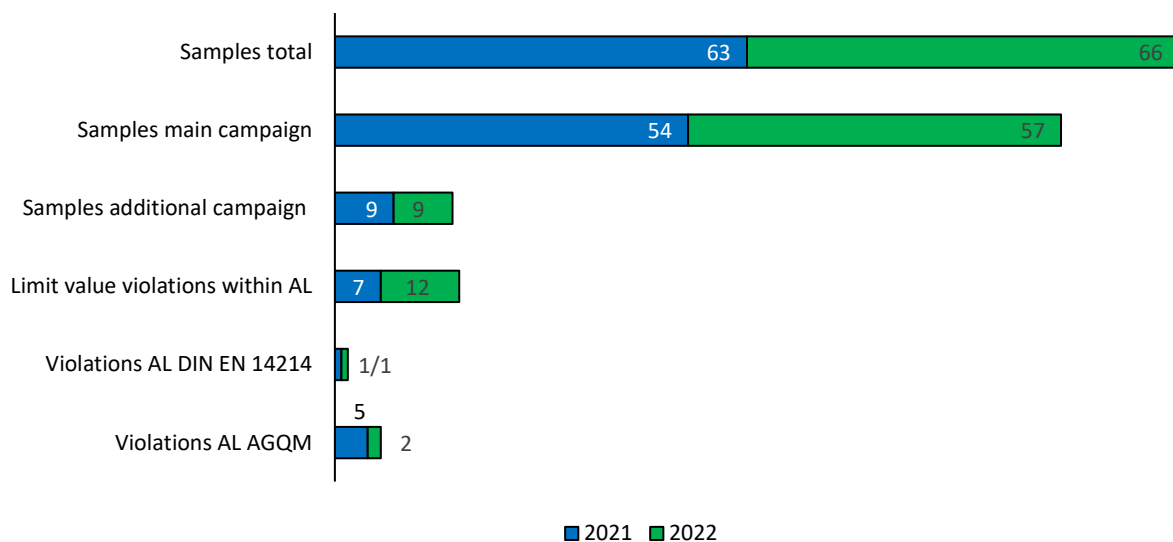


Figure 24: Comparison of the number of samples for main and additional campaigns as well as the number of limit value violations within and outside the acceptance limit values (AL) for the years 2021 and 2022.

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/m ³	860	900	859.7	900.3
Sulphur content (UV)	DIN EN ISO 20846	2011	mg/kg	-	10.0	-	113
Water content K.-F.	DIN EN ISO 12937	2000	% (m/m)	-	0.050	-	0.059
Total contamination	DIN EN 12662	1998 ¹	mg/kg	-	24	-	28
Oxidation stability (at 110 °C)	DIN EN 14112	2016	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	-	124.4
Content of linolenic acid methyl ester	DIN EN 14103	2011	% (m/m)	-	12.0	-	12.4
Content of free glycerol				-	0.02	-	0.026
Content of monoglycerides	DIN EN 14105	2011	% (m/m)	-	0.70	-	0.82
Content of diglycerides				-	0.20	-	0.24

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



Test Parameter	Method	Year of Publication	Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Content of triglycerides	DIN EN 14105	2011	% (m/m)	-	0.20	-	0.27
Content of total glycerol				-	0.25	-	0.285
Content of alkali metals (Na+K)				-	5.0	-	6.1
Content of earth alkaline metals (Ca+Mg)	DIN EN 14538	2006	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.	0	-	1.8
				from 01.10. to 15.11.	-5	-	-3.1
				from 16.11. to 28/29.02.	-10	-	-7.9
				from 01.03. to 14.04	-5	-	-3.1
Cloud point (if used as blend component for diesel fuel)	DIN EN 23015	1994	°C	from 15.04. to 30.09.	5	-	7.4
				from 01.10. to 15.11.	0	-	2.4
				from 16.11. to 28/29.02.	-3	-	-0.6
				from 01.03. to 14.04	0	-	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	% (m/m)	-	0.027	-	0.034
Water content (for traders)	DIN EN ISO 12937	2002	% (m/m)	-	0.032	-	0.039
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

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



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	-	20	-	20
Cloud point	DIN EN 23015	1998	°C	-	15	-	17.4
CFPP	DIN EN 116	2015	°C	-	10	-	11.4