Biodiesel Quality in Germany

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

2019

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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.

Biodiesel or FAME (fatty acid methyl ester) is used in the member states of the European Union via an admixture with diesel fuel in order to reduce CO_2 emissions and the use of fossil fuels. In Germany, admixtures of up to 7% (B7) are available. The European Standard EN 14214 describes the qualitative requirements a biodiesel have 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 in order 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 2019.

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 a main campaign have to take part in an additional campaign.

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

In 2019, 16 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:

C1:	14. January to 25. January	Winter grade
C2:	13. May to 24. May	Summer grade
C3:	07. October to 18. October	Intermediate grade

54 samples in the main campaigns and 5 samples in the resulting additional campaigns were taken and analysed. Three samples are taken each time a sampling takes place. One sample is used for analysis, the other two serve 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 may receive sanction points and have to participate in the next unannounced additional campaign.

The implementation of the QM system of the AGQM is evaluated for each member on the basis of 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

Basically, the requirements of the quality standard DIN EN 14214 apply. 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 in the course of sampling.

The valid version of DIN EN 14214 is used for the analysis. In 2019, 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 + A1:2014 and DIN EN 14214:2012 + A2:2019. Table 1: Limits and Test Methods for the Parameters tested according to DIN EN 14214:2014 in the Annex shows the parameters to be tested with their limit values in accordance with DIN EN 14214.

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. The AGQM limit values for these parameters are listed in Table 2: Limits and Test Methods for the Parameters tested according to the QM-System of AGQM. in the Annex. Separate limit values for warehouse operators were also defined in the AGQM guidelines for the parameter water content. Since only one warehouse operator with two warehouses was sampled in 2019, the results are not shown in the graphic representation for the water content in order to preserve the anonymity of the company.

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 alternative raw materials is generally used exclusively as a blend component for biodiesel produced from classic raw materials (especially rapeseed oil) and is not marketed as neat fuel. As a result of the blending these components, a biodiesel conforming to the standards is generated.

Manufacturers of biodiesel from these alternative fuels also belong to the member group of AGQM. For this reason, a separate chapter for blend components for biodiesel was implemented in the QM system in autumn 2017, in which special AGQM limit values for the parameters sulfur content, CFPP and cloud point were included. These three parameters are strongly dependent from the fatty acid composition or impurities in the raw material and can hardly be influenced in the manufacturing process. If a producer requests a corresponding exception from AGQM, the limit values to be observed are not the values of DIN EN 14214, but the specific limit values (see Annex Table 3: Limits and Test Methods for the tested Parameters for blend component for biodiesel according to the QM-System of AGQM.) for blend components. The corresponding samples are marked with an X in the diagrams.

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 with regard to 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 a black line, the acceptance limits, which are calculated taking into account the precision of the method, by a red line. Under customs law, but also with regard to the award of sanction points under the QM system, these acceptance limits are decisive.

The graphs of the parameter total contamination and water content also show the stricter AGQM limit and the AGQM acceptance limit. The diagrams for the parameters sulfur content, CFPP and cloud point were supplemented by the specific limit values and acceptance limit values for blend components for biodiesel.

4.1. Fatty Acid Methyl Ester Content

Test method:	*DIN EN 14103:2015*
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 % by mass [% (m/m)]. EN 14214 requires an ester content of at least 96.5% (m/m). A distilled end product after the transesterification usually has a higher ester content, since undesirable substances can be separated in this way.

In Diagram 1 it can be seen that all tested samples comply with the standard limit for the ester content. 95% of the samples have an ester content of over 97.9% (m/m).

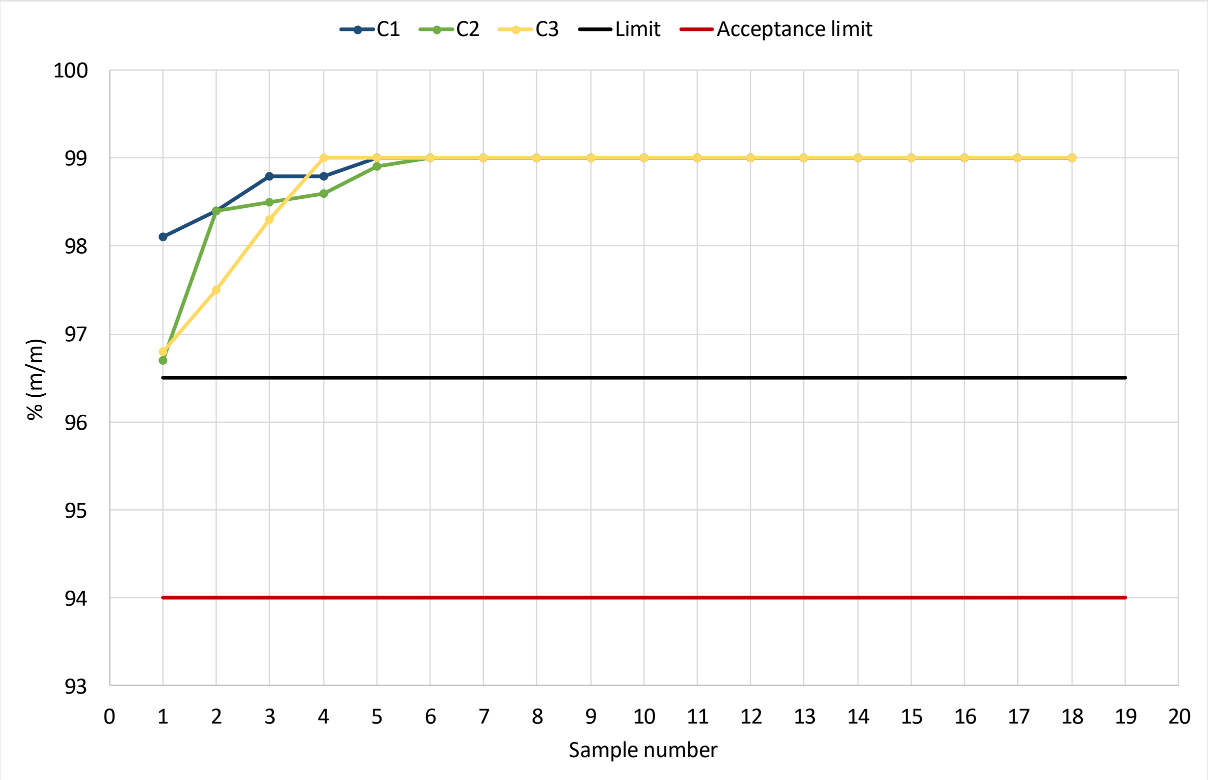

Diagram 1: Fatty Acid Methyl Ester Content acc. to DIN EN 14103.

4.2. Density at 15 °C

Test method:	*DIN EN ISO 12185:1997*
Limit of DIN EN 14214:	*min. 860 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.

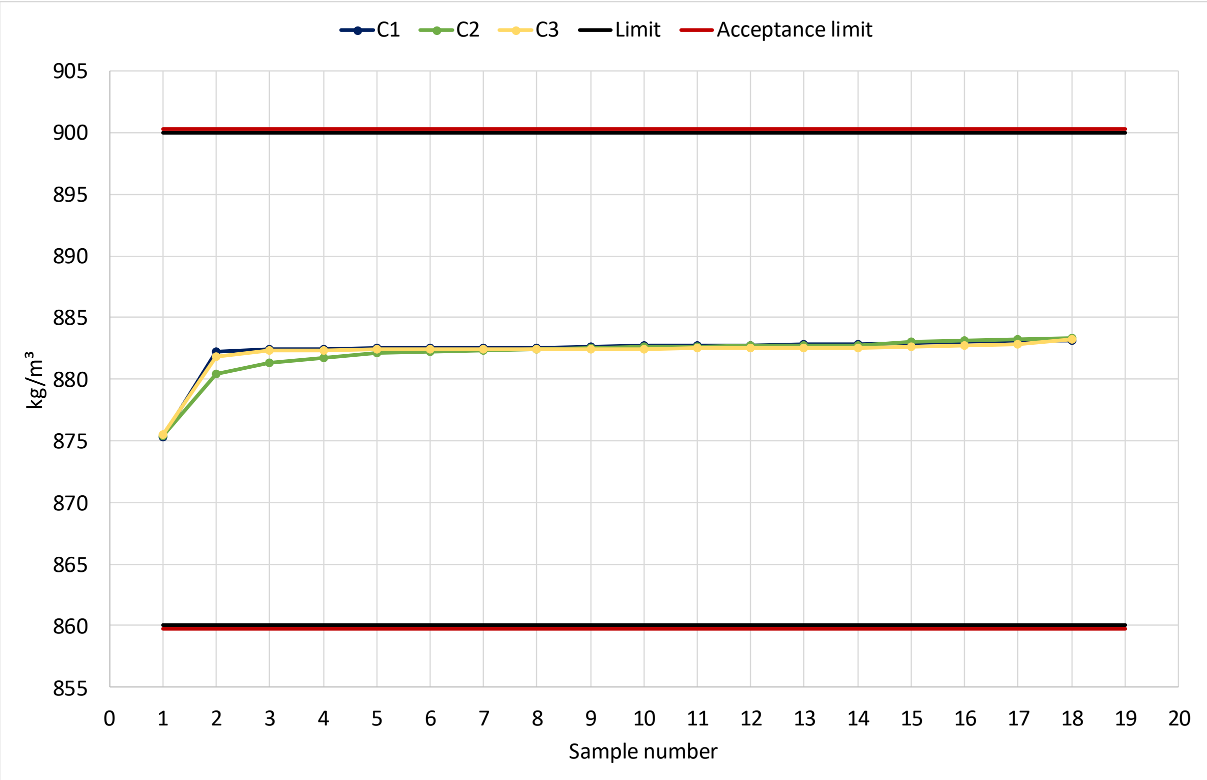

Diagram 2: Density at 15 °C acc. to DIN EN ISO 12185.

Diagram 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 881 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.

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 ppm*
AGQM acceptance limit for blend components for biodiesel:	*max. 14.5 ppm*

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.

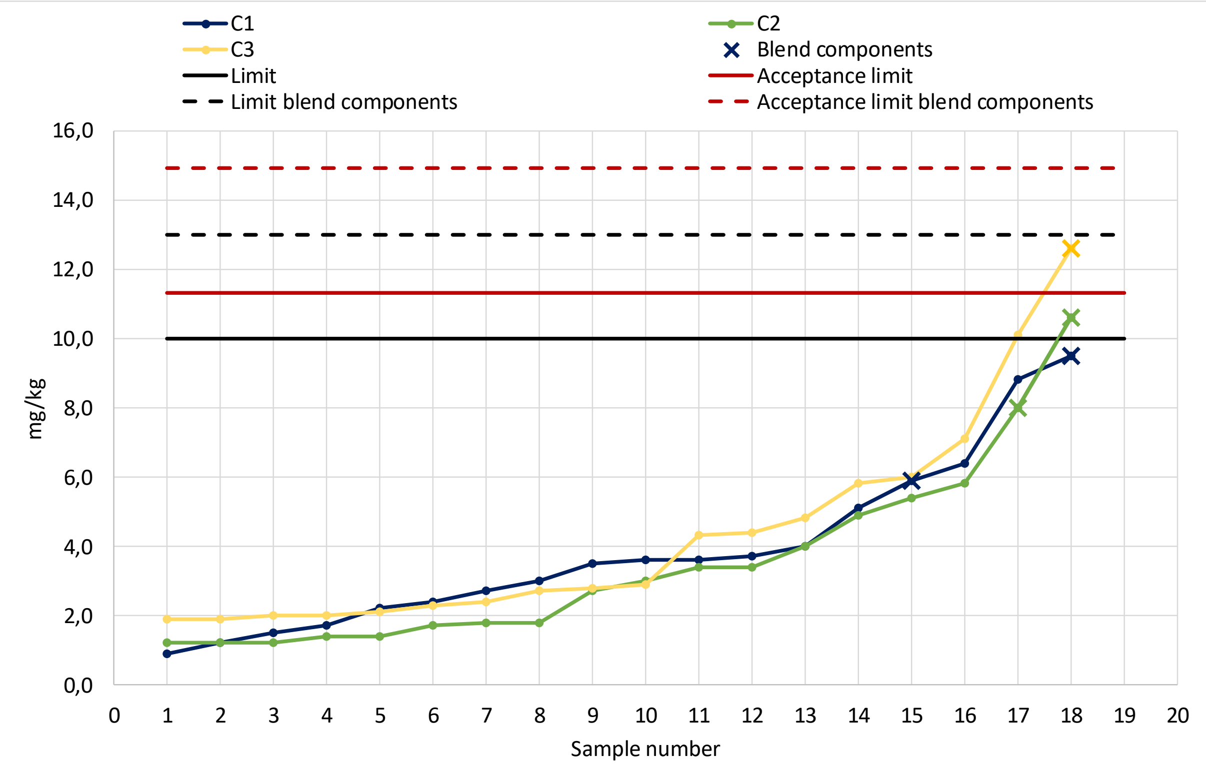

Diagram 3: Sulphur Content acc. to DIN EN ISO 20846.

As can be seen in Diagram 3, all but one sample comply with the required limits. A sample in C3 exceeds the limit (10 mg/kg) at 10.1 mg/kg within the acceptance limit (11.3 mg/kg). The samples marked with an X are blend components for biodiesel, for which according to the QM system deviating limit values apply. All samples of blend components for biodiesel were within the specific limit. 95% of the samples (without blend components for biodiesel) have a sulfur content below 6.82 mg/kg.

4.4. Water Content

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

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 need to be dried at the end of the biodiesel production. Subsequently, the storage conditions must be selected accordingly, in order 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 water dissolved in it can precipitate. In winter, freezing of potential free water can block filters or piping, in summer it can cause corrosion or promote microbial growth. DIN EN 14214 requires a maximum water content of 500 mg/kg. The AGQM has stricter quality guidelines due to the issues outlined above and requires from its members a maximum water content of 220 mg/kg ex works.

Diagram 4 shows the values for the water content. It can be seen that all tested samples are well below the standard limit. Except for one sample in C3, all values are within the AGQM limit for manufacturers.

In campaign 3 one sample exceeded the AGQM limit for producers (220 mg/kg) with 290.5 mg/kg outside the AGQM acceptance limit (280 mg/kg). The company itself discovered a technical malfunction in the distillation plant and therefore did not request an arbitration analysis. A sanction point was awarded. In the resulting additional campaign, the member was inconspicuous.

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

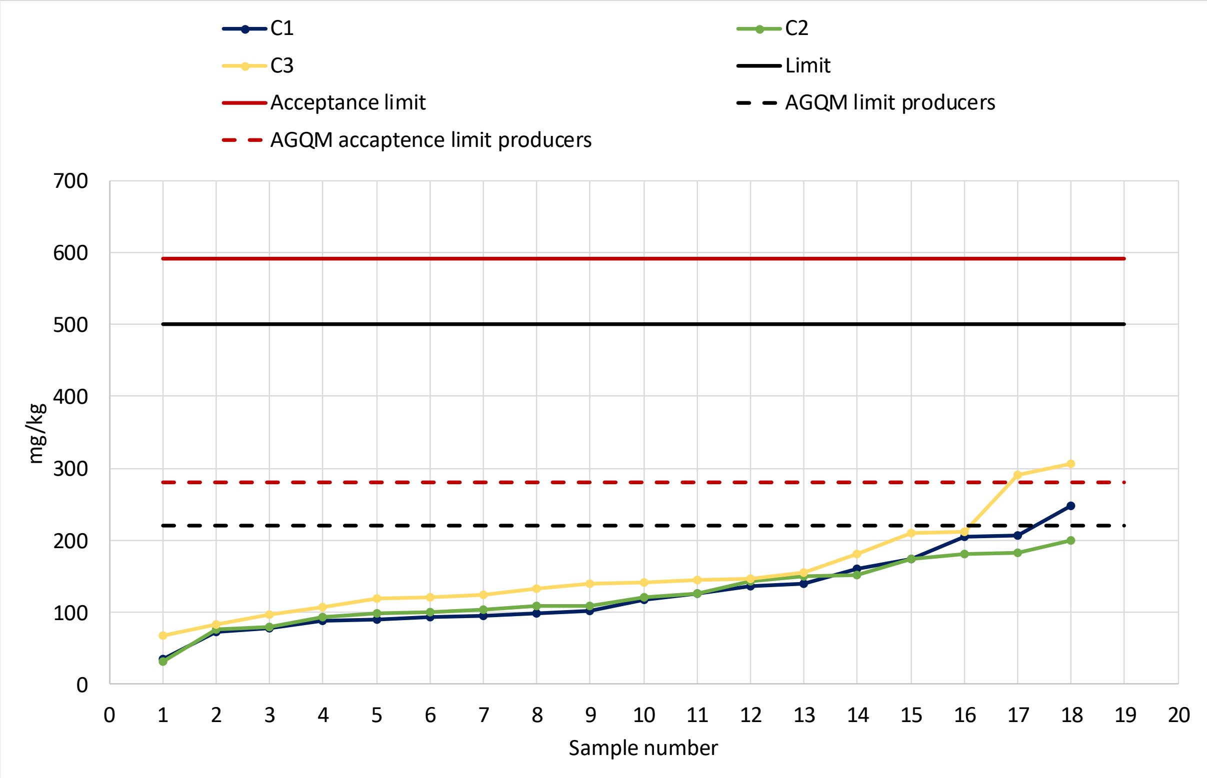

Diagram 4: Water Content acc. to DIN EN ISO 12937.

4.5. Total Contamination

Test method: *DIN EN 12662:1998*

Due to the fact that the current version of DIN EN 12662:2014 is unsuitable for pure FAME (B100) concerning the determination of parameter 'Total Contamination', DIN EN 12662:1998 applies for AGQM's checks. This procedure is based on a recommendation by CEN TC19 – JWG 1 of 13 July 2014.

Limit of DIN EN 14214: *max. 24 mg/kg*

Acceptance limit: *max. 31 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 here. 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.

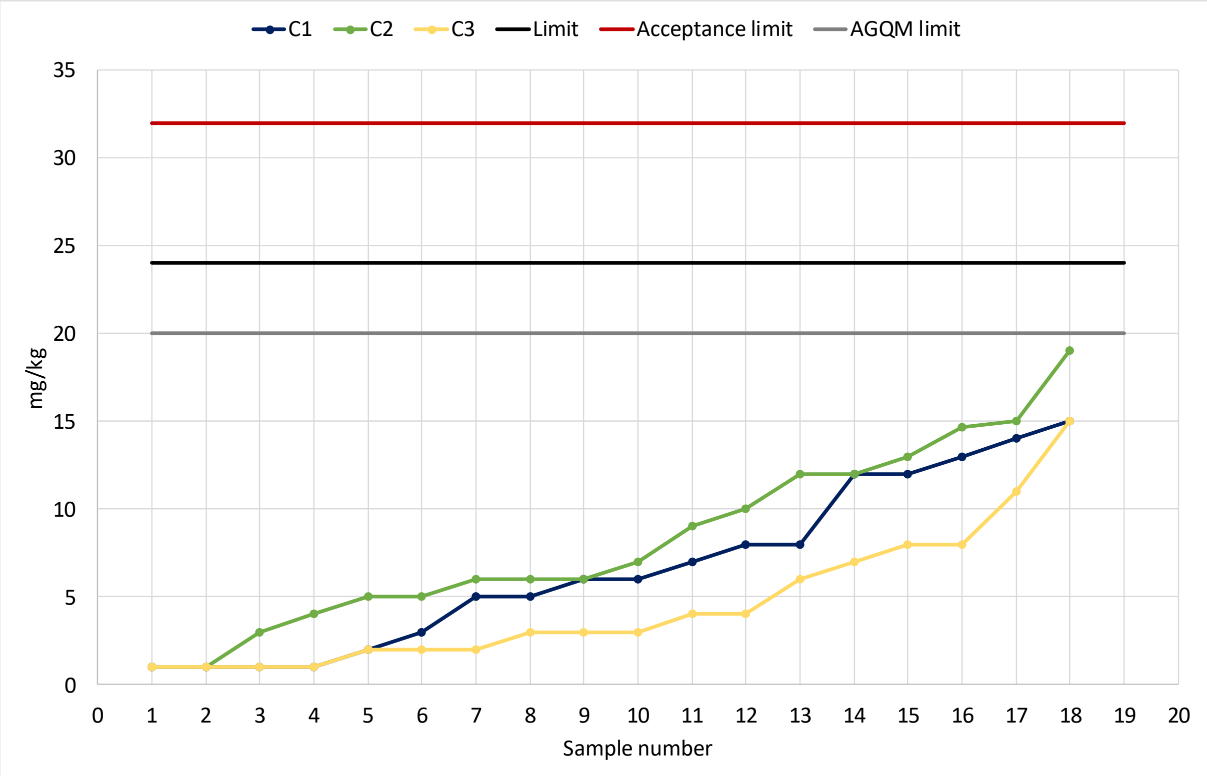

Diagram 5: Total Contamination acc. to DIN EN 12662.

Diagram 5 shows that all samples can meet the stricter AGQM limit for total contamination.

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 to detection of these oxidation products is referred to as induction time or oxidation stability. DIN EN 14214 requires a minimum oxidation stability of 8 hours.

Diagram 6 shows the oxidation stabilities of the tested samples. All but one sample meet the requirements of the standard. One sample from campaign 3 falls below the limit (8.0 h) at 7.85 h within the acceptance limit (6.6 h). 95% of the samples have an oxidation stability over 8.4 h.

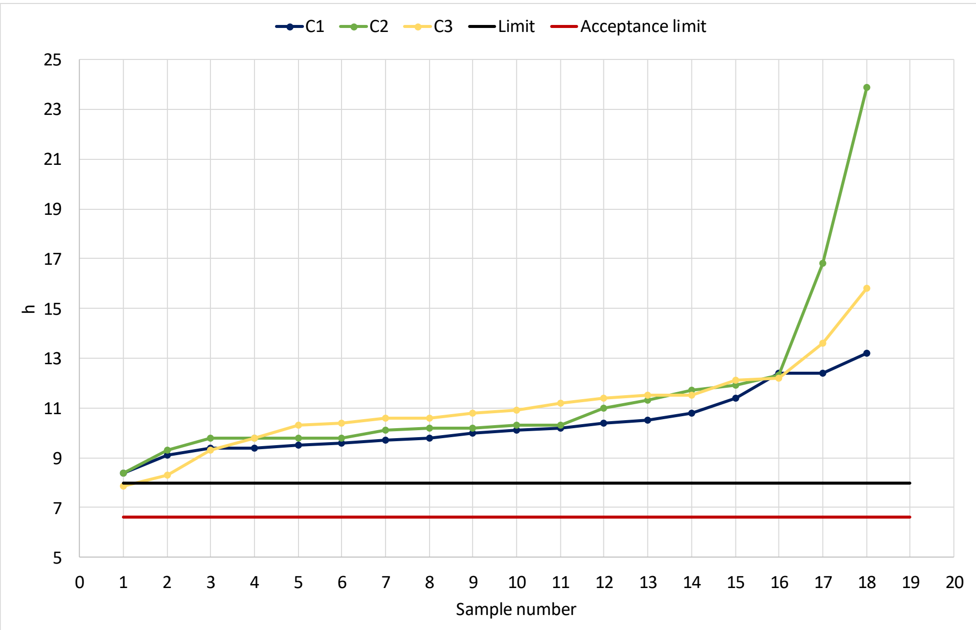

Diagram 6: Oxidation Stability acc. to DIN EN 14112.

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.

Diagram 7 shows the measured values for the acid number. All samples meet the requirements of the standard.

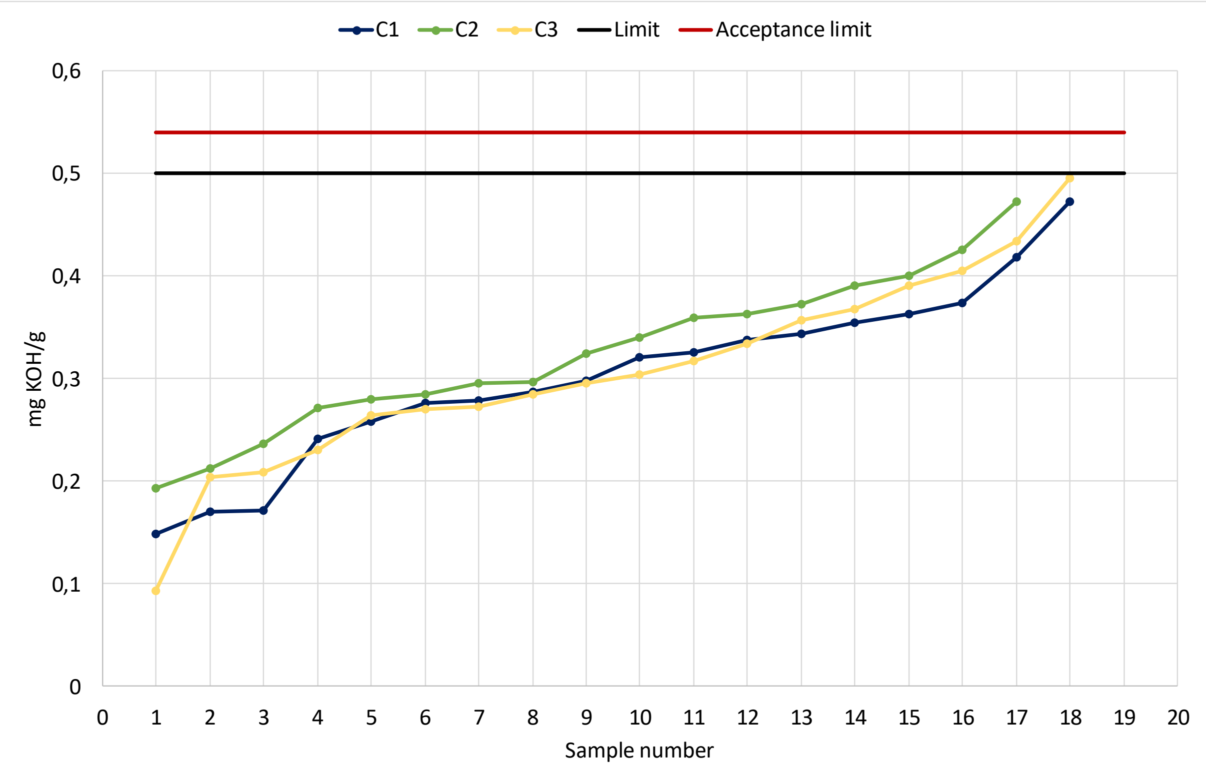

Diagram 7: Acid Number acc. to DIN EN 14104.

4.8. Iodine Number

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

The iodine value is a measure of the proportion of double bonds which is present in fats and oils and also in the 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 value. 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, the iodine value is determined by calculation from the gas chromatographically measured fatty acid profile according to DIN EN 16300. The result is given in g iodine/100 g biodiesel.

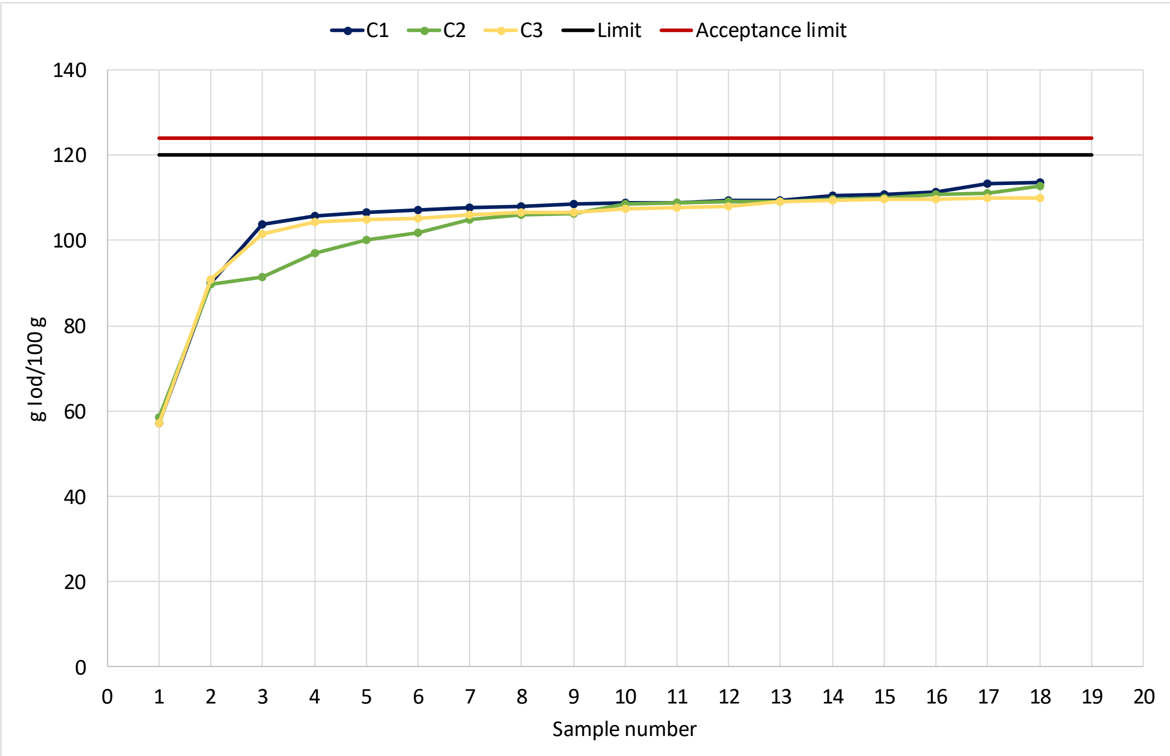

Diagram 8: Iodine Number acc. to DIN EN 16300.

Diagram 8 shows the results for the iodine number. All examined samples are below the standard limit value. Three samples show iodine numbers below 60 g iodine/100 g biodiesel all year round, which is also due to the raw material used.

4.9. Mono-, Di-, und Triglycerides, free Glycerol

Test method:	*DIN EN 14105:2011*
<u>Monoglycerides</u>	
Limit of DIN EN 14214:	*max. 0.70% (m/m)*
Acceptance limit:	*max. 0.82% (m/m)*
<u>Diglycerides</u>	
Limit of DIN EN 14214:	*max. 0.20% (m/m)*
Acceptance limit:	*max. 0.24% (m/m)*
<u>Triglycerides</u>	
Limit of DIN EN 14214:	*max. 0.20% (m/m)*
Acceptance limit:	*max. 0.27% (m/m)*
<u>Free Glycerol</u>	
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 (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.

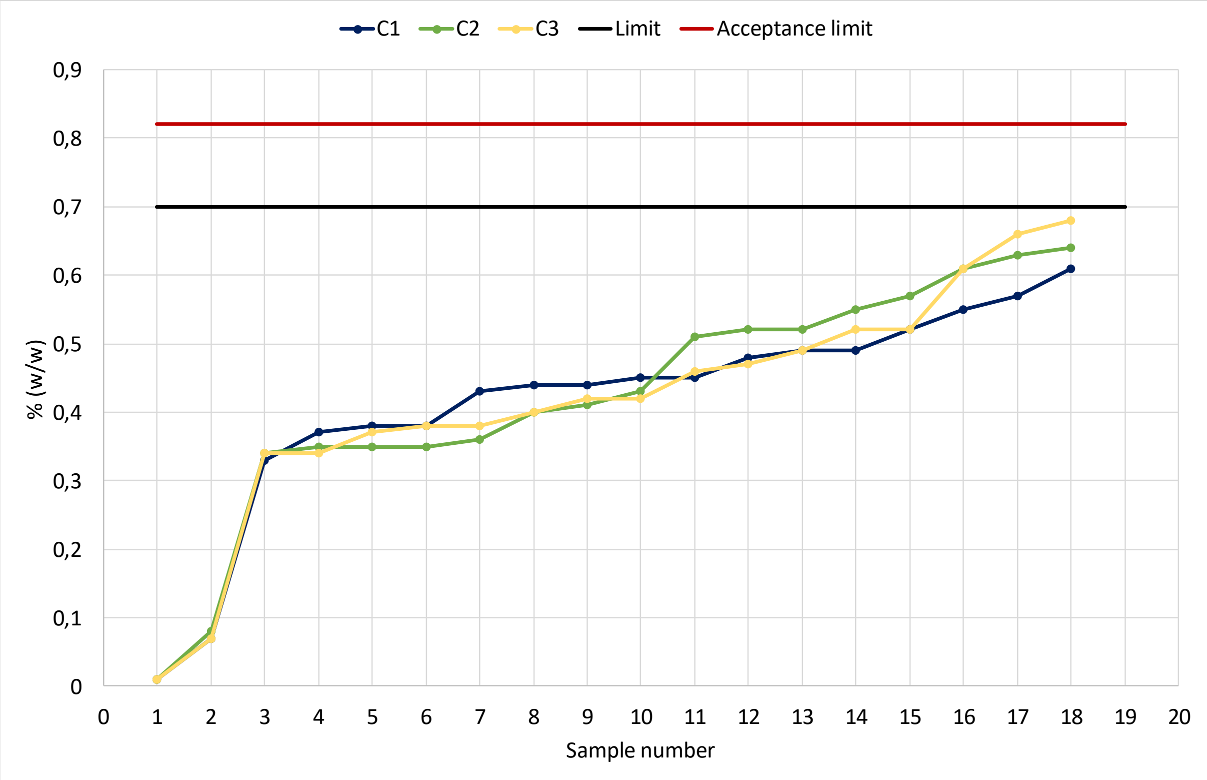

Diagram 9: Monoglycerides acc. to DIN EN 14105.

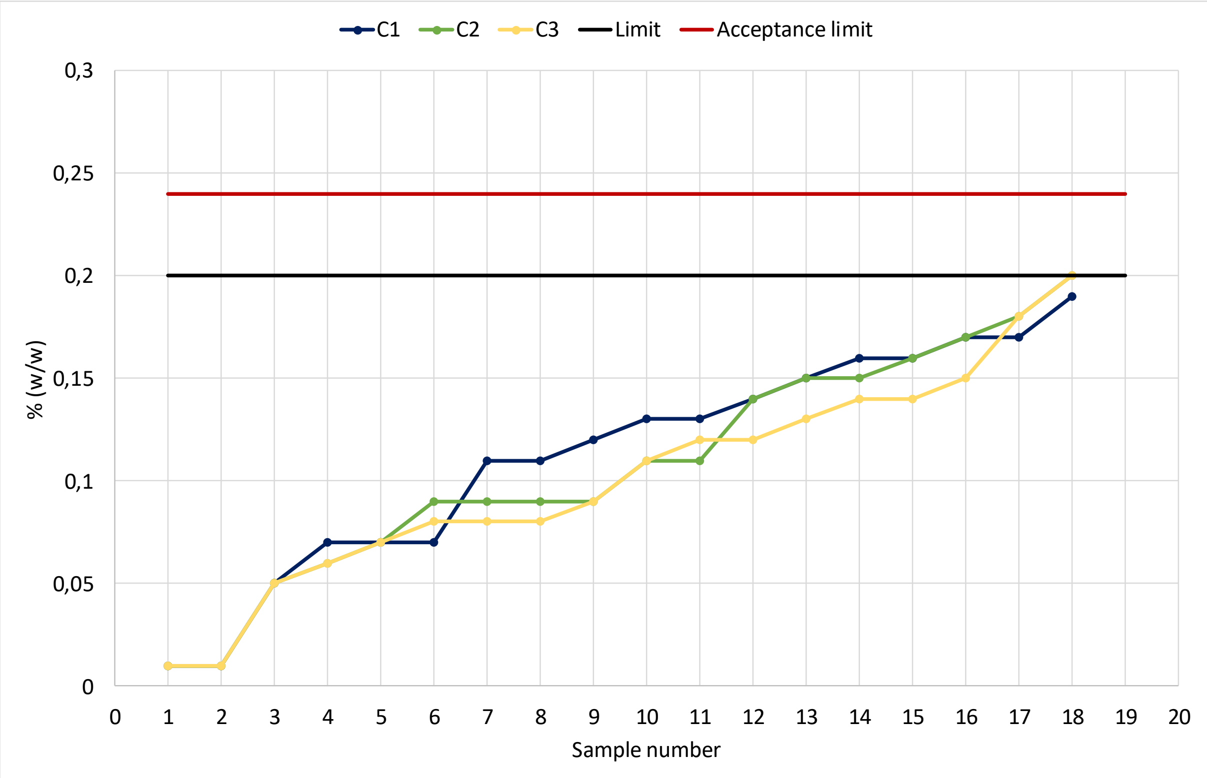

Diagram 10: Diglycerides acc. to DIN EN 14105.

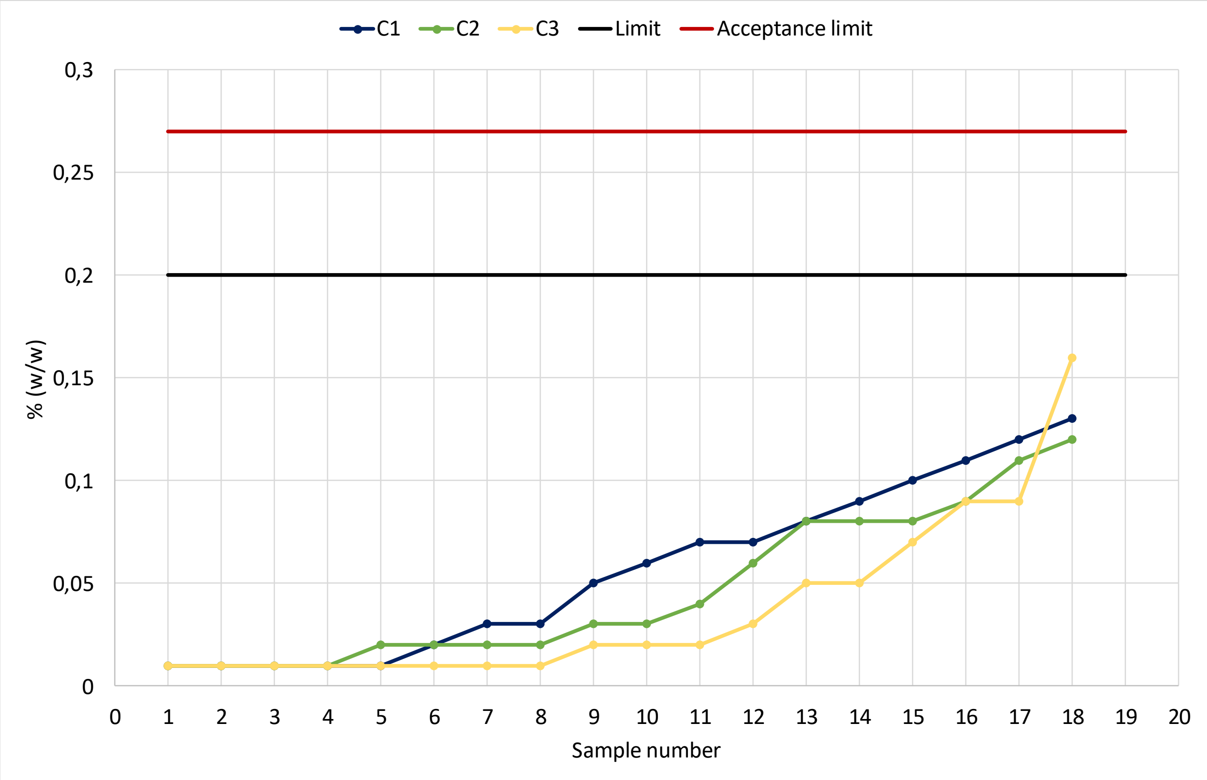

Diagram 11: Triglycerides acc. to DIN EN 14105.

Diagrams 9 to 11 show the results of the investigations for mono-, di- and trigycerides. All samples are within the standard limits. For the monoglycerides, some samples even show values lose to 0.0% (m/m), which suggests that the production process involves a distillation step.

Diagram 12 shows the free glycerin content. A sample in C1 exceeds the limit (0.020% (m/m)) at 0.051% (m/m) outside the acceptance limit (0.026% (m/m)). This value is the result of an arbitration analysis and the member received a sanction point. The member carried out measures, so that the member was inconspicuous in the next campaigns. All other samples comply with the standard limit.

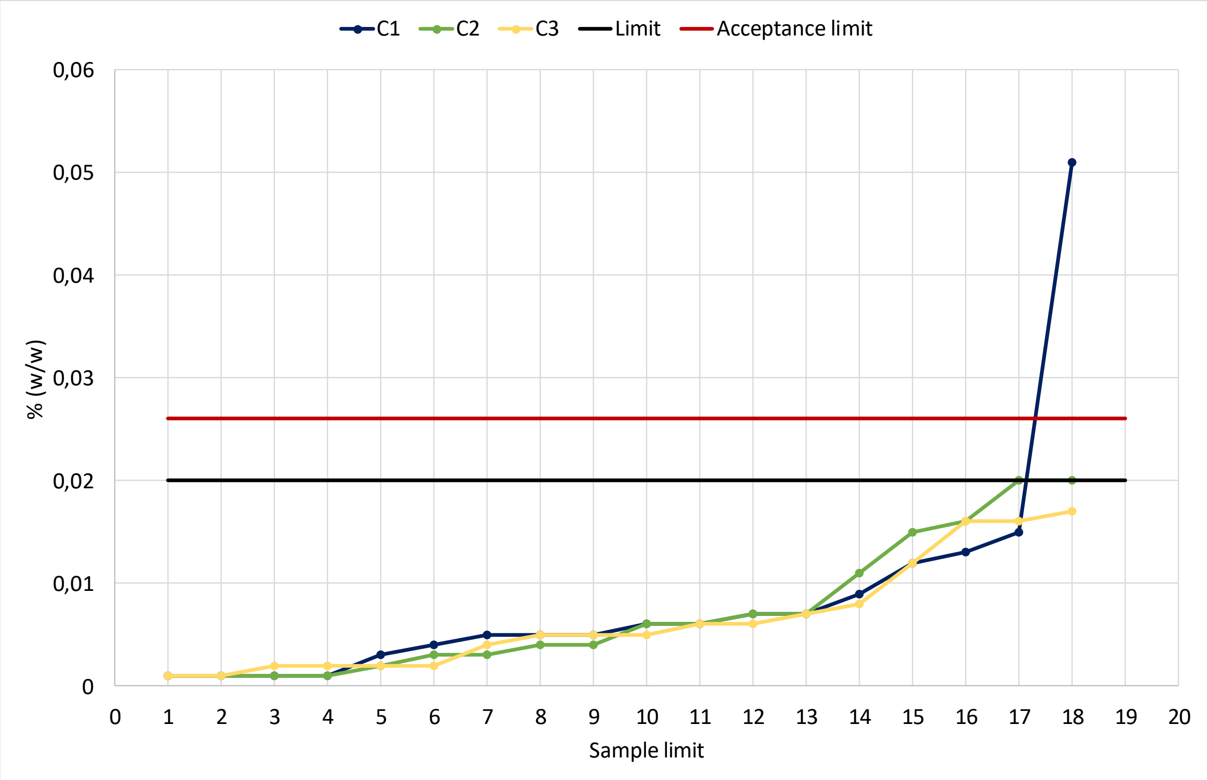

Diagram 12: Free Glycerol acc. to DIN EN 14105.

4.10. Alkali 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*
Acceptance limit:	*max. 6.1 mg/kg*

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. Alkali metals are also associated with ash formation. Sodium and potassium can agglomerate on the surface of particle filters and oxidation catalysts, reducing the effectiveness and lifetime of the systems.

The alkaline earth metals calcium and magnesium are either added to the raw material in the process or can reach the end 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 alkali metal soaps. The use of softened water can prevent the entry of alkaline earth metals into the biodiesel.

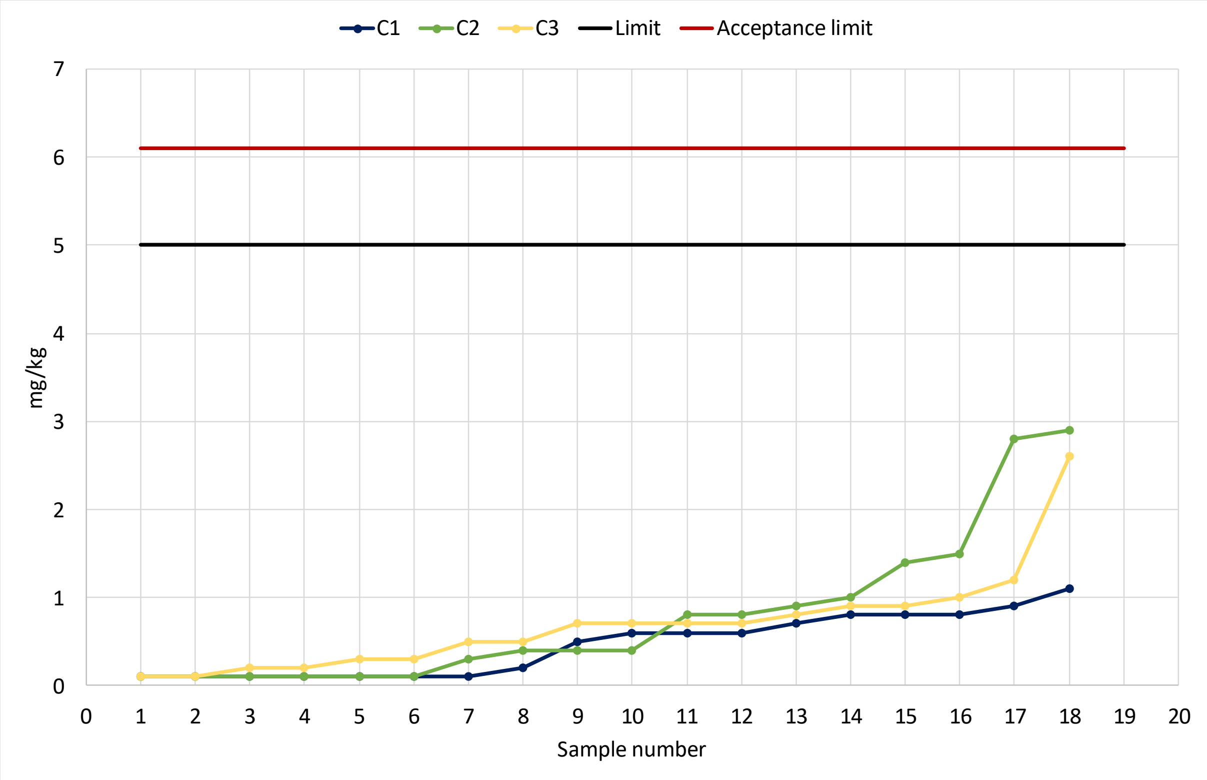

Diagram 13: Sum of Alkali Metals Sodium and Potassium acc. to DIN EN 14538.

Diagram 13 and Diagram 14 clearly show that biodiesel producers attach great importance to low levels of alkali and alkaline earth metals. The contents of the alkali metals sodium and potassium are, except for three samples, all below 2 mg/kg (maximum limit 5 mg/kg). 95% of the samples have alkali contents below 1.89 mg/kg. The levels of the alkaline earth metals magnesium and calcium are even well below the limit of quantification of 1 mg/kg.

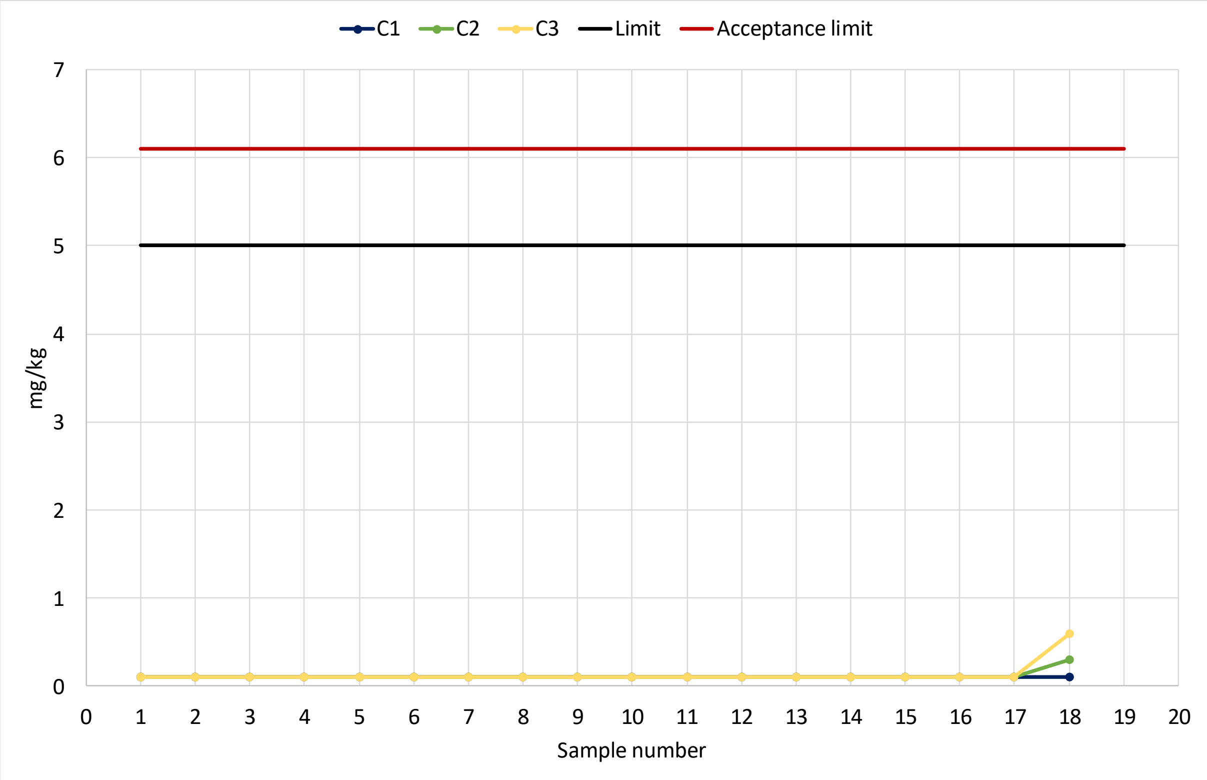

Diagram 14: Sum of Alkaline Earth Metals Calcium and Magnesium acc. to DIN EN 14538.

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. The limit is a maximum of 4 mg/kg, the precision of the method does currently not allow a further tightening.

Diagram 15 shows the values for the phosphorus content. The almost consistently slightly increased values in campaign 2 are due to a test equipment calibration. 95% of the values are below 1.7 mg/kg and thus far below the limit.

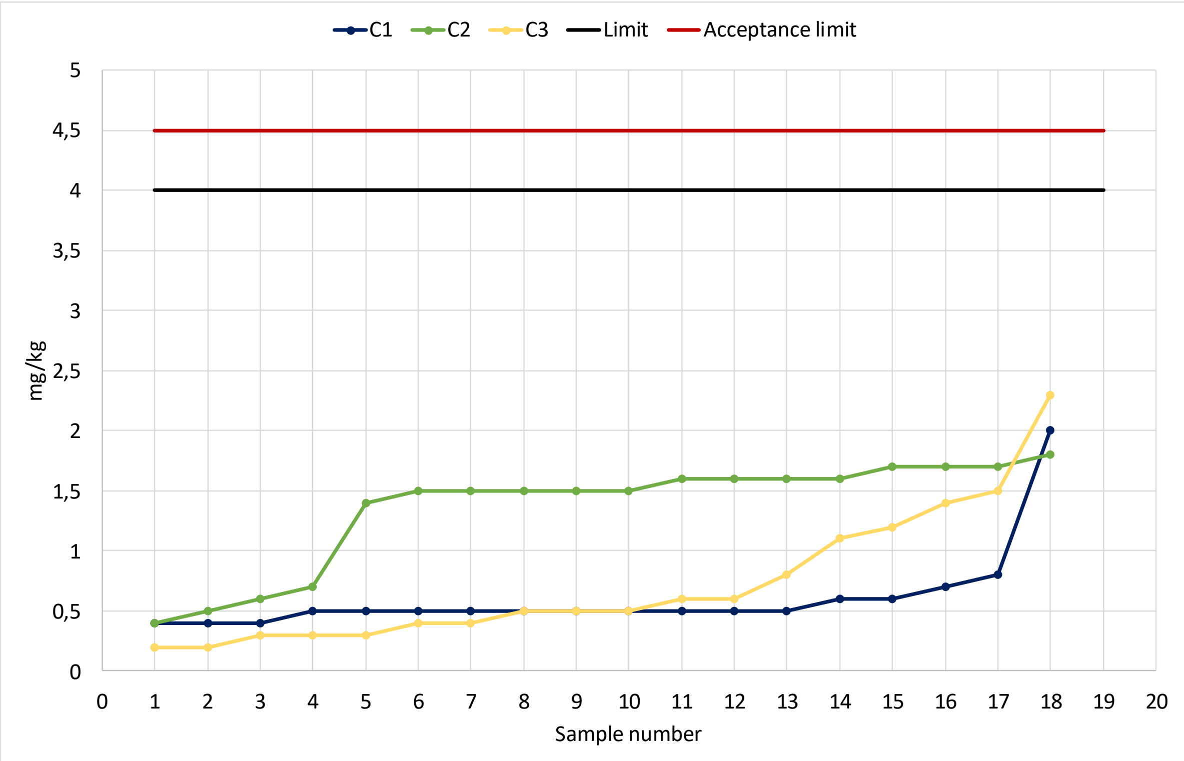

Diagram 15: Phosphorous Content acc. to DIN EN 14107.

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.

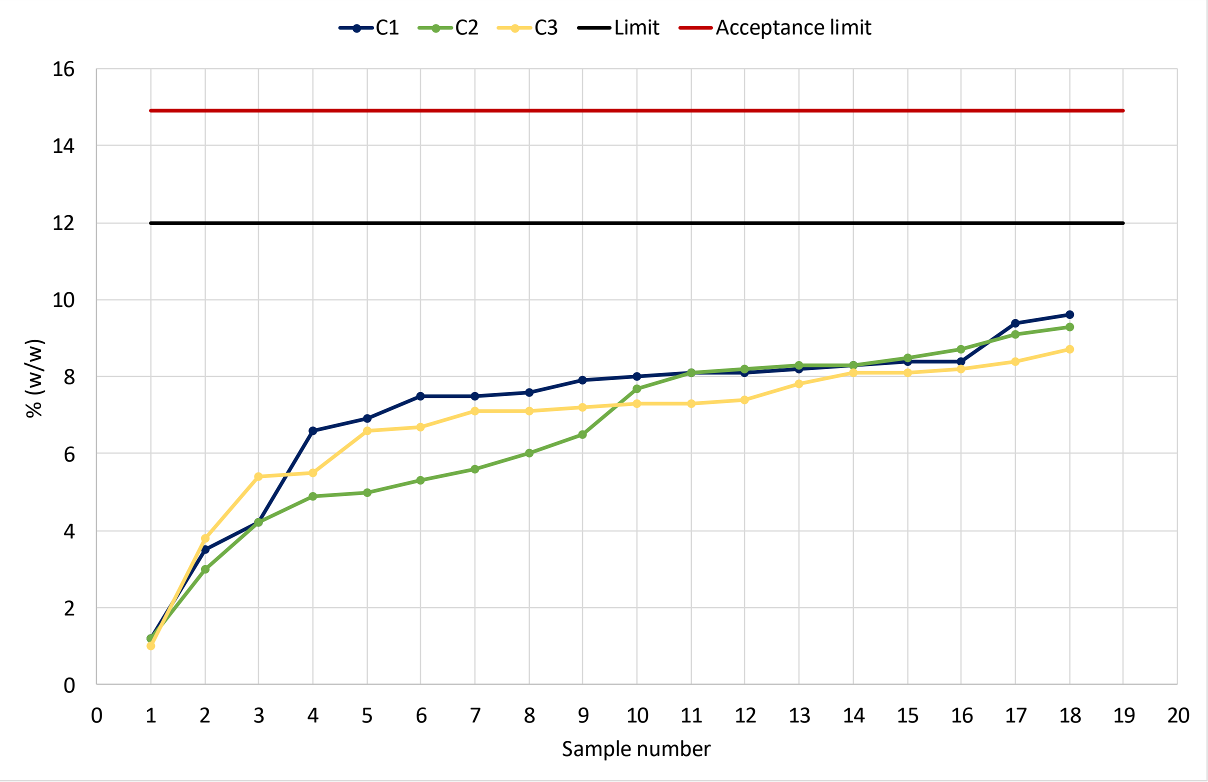

Diagram 16: Content of Linolenic Acid Methyl Ester acc. to DIN EN 14103.

As shown in Diagram 16, 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 8% and 10%. The lower contents of a large proportion of the samples in the summer campaign C2 show that the raw material rapeseed oil commonly used in biodiesel production was at least partially replaced by other oils.

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. Similar to diesel fuel, different requirements for summer, intermediate and winter quality apply.

In Germany, with regard to the cold properties, the legal regulation that biodiesel as a blend component for diesel fuel between 16.11. and the 28./29.02. have to comply with a CFPP of -10 °C, if the -20 °C required in DIN EN 14214 can be achieved by adding additives. The additivation of the mixture of diesel fuels and biodiesel then takes place in the refineries. The market for pure biodiesel (B100) has nearly come to a standstill due to the statutory regulations on mineral oil tax, so that almost exclusively biodiesel is supplied as a blend component for diesel fuel.

Diagram 17 shows the values and various limits for the CFPP. The winter limit is indicated by a dotted line, the intermediate limit by a dashed line and the summer limit by a solid line. In addition, the AGQM limit value for blend components for biodiesel is represented by a solid grey line.

In C1, two samples exceed the winter limit (-10 °C) at -9 °C and -8 °C within the acceptance limit (-7.9 °C). The samples marked with an X are blend components for biodiesel, for which according to the QM system item 2.1.1 deviating limit values apply. All analysed blend component samples are within the specific limit.

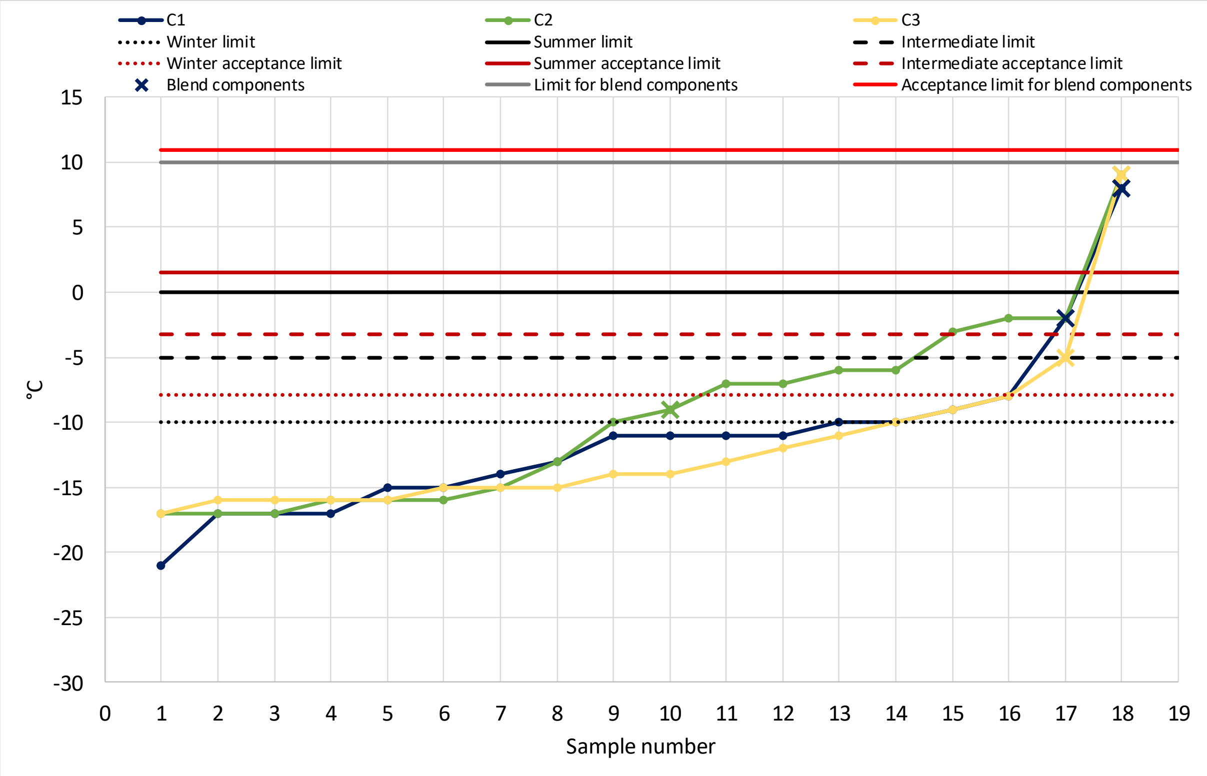

Diagram 17: CFPP acc. to DIN EN 116.

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, °C4*	*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.

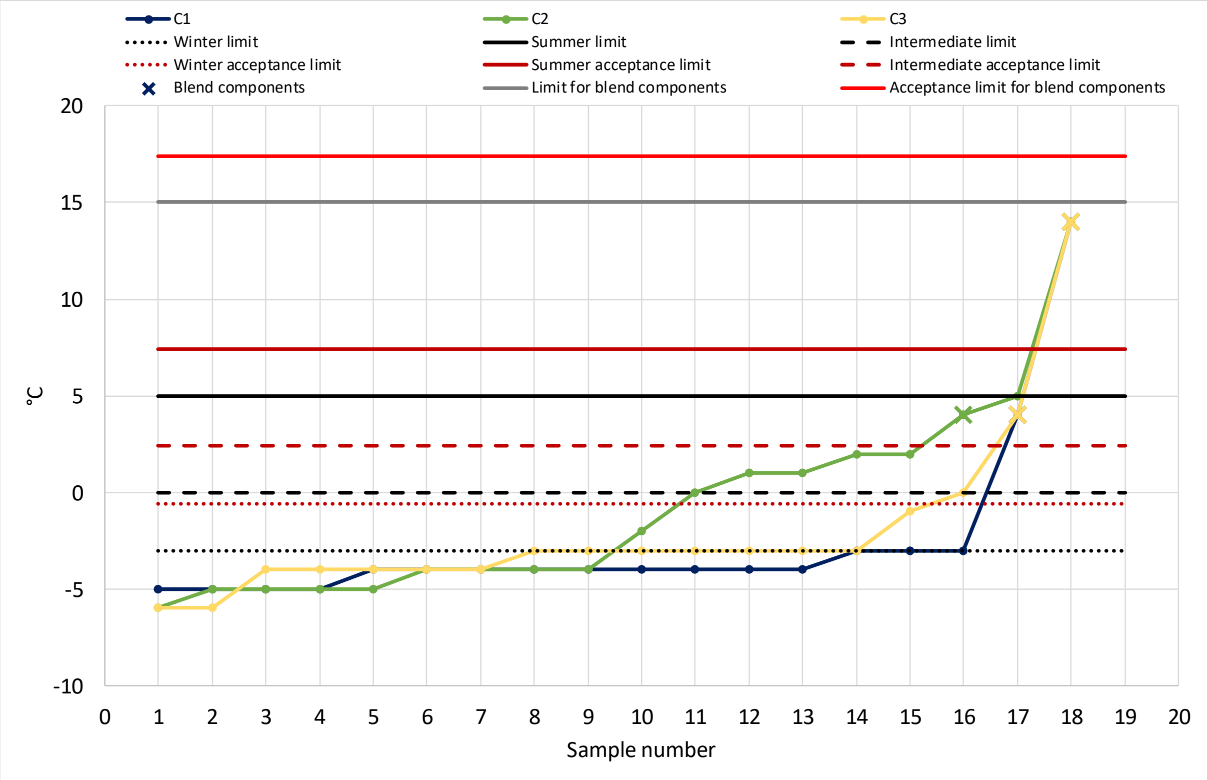

Diagram 18: Cloud point acc. to DIN EN 23015.

Diagram 18 shows the measurements for the Cloud point. All examined samples are within the corresponding limit values. The samples marked with an X are blend components for biodiesel, for which according to the QM system item 2.1.1 deviating limit values apply. These products must not be marketed as pure fuel. All analysed blend component samples are within the specific limit.

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 campaign. According to this principle, two additional campaigns were carried out in 2019, in which 5 samples were taken.

Three companies participated in additional campaign 1. A company was found to exceed the limit within the acceptance limit. A second company showed four abnormalities, including two violations of the acceptance limit. The member had already determined the limit violations as part of internal quality assurance and had taken appropriate countermeasures. No arbitration analysis was commissioned. Since no abnormalities were found in campaign 2, additional campaign 2 could be omitted.

Two companies took part in additional campaign 3, for both companies all analysis results were within the relevant limit values.

5 Summary

Since 2010, the AGQM has been publishing an annual report on the quality of the biodiesel produced and traded by its members. This report presents the results of the unannounced sampling of each year.

In summary, the samples carried out in 2019 show a positive development compared to the previous year 2018. The number of abnormalities found was significantly lower, as can be seen in Figure 19.

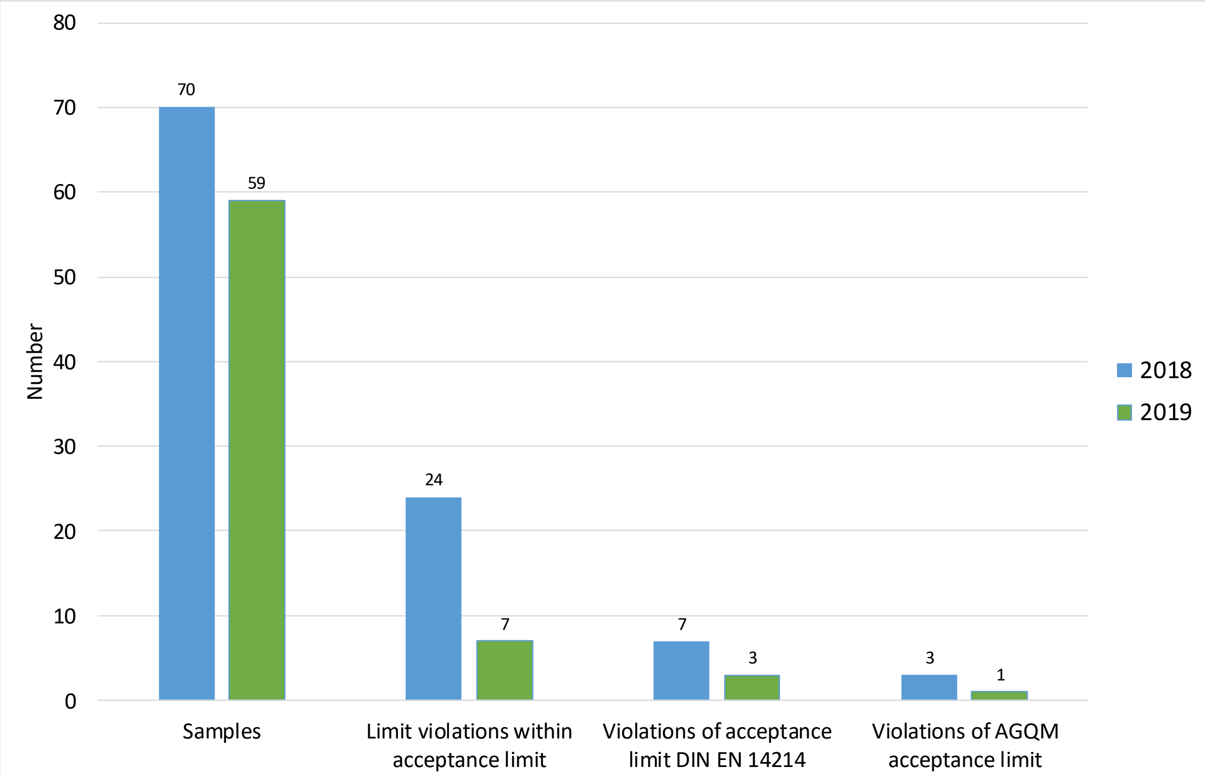

Figure 19 Overview of abnormalities compared to 2018.

Based on the 17 sampled member companies, it is noticeably that 14 members were unremarkable in all campaigns. The abnormalities found relate to 3 companies. These companies also detected the deviations as part of their own quality control monitoring so that the goods could be effectively prevented 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.

It is very remarkable that although the summer campaign C2 had a particularly large number of limit violations in 2018 (17 in total), there was not a single limit violation in C2 in 2019. The members thus learned from the hot summer of 2018 and adjusted their quality assurance measures accordingly in 2019.

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 14214:2014

Test Parameter	Method	Year of Publication	Unit	Standard Limits		Acceptance Limits	
				min.	max.	min.	max.
Fatty Acid Methyl Ester Content	DIN EN 14103	2015	% (m/m)	96.5	-	94.0	-
Density at 15 °C	DIN EN ISO 12185	1997	kg/m³	860	900	859.7	900.3
Sulphur Content (UV)	DIN EN ISO 20846	2011	mg/kg	-	10.0	-	11.3
Water Content K.-F.	DIN EN ISO 12937	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/ 100g	-	120	-	124
Content of Linolenic Acid Methyl Ester	DIN EN 14103	2015	% (m/m)	-	12.0	-	12.4
Content of Free Glycerol	DIN EN 14105	2011	% (m/m)	-	0.02	-	0.026

[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 Monoglycerides	DIN EN 14105	2011	% (m/m)	-	0.70	-	0.82
Content of Diglycerides		2011	% (m/m)	-	0.20	-	0.24
Content of Triglycerides		2011	% (m/m)	-	0.20	-	0.27
Content of Alkali Metals (Na+K)	DIN EN 14538	2006	mg/kg	-	5.0	-	6.1
Sodium Content		2006	mg/kg	-	5.0	-	6.1
Potassium Content		2006	mg/kg	-	5.0	-	
Content of Earth Alkali Metals (Ca+Mg)		2006	mg/kg	-	5.0	-	6.1
Calcium Content		2006	mg/kg	-	5.0	-	6.1
Magnesium Content		2006	mg/kg	-	5.0	-	
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 (if used as blend component for biodiesel)	DIN EN 23015	2013	°C		15		17.4
CFPP (if used as blend component for biodiesel)	DIN EN 116	2015	°C		10	-	10.9

[2]2 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.

6.2 Abbreviations

AC1	Additional Campaign 1
AC2	Additional Campaign 2
AC3	Additional Campaign 3
AGQM	Association Quality Management Biodiesel
BImSchV	Bundes-Immissionsschutzverordnung (German Federal Emission Protection Directive)
C1	Campaign 1
C2	Campaign 2
C3	Campaign 3
CEN	Comité Européen de Normalisation (European Standardisation Committee)
CFPP	Cold Filter Plugging Point
DIN	Deutsches Institut für Normung (German Institute for Standardisation)
EN	European Standard
FAME	Fatty Acid Methyl Ester
QM system	Quality Management System
TC	Technical Committee