Biodiesel Quality in Germany

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

2018

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1 Introduction

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 must meet in order to market it as a ready-to-use product. The Association Quality Management Biodiesel (AGQM) was founded in 1999 by German biodiesel producers and warehouse operators in order to meet the quality requirements of the resulting German standard DIN EN 14214.

Among other things, AGQM carries out annual unannounced sampling of its members as a quality assurance measure. Sampling without prior notice will ensure that the results are consistent with the actual operations of the producers and warehouse operators. This report summarizes the results of the samplings conducted in 2018. Furthermore, the measured quality parameters and their significance in the biodiesel production process are briefly explained.

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 irregularities (violation of a limit or acceptance limit) in a main campaign must take part in an additional campaign. Furthermore, it is specified that all quality parameters listed in the statutory requirement of the 36th BImSchV (Federal Emission Control Ordinance) for the proof of biofuel properties are examined.

Sampling and analysis are performed by an independent laboratory accredited for biodiesel analysis. The analyses are based on the valid version of DIN EN 14214. In 2018, the required standard limits as well as the associated acceptance limits resulting from the precision of the respective method, complied with DIN EN 14214:2014. The annex (5.1) lists the parameters to be tested with their limit values according to DIN EN 14214 in table 2. For the parameters water content, total contamination and cold filter plugging point (CFPP), the AGQM places higher demands on the biodiesel quality of its members than required by law, which document the special quality requirements of the AGQM. Table 3 (annex 5.1) shows the parameters with AGQM limits. Due to the market development in recent years, quality parameters for so-called blend components for biodiesel have been defined by AGQM, which allow a deviation from the standard limit values of DIN EN 14214 for certain products. Further details can be found under point 3. The deviant limit values for blend components for biodiesel are listed in the annex in table 4.

In 2018, 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:	08. January to 19. January	Winter grade
C2:	04. June to 15. June	Summer grade
C3:	22. October to 02. November	Intermediate grade

A total of 54 samples in the main campaigns and 16 samples in the resulting additional campaigns were taken and analysed.

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 must 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 Results of the sampling and evaluation

In the following section you will find the test method, the limit value according to DIN EN 14214, if applicable the AGQM limit value, the acceptance limit value as well as a description of each parameter. This is followed by a graphical representation of the measured values and their evaluation.

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 rejection limits are decisive. The total contamination and water content graphs also show the stricter AGQM limit and the AGQM acceptance limit.

In addition to the 16 biodiesel producers, one warehouse operator participated in AGQM's annual sampling.

Blend Components for Biodiesel

In order to improve the greenhouse gas balance and to support the circular economy, increasing amounts of alternative raw materials, e.g. used cooking oils and fats (UCO) and fatty acids are used for the production of biodiesel. AGQM also supports producers of such products in the context of quality assurance. Biodiesel from these raw materials is used exclusively as a blend component for biodiesel from classic raw materials (especially rapeseed oil) and is not marketed as neat fuel. Since the mixing of such blend components with other goods results in an overall standard-compliant biodiesel, there is scope for specification.

In 2013, a special regulation for blend components for biodiesel was created in the AGQM QM system. Initially, these fuels were exempted from the determination of the parameters sulphur content, CFPP and cloud point, if the producer had previously requested a corresponding exemption. These three parameters are strongly dependent on the fatty acid composition or impurities in the raw material and can hardly be influenced in the production process. Therefore, limit violations were not sanctioned for the above parameters in the case of blend components for biodiesel.

In autumn 2017, a separate chapter was implemented for blend components for biodiesel in the QM system, which includes specific AGQM limits for sulphur content, CFPP and cloud point, which will be reviewed during the unannounced sampling. In the diagrams, the corresponding samples are marked with an X. An overview of the specific limit values for blend components for biodiesel can be found in annex 5.1 in table 4.

3.1. Fatty Acid Methyl Ester Content

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

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 [% (w/w)]. EN 14214 requires an ester content of at least 96.5% (w/w). 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.

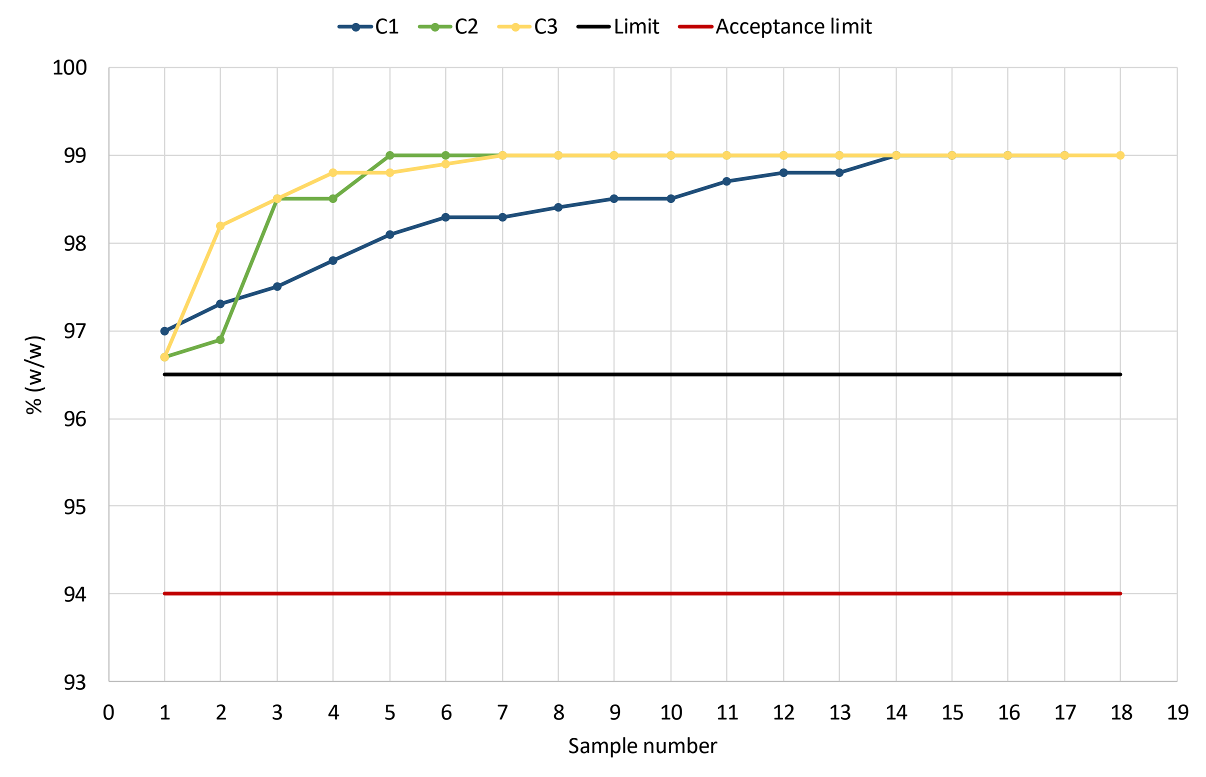

Diagram 1: Fatty Acid Methyl Ester Content acc. to DIN EN 14103.

3.2. Density at 15 °C

Test method:	*DIN EN ISO 12185:1997*
Limit of DIN EN 14214:2014:	*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.

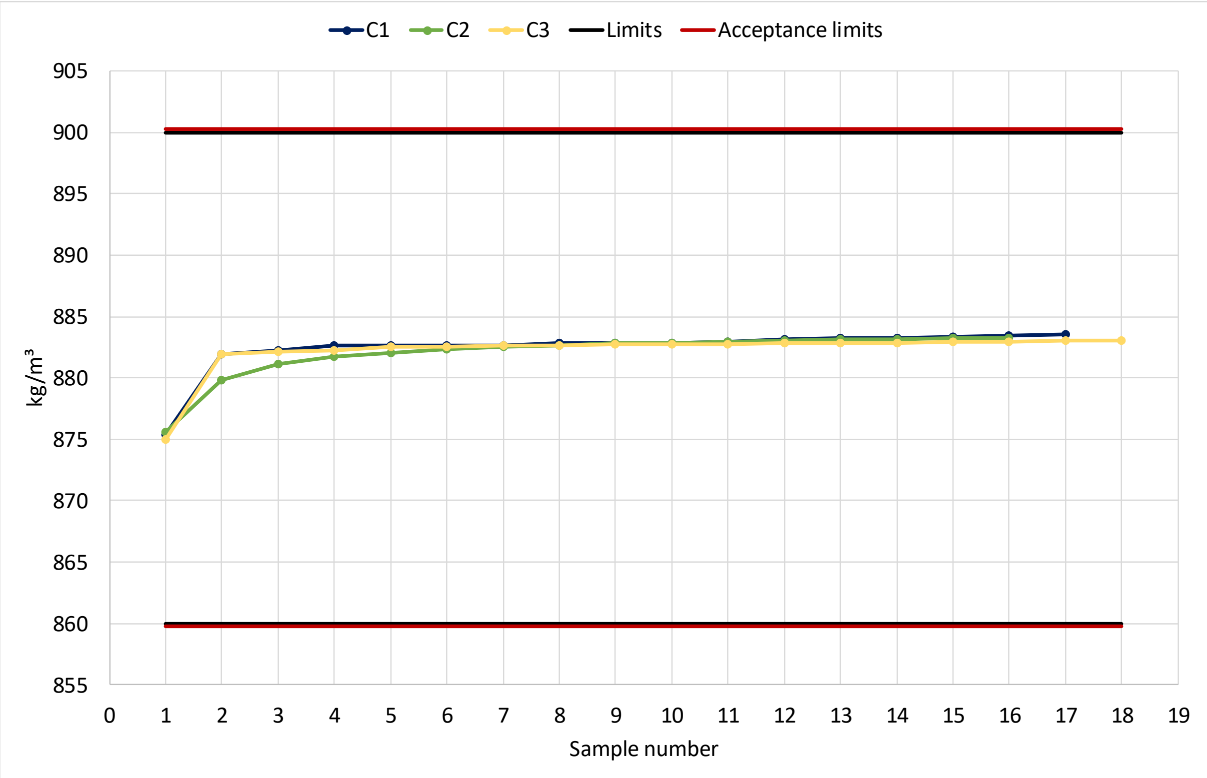

Diagram 2: Density at 15 °C acc. to DIN EN ISO 12185.

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 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.

3.3. Sulphur Content

Test method:	*DIN EN ISO 20846:2011*
Limit of DIN EN 14214:2014:	*max. 10 mg/kg*
Acceptance limit:	*max. 11.3 mg/kg*
AGQM limit for blend components for biodiesel:	*max. 13 ppm*
AGQM acceptance limit for blend components for biodiesel:	*max. 14.9 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 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.

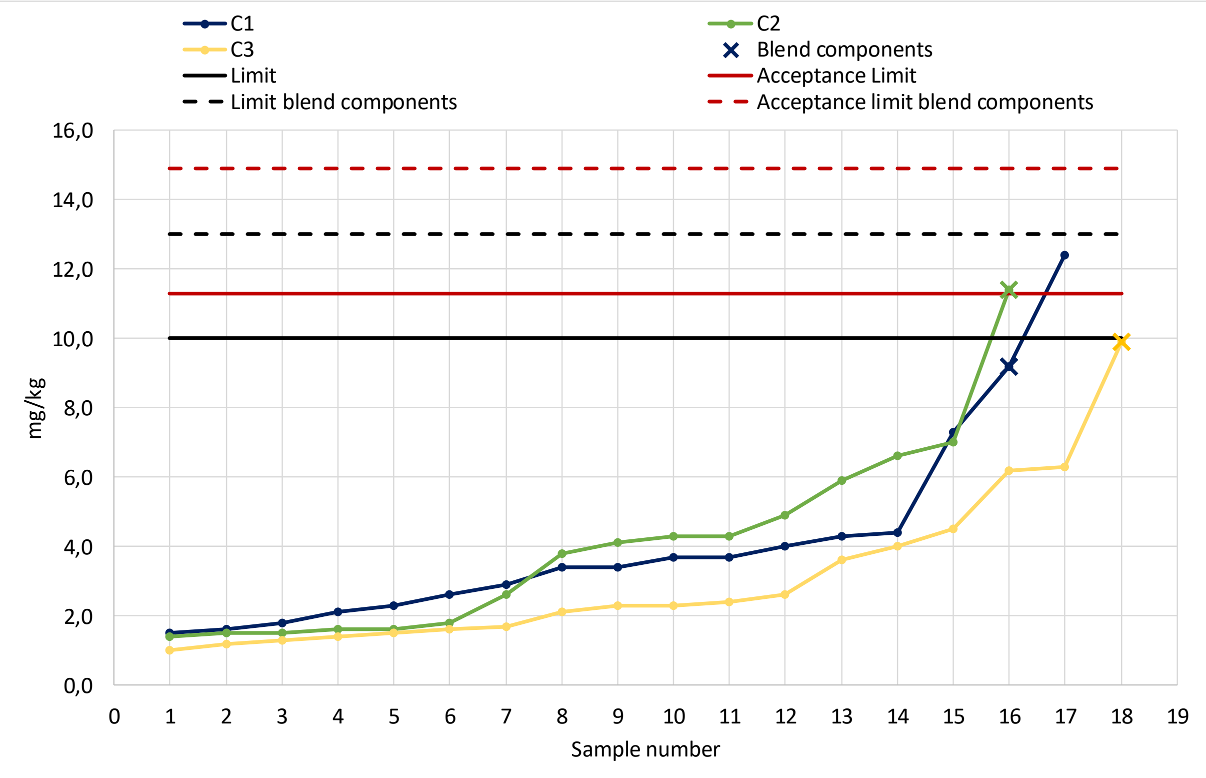

Diagram 3: Sulphur Content acc. to DIN EN ISO 20846.

As can be seen in Diagram 3, all but one sample comply with the required limits. A sample in C1 exceeds the limit (10 mg/kg) at 12.4 mg/kg outside the acceptance limit (11.3 mg/kg). This value is the result of an arbitration analysis. The member received a sanction point for the exceeding of the limit and showed no deviation in the next campaigns. The samples marked with an X are blend components for biodiesel, for which according to the QM systemdeviating limit values apply. These products must not be marketed as pure fuel. All samples of blend components for biodiesel were within the specific limit.

3.4. Water Content

Test method:	*DIN EN ISO 12937:2002*
Limit of DIN EN 14214:2014:	*max. 500 mg/kg*
Acceptance limit:	*max. 591 mg/kg*
AGQM limit:	*max. 220 mg/kg for producers*
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 must be dried as last step 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 absorb only very small amounts of water, so when mixing with biodiesel, 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. However, in Campaign 1 and Campaign 3 there were one each and in Campaign 2 two samples that exceeded the AGQM limit of 220 mg/kg but with a maximum of 257 mg/kg well below the AGQM rejection limit of 280 mg/kg.

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

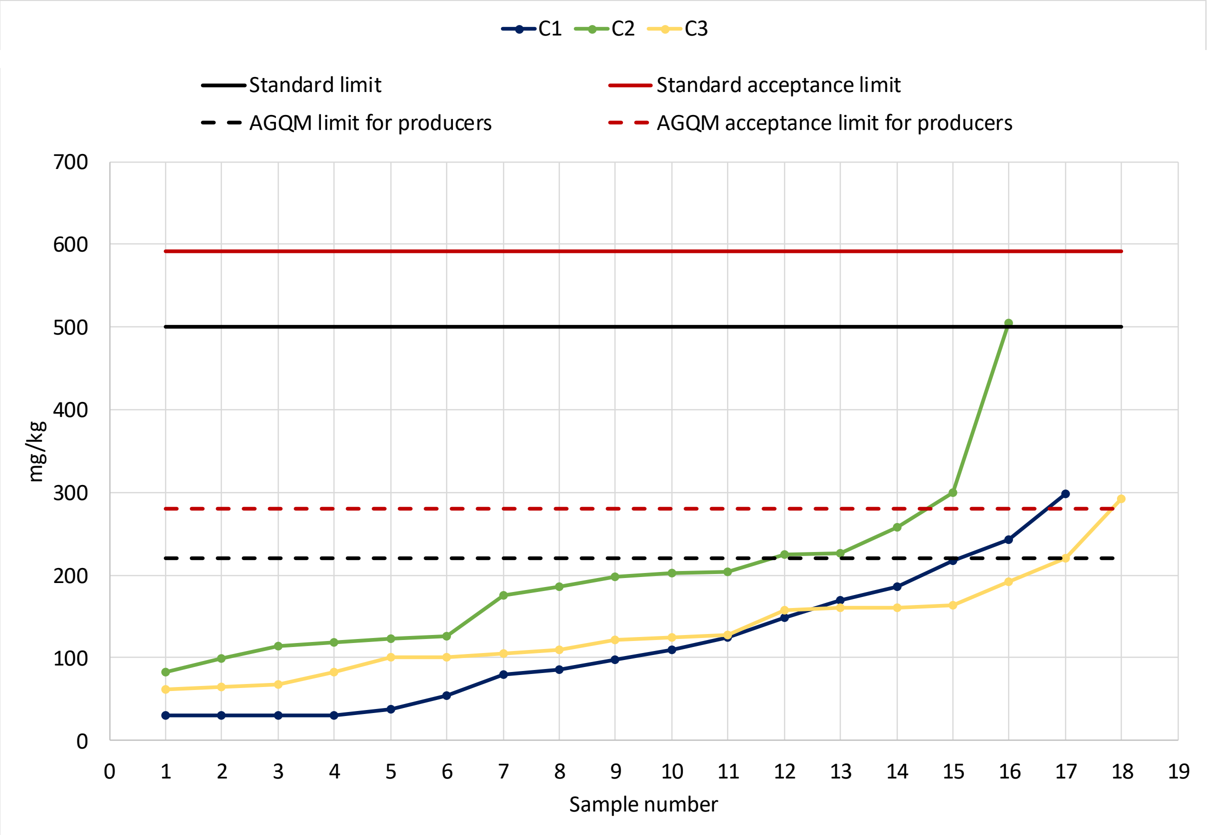

Diagram 4: Water Content acc. to DIN EN ISO 12937.

3.5. Total Contamination

Test method: *DIN EN 12662:1998*

Due to the fact that the current version of DIN EN 12662:2014 is unsuitable for 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 14214:2014: *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.

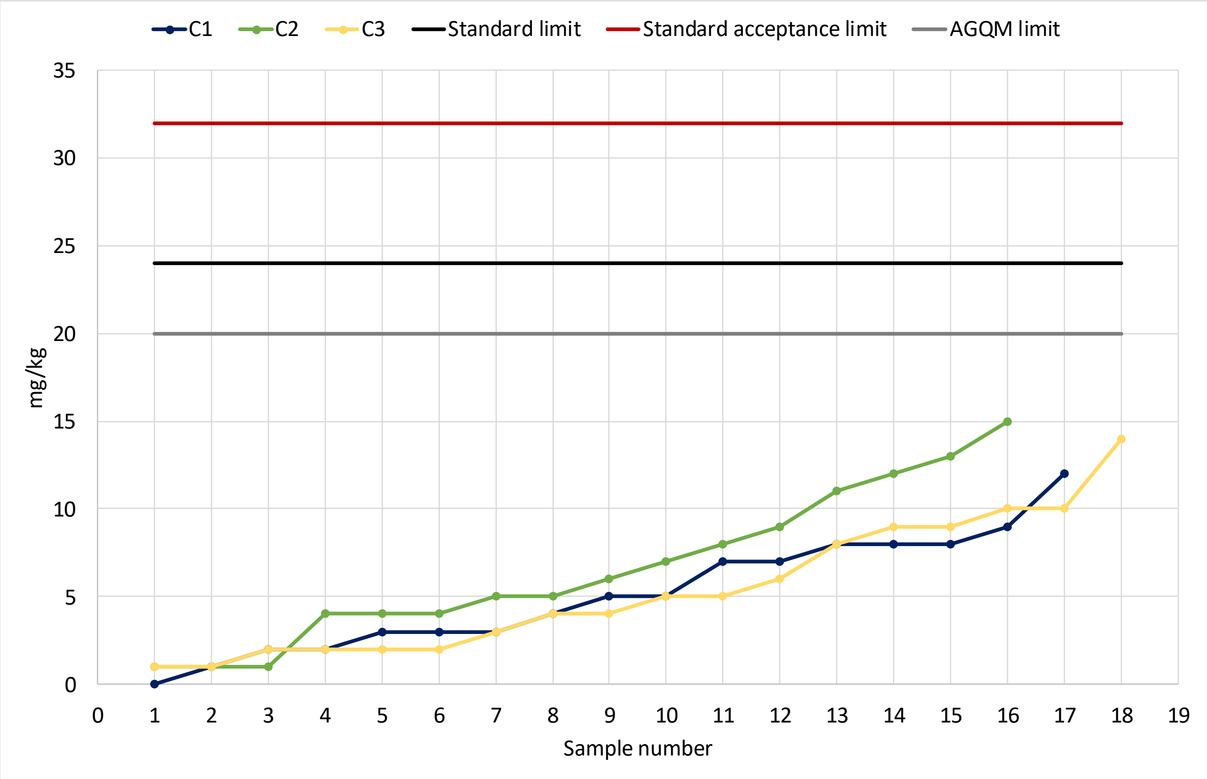

Diagram 5: Total Contamination acc. to DIN EN 12662.

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

3.6. Oxidation Stability

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

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 7.5 h within the acceptance limit (6.6 h).

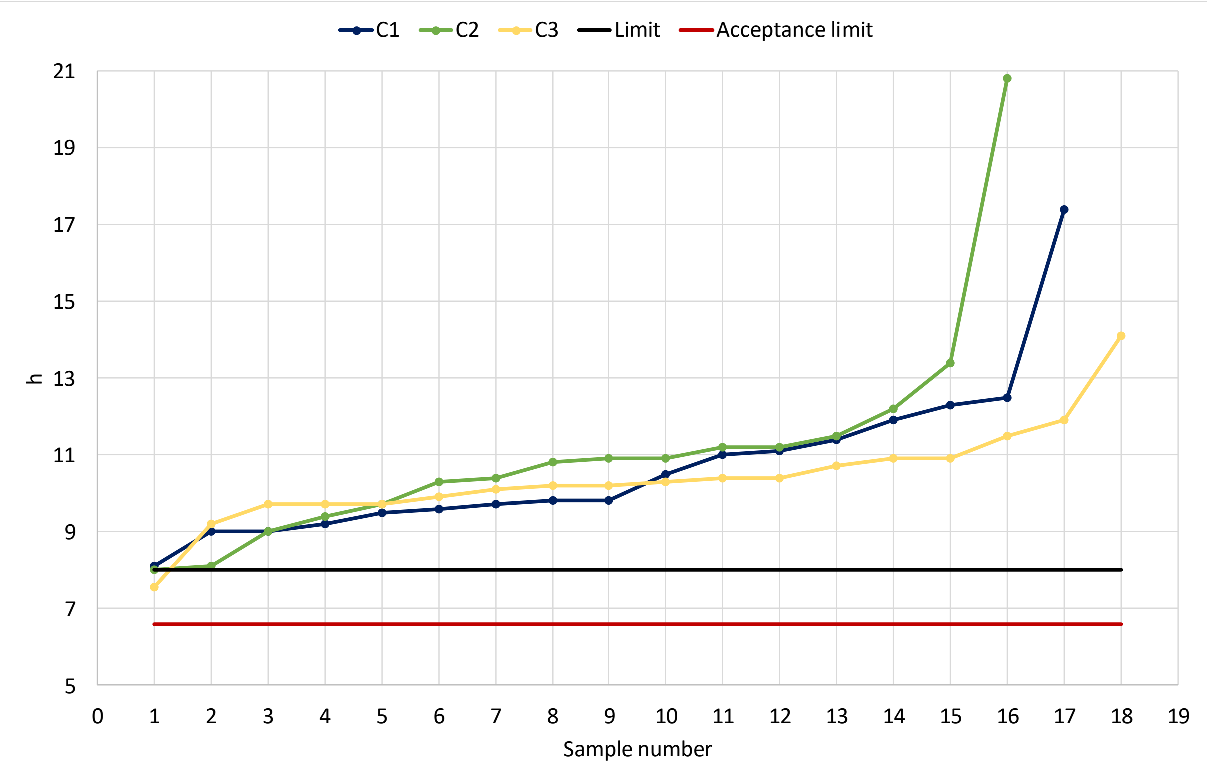

Diagram 6: Oxidation Stability acc. to DIN EN 14112.

3.7. Acid Number

Test method:	*DIN EN 14104:2003*
Limit of DIN EN 14214:2014:	*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.

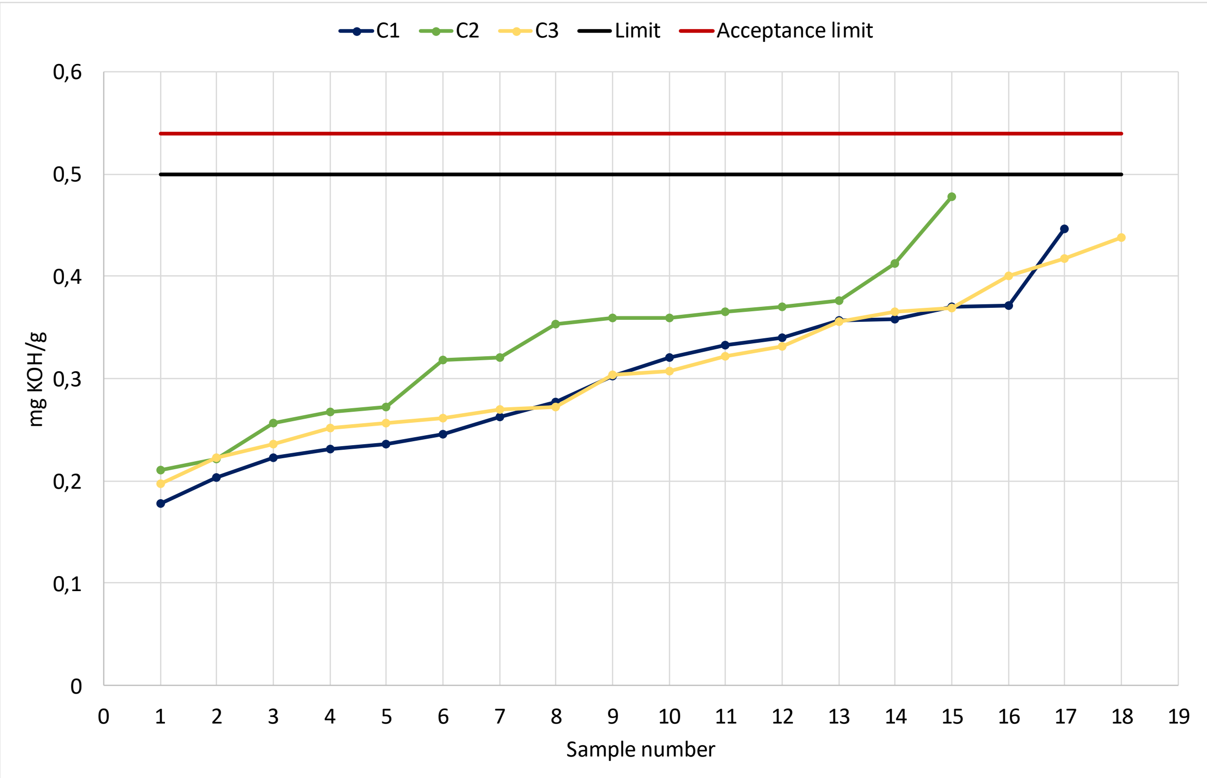

Diagram 7: Acid Number acc. to DIN EN 14104.

3.8. Iodine Number

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

The iodine 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.

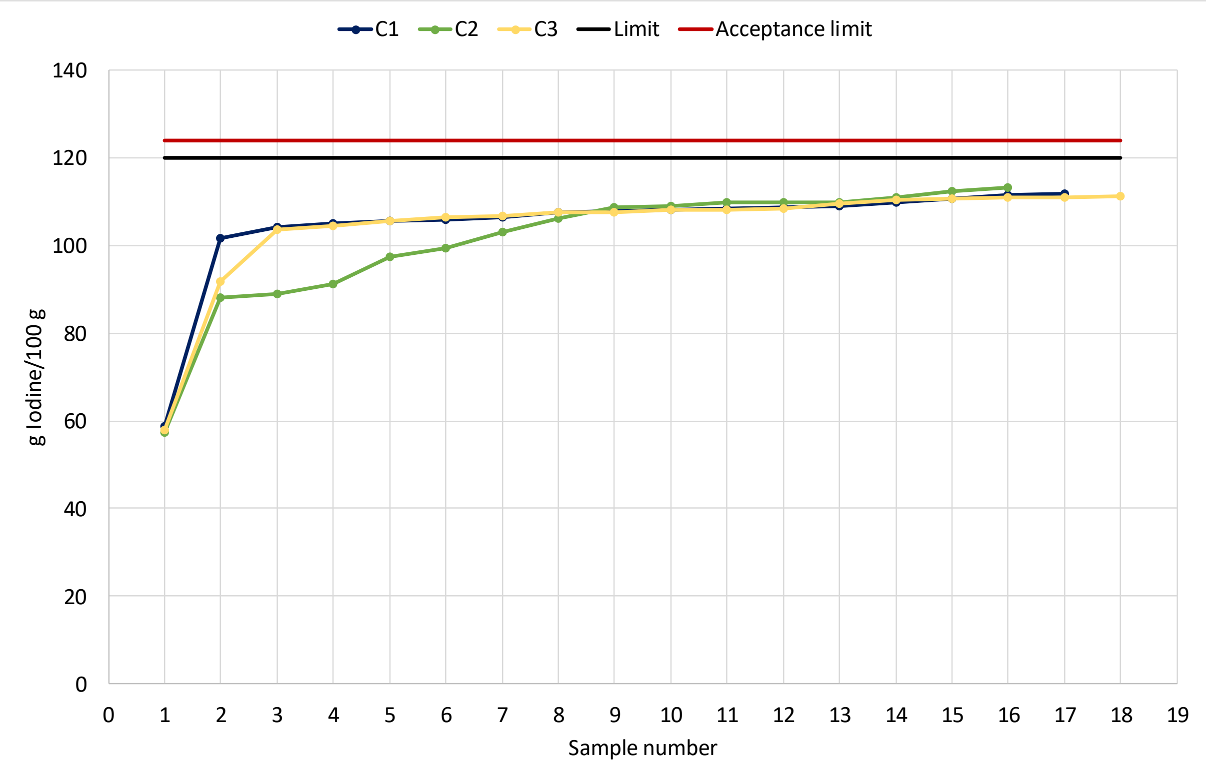

Diagram 8: Iodine Number acc. to DIN EN 16300.

Diagram 8 shows the results for the iodine number. All examined samples are below the standard limit value. It is noticeable that in the second campaign in part lower iodine numbers are measured, which suggests the use of raw materials with a higher degree of saturation. A high degree of saturation causes poor cold properties (with regard to CFPP and cloud point), which is of less importance in the summer months. Three samples show iodine numbers below 60 g iodine/100 g biodiesel all year round, which is also due to the raw material used.

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

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

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. In addition, 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% (w/w) than that for di- and triglycerides at 0.20% (w/w). 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 adjusts. The almost complete removal of the glycerides is possible only by distillation.

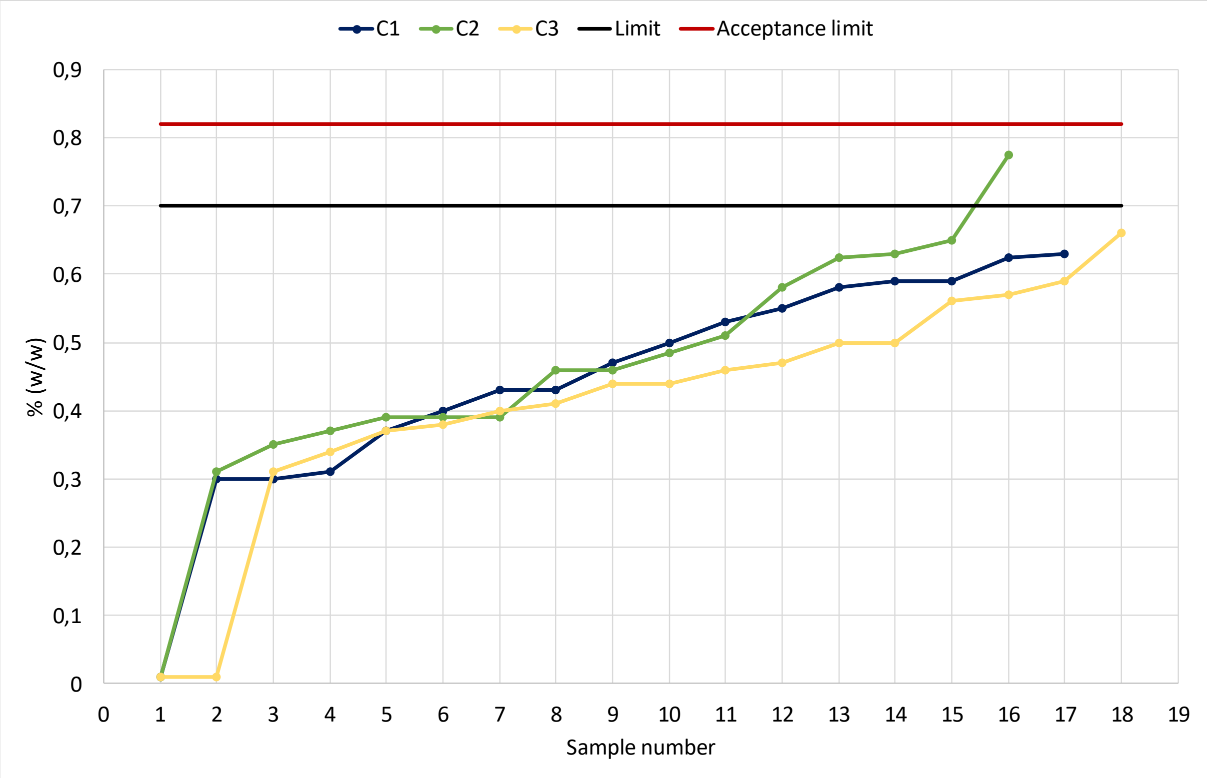

Diagram 9: Monoglycerides acc. to DIN EN 14105.

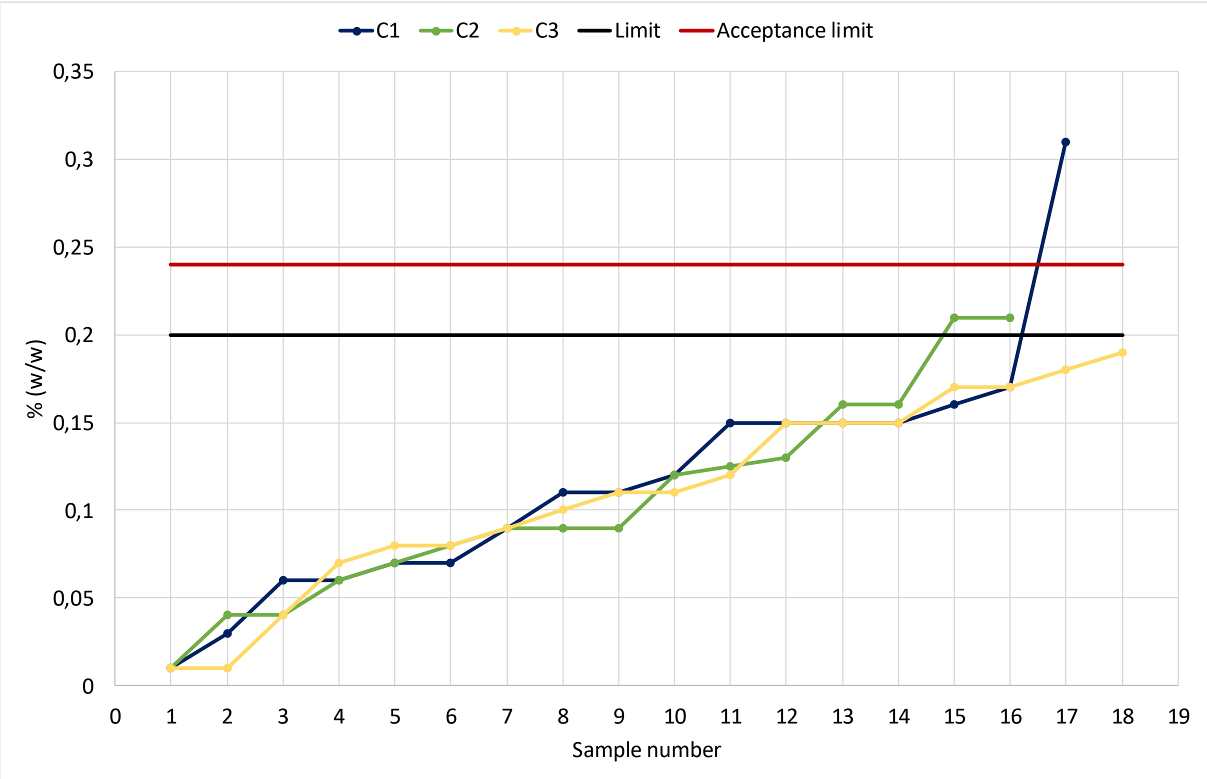

Diagram 10: Diglycerides acc. to DIN EN 14105.

Diagram 9 shows the measured values for the monoglycerides. One sample in Campaign 2 exceeds the limit (0.70% (w/w)) at 0.77% (w/w) within the rejection limit (0.82% (w/w)). All other samples are below the limit, and some samples even have values close to 0% (w/w), suggesting that the production process involves a distillation step.

Diagram 10 shows the measurements for the content of diglycerides. In Campaign 1, a sample at 0.31% (w/w) exceeds both the standard limit (0.20% (w/w)) and the rejection limit (0.24% (w/w)). The member had determined the excess itself and did not want an arbitration, therefore, a sanction point was assigned. In Campaign 2, two samples at 0.21% (w/w) exceed the limit within the acceptance limit.

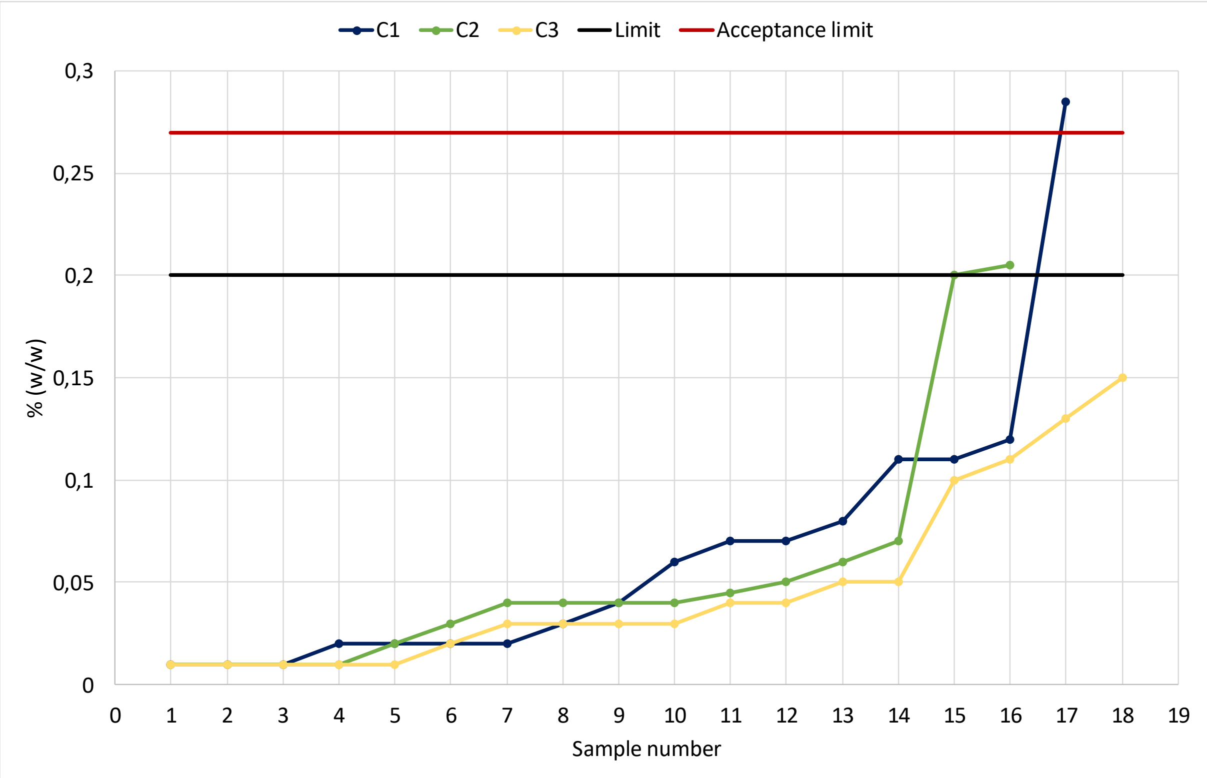

Diagram 11: Triglycerides acc. to DIN EN 14105.

Diagram 11 shows the results of triglyceride content measurements. In Campaign 1, a sample at 0.28% (w/w) exceeds both the limit (0.20% (w/w)) and the acceptance limit (0.27% (w/w)). The same member had also exceeded the limit for the diglycerides in Campaign 1 and did not want an arbitration test, which is why a second penalty point had to be awarded here. In Campaign 2, a member crossed the limit within the acceptance limit at 0.21% (w/w).

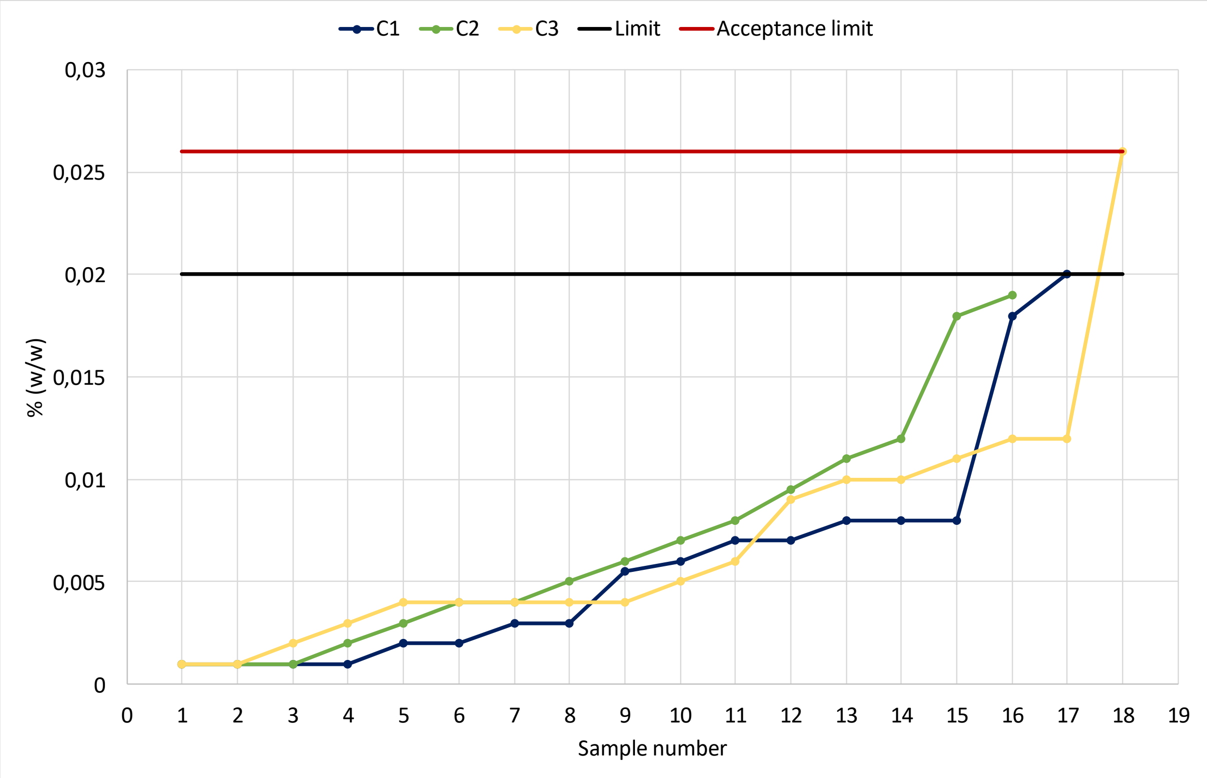

Diagram 12: Free Glycerol acc. to DIN EN 14105.

Diagram 12 shows the content of free glycerol. A sample in C3 exceeds the limit (0.020% (w/w)) at 0.026% (w/w) just within the acceptance limit (0.026% (w/w)). All other samples comply with the standard limit.

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

Test method:	*DIN EN 14538:2006*
Limit of DIN EN 14214:2014:	*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 laundry, 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 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) and the contents of the alkaline earth metals magnesium and calcium are even well below the determination limit of 1 mg/kg.

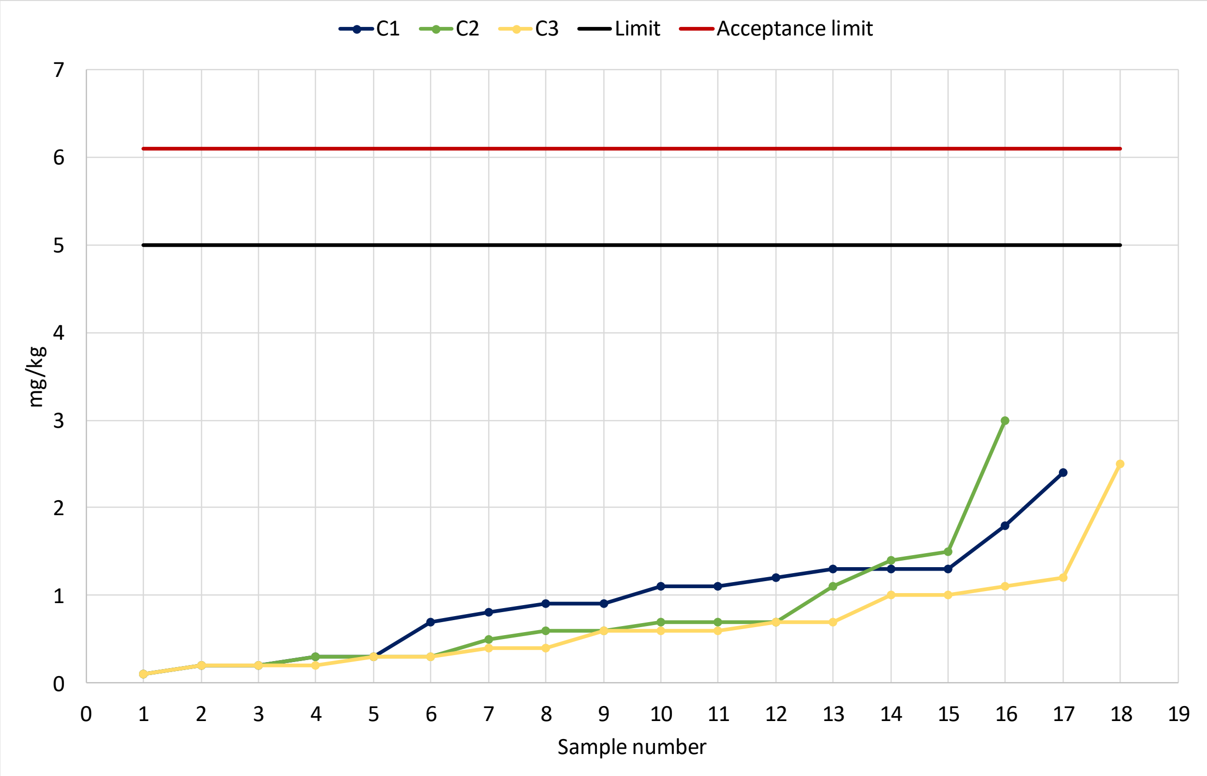

Diagram 13: Sum of Alkali Metals Sodium and Potassium acc. to DIN EN 14538.

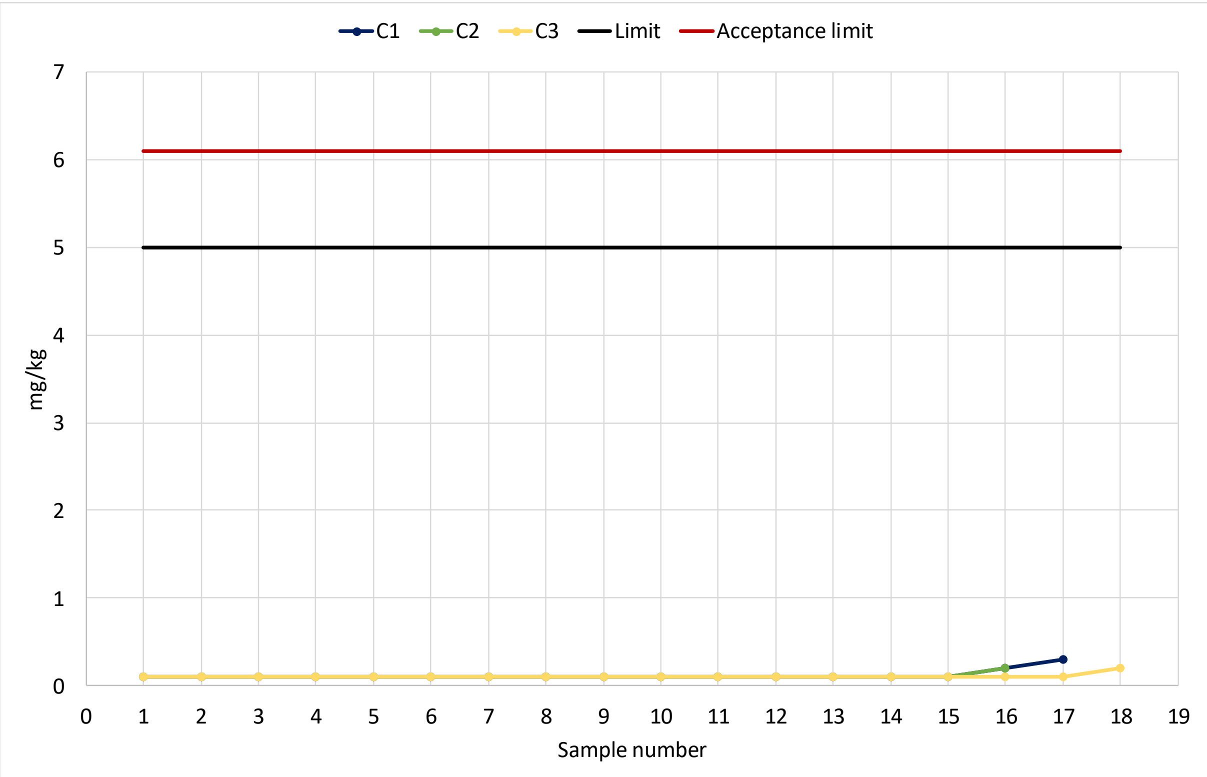

Diagram 14: Sum of Alkaline Earth Metals Calcium and Magnesium acc. to DIN EN 14538.

3.11. Phosphorous Content

Test method:	*DIN EN 14107:2003*
Limit of DIN EN 14214:2014:	*max. 4.0 mg/kg*
Acceptance limit:	*max. 4.5 mg/kg*

The phosphorus content must already be taken into account 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. All values lie with max. 1 mg/kg well below the limit.

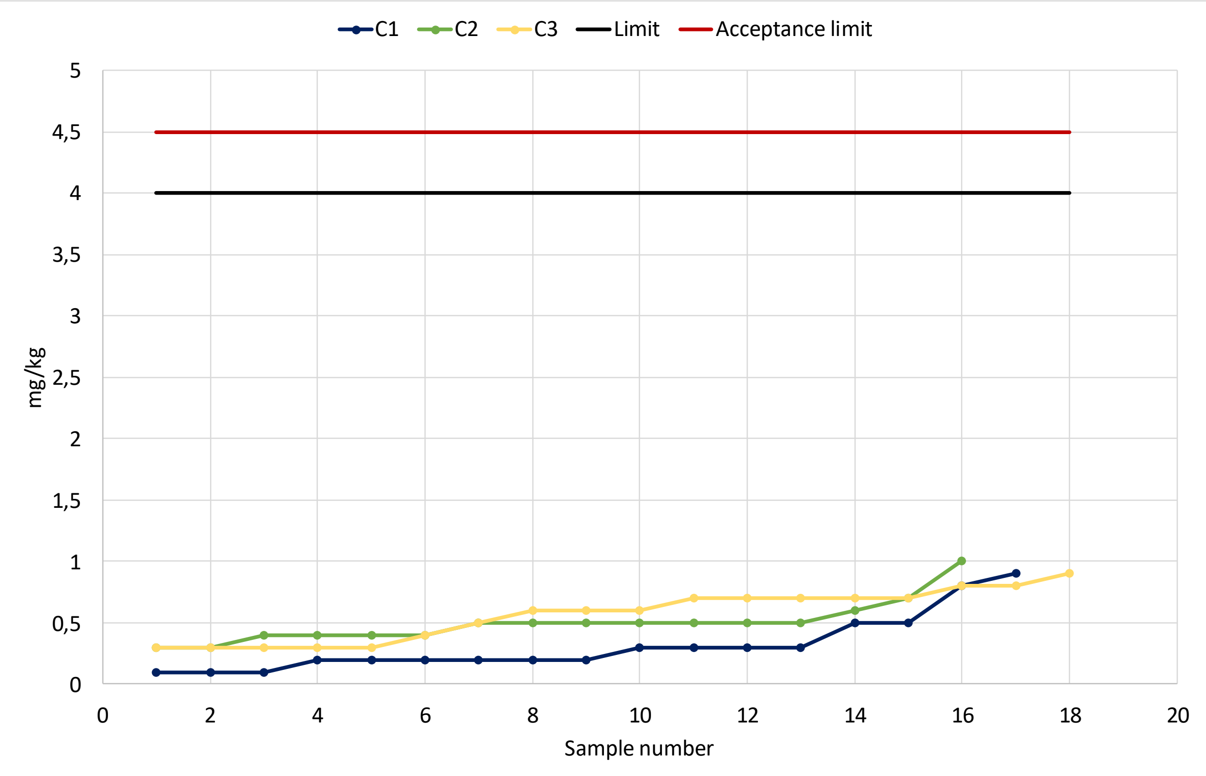

Diagram 15: Phosphorous Content acc. to DIN EN 14107.

3.12. Content of Linolenic Acid Methyl Ester

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

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% (w/w). It is determined from the fatty acid profile by gas chromatography.

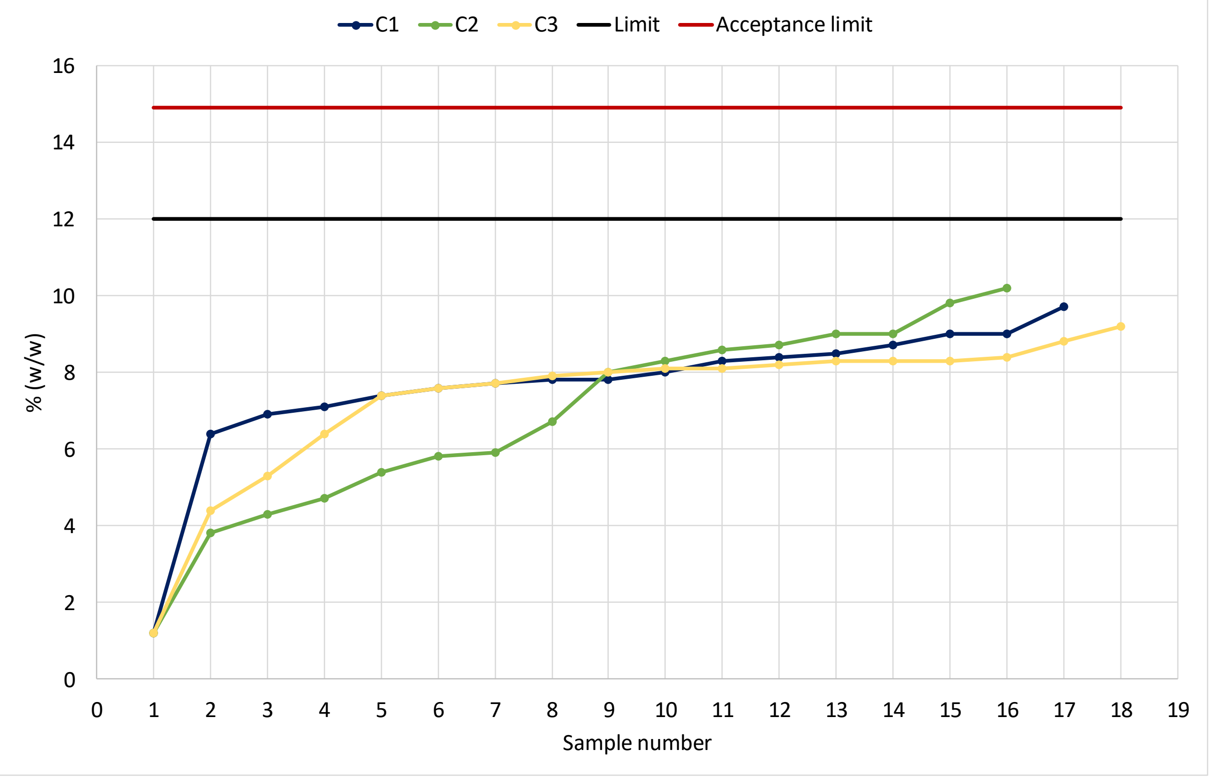

Diagram 16: Content of Linolenic Acid Methyl Ester acc. to DIN EN 14103.

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.

3.13. Cold Filter Plugging Point (CFPP)

Test method: *DIN EN 116:2015*
Limits according to DIN EN 14214:2014 for biodiesel as blend component in diesel fuel:

Period	*Limit*	*Acceptance limit*	
from 15.04. to 30.09.	*0 °C*	*+1.8 °C*	*Summer period*
from 01.10. to 15.11.	*-5 °C*	*-3.1 °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*	*-3.1 °C*	*Intermediate period*
AGQM limit for blend components for biodiesel	*+10 °C*	*+10.9*	*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. must comply with a CFPP of -10 °C, if the -20 °C required in DIN EN 14214 can be achieved by adding additives. The additivation then takes place in the refineries of the oil companies for the mixture of diesel fuel and biodiesel. 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 plots 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, three samples exceed the winter limit (-10 °C) at -9 °C, but 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 deviating limit values apply. These products must not be marketed as pure fuel. All analysed blend component samples are within the specific limit.

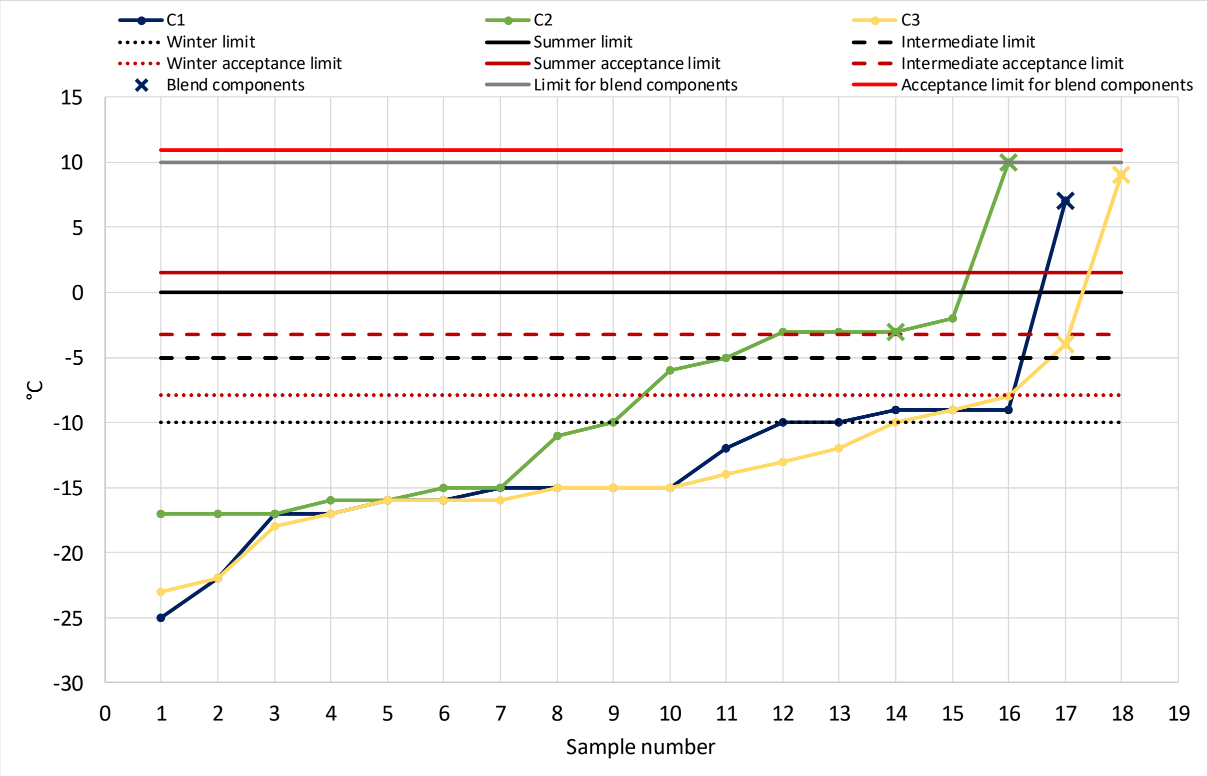

Diagram 17: CFPP acc. to DIN EN 116.

3.14. Cloud point (CP)

Test method: *DIN EN 23015:2013*

Limit according to DIN EN 14214:2014 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*	*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.

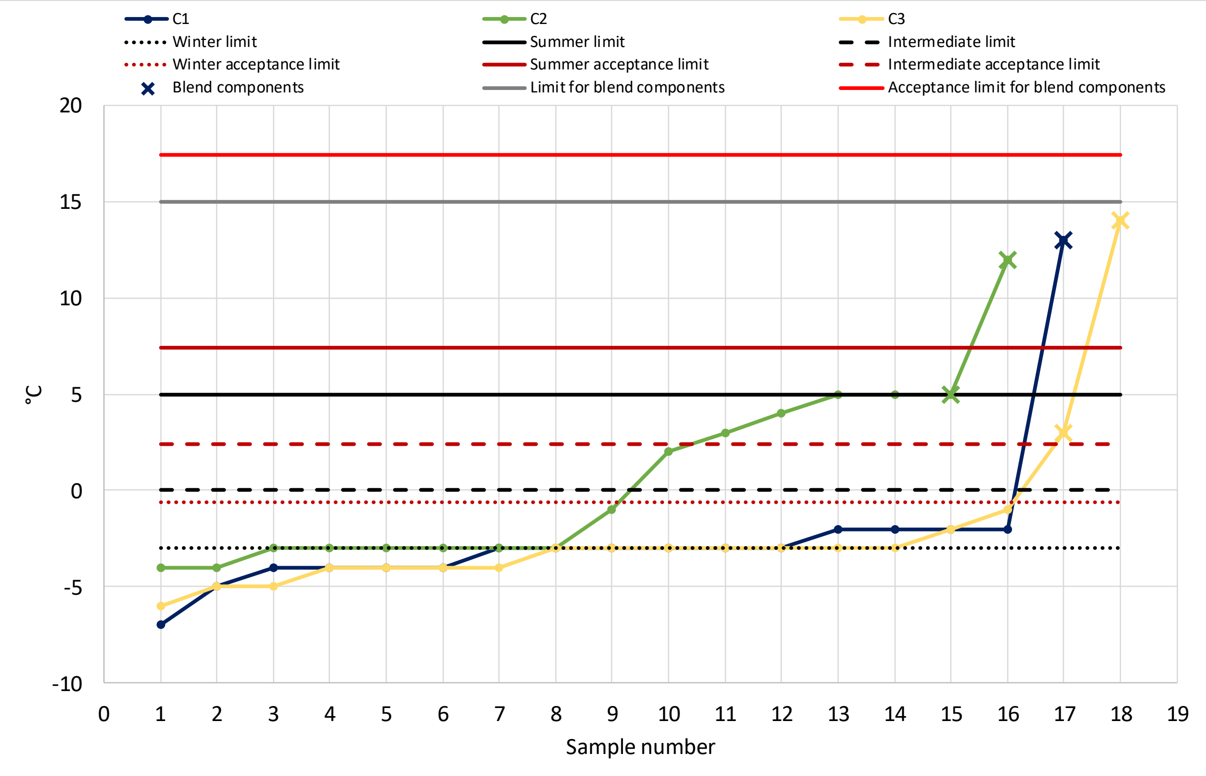

Diagram 18: Cloud point acc. to DIN EN 23015.

Diagram 18 shows the measurements for the Cloud point. In C1, four samples exceed the winter limit (-3 °C) at -2 °C, but within the acceptance limit (0.6 °C). The samples marked with an X are blend components for biodiesel, for which according to the QM system 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. In the three additional campaigns carried out, 16 samples were taken.

Additional Campaign 1 involved seven companies. Here, fortunately, no limit violations were found.

Additional Campaign 2 also sampled seven companies. Four members were found to have violations in the parameters water content, oxidation stability, diglyceride content and CFPP, all within the corresponding acceptance limit. One member, at 0.59 mg KOH/g, exceeded the limit for the acid number outside the acceptance limit (0.54 mg KOH/g). Since the member himself had determined the excess, no arbitration was requested and a sanction point was assigned. One member exceeded the AGQM limit and acceptance limit for total contamination (20 mg/kg) at 27 mg/kg, again no arbitration was requested, therefore, a sanction point was also assigned here. For one member, who was already conspicuous in previous campaigns, massive violations were found. The AGQM limit for water content for producers (220 mg/kg) was exceeded at 287 mg/kg outside the acceptance limit. Also, both the AGQM (20 mg/kg) and the standard limit (24 mg/kg) for total contamination were exceeded at 52.5 mg/kg well beyond the acceptance limit. In addition, the limit value for the alkali metal content (5 mg/kg) was exceeded at 9.2 mg/kg outside the acceptance limit. For these three limit violations, the member received 2 sanction points, which is the maximum number of sanction points per campaign, and the member was asked to clarify the issue.

The additional campaign 3 was attended by two companies. One member was found to have a high water content but still within the AGQM acceptance limit for producers. For the second member a sulphur level of 11.3 mg/kg was found, which is still within the acceptance limit (11.3 mg/kg). In addition, the member exceeded the limit for free glycerol (0.020% (w/w)) outside the acceptance limit (0.026% (w/w)). Since the member did not want to have an arbitration, a sanction point was assigned.

4 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.

Table 1 summarizes the limit violations in 2018. There were a total of 26 limit violations, with 9 outside the corresponding acceptance limit. A member who was responsible for a total of six limit violations (C1: di- and triglyceride content, C2: diglyceride content, AC2: water content, total contamination, alkali metal content) had to leave the AGQM on 31.12.2018.

Table 1: List of samples that have limit value violations in 2018.

Parameter	Method	Samples per campaign					
		C1	AC1	C2	AC2	C3	AC3
Sulphur content	DIN EN ISO 20846	1					1
Water content	DIN EN ISO 12937	1		3	2	1	1
Total contamination	DIN EN 12662				2		
Oxidation stability	DIN EN 14112			1	2		
Acid number	DIN EN 14104				1		
Monoglyceride content	DIN EN 14105			1			
Diglyceride content	DIN EN 14105	1		2	1		
Triglyceride content	DIN EN 14105	1		1			
Content of free glycerol	DIN EN 14105					1	1
Alkali metal content	DIN EN 14538				1		
CFPP	DIN EN 116	3			1		
Cloudpoint	DIN EN 23015	4					
Limit violations within the acceptance limit							
Violations of the acceptance limit of DIN EN 14214							
Violations of the AGQM acceptance limit							

It is noticeable that in the summer campaigns C2 and AC2 particularly many limit value violations occurred (altogether 17). One explanation is, that the extremely hot summer had an impact on the production and storage conditions. Another possible cause could be that, due to an increasingly difficult market environment, producers are forced to increasingly run their processes in the marginal area of profitability.

That is why it is all the more important that the producers in the AGQM continuously deal with the subject of product quality and quality management. Discrepancies are consistently uncovered through unannounced sampling, and AGQM assists concerned members with various measures (such as audits or coaching) in root cause research. In addition, statements are requested in the case of ongoing irregularities and the implementation of measures in the companies is accelerated. This procedure usually leads to inconspicuous members in follow-up campaigns. This ensures that products that are present on the market as AGQM products meet a high quality standard and that labelling is a reliable quality feature for customers and traders.

5 Annex

5.1 Limits and Test Methods

Table 2: 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	% (w/w)	96.5	-	94.0	-
Density at 15 °C	DIN EN ISO 12185	1997	kg/m³	860	900	859.7	900.3
Sulphur Content (UV)	DIN EN ISO 20846	2011	mg/kg	-	10.0	-	11.3
Water Content K.-F.	DIN EN ISO 12937	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	% (w/w)	-	12.0	-	12.4
Content of Free Glycerol	DIN EN 14105	2011	% (w/w)	-	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		2011	% (w/w)	-	0.70	-	0.82
Content of Diglycerides	DIN EN 14105	2011	% (w/w)	-	0.20	-	0.24
Content of Triglycerides		2011	% (w/w)	-	0.20	-	0.27
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 3: 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 4: 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.

5.2 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)
DIN EN 14214:2014	German Version of EN 14214:2012+A1:2014
EN	European Standard
FAME	Fatty Acid Methyl Ester
QM system	Quality Management System
TC	Technical Committee