Biodiesel Quality in Germany

Results of Sampling Operation of Manufacturer and Storage Operator of AGQM 2013

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1. Introduction

Biodiesel remains the key fuel based on renewable raw materials in Germany and will play an important role in the future for maintenance of mobility and in the fight against climate change. Considering the unstable political situation in some of the oil producing countries the aspect of security of supply gets into focus. Hence biodiesel can provide a fundamental contribution to decrease dependency in Europe.

The aim of AGQM (Arbeitsgemeinschaft Qualitätsmanagement Biodiesel e.V.) and associated biodiesel manufacturer and supplier is to merchandise a product that not only fulfills the minimum requirement standards of the norm but also complies with the demanding quality criteria that AGQM sets to provide fuel at its best for the customer. Especially blending of diesel fuel, which constitutes the substantial market, shows the advantage of the above mentioned approach: while lots of European countries complain about problems, in Germany it's practically running smoothly. The quality assurance actions, which are successfully implemented by the AGQM members since a long time of period, are based on the quality management system (QM-System) of the AGQM. An essential element of the system are quality controls on a regular basis of the members. The system is regularly revised by the board of quality assurance to cope with the permanent changes and constantly increasing requirements.

The members of the quality assurance committee are experts on this field and have been provided mostly by the AGQM firm members, but also by specialists of outside companies' e.g. Testing laboratories.

Todays produced biodiesel is exclusively put on the market as blend of B7 and approximately 90 % of the in Germany sold amount comes from AGQM members.

Over the years priorities of manufacturers have been shifted regarding the selection of raw materials for the biodiesel production. This shifting has different reasons:

On the one hand wild fluctuations of raw material prices, that forces the producers to disposition during raw material procurement and on the other hand the political frame conditions that manipulate and influence the raw material selection. While biodiesel was produced almost exclusively from rapeseeds in the past, today waste edible oils are used for production, since they will be credited twice regarding the quota obligation.

Meanwhile the results of the regular monitoring have created an important and internationally unique data base proving the positive development of the Biodiesel quality of AGQM's members. In 2011 the results of the unannounced sampling of AGQM members were published in a quality report[2] for the first time. They substantiate the high quality level of the Biodiesel volume marketed by all AGQM members.

2. Quality Assurance Action: unannounced Sampling of AGQM- Members

In 2013, six sampling campaigns (campaigns 1 to 6) were stipulated. AGQM does not execute any sampling and analytics but – following a tender – annually assigns it to a laboratory accredited for Biodiesel analytics, which must have successfully participated in the annual round robin test on fatty acid methyl ester (FAME), jointly organized by AGQM and "Fachausschuss für Mineralöl- und Brennstoffnormung (FAM) im DIN".

Biodiesel samples are taken unannounced from production plants and trading companies for testing with regard to the parameters stipulated in the Quality Management System, attachment A. Therefore the latest version of DIN EN 14214 forms the basis, although according to the stipulations of AGQM's QM System, more stringent limits apply for some parameters compared to those of the standard. Independent of the quality parameters laid down by legal stipulations in force – which refer to the 10. BImSchV – the current standard requirements always apply for AGQM quality checks.

In the appendix (table 2) the tested parameters with limit values are listed according to DIN EN 14214:2012. All parameters that have been changed since DIN EN 14214:2010 are marked in blue. Table 3 shows the required parameter of AGQM that are above the standard specification.

Regarding the sampling time points it has been considered, that the AGQM-members were sampled in summer and winter time, since different requirements are stipulated according to the parameter Cold Filter Plugging Point (CFPP) and Cloud Point. These requirements are determined in the national appendix of the norm and differ from country to country dependent of climatic conditions.

The campaigns are named C1 to C6. The following table shows the periods of sampling:

C 1:	11. February to 22. February	winter goods
C 2:	8. April to 19. April	intermediate- and summer goods
C 3:	21. May to 31. May	summer goods
C 4:	1. July to 12. July	summer goods
C 5:	16. September to 27. September	summer goods
C 6:	11. November to 21. November	intermediate- and winter goods

Altogether 108 biodiesel samples of AGQM members have been tested. Only 10 % of the samples did not fulfill the specifications.

3. Individual Results

The following results show, beside a short description of the parameters, the limit values given by the norm, the applied rejection limits, the applied test method and the graphical presentation of the measured data.

The results are anonymized and give no indication of the sample source. The samples get an internal number to be responsive to distinctive features. But only the numbers that indicate out of specification values will be mentioned in the report (table1).

For clarification of the distribution the values of the diagrams of each campaign are pictured in ascending order. Additionally the limit values are marked by a black line and the rejection limits are marked by a red line. Some of the graphics contain additional information: In the graphics of the parameter total contamination, water content and CFPP the AGQM limit values (table 3) will be marked by a grey line and the rejection limit values marked by a light red line.

Another noteworthiness, coming from a special regulation, is to find in the diagrams of sulfur content, CFPP and Cloud Point regarding the biodiesel production out of waste edible oils. The production of goods coming out of waste edible oils that are conforming to standards are extremely difficult and not always successful, hence there is the possibility to apply for an exception regulation at AGQM for the certain parameters. Biodiesel coming out of waste edible oils is not allowed to be released to the market directly. It has to be blended with goods conforming to standard before and therefore will be marked in the graphics of this report.

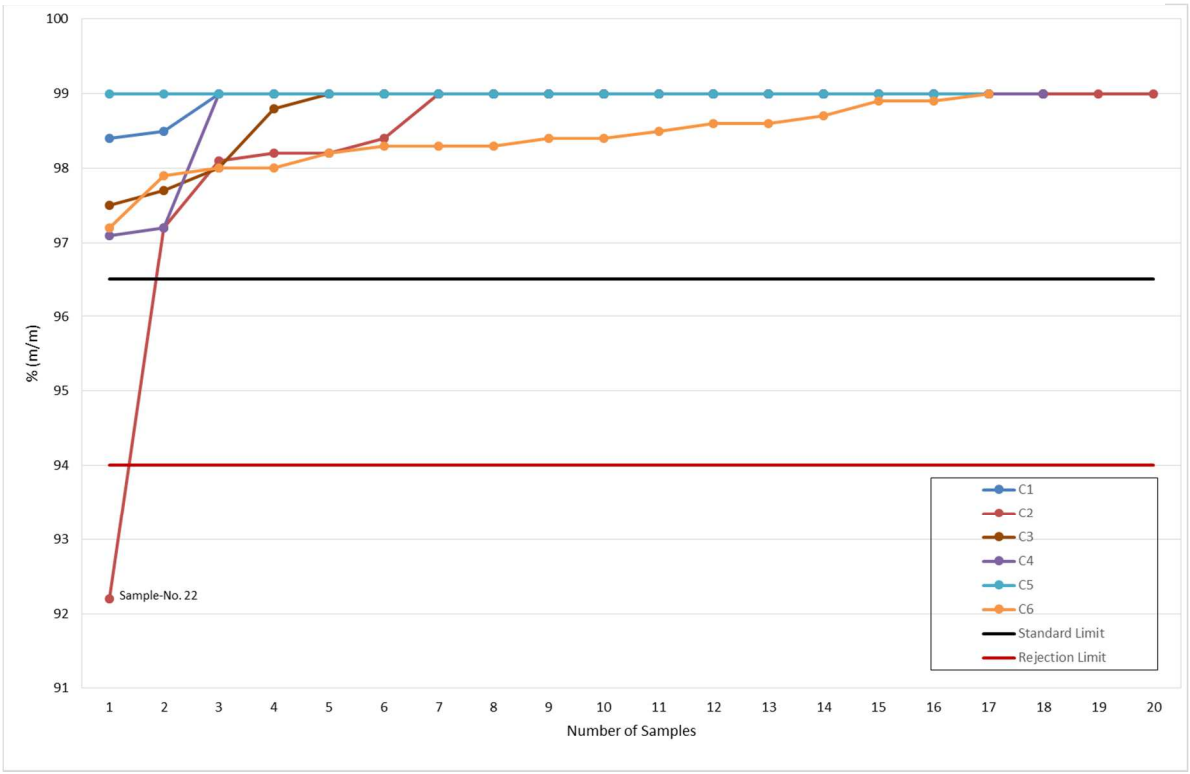
A comment is added to each parameter especially regarding deviations and abnormal characteristics.

3.1 Content of Fatty Acid Methyl Ester („FAME“)

Test method: DIN EN 14103:2011
Limit: DIN EN 14214:2012: ≥ 96.5 % (m/m),
Rejection limit at least: 94.0 % (m/m)

The content of fatty acid methyl esters – usually abbreviated ‘ester content’ – is an indication of the degree of transesterification and the purity of the Biodiesel; the higher the value, the better the quality.

Figure 1: Content of Fatty Acid Methyl Ester DIN EN 14103



Measured values given as > 99.0 % are illustrated in the graph (fig.1) as ester content of 99%. Sample 22 derives from a member that repeatedly did not fulfill the quality standards for the produced goods, which will also be shown for parameters of sample 4 and 42 from the same member. The reason for that could not yet be clearly identified and the member resigned of the AGQM in 2013

3.2 Density at 15 °C

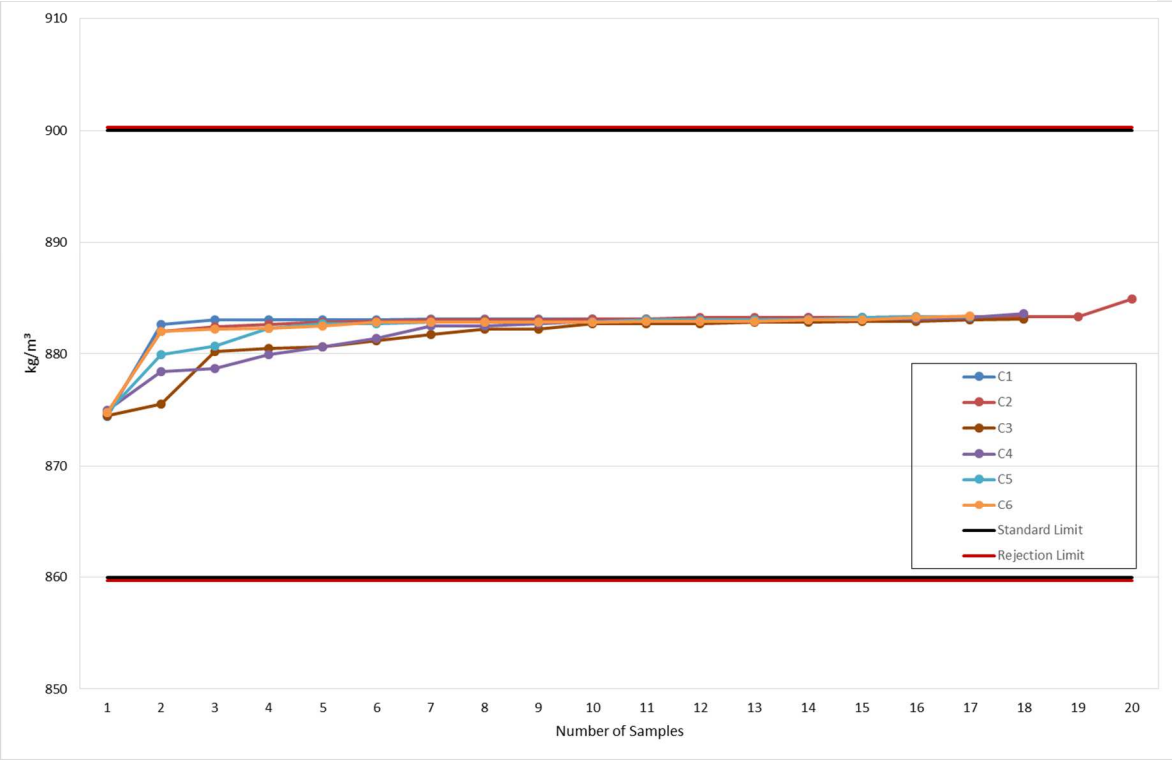
Test method: DIN EN ISO 12185:1997

Limit: DIN EN 14214:2012: between 860 and 900 kg/m³

Rejection limit at least: 859.7 kg/m³; Rejection limit at the most: 900.3 kg/m³

The density of a substance is the quotient of its mass and its volume at a certain temperature; it is a property specific for a substance.

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



Density of the samples occur in a very tight range and show a value of 883 kg/m³ (fig.2) in 2/3 of the tested samples. This might be the result of the comparatively tight fatty acid spectrum of the used raw materials.

3.3 Sulphur Content

Test method: DIN EN ISO 20846:2011
Limit: DIN EN 14214:2012: ≤ 10 mg/kg,
Rejection limit at the most: 11.3 mg/kg

While oil plants usually contain just traces of sulphur (with the exception of high erucic acid rapeseed), animal fats contain sulphur as accompanying substance of protein compounds which may get into the Biodiesel during the production process. Also, sulphur can get into the product when sulphur-containing catalysts are used for esterification. The following graph shows the sulphur values of the tested production samples.

Figure 3: Sulphur Content DIN EN ISO 20846

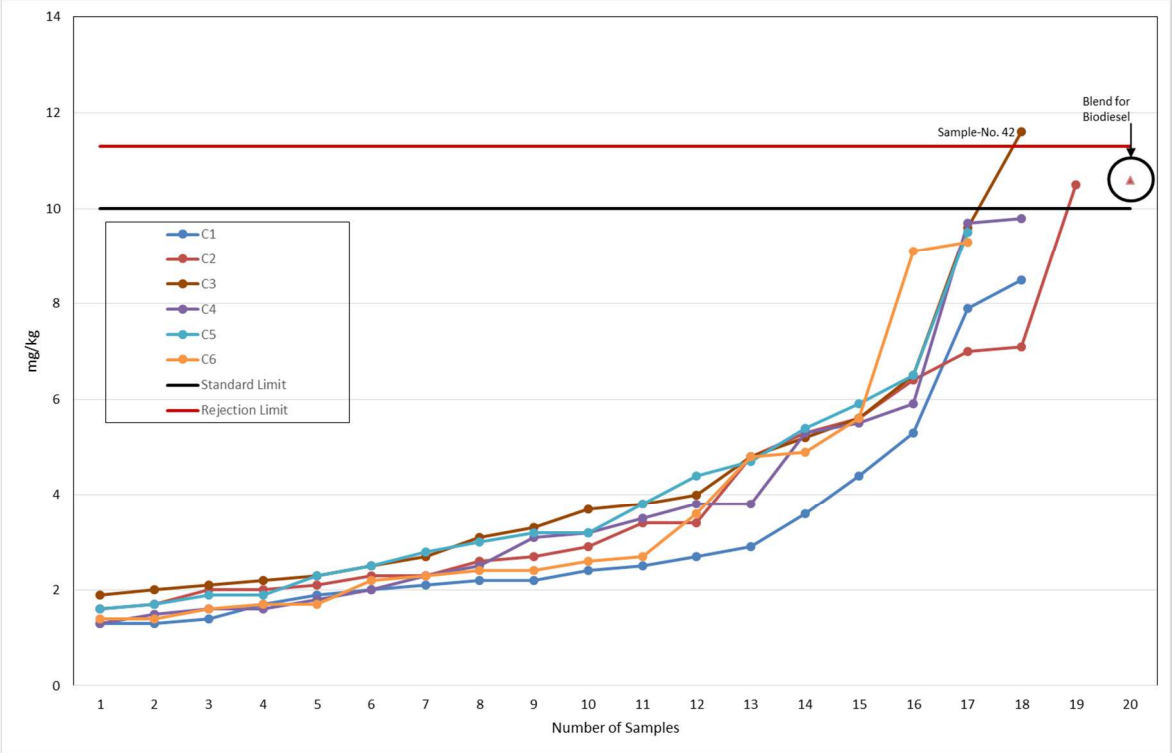


Fig. 3 shows, that 97 % of all tested samples comply with the sulphur limits. Only sample no 42 violates the limit, but the reason for that could not yet be clearly identified.

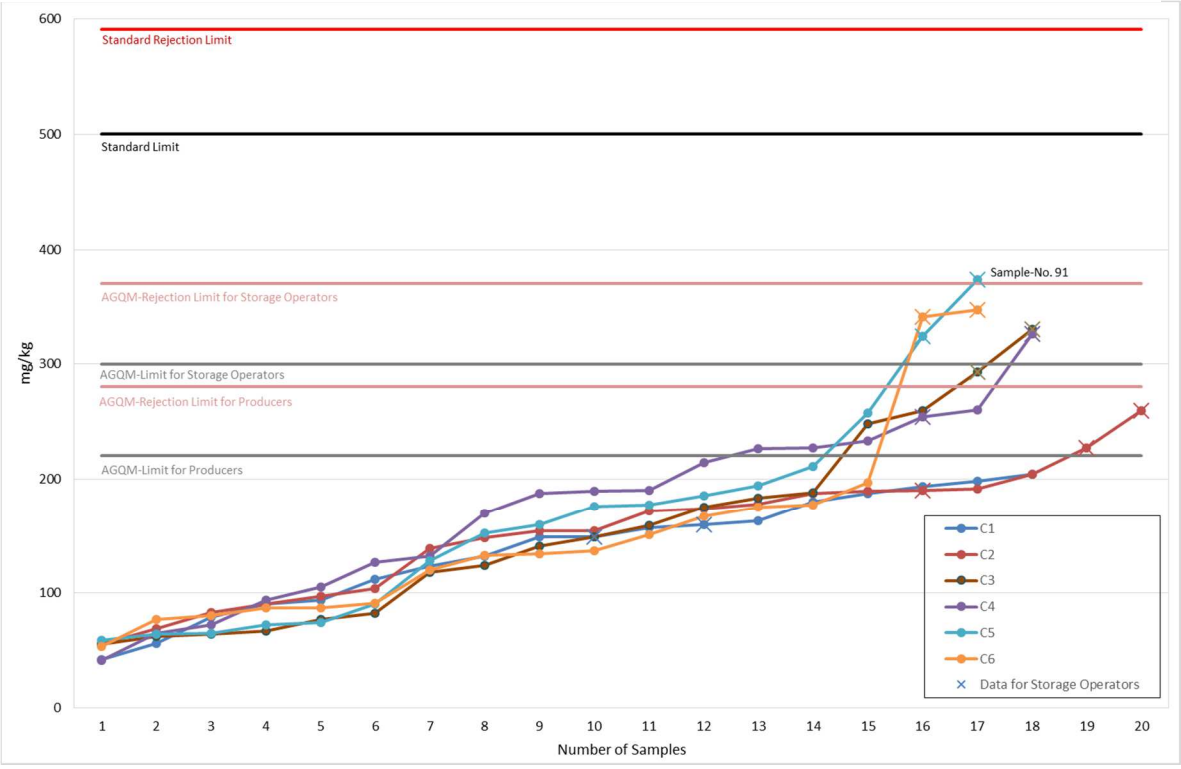
3.4 Water Content

Test method: DIN EN ISO 12937:2000
Limit: DIN EN 14214:2012: ≤ 500 mg/kg,
Rejection limit at the most: 591 mg/kg

Limit AGQM: ≤ 220 mg/kg for producers,
Rejection limit: 280 mg/kg
Limit AGQM: ≤ 300 mg/kg for storage operators,
Rejection limit: 370 mg/kg

Three limits must be considered for the assessment of the water content: first the maximum content of 500 mg/kg as stipulated by DIN EN 14214 and secondly the values defined by AGQM's quality management system with 220 mg/kg for producers and 300 mg/kg for storage operators. Bearing in mind that Biodiesel is hygroscopic, the rising water content along the transport chain is thus taken into consideration.

Figure 4: Water Content DIN EN ISO 12937



All tested samples show values below the standard specification limit (fig.4). 99 % even fulfill the more restricted AGQM requirements. Only sample no 91 exceeds the rejection limit for storage operators and therefore the goods are not allowed to be released to the market as AGQM declared goods. Nevertheless it can be released to the market as standard compliant goods.

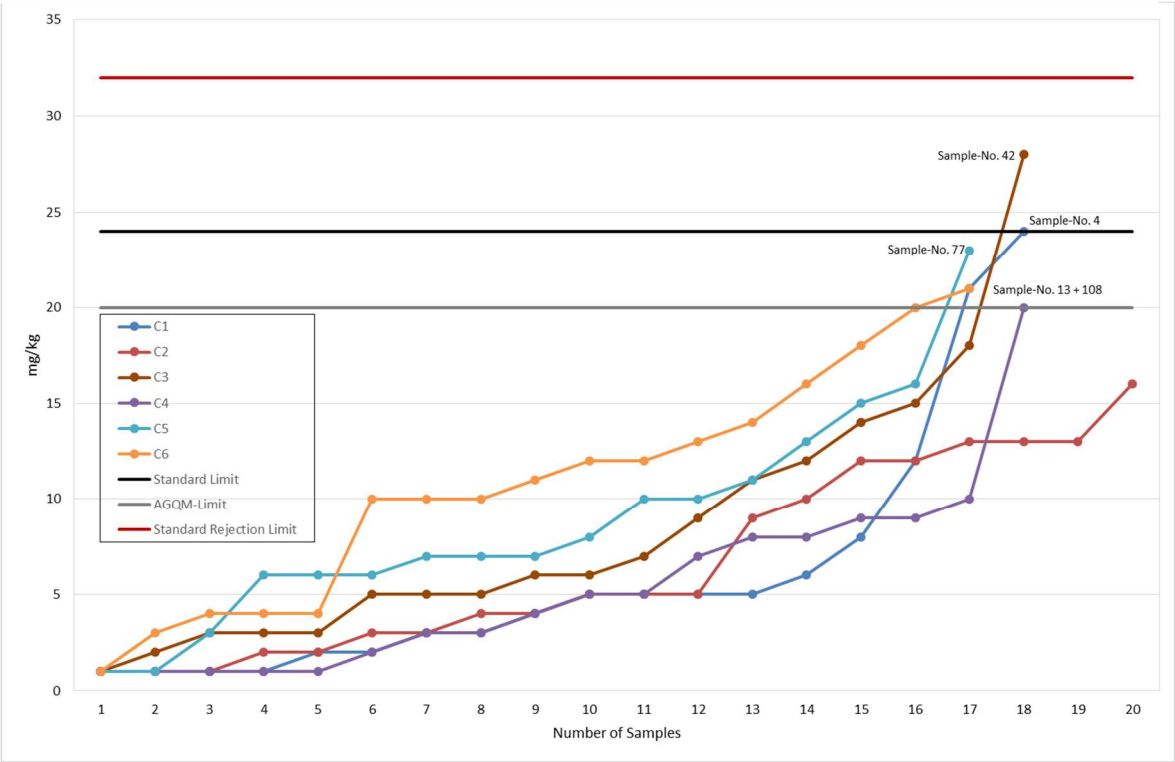
3.5 Total Contamination

Test method: DIN EN 12662:1998
Limit: DIN EN 14214:2012: ≤ 24 mg/kg,
Rejection limit at the most: 32 mg/kg
Limit AGQM: ≤ 20 mg/kg (The AGQM limit for total contamination already functions as rejection limit.)

Due to the fact that the current version of DIN EN 12662 is not suitable for the determination of the total contamination, DIN EN 12662:1998 is used by AGQM for this sampling campaign subsequent to a recommendation by CEN TC 19 – JWG 1 (CEN = European Committee for Standardization), dated 8 March 2012.

The total contamination is a measure for the volume of non-filterable, solid particles contained in Diesel or Biodiesel; it is determined gravimetrically by filtration and weighing of the filters. For that AGQM stipulated its own, more stringent limit of 20 mg/kg in order to improve the implementation security of Biodiesel and to account for the imprecision of the method.

Figure 5: Total Contamination DIN EN 12662



All samples but one (no 42) met the requirements of the DIN EN 14214 (fig. 5). Four samples (no 4, 13, 77 and 108) did not fulfill the AGQM limit, which therefore means that the goods are not allowed to be released to the market as AGQM declared goods. In all four cases quality assurance actions have been performed.

3.6 Oxidation Stability

Test method: DIN EN 14112:2003
Limit: DIN EN 14214:2012: ≥ 8 h,
Rejection limit at least: 6.6 h

The oxidation stability of Biodiesel is defined by its induction time. According to DIN EN 14214:2010 the stipulated time for oxidation stability is 6 h. In November 2012 the new revision DIN EN 14214:2012 was published in which the limit for the oxidation stability was raised to 8 h.

Figure 6: Oxidation Stability DIN EN 14112

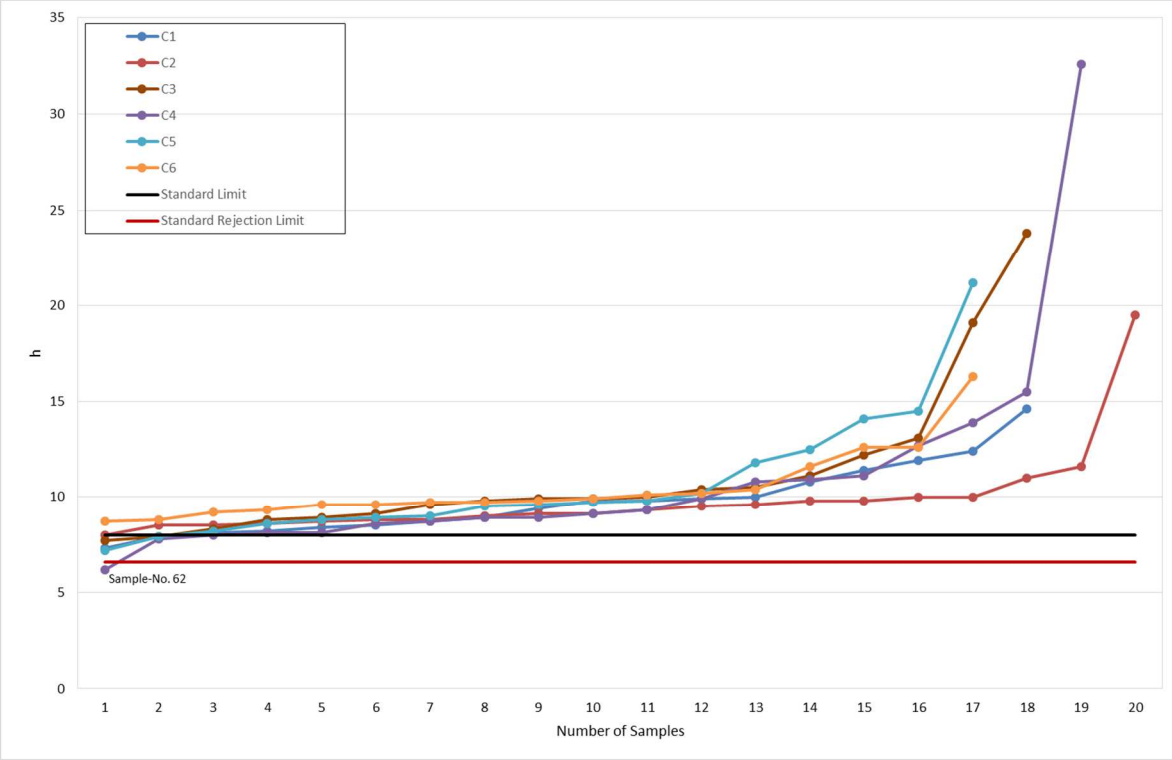


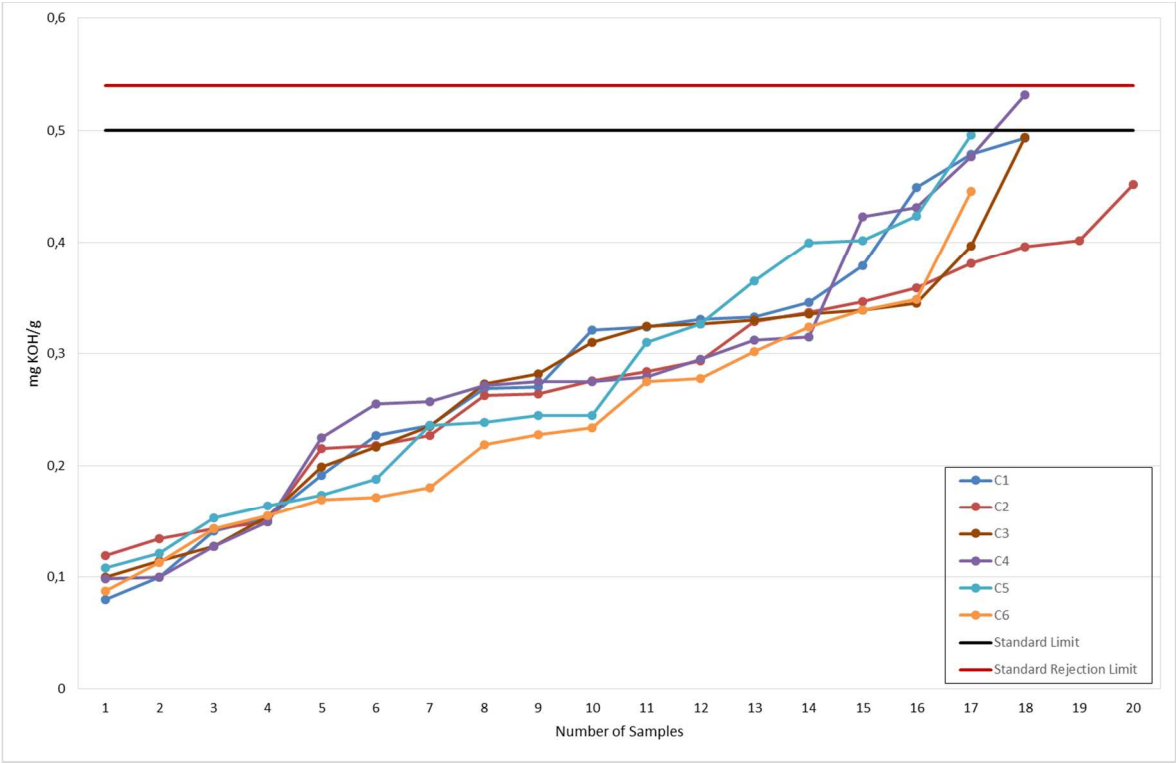
Figure 6 shows that the increased requirements to the oxidation stability were no problem for the members at all. Considering the precision of the test method only one sample (no 62) was not able to fulfill the tightened requirements to the oxidation stability of 8 h. With a value of 6,2 h no 62 still fulfilled the requirements of the DIN EN 14214:2010 and therefore was allowed to release the goods to the market.

3.7 Acid Number

Test method: DIN EN 14104:2003
Limit: DIN EN 14214:2012: ≤ 0.5 mg KOH/g,
Rejection limit at the most: 0.54 mg KOH/g

Free fatty acids in Biodiesel may cause corrosion and also form soaps with alkaline components which may result in gumming and filter plugging. In addition, caused by the Biodiesel's ageing, short-chain carboxylic acids (formic acid, acetic acid) may form which have an even stronger corrosive effect. Therefore, the limit of DIN EN 14214 is stipulated at 0.5 mg KOH/g.

Figure 7: Acid Number DIN EN 14104



The result is pleasing. The limit of DIN EN 14214 was complied with in all cases (fig. 7).

3.8 Iodine Number

Test method: DIN EN 14111:2003
Limit: DIN EN 14214:2012: 120 g Iod/100g,
Rejection limit at the most: 123 g Iod/100g

Test method: DIN EN 16300:2012
Limit: DIN EN 14214:2012: 120 g Iod/100g,
Rejection limit at the most: 124 g Iod/100g

The Iodine number is a measure for the proportion of unsaturated fatty acids in Biodiesel and is limited to 100 g of Iodine/100 g by DIN EN 14214. It is a generally accepted rule that the stability of Biodiesel decreases due to a rising number of double bonds – thus also the increasing iodine consumption. Therefore, along with the oxidation stability, the iodine number is considered indicator for the stability of biodiesel.

This parameter can either be measured with DIN EN 14111, being a wet-chemical method (titration), or it can be calculated based on its methyl ester composition according to DIN EN 14214, annex resp. DIN EN 16300. The calculation method is described in the annex of DIN EN 14214:2010. Upon publication of DIN EN 14214:2012, DIN EN 16300 was also published, replacing the annex for the calculation of the iodine number. The determination was carried out with both methods.

Figure 8: Iodine Number DIN EN 16300 (calculated based on its methyl ester composition)

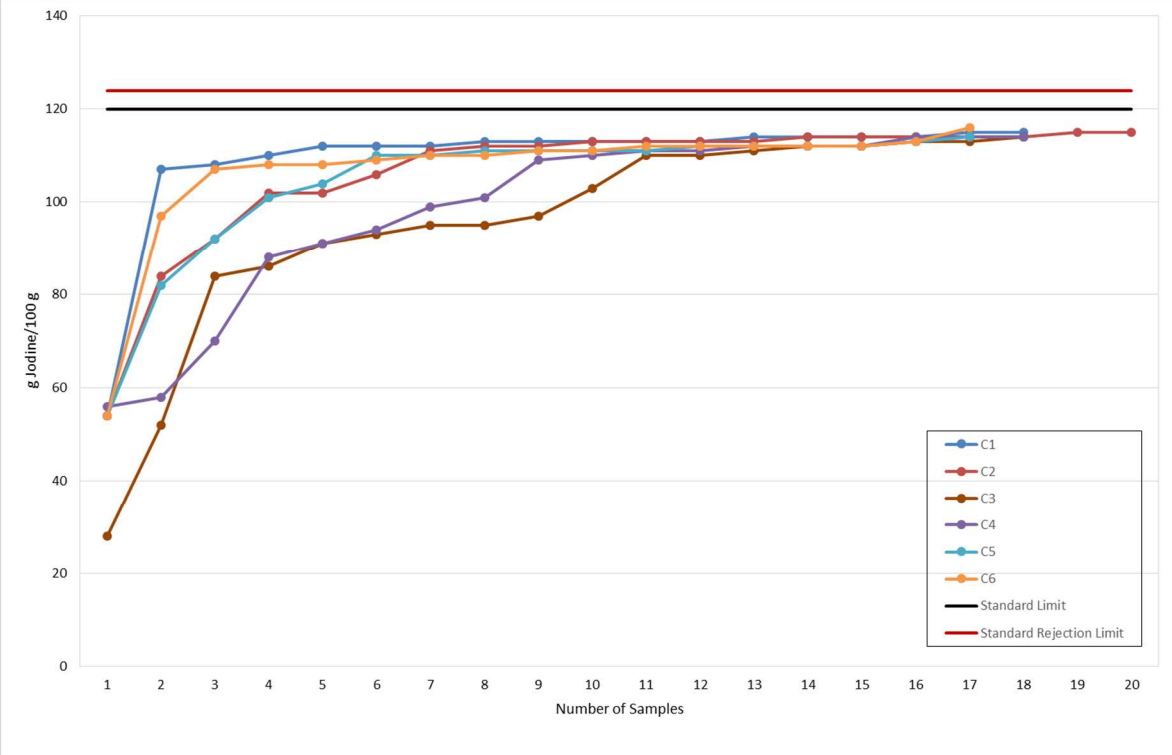
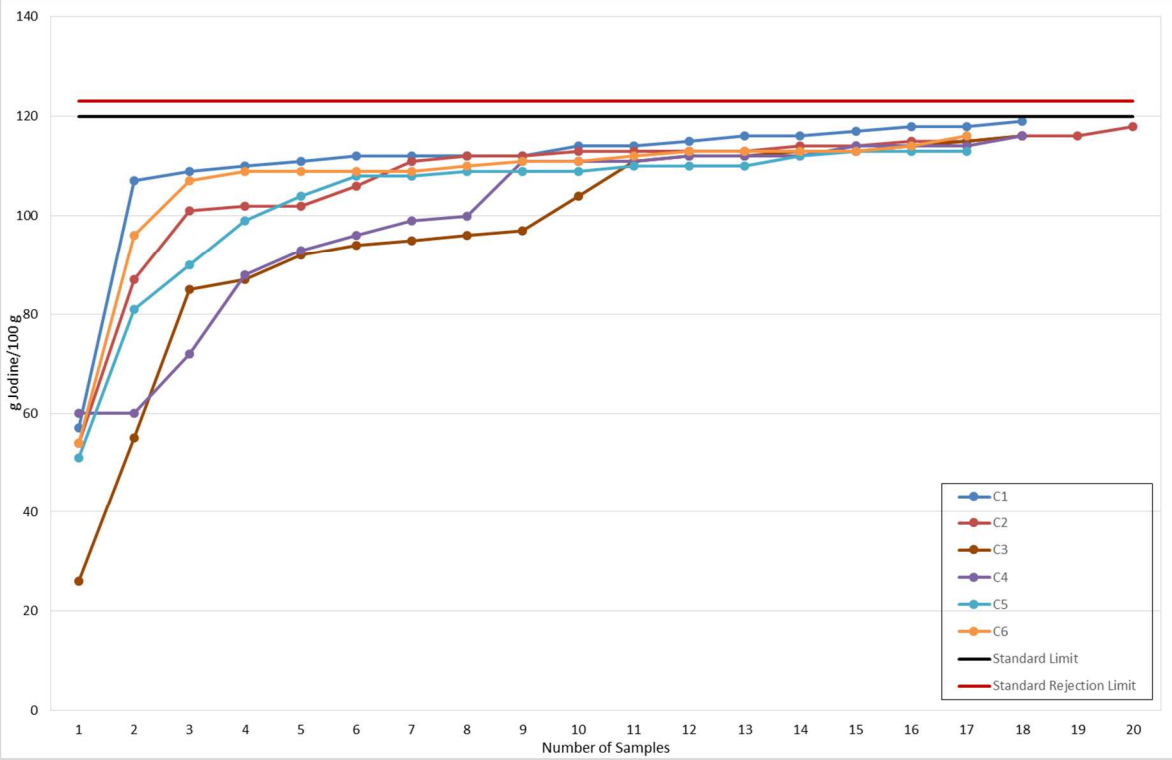


Figure 9: Iodine Number DIN EN 14111 (titration)



The two graphs (fig. 8 and 9) illustrate that there is no remarkable difference between the results of the two methods. All samples fulfill the DIN EN14214:2012

During the entire course of the campaigns limit violations with regard to the iodine number were not identified. For several producers significantly lower values can be noticed in campaigns C3 and C4. The reason is that in summer a limited proportion of palm oil – which has a much lower iodine number – is used for the production of Biodiesel (requirement for CFPP: 0 °C), while in the winter months rapeseed oil methyl ester and blends with soybean methyl ester can be used almost exclusively. However, the iodine number cannot be implemented as the sole factor for the identification of feedstock, this is only possible considering the fatty acid pattern. During C3 one sample showed a significant small value of the iodine (27 mg/kg). With the help of the fatty acid pattern, coconut oil was identified as raw material.

3.9 Glycerides / Free Glycerol

Test method: DIN EN 14105:2003-10,
Test method: DIN EN 14105:2011-07

Partial glycerides and triglycerides are an indication for complete transesterification. Their contents can be influenced by the process conditions; usually they occur in the order triglycerides < diglycerides < monoglycerides because the split-off of the last fatty acid is the slowest transesterification step. A high triglyceride concentration, despite correspondingly lower mono and di values, is mostly an indication for contamination with oils and fats, e.g. along the logistics chain. The data for the individual components of the sampling campaigns was evaluated as follows below.

Upon publication of DIN EN 14214:2012 the test method was changed. The precision of the method was improved and the internal standards changed. All samples of campaigns 1 to 5 were assessed according to DIN EN 14105:2003-10, the samples of campaign 6 according to DIN EN 14105:2011-07.

3.9.1 Monoglycerides

Limit: DIN EN 14214:2012: ≤ 0.70 % (m/m),

Rejection limit at the most: 0.82 % (m/m)

Figure 10: Monoglycerides DIN EN 14105

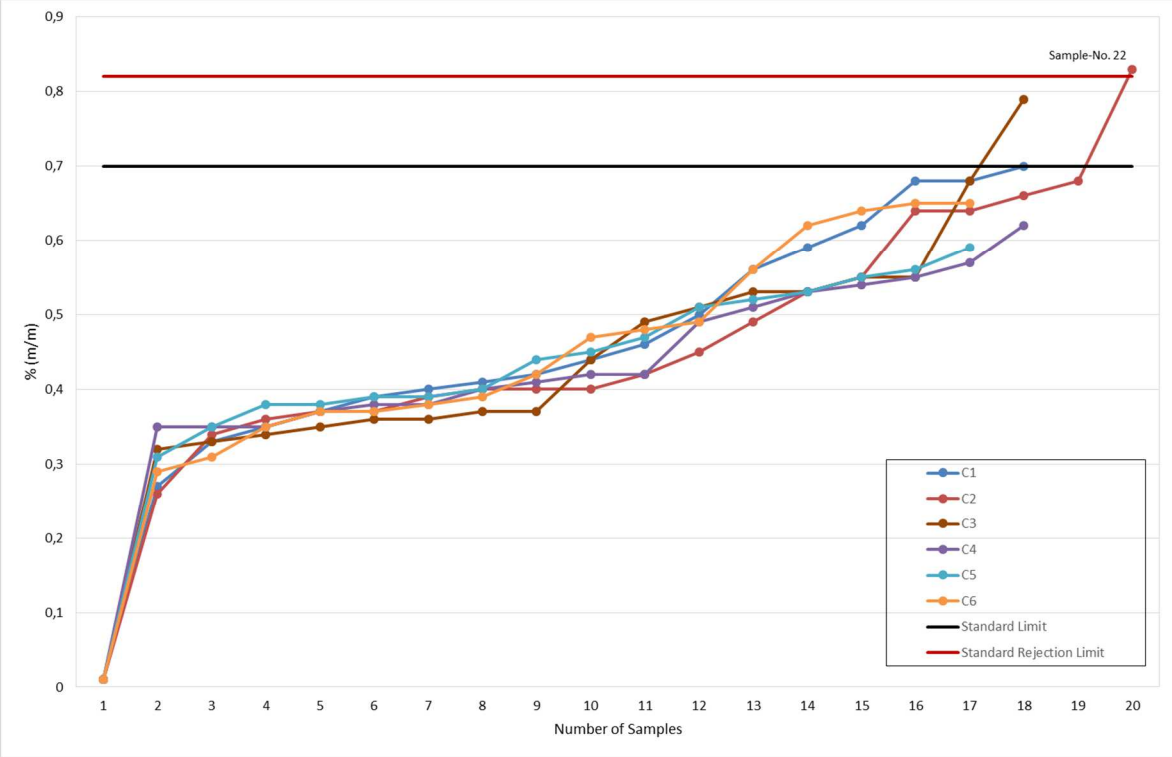


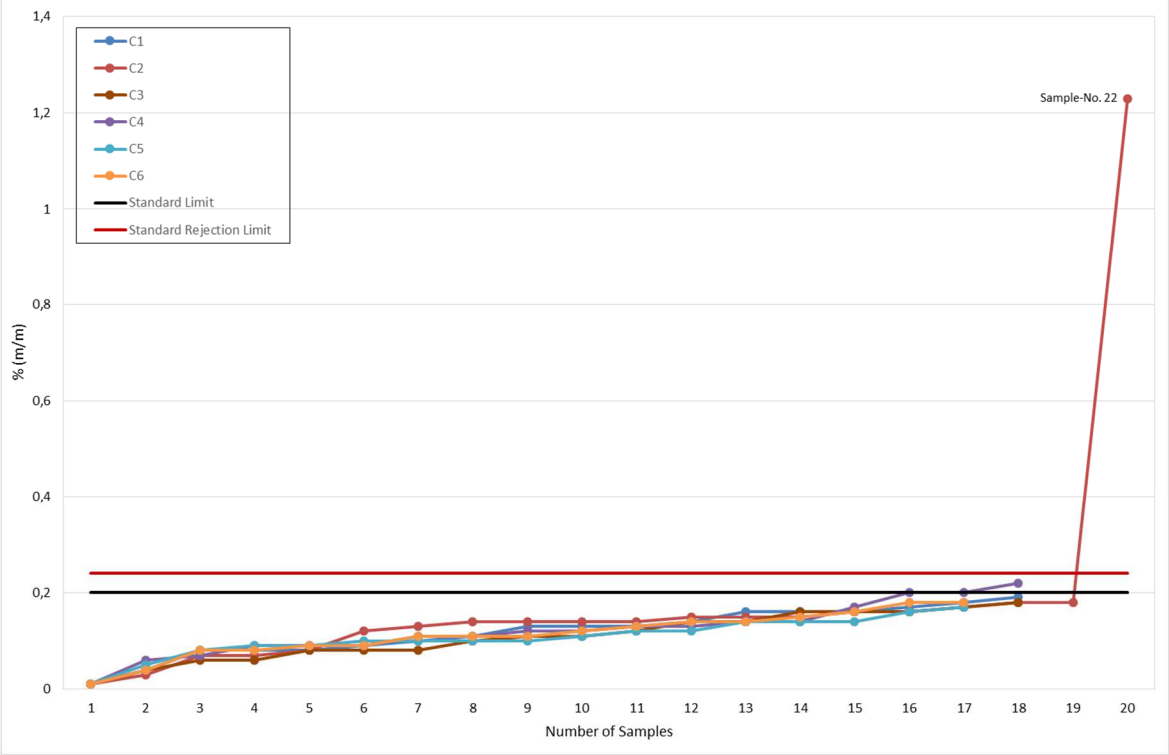
Figure 10 shows that all samples, except sample 22, fulfilled the requirements of the norm.

3.9.2 Diglycerides

Limit: DIN EN 14214:2012: ≤ 0.2 % (m/m),

Rejection limit at the most: 0.24 % (m/m)

Figure 11: Diglycerides DIN EN 14105



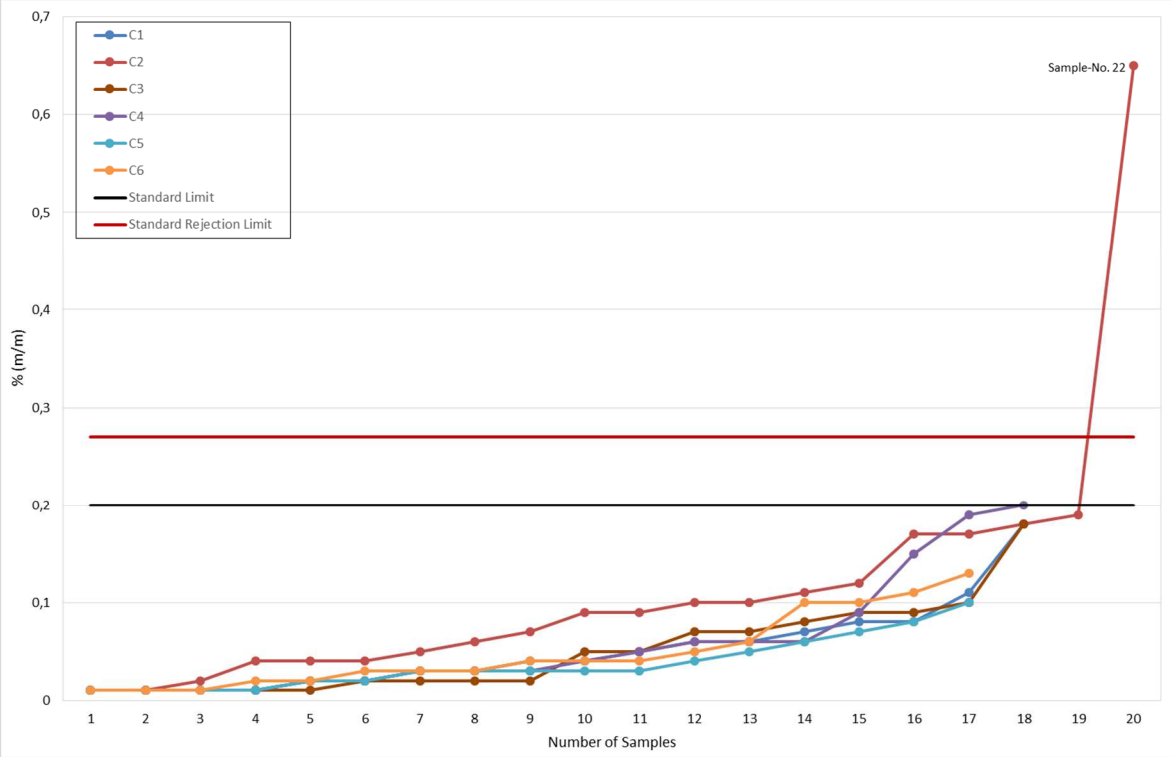
Also figure 11 shows that sample 22 is noticeable because of limit exceedance. All other samples fulfill the norm.

3.9.3 Triglycerides

Limit: DIN EN 14214:2012: ≤ 0.2 % (m/m),

Rejection limit at the most: 0.27 % (m/m)

Figure 12: Triglycerides DIN EN 14105



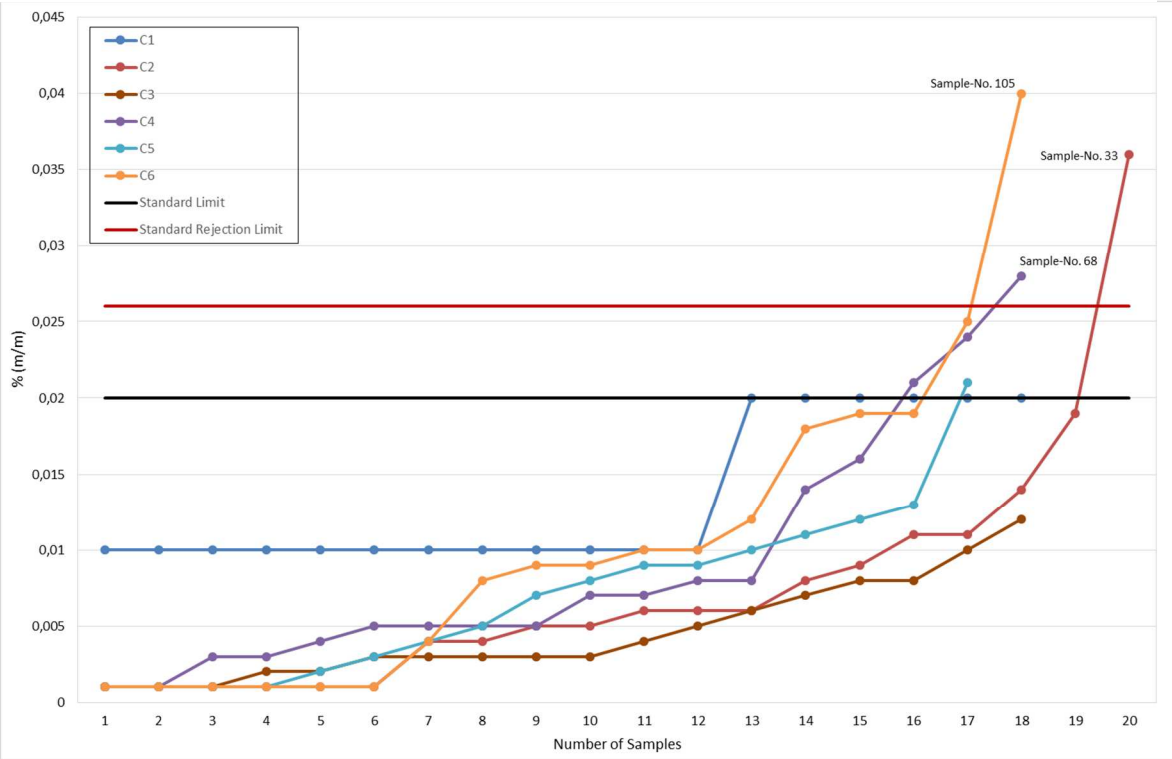
Again the evaluation shows (fig. 12) that generally the contents of triglycerides are way below the permissible limit. Only sample no 22 exceeds the limit of 0.2 % (m/m).

3.9.4 Free Glycerol

Limit DIN EN 14214:2012: ≤ 0.02 % (m/m),

Rejection limit at the most: 0.026 % (m/m)

Figure 13: Free Glycerol DIN EN 14105



With exception to the samples 33, 68 and 105 all other test samples fulfill the requirements of the norm. The limit exceeds of these three samples can be commented as follows:

- Sample 33: The reason for limit exceed was not clearly solved, but the problem was solved during the next campaign.
- Sample 68: This sample showed a limit exceed during the regular measurements in the 4th campaign. The affected member decided for an analysis by the court of arbitration. During that time the effective legal regulation forced the analysis to use the old method according to DIN EN 1405:2003. Taking the limit of 0.02 % (m/m) as a basis to the rejection limit of 0.032 % (m/m) respectively, it becomes clear, that the sample fulfilled the legal requirements according to the precision of the test method and was allowed to be released to the market.
- Sample 105: All goods of which the testing sample originates, significantly exceed the limit of DIN EN 14214:2012. The reason is a defect to the stirrer that occurred during

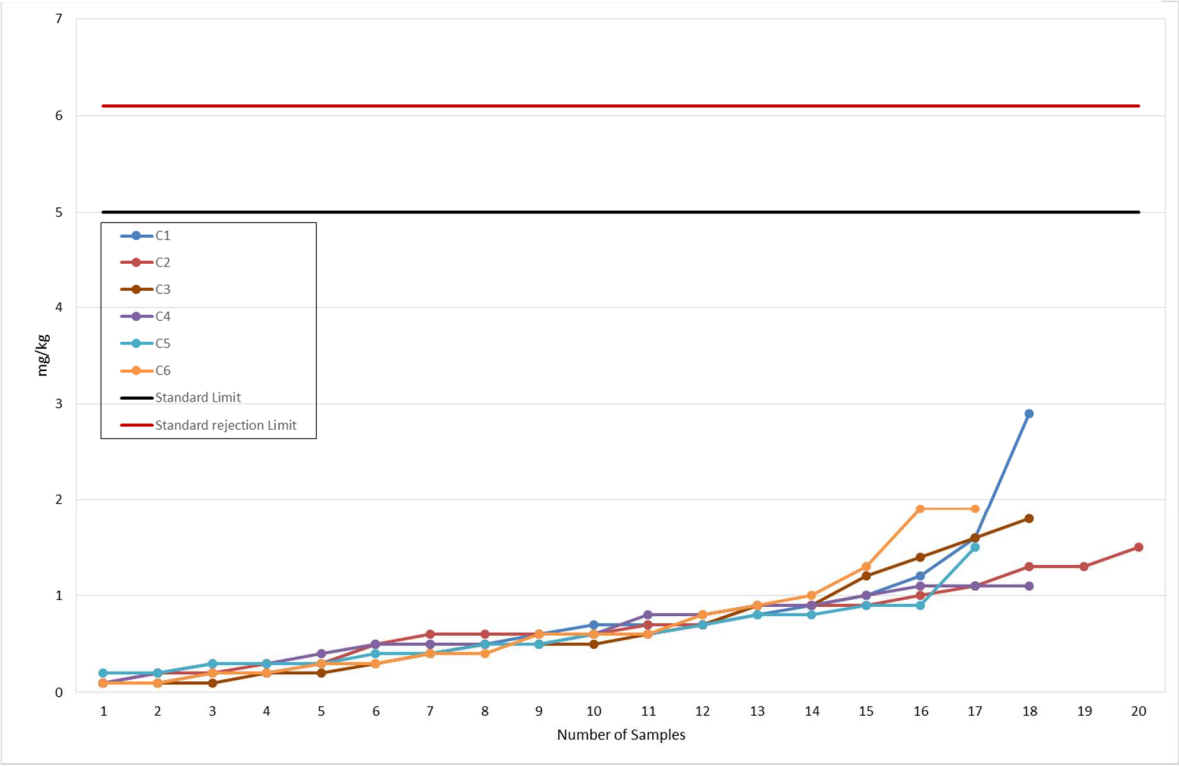
the biodiesel production. The corresponding product batch was not released to the market. After the reparation of the mixer the biodiesel was processed again.

3.10 Alkali Metals: Sodium / Potassium

Test method: DIN EN 14538:2006
Limit: DIN EN 14214:2012: ≤ 5 mg/kg,
Rejection limit at the most: 6.1 mg/kg

The Alkali metals sodium and potassium result from the catalyst used for the Biodiesel production. The soaps forming during the reaction must be removed from the final product by suitable cleaning steps.

Figure 14: Sum of Alkali Metals: Sodium and Potassium DIN EN 14538



Fears of the automotive industry assuming that a high sodium proportion may result in ash forming and thus deposits on the surface of particle filters and oxidation catalysts might occur – which in turn might have an impact on the effectiveness and duration of the systems – are unfounded. Also, fears that the sodium proportion in Biodiesel may lead to deposits on the injection nozzles and thus to deteriorated values of the exhaust case emissions could be

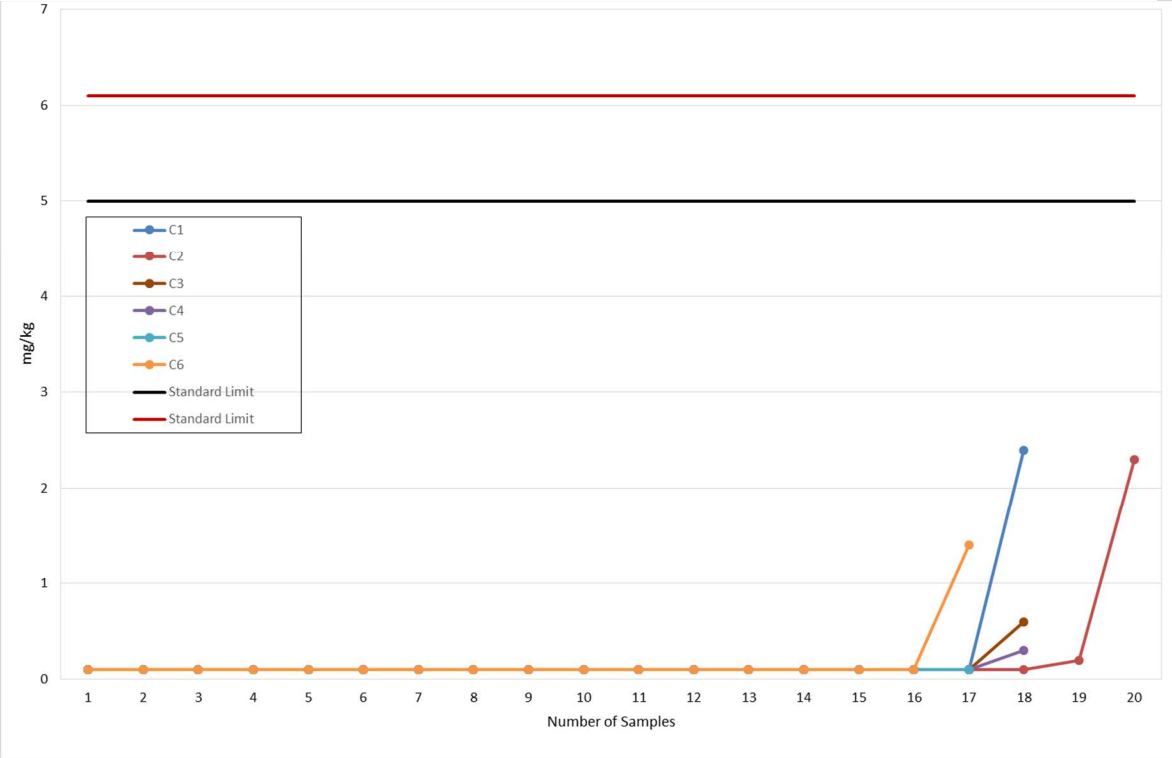
disproved. The results show (fig. 14) that all measured values are way below the required standard limit and therefore, Alkali elements are completely harmless.

3.11 Earth Alkali Metals: Calcium / Magnesium

Test method: DIN EN 14538:2006
Limit: DIN EN 14214:2010/2012: ≤ 5 mg/kg,
Rejection limit at the most: 6.1 mg/kg

The Alkali earth metals calcium and magnesium get into the final product if 'hard' water is used for the washing process: their reaction with free fatty acids leads to the forming of Ca and Mg soaps. Such soaps can cause filter plugging and the gumming of injection nozzles.

Figure 15: Sum of Earth Alkali Metals: Calcium and Magnesium DIN EN 14538



As it is the case with Alkali metals the sum of the Alkali earth metals does not reach the limit in any case (fig. 15). All cumulative values stay below 2.5 mg/kg. The content of Alkali earth metals is even lower than 1 mg/kg in more than 95 % of the samples. Furthermore it is possible to decrease the limit value by modern analysis techniques. But is has to be mentioned that the precision data are only valid for the specific measurement range until 1 mg/kg.

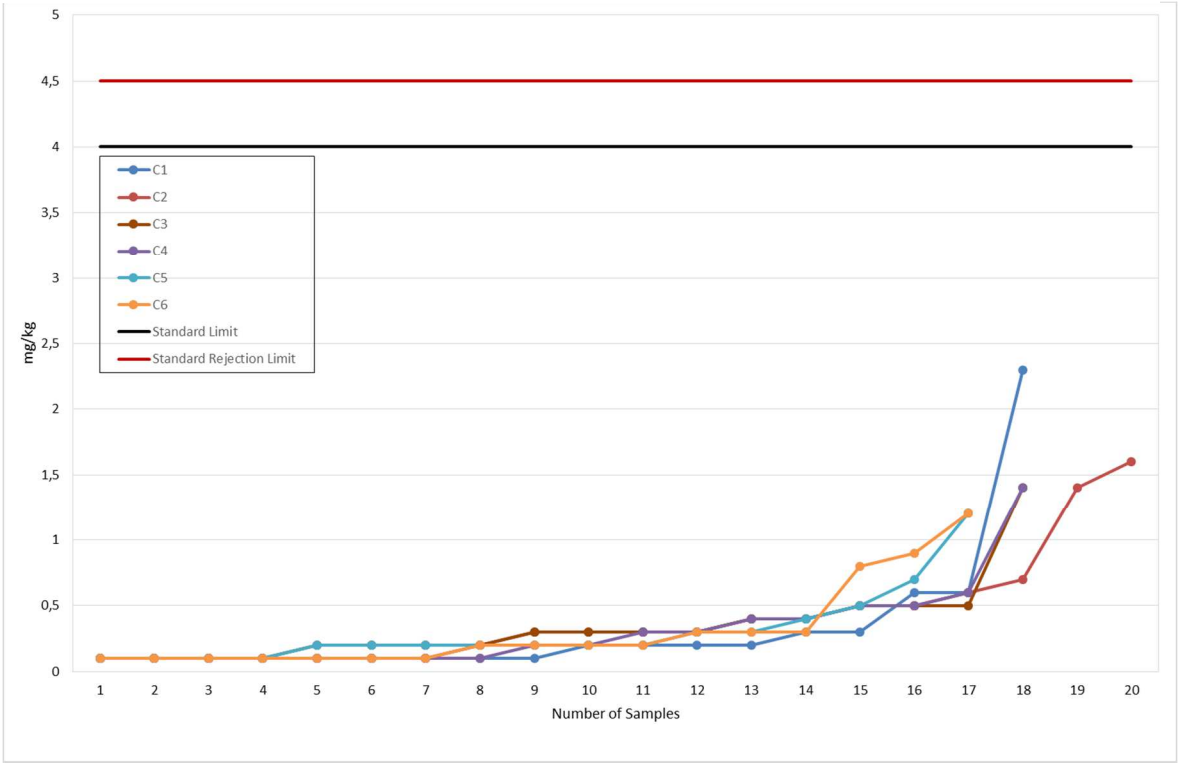
These results illustrate that even the lower limits called for by the automotive industry are considerably undercut.

3.12 Phosphorus Content

Test method: DIN EN 14107:2003
Limit: DIN EN 14214:2012: ≤ 4 mg/kg,
Rejection limit at the most: 4.5 mg/kg

Traces of phosphorus in Biodiesel mostly result from phospholipids which are a natural part of the vegetable oils used. The phosphorus content must already be considered when choosing the feedstock because, if too high, it interferes with the transesterification process. During the normal transesterification process, jointly with the watery glycerol phase, any existing phosphatides are separated from the Biodiesel to the greatest possible extent. However, phosphatides can impede the procession of the glycerol phase to pharmaceutical glycerol which is another reason for the best possible limitation of the phosphorous content in feedstock.

Figure 16: Phosphorus Content DIN EN 14107



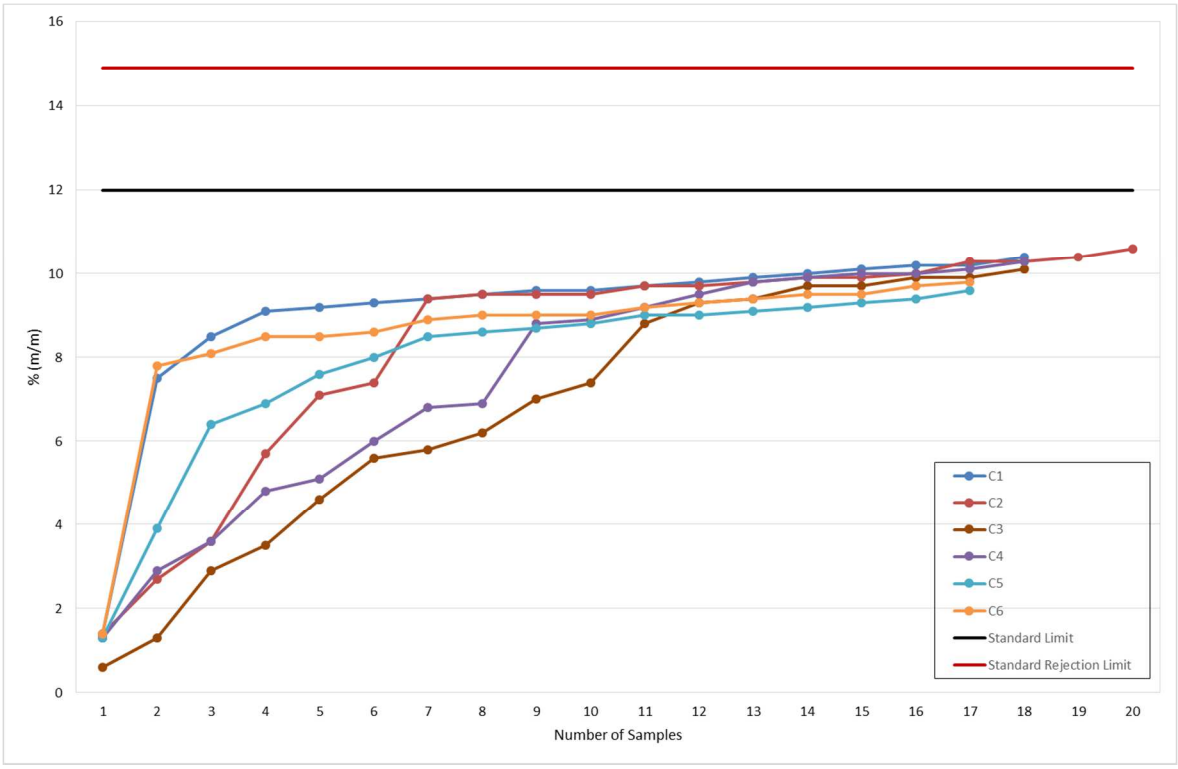
None of the samples shows a phosphorus content of more than 2.5 mg/kg of FAME. In 85 % of the samples the phosphorus content stays even below 0.5 mg/kg of FAME (fig. 16). As it is the case with Alkali and Alkali earth metals, the precision of the method does not allow any reduction of the limit. Low phosphorus content in Biodiesel is of great importance for the automotive industry and its suppliers since phosphorus is a catalyst poison which may irreversibly damage the exhaust gas after treatment system. The low values found here show that AGQM's Biodiesel producers provide Biodiesel of a quality which stays even significantly below the required limit.

3.14 Content of Linolenic Acid

Test method: DIN EN 14103:2011
Limit: DIN EN 14214:2012: ≤ 12.0 % (m/m),
Rejection limit at the most: 14.9 % (m/m)

Linolenic acid is a triple unsaturated fatty acid with 18 carbon atoms. The chemical structure is susceptible for oxidative attacks, hence the content of linolenic acid in biodiesel is limited to 12 % (m/m). The linoletic acid content in rapeseed oil is 8 to 10 %. Gas chromatography is implemented for the detection and assay of this acid.

Figure 17: Content of Linolenic Acid DIN EN 14103



The graph shows that all products could meet the requirements of DIN EN 14214 without any problems. Once again it can be seen – as in the cases of iodine number and CFPP – that during the summer months (campaigns 3 and 4) rapeseed oil as feedstock for the production of Biodiesel was partially substituted by other oils.

3.14 Cold Filter Plugging Point (CFPP)

Test method: DIN EN 116:1997

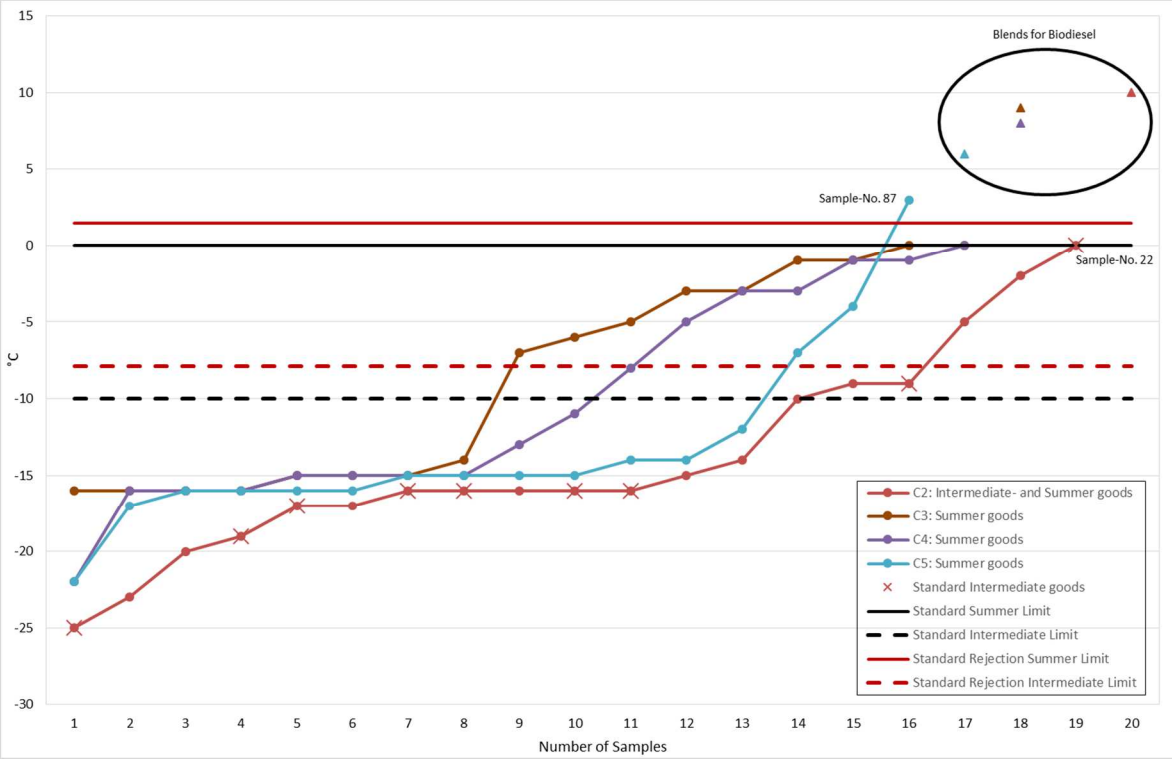
Limit: DIN EN 14214:2012

	Limit	*Rejection limit*
from 15.04. to 30.09.	*0° C*	*1.5 °C*
from 01.10. to 15.11.	*-10 °C*	*-7.9 °C*
from 16.11. to 28./29.02.	*-20 °C*	*-17.3 °C*
from 01.03. to 14.04.	*-10 °C*	*- 7.9 °C*

AGQM-limits: -20°C max. from 19.10. to 28./29.02

The CFPP is a measure for the cold properties of Biodiesel. Due to the applicable climate situation the requirements for 'cold properties' are stipulated nationally. Analogous to Diesel fuel, different requirements apply for summer, intermediate and winter qualities.
Due to specific regulations of the Energy Tax Law, special requirements apply in Germany: although for the use of FAME as blend component the limit of only -10 °C applies for winter quality, FAME must yet be able to achieve a CFPP of -20 °C by means of suitable additivation.
To provide a better overview the results of the summer and winter campaign have been represented in two different graphics. Samples that have been taken from the intermediate phase are marked with an "x".

Figure 18: (Intermediate- and summer goods) DIN EN 116 CFPP

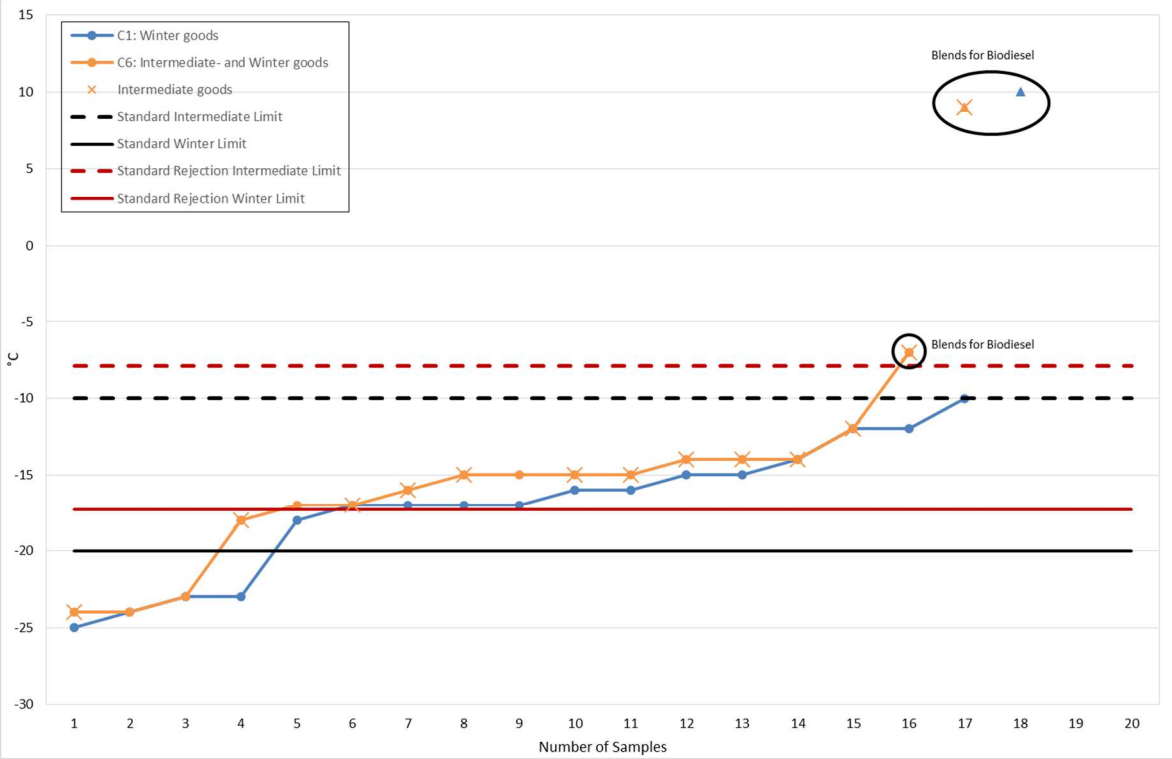


The sampling of the second campaign was performed during the 8th and19th of April, which means that the samples have been taken during the intermediate- and summer phase. The limit for the summer phase has been marked by a continuous line and the limit for the intermediate phase with a dashed line. Samples that have been taken from the intermediate phase are marked with an “x”.

Over 90 % of all tested samples fulfilled the requirements of the DIN EN 14214. The limit exceeds can be explained as follows:

- Sample 22: It is about the sample that already showed conspicuous pattern.
- Sample 87: This sample shows limit exceeds of the summer limit for unknown reasons.
- The four encircled samples represent blends for biodiesel, that have to be mixed with pure goods before released to the market, as already described in the diagram.

Figure 19: (Intermediate- and winter goods) DIN EN 116 CFPP



The sampling of the sixth campaign was performed during the 11th and 21st of November, which means that the samples have been taken during the intermediate- and winter phase.

The limit for the winter phase has been marked by a continuous line and the limit for the intermediate phase with a dashed line. Samples that have been taken from the intermediate phase are marked with an “x”.

As the diagram shows (fig.19), three limit violations occur. In that case it concerns blend samples for biodiesel that will not be released to the market until they are mixed with standard compliant goods.

3.16 Cloudpoint (CP)

Test method: DIN EN 23015:1994

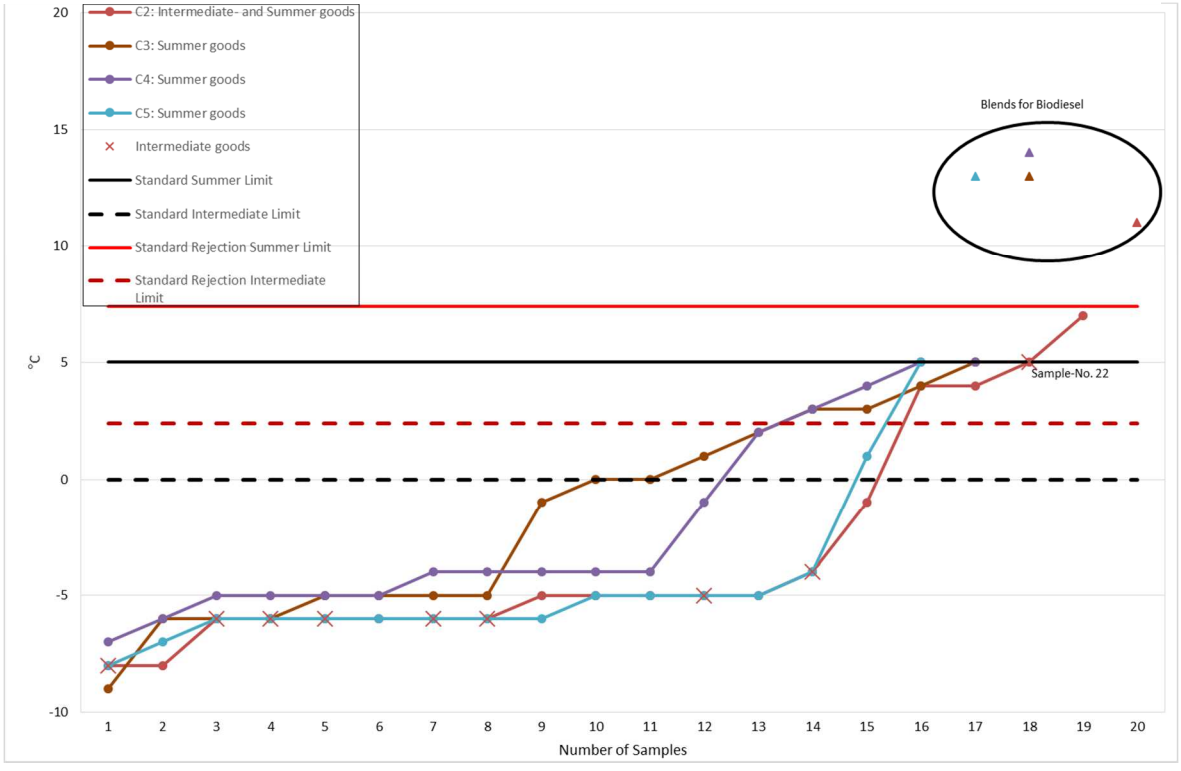
Limit: DIN EN 14214:2012:

	Limit	*Rejection limit*
from 15.04. to 30.09.	*5° C*	*7.4 °C*
from 01.10. to 15.11.	*0 ° C*	*2.4 °C*
from 16.11. to 28./29.02.	*-3° C*	*-0.6 °C*
from 01.03. to 14.04.	*0° C*	*2.4 °C*

The Cloudpoint is the temperature at which, in a clear liquid product, temperature-related clouding (‘clouds’) first sets in during cooling according to stipulated test conditions. Since November 2012, upon publication of DIN EN 14214:2012, the Cloudpoint has been part of the requirements for Biodiesel as blend component. In Germany the cloud point does not assume a role at the time of sampling, since it was not anchored in the 10. BImSchV and the corresponding DIN EN 14214:2010 at that time.

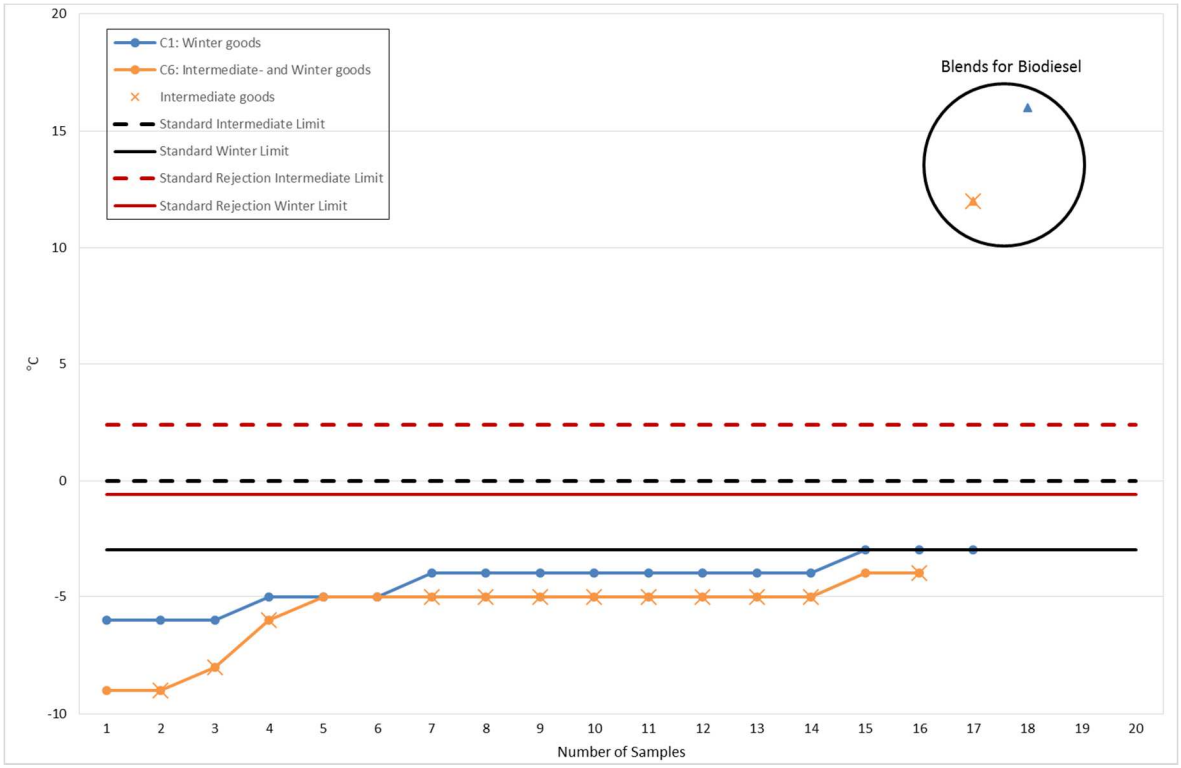
To provide a better overview the results of the summer and winter campaign have been represented in two different graphics. Samples that have been taken from the intermediate phase are marked with an “x”.

Figure 20: Cloudpoint (Intermediate- and summer goods) DIN EN 23015



As already described for the parameter CFPP, the second campaign has been performed partially during the summer- and partially during the intermediate phase. More than 90 % of all tested samples fulfill the requirements of the DIN EN 14214:2012. In 4 of 5 limit exceeds it is about blends for biodiesel, as the fifth sample is no 22, which occurs in limit exceeding lots of times over the testing rounds.

Figure 21: Cloudpoint (Intermediate- and winter) DIN EN 23015



During the winter- and intermediate phase only two limit exceeds occurred, caused by blends for biodiesel, that are not allowed for direct release to the market.

4. Summary

As an essential element the AGQM quality management system encompasses regular product quality checks by means of unannounced sampling. By doing so the observance of the specifications is supervised and also the member companies' self-monitoring is supported.

At the same time the measuring information gathered over the years forms the basis of a database unique in the world concerning the development of the quality of Biodiesel, which demonstrates impressively the continuing improvement and optimisation of production processes and quality assurance measures.

In 2013 nineteen biodiesel producers and two trading firms attended the quality assurance measures. Aside from the production side three storage tanks and one supplier have been tested. Through the year 6 campaigns have been performed along different seasons and a total of 110 biodiesel samples (22 less than 2012) were tested and analyzed. The decrease of tested samples corresponds to production stops of some sites.

For the parameters ‚water content', ‚total contamination' and ‚CFPP' AGQM introduced more stringent limits than those of the standard. On the other hand AGQM responds to their members and therefore accomplished a special regulation in 2013 for biodiesel that has been produced from waste edible oil. This regulation implies that members, who produced biodiesel out of waste edible oils, are allowed to release their product to the market once it is processed and therefore mixed as blend product. Such biodiesel will not be evaluated for the parameters sulphur content, CFPP and Cloud Point, hence there will be no sanctions respectively.

The result of the sampling shows, that except for six biodiesel samples all requirements of the DIN EN 14214 have been fulfilled. Further six samples were handled according to the regulation of waste edible oils. Excluding these six samples of the total evaluation and considering each precision of testing method, approximately 95 % of all tested samples fulfilled the requirements of the DIN EN 14214. An overview of all limit violations is shown in the following table.

Parameter	Method	Sample Number										
		4	13	22	33	42	62	68	77	87	91	108
Ester Content	DIN EN 14103											
Sulphur Content (UV)	DIN ISO 20846											
Water Content K.-F.	DIN EN ISO 12937											
Total Contamination	DIN EN 12662											
Oxidation Stability at 110 °C	DIN EN 14112											
Content of free Glycerol	DIN EN 14105											
Monoglyceride Content	DIN EN 14105											
Diglyceride Content	DIN EN 14105											
Triglyceride Content	DIN EN 14105											
Total Glyceride Content	DIN EN 14105											
CFPP	DIN EN 116											
Cloudpoint	DIN EN 23015											

Overview Rejection Limit Violation of DIN EN 14214:2012
AGQM-Overview Rejection Limit Violation

Table 1: List of samples that contain limit violations.

The samples 22 and 42, from the same member were especially conspicuous. More than one limit violation occurred, without knowing the exact reason, but it has been assumed that the producing progress might be the problem. The relevant member resigned from AGQM meanwhile. All other samples that showed limit violations, only contained one deviation at all. Five of the samples were not able to fulfill the stringent requirements of AGQM regarding the parameter total contamination and water content. Anyway the corresponding goods in these cases were allowed to be released to market, since legal limits were not violated. But it's prohibited to the members to declare those goods as AGQM conform goods. From the rest of the four samples, no 68 is allowed to be released the market under AGQM standards even if there was a limit violation, as explained in 3.9.4.

The overall result of sampling the producer and storage operators in 2013 substantiate again impressively the high quality standard that AGQM members can provide.

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5. Appendix

Table 2: Limits and testing methods for the tested parameters according to DIN EN 14214:2012

Test Parameter	Method	Year of publication	Unit	Standard Limits		Rejection Limits	
				min.	max.	min.	max.
Ester Content	DIN EN 14103	2011	% (m/m)	96.5	-	94.0	-
Density 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	2000	mg/kg	-	500	-	591
Total Contamination	DIN EN 12662	1998[1]	mg/kg	-	24	-	32
Oxidation Stability at 110 °C	DIN EN 14112	2003	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 Iod/100g	-	120	-	124
Iodine Number	DIN EN 14111	2003	g Iod/100g	-	120	-	123
Linolenic Acid Content	DIN EN 14103	2011	% (m/m)	-	12.0	-	14.9
Content of free glycerol	DIN EN 14105	2011	% (m/m)	-	0.02	-	0.026
Monoglyceride Content		2011	% (m/m)	-	0.70	-	0.82
Diglyceride Content		2011	% (m/m)	-	0.20	-	0.24
Triglyceride Content		2011	% (m/m)	-	0.20	-	0.27
Total Glyceride Content		2011	% (m/m)	-	0.25	-	0.28
Alkali Content (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	-		-	
Earth Alkali Content (Ca + Mg)		2006	mg/kg	-	5.0	-	6.1
Calcium Content		2006	mg/kg	-	5.0	-	6.1
Magnesium Content		2006	mg/kg	-		-	
Phosphorus Content	DIN EN 14107	2003	mg/kg	-	4.0	-	4.5
CFPP	DIN EN 116	1997	°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 -10 -20 -10	- - - -	1.5 -7.9 -17.3 -7.9
Cloudpoint	DIN EN 23015	1994	°C	from 15.04. to 30.09. from 01.10. to 15.11 from 16.11. to 28/29.02 from 01.03. to 14.04	5 0 -3 0	- - - -	7.4 2.4 -0.6 2.4

[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 will be applicable until further notice.

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Table 3: Limits and testing methods for the tested parameters according to QM-System of AGQM

Test Parameter	Method	Year of publication	Unit	AGQM-Limits		Rejection limits	
				min.	max.	min.	max.
Water Content K.-F.(for producer)	DIN EN ISO 12937	2000	mg/kg	-	220	-	280
Water Content K.-F.(for storage operators)	DIN EN ISO 12937	2000	mg/kg	-	300	-	370
Total Contamination	DIN EN 12662	1998[2]	mg/kg	-	20	-	20
CFPP	DIN EN 116	1997	°C	from 19.10. to 28/29.02	-20 (valid for application as pure fuel (B100))	- - - -	-17.3

[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 will be applicable until further notice.

6. Abbreviation

AGQM	Arbeitsgemeinschaft Qualitätsmanagement Biodiesel e.V. (Assoziation Quality Management Biodiesel)
B7	Short for blend fuel according to DIN EN 590 with a percentage of 7 % biodiesel.
C 1	Campaign 1
C 2	Campaign 2
C 3	Campaign 3
C 4	Campaign 4
C 5	Campaign 5
C 6	Campaign 6
CEN	European Committee for Standardization
CFPP	Cold Filter Plugging Point
DIN	Deutsches Institut für Normung (German Institute of Standardization)
e.g.	For example
EN	Europäische Norm (European Standard)
e.V.	eingetragener Verein (Registered Assoziation)
FAM	Fachausschuss für Mineralöl- und Brennstoffnormung im DIN (Technical Committee for Mineral Oil and Fuel Standard)
FAME	Fatty Acid Methyl Ester
Fig.	Figure
JWG	Joint working group
K.-F.	Karl Fischer
Mio.	Millions
No.	Number
QM-System	Quality Management System