Arbeitsgemeinschaft
Qualitaetsmanagement Biodiesel e.V.
Claire-Waldoff-Strasse 7
D-10117 Berlin

Biodiesel Quality in Germany

AGQM Sampling Campaigns 2012

Introduction

Since its foundation in 1999, Arbeitsgemeinschaft Qualitaetsmanagement Biodiesel e.V. (AGQM) has taken a large number of measures to assure the continuous improvement of the quality of 'Biodiesel'. Apart from implementing a quality assurance system – which the association makes available to its members and the observance of which it monitors – the most important measure were regular checks of the Biodiesel quality at filling stations and member companies, both trading companies and production plants.

Over the years the monitoring of the fuel quality underwent a major priority change. When the network of Biodiesel filling stations ceased to exist, the originally paramount objective of monitoring filling stations became obsolete. Today Biodiesel is almost exclusively marketed as blend component for B7 fuel, so consequently AQGM's quality monitoring focuses on producing and trading member companies which meanwhile represent about 90 % of the entire fuel volume sold in Germany.

Despite this market shift caused by legal regulations in Germany, Biodiesel will still remain to be the most important fuel on the basis of renewable feedstock as well as a significant factor to maintain mobility and to fight the climate change in the future.

Even though Diesel fuel now only contains Biodiesel of just up to 7 % (v/v), in recent years quality requirements have become considerably more stringent in certain aspects due to increasing demands by engine manufactures and legislation concerning emissions, which is reflected by the increasing requirements of DIN EN 14214, a standard derived from the European Fuel Quality Directive. Its observance is the prerequisite for a failure-free operation of vehicles and at the same time it is the condition on which the eligibility of Biodiesel as blend component with regard to the quota obligation is based.

In Germany Biodiesel is subject to monitoring by the Customs authorities with regard to meeting quality requirements. All quality parameters checked by Customs are stipulated by the *36. Bundesimmisionsschutzverordnung* (BImSchV). Customs authorities are instructed to make random checks of the quality of Biodiesel. If the result of the analytics shows any violation of limits this is not just considered an administrative but a tax offence and punished as such. AGQM members benefit in particular from the quality assurance measures: in negotiations with the German Federal Ministry of Finance, AGQM could achieve that such checks may be foregone if member companies are regularly sampled and monitored by AGQM (Vorschriftensammlung Bundesfinanzverwaltung-Nachrichten[1]: Biokraftstoffquoten-gesetz N 59 2007 Nr. 320, BImSchV §4).

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 2010 the results of the unannounced sampling of AGQM members were

[1] http://www.agqm-biodiesel.de/de/gesetzliche-grundlagen/gesetze/
(Link dated 3 March 2013)

published in a quality report[2] for the first time. They substantiate the high quality level of the Biodiesel volume of about 2.5 million tons marketed by all AGQM members.

Sampling – Execution and Scope

In 2012, 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 at production plants and trading companies and tested with regard to the parameters stipulated in the Quality Management System, attachment A. For that the latest version of DIN EN 14214 forms the basis, although according to the stipulations of AGQM's QM System, table 1, 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.

Upon the publication of DIN EN 14214:2012-11 (hereafter called DIN EN 14214:2012) in November 2012, there were changes with regard to test methods, limits and thus also to the rejection limits which are the benchmark relevant for the sampling of AGQM members.

Therefore, all samples were assessed according to the standard applicable at the time of sampling; those were the requirements of DIN EN 14214:2010 (see attachment, table 1) for campaigns 1 to 5 and of DIN EN 14214:2012 (see attachment, table 2) for campaign 6. In table 2 all changed standard values are written in blue. The AGQM limits, more stringent in some cases, are listed next to the relevant parameters. Furthermore, parameter 'Cloudpoint', included additionally into DIN EN 14214:2012, was also tested from campaign 5.

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

C1: January/February	C3: May/June	C5: October
C2: March	C4: August	C6: November/December

The following evaluation describes the individual parameters tested; the results are illustrated graphically. They are anonymous and do not give any indication as to the samples' origin.

For every campaign the results are given in rising order to illustrate the spread of the results; the individual limits are shown by a red line.

[2] http://www.agqm-biodiesel.de/files/6913/2999/1435/20110530_Herstellerbepr_Final_eng.pdf
(Link dated 3 March 2013)

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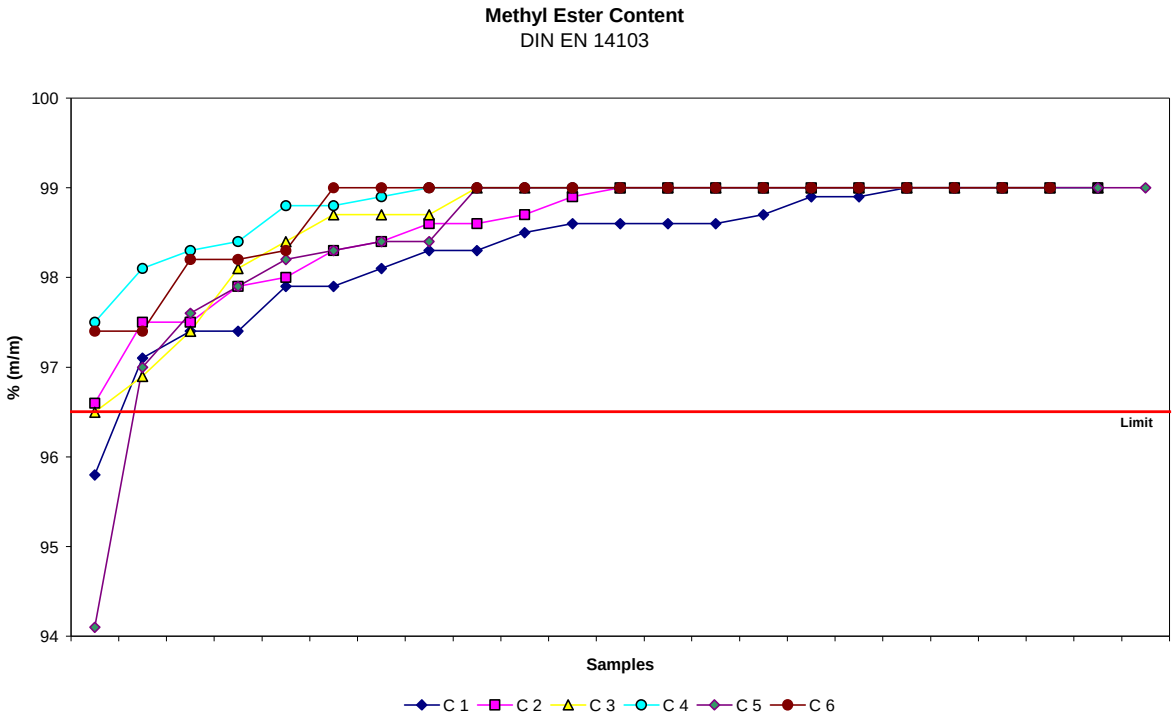
Individual Results

Content of Fatty Acid Methyl Ester (‚FAME')

Test method: DIN EN 14103:2003
Limit DIN EN 14214:2010: ≥ 96.5 % (m/m), rejection limit at least 94.7 % (m/m)

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. DIN EN 14214 stipulates a FAME content of at least 96.5 %.

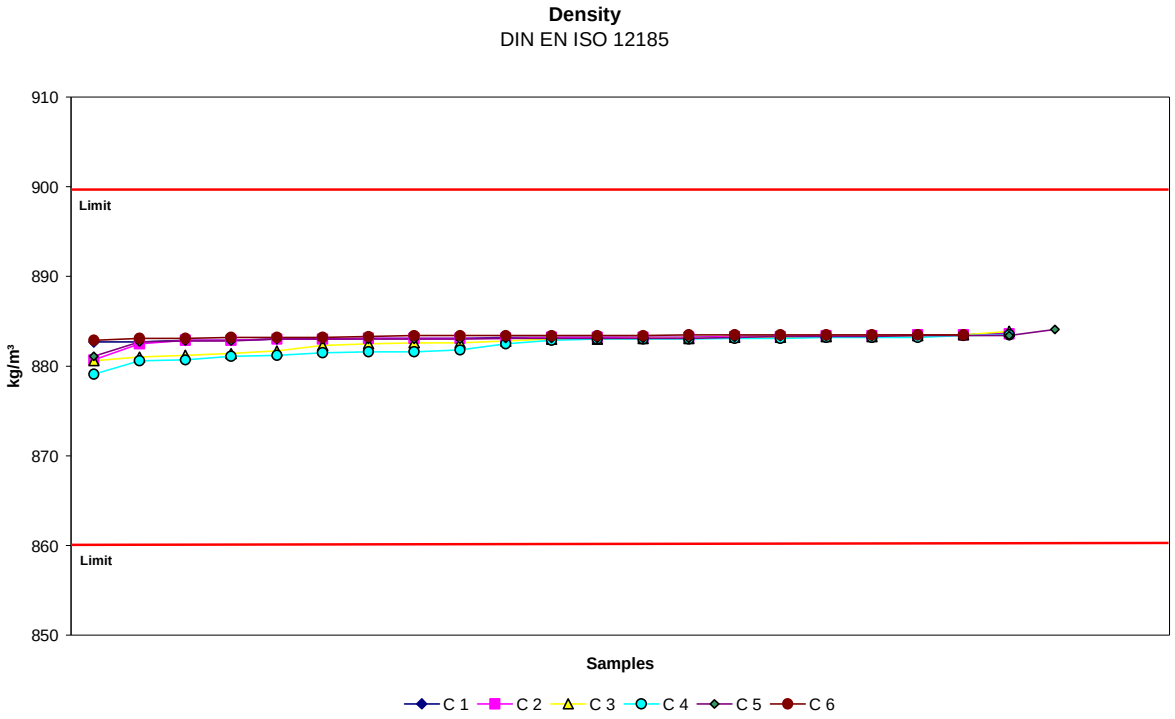


Measured values given as ‚> 99.0 % are illustrated in the graph as ester content of 99 %. The graph illustrates that the standard limit is observed with the exception of two measured values. Considering the precision of the test method, the measured value of campaign 1 also complies with the requirements of DIN EN 14214, whereas the measured value of campaign 5 still remains faulty by staying below the limit. The reason for that could not yet be clearly identified. This Biodiesel sample of campaign 5 also violates the limits of two other parameters. Correction measures are currently elaborated.

Density at 15 ℃

Test method: DIN EN ISO 12185:1997
Limit DIN EN 14214:2010/2012: 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.

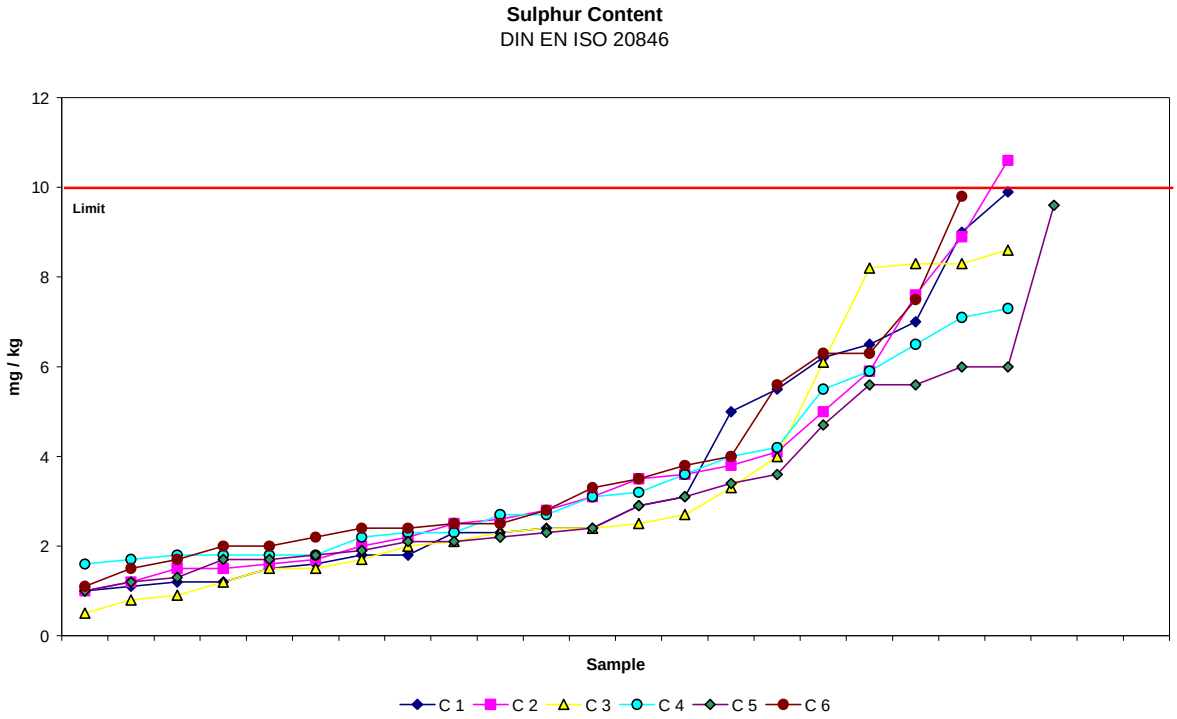


Almost all measuring results show a density of 883 kg/ m³. All samples comply with DIN EN 14214.

Sulphur Content

Test method: DIN EN ISO 20846:2004/2011
Limit DIN EN 14214:2010/2012: ≤ 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.



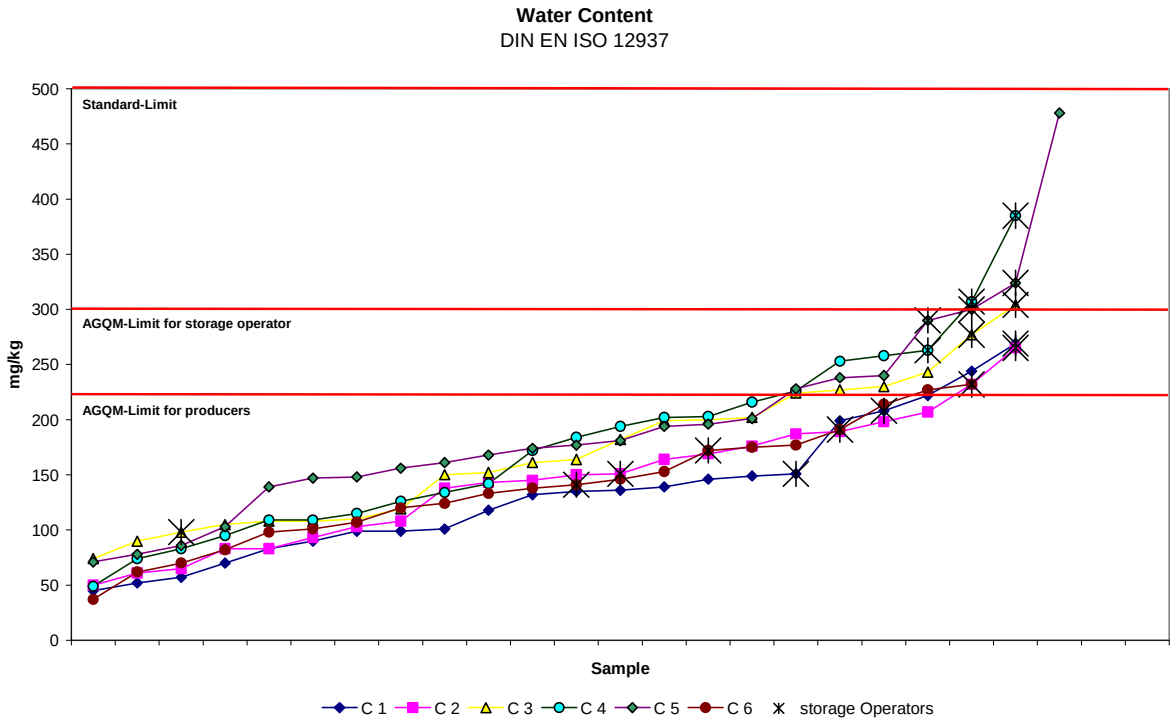
The sulphur content of Biodiesel is obviously no problem. The graph clearly shows that only one value violates the limit of DIN EN 14214. Considering the precision of the test method this value also complies with the requirement of the standard. It is pleasing that more than 75 % of all samples have a sulphur content of < 5 mg/kg.

Water Content

Test method: DIN EN ISO 12937:2000
Limit DIN EN 14214:2010/2012: ≤ 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.



The majority of samples contain less than 200 mg of water/kg of FAME which is still significantly below AGQM's stipulation. Considering the precision of the test method all samples tested, apart from one exception each of producers and storage operators, meet the more stringent AGQM limits given above. The graph shows that all measuring results comply with the limit of DIN EN 14214.

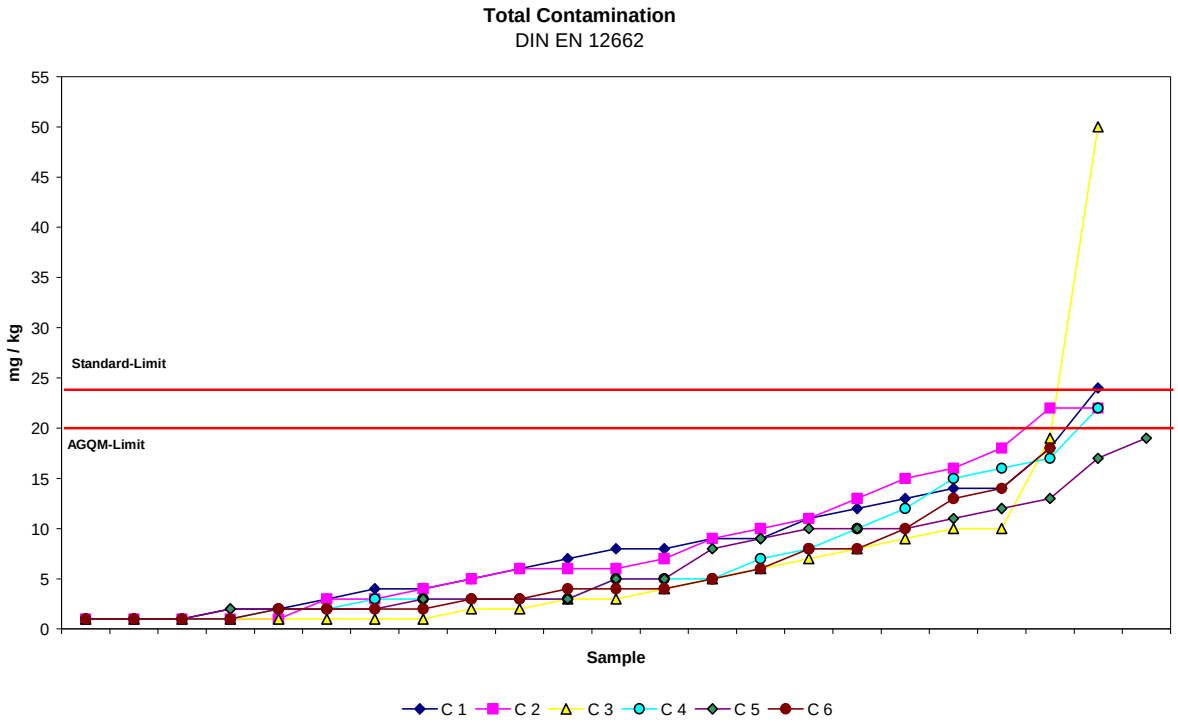
Total Contamination

Test method: DIN EN 12662:1998
Limit DIN EN 14214:2010/2012: ≤ 24 mg/kg, rejection limit at the most: 32 mg/kg

Limit AGQM: ≤ 20 mg/kg for producers (the AGQM limit already functions as rejection limit for total contamination)

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.

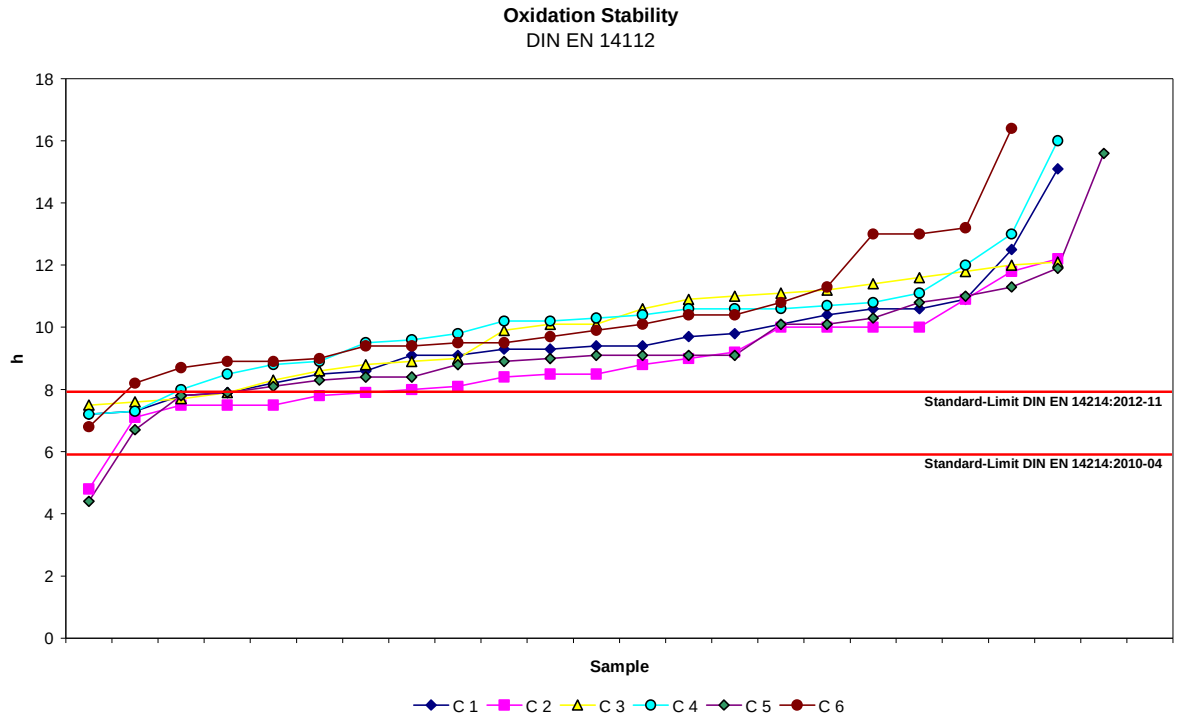


All samples but one met the requirements of DIN EN 14214. The reason for this outlier was welding work to the sampling pipe previous to sampling. This example illustrates that even comparably minor perturbation to the production plant or process may quite easily lead to an impairment of the Biodiesel quality. Another four samples exceeded the AGQM limit. It is a positive aspect that more than 2/3 of all samples show a particle volume of less than 10 mg/kg.

Oxidation Stability

Test method: DIN EN 14112:2003
Limit DIN EN 14214:2010: ≥ 6 h, rejection limit at least: 4.9 h
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. This limit applies to campaigns 1 to 5. 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. For that reason the limit of 8 h applies to campaign 6.

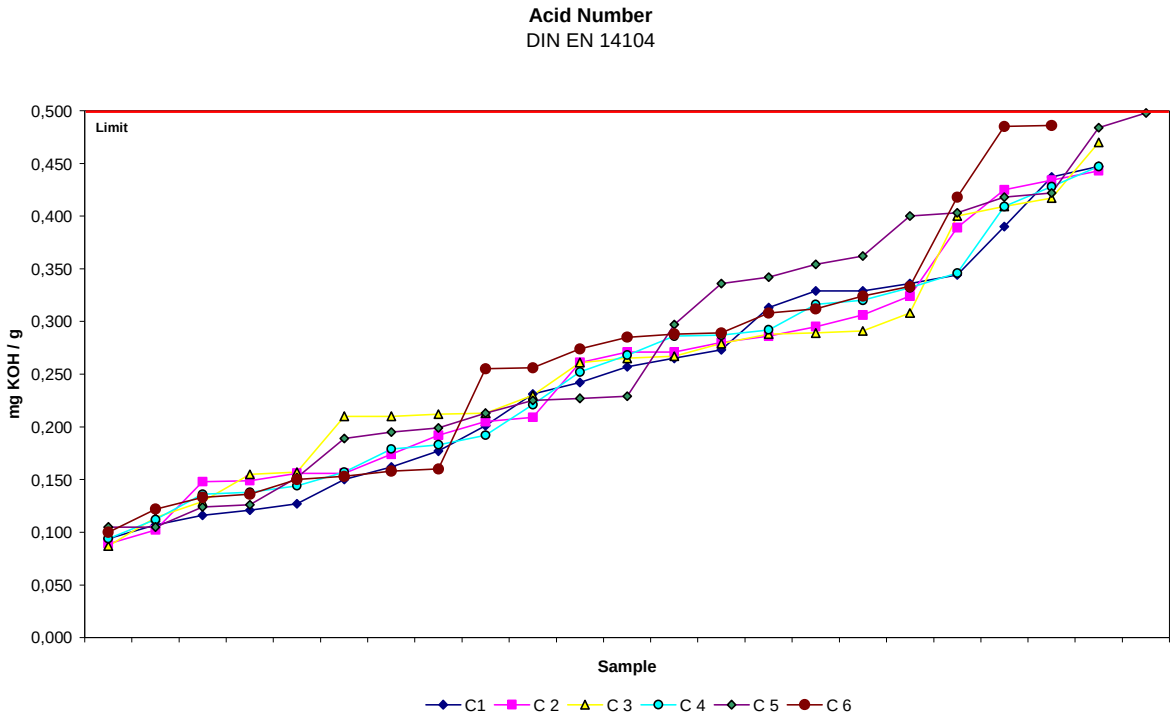


The graph shows that in campaigns 2, 5 and 6 one sample each did not reach the standard limit; considering the precision of the test method the sample of campaign 6 can be considered standard-conform. It is assumed that in campaign 6 the value measured stayed below the limit due to insufficient adaptation to DIN EN 14214:2012 with regard to the tightened requirement of 8 h. In campaign 2 belated additivation with the oxidation stabilizer during the tanking process could be identified as cause for staying below the limit. Here a correction measure could be carried out successfully. The reason for staying below the limit in campaign 5 is yet unresolved. It was the same Biodiesel sample which already showed irregularities with regard to its ester content and CFPP. Considering the precision of the test method all samples of campaigns 1 to 5 met the more stringent requirements of DIN EN 14214:2012, except for the two limit violations of campaigns 2 and 5. It can thus be concluded that in the future the raised limit of 8 hours can be complied with without any problems.

Acid Number

Test method: DIN EN 14104:2003
Limit DIN EN 14214:2010/2012: ≤ 0.5 mg KOH/g,
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.



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

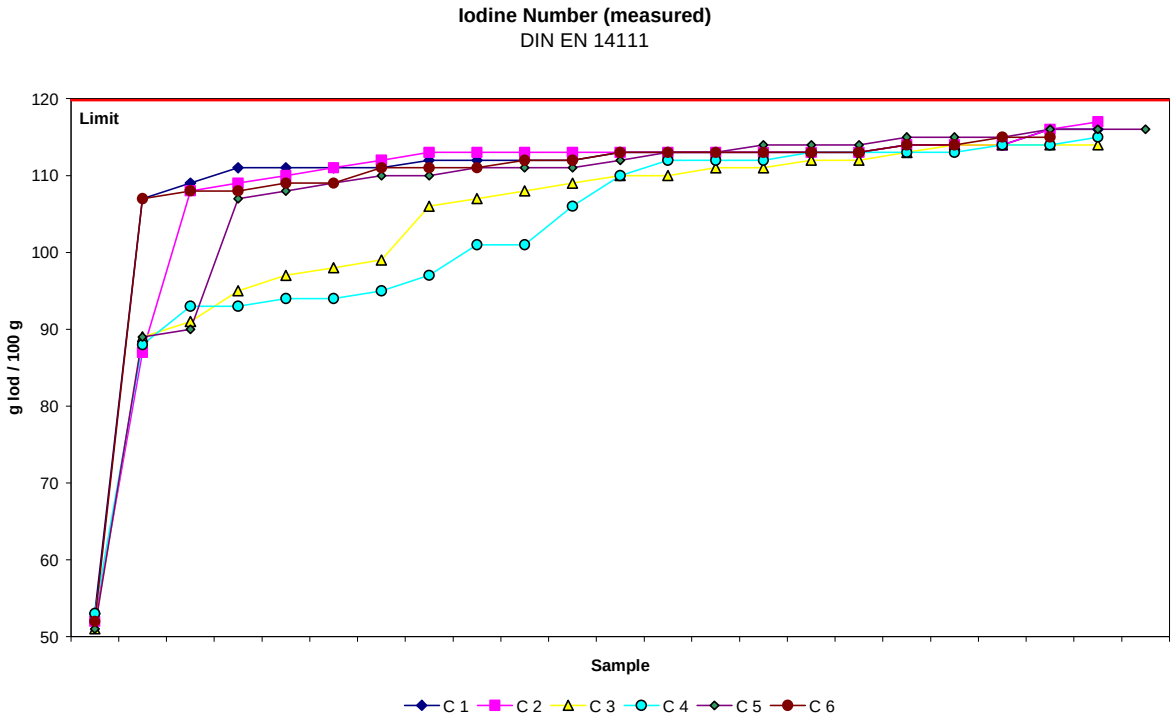
Iodine Number

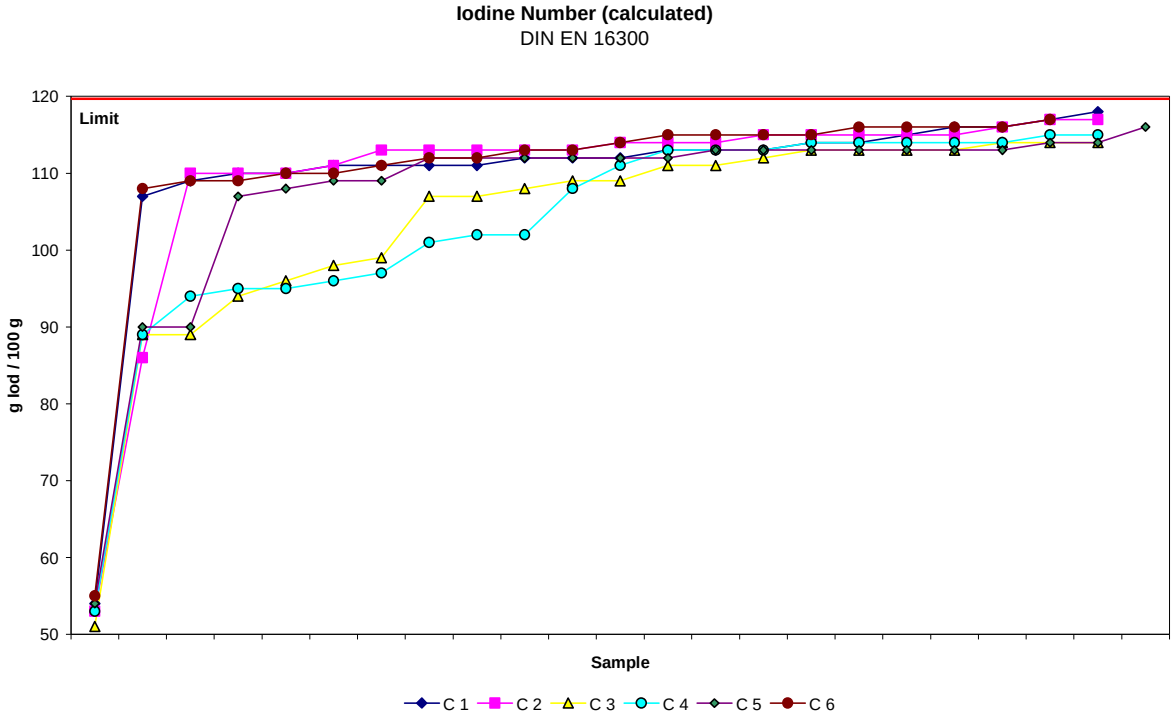
Test method: DIN EN 14111:2003
Limit DIN EN 14214:2010/2012: 120 g of Iodine/100g
Rejection limit at the most: 123 g of Iodine/100g

Test method: DIN EN 14214 Annex B.5/ DIN EN 16300:2012
Limit DIN EN 14214:2010/2012: 120 g of Iodine/100g,
Rejection limit at the most: 124 g of Iodine/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, 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.





The two graphs illustrate that there is no remarkable difference between the results of the two methods.

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 ℃), while in the winter months rapeseed oi l 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.

Glycerides / Free Glycerol

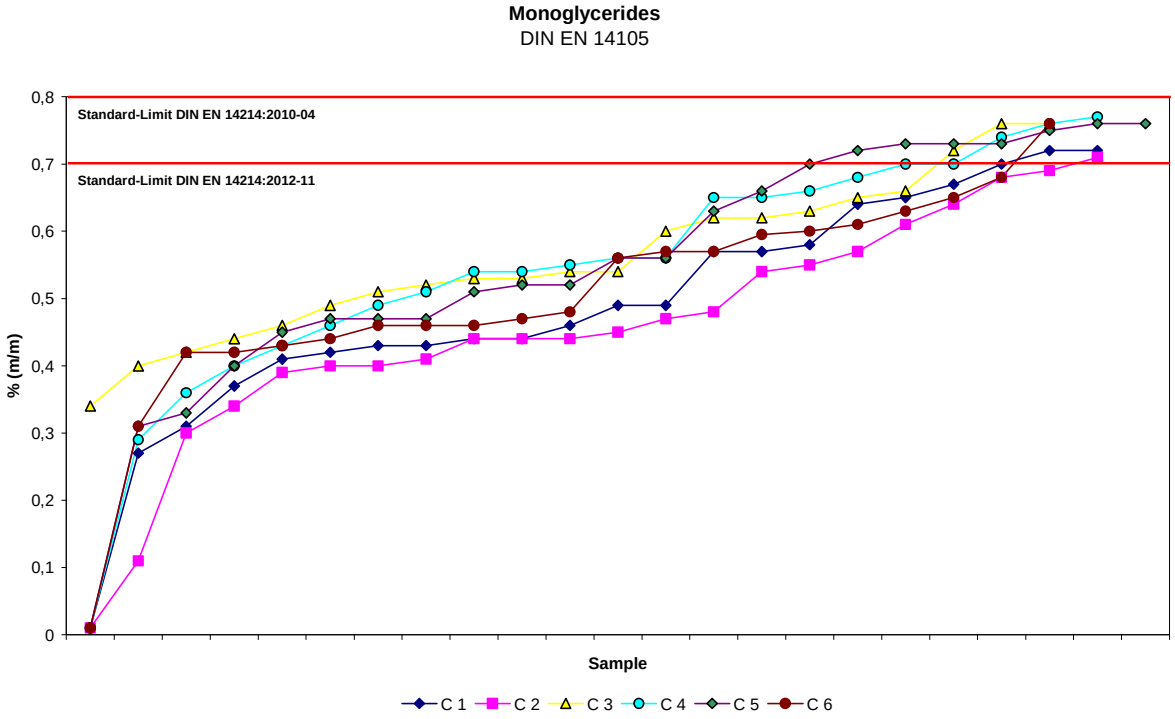
Test method: DIN EN 14105:2003-10
Test method: DIN EN 14105:2011-07

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.

Monoglycerides

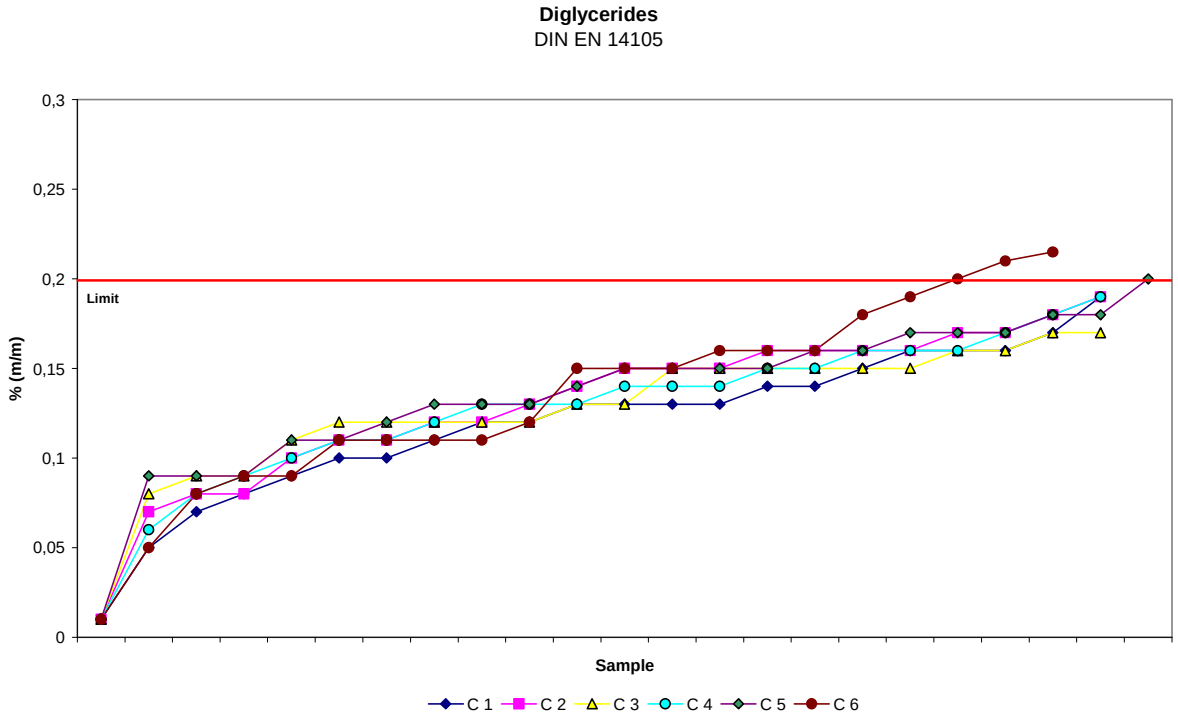
Limit DIN EN 14214:2010: ≤ 0.80 % (m/m), rejection limit at the most: 0.94 % (m/m)
Limit DIN EN 14214:2012: ≤ 0.70 % (m/m), rejection limit at the most: 0.82 % (m/m)



FAME according to DIN EN 14214:2010 may contain monoglycerides to a maximum of 0.80 % (m/m). The graph shows that all tested samples stayed below the limit. Campaign 6 was assessed according to the current version of DIN EN 14214, in which the limit for monoglycerides was lowered to 0.70 % (m/m). There is one outlier which only meets the standard with regard to the precision of the test method; otherwise all tested products are within the permissible range.

Diglycerides

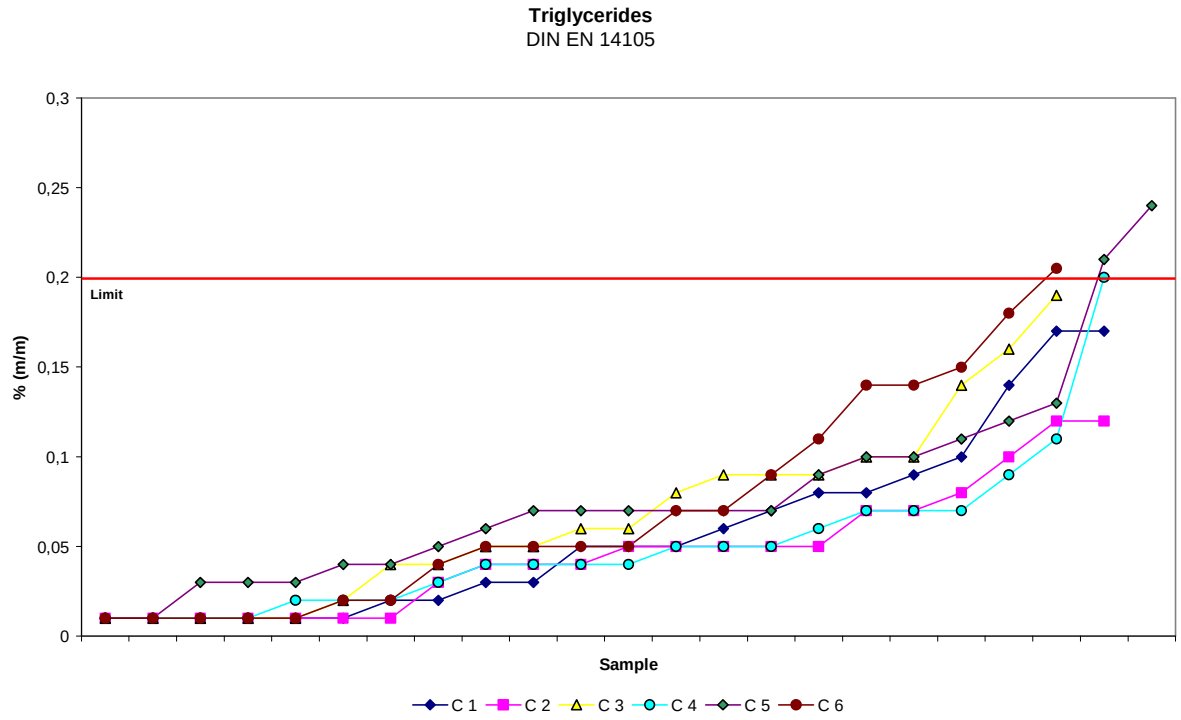
Limit DIN EN 14214:2010/2012: ≤ 0.2 % (m/m),
Rejection limit at the most: 0.24 % (m/m)



As it is the case with monoglycerides, the graph reflects that AGQM's Biodiesel producers have the transesterification process under control. Failure and ensuing exceedance of limits almost only occur when the production plants are started and/or in case of technical faults. Generally such problems are detected during in-house operational checks. The two identified cases of limit exceedance are within the precision of the test method.

Triglycerides

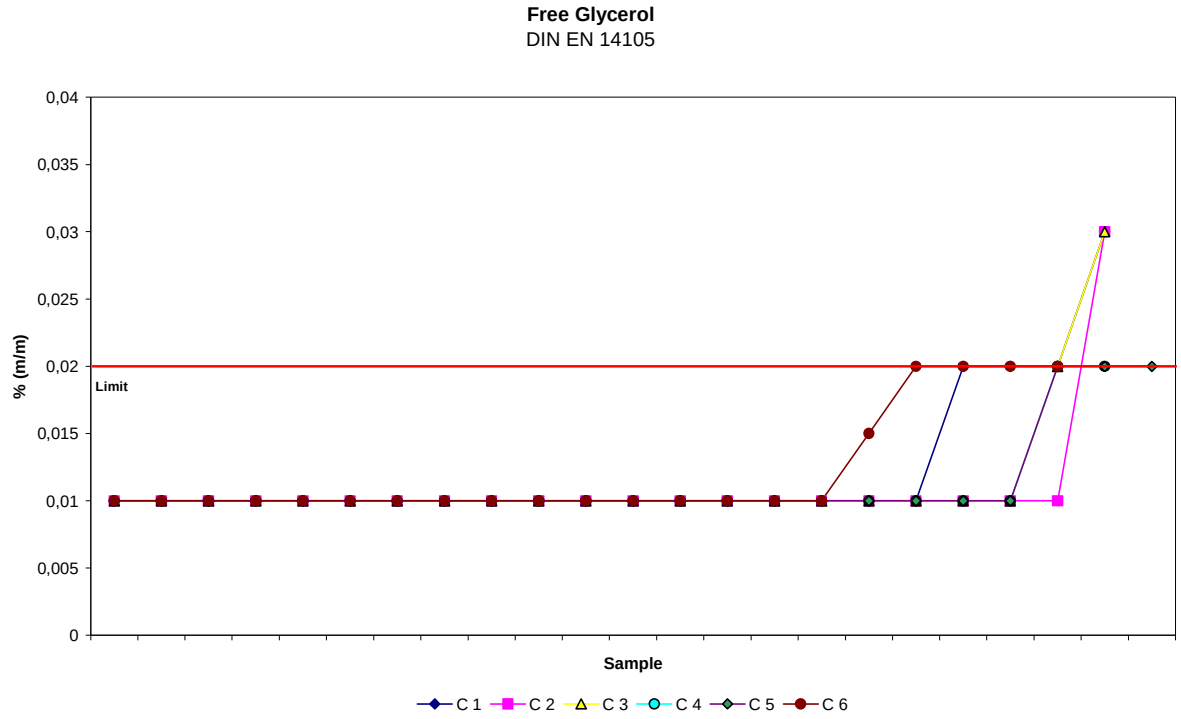
Limit DIN EN 14214:2010: ≤ 0.2 % (m/m), rejection limit at the most: 0.26 % (m/m)
Limit DIN EN 14214:2012: ≤ 0.2 % (m/m), rejection limit at the most: 0.27 % (m/m)



Again the evaluation shows that generally the contents of triglycerides are way below the permissible limit. The measured values of the three cases exceeding the limit are within the precision range of the test method.

Free Glycerol

Limit DIN EN 14214:2010: ≤ 0.02 % (m/m), rejection limit at the most: 0.032 % (m/m)
Limit DIN EN 14214:2012: ≤ 0.02 % (m/m), rejection limit at the most: 0.026 % (m/m)

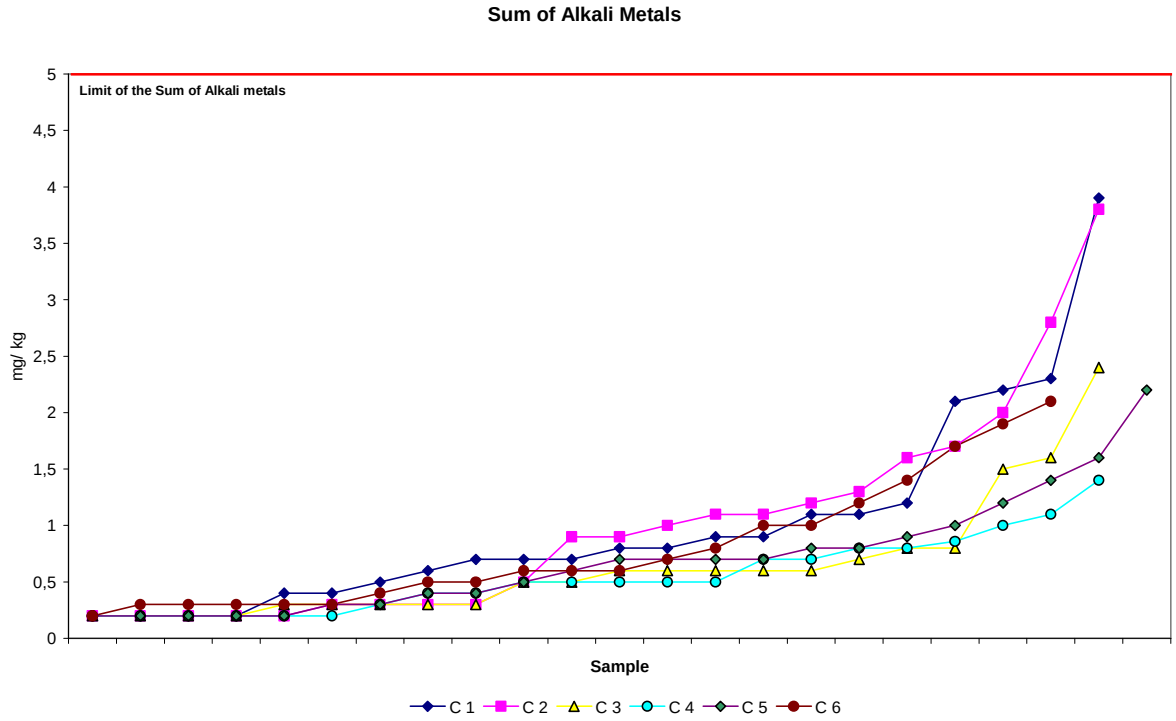


More than 85 % of the tested samples show a measuring value of ≤ 0.01 % (m/m) of free glycerol. Despite two samples exceeding the limit, the standard is complied with in all cases considering the precision of the test method.

Alkali Metals: Sodium / Potassium

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 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. DIN EN 14214 restricts the sum of the sodium and potassium contents to 5 mg/kg (Na + K: ≤ 5 mg/kg).

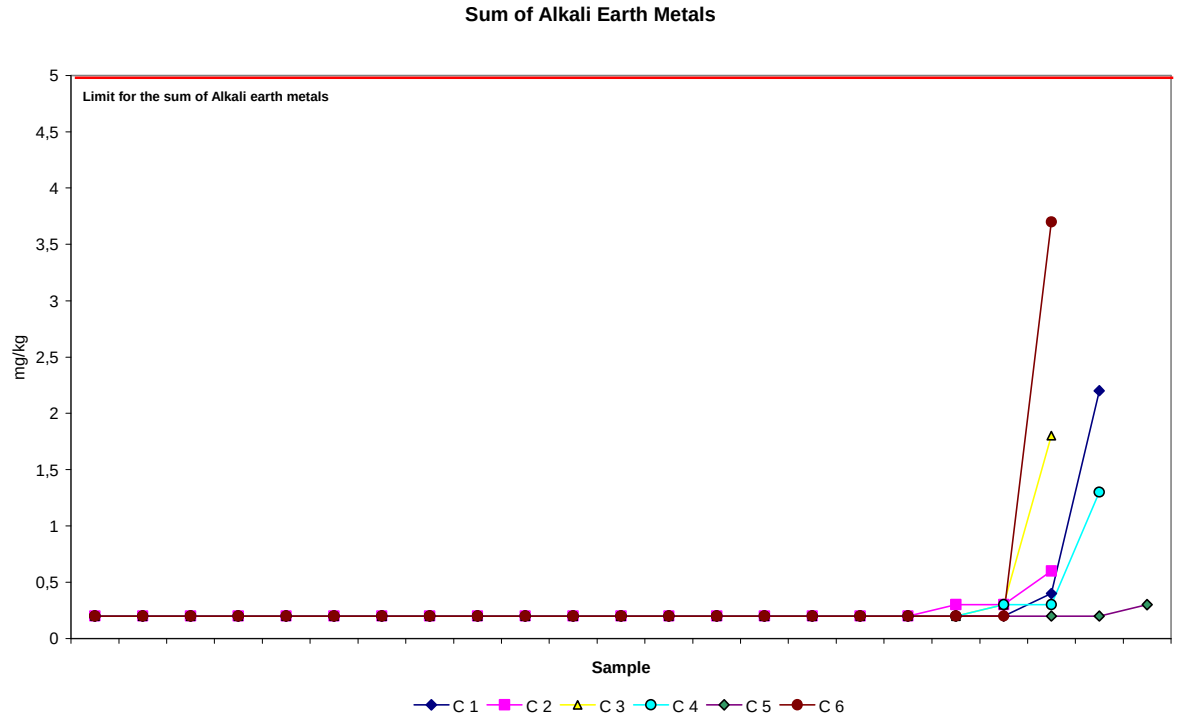


In none of the cases the sum of the Alkali metals reached the limit of 5 mg/kg. 90 % of the samples show a content of less than 2 mg/kg. Fears of the automotive industry assuming that a high sodium proportion may result in ash forming and thus deposits on the surface of particle filters and oxidation 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 that all measured values are way below the required standard limit and therefore, Alkali elements are completely harmless.

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. DIN EN 14214 restricts the sum of the calcium and magnesium contents to 5 mg/kg Ca + Mg: ≤ 5 mg/kg).

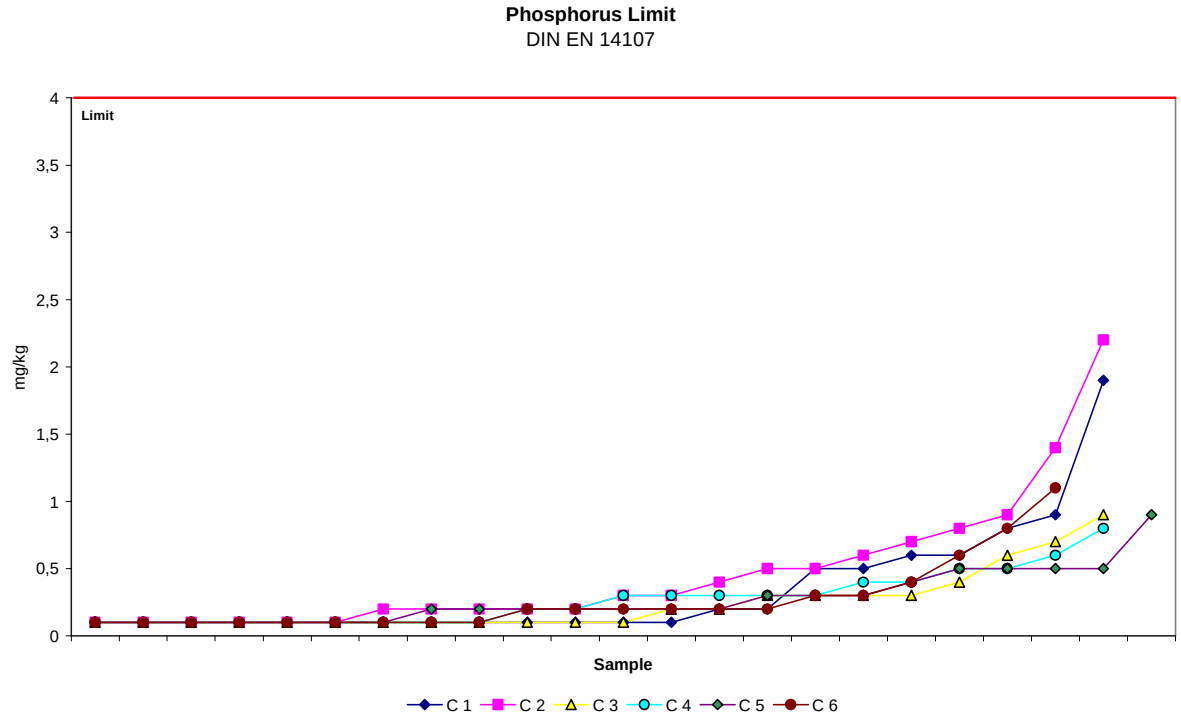


As it is the case with Alkali metals the sum of the Alkali earth metals does not reach the limit in any case. All cumulative values stay below 4 mg/kg. The content of Alkali earth metals is even lower than 1 mg/kg in more than 95 % of the samples. These results illustrate that even the lower limits called for by the automotive industry are considerably undercut.

Phosphorus Content

Test method: DIN EN 14107:2003
Limit DIN EN 14214:2010/2012: ≤ 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.



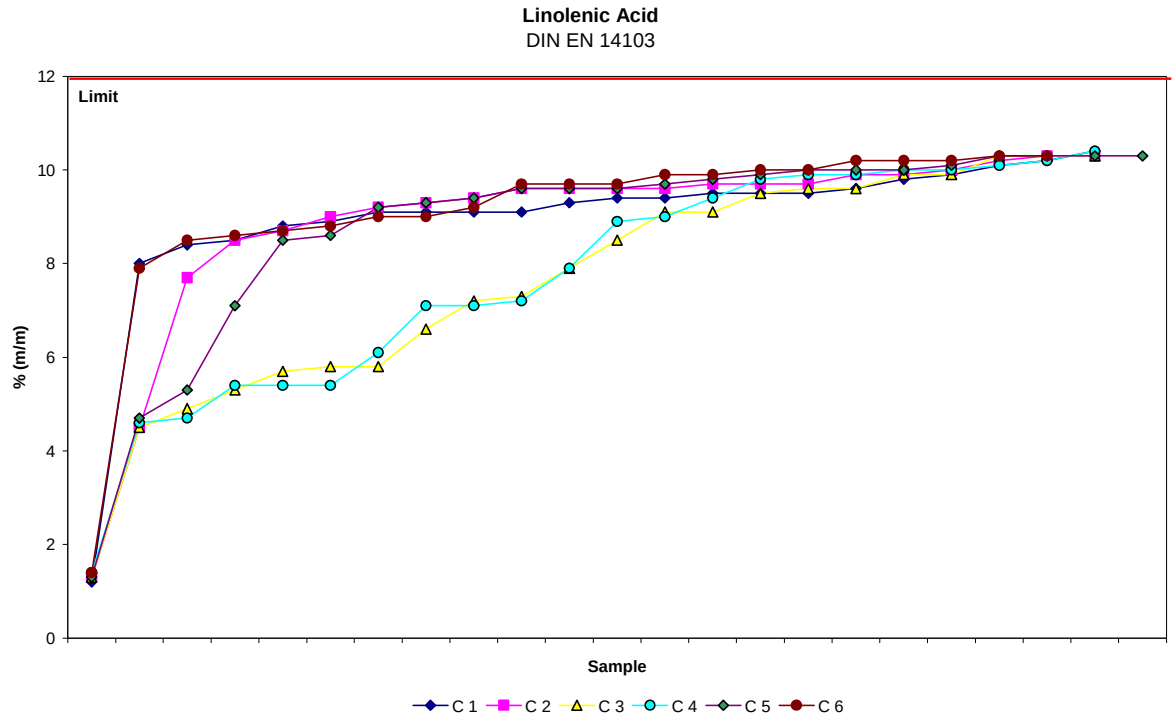
None of the samples shows a phosphorus content of more than 2.2 mg/kg of FAME. In 97 % of the samples the phosphorus content stays even below 1 mg/kg of FAME. As it is the case with Alkali and Alkali earth metals, the precision of the method does not allow any reduction of the limit. Low phosphorus content 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.

Linolenic Acid

Test method: DIN EN 14103:2003
Limit DIN EN 14214:2010: ≤ 12.0 % (m/m), rejection limit at the most: 14.2 % (m/m)

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 linoletic acid content in rapeseed oil is 8 to 10 %. Gas chromatography is implemented for the detection and assay of this acid.



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.

Cold Filter Plugging Point (CFPP)

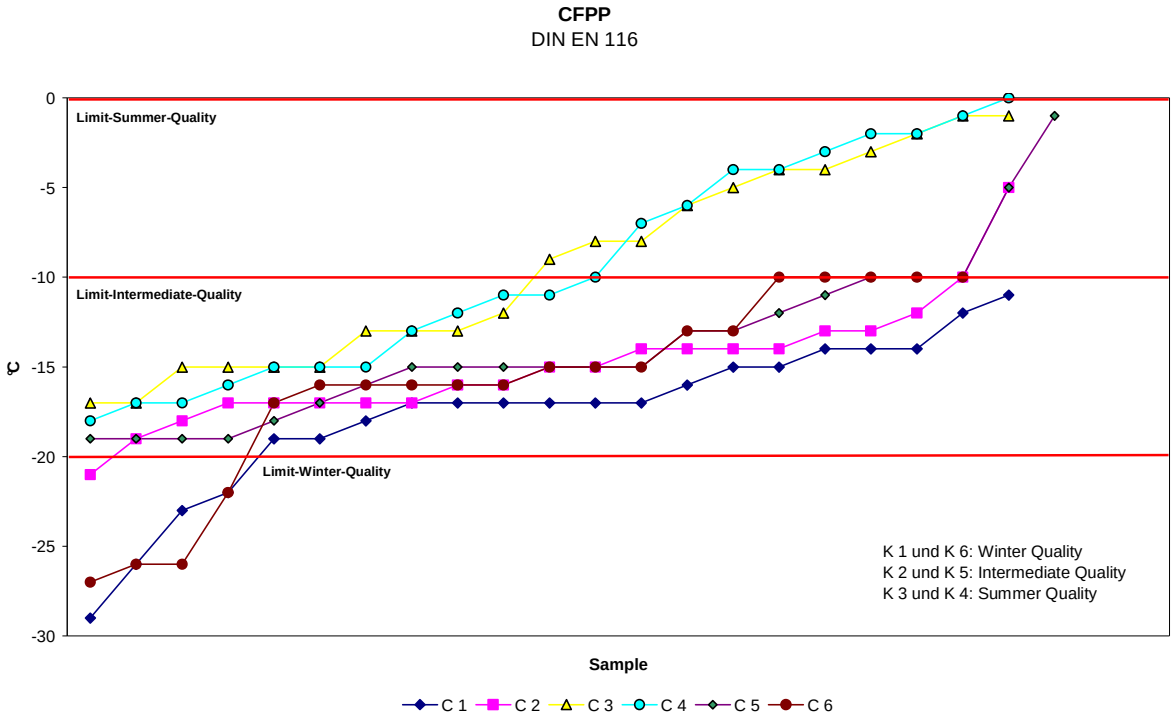
Test method: DIN EN 116:1997

Limit according to DIN EN 14214:2010/2012:		*Rejection limits:*
from 15.4. to 30.9.	*0°C*	*1.5 ℃*
from 1.10. to 15.11.	*-10 ℃*	*-7.9 ℃*
from 16.11. to 28./29.2.	*-20 ℃*	*-17.3 ℃*
from 1.3. to 14.4.	*-10 ℃*	*7.9 ℃*

AGQM Limits: -20℃ (maximum) from 19.10. to 28./29. 2.

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 ℃ applies for winter quality, FAME must yet be able to achieve a CFPP of -20 ℃ by means of suitable additivation.



Considering the individual sampling periods, the winter and summer qualities comply with the requirements of the standard. There are three limit violations for intermediate quality which can be explained as follows:

Sample 1: The sample was produced during the intermediate period; however, it had been intended as summer quality and was only marketed later in the summer time.

Sample 2: The sample served as blend component for Biodiesel; i.e. the tested Biodiesel had been mixed with other Biodiesel before it was marketed.

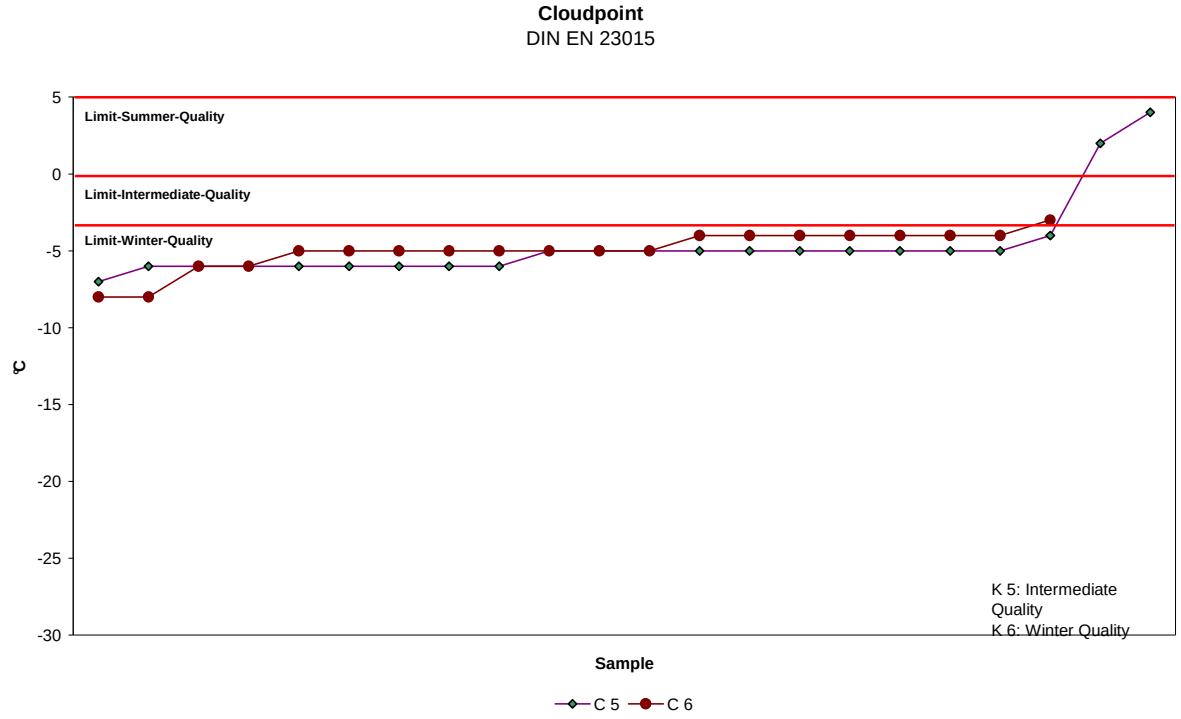
Sample 3: This sample is the sample repeatedly mentioned before which could not comply with the limits for ester content and oxidation stability either. The reasons for that are still unresolved.

Cloudpoint (CP)

Test method: DIN EN 23015:1994

Limit according to DIN EN 14214:2012:		*Rejection limits:*
from 15.4. to 30.9.	*5°C*	*7.4 °C*
from 1.10. to 15.11.	*0 °C*	*2.4 °C*
from 16.11. to 28./29.2.	*-3°C*	*-0.6 °C*
from 1.3. to 14.4.	*-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. As described before, from campaign 5 the Cloudpoint was determined additionally. Since DIN EN 14214:2010, which did not comprise the Cloudpoint, was still in force at the time of campaign 5, there was no limit applicable at the time of sampling. It was only after DIN EN 14214:2012 was published that varying limits became applicable for the Cloudpoint dependent on the time of year. The diagram shows that the limit of DIN EN 14214 was not exceeded.

Summary

As an essential element the AGQM quality management system encompasses regular product quality checks by means of unannounced sampling. The results of 132 samples thus obtained in 2012 were assessed by AGQM's head office; 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.

Despite the excellent performance of our member companies, there were some cases of limit and standard violation. The following table shows the number of standard violations as well as AGQM limit violations in 2012 in absolute figures and percentages.

Parameter	Number of Limit Violations (DIN EN 14214)	Limit Violations in % (DIN EN 14214)	Number of AGQM Limit Violations	AGQM Limit Violations in %
Ester Content	1	0.8	-	-
Density (15 ℃)	0	0	-	-
Sulphur Content	0	0	-	-
Water Content	0	0	2	1.5
Total Contamination	1	0.8	5	3.8
Oxidation Stability at 110 ℃	2	1.5	-	-
Acid Number	0	0	-	-
Iodine Number	0	0	-	-
Glycerol/Glycerides	0	0	-	-
Alkali Content (Na + K)	0	0	-	-
Earth Alkali Content (Ca + Mg)	0	0	-	-
Phosphorus Content	0	0	-	-
Linolenic Acid	0	0	-	-
CFPP	1	0.8	0	0
Cloud Point	0	0	-	-

The evaluation of all 132 tested samples shows that considering the relevant precisions of the test methods 129 samples, almost 98 %, comply with the requirements of DIN EN 14214. Just 3 samples could not meet the requirements with regard to one or more parameters.

For the parameters ‚water content', ‚total contamination' and ‚CFPP' AGQM introduced more stringent limits than those of the standard. The evaluation shows that principally German Biodiesel producers have no problem to also comply with those more rigid requirements. Still, the AGQM limits were exceeded in two cases with regard to the water content and in five cases concerning the total contamination. The sources for failing were identified in all cases and corrective measures could be implemented successfully as the tests of the subsequent campaigns prove.

For the automotive industry and its suppliers it is especially important that the concentration of Alkali and Earth Alkali in Biodiesel is as low as possible because those elements form soaps with free fatty acids and may thus cause filter plugging and gumming of the injection pumps and jet needles. Ash forming is another important aspect: sodium in particular deposits on the surfaces of particle filters and oxidation catalysts and thus reduces the effectiveness and duration of the systems. If too high, the phosphorus content may also cause damage. Phosphorus is a typical catalyst poison which may destroy the effect of exhaust gas after-treatment systems irreversibly. The results of the sampling illustrates that the measuring values stay way below the standard limits and the concentrations of Alkali, Earth Alkali and phosphorus are therefore completely harmless.

Once again the overall result of the sampling campaign of production plants and storage operators in 2012 impressively proves the high quality level of AGQM's member companies.

Arbeitsgemeinschaft
Qualitaetsmanagement Biodiesel e.V.
Claire-Waldoff-Strasse 7
D-10117 Berlin

Annex

Table 1: Limits and Test Methods for the Parameters Tested of the Samples of Campaigns 1 to 5 according to DIN EN 14214:2010

Test Parameter	Method	Year of Publication	Unit	Standard Limits		Rejection Limits	
				min.	max.	min.	max.
Ester Content	DIN EN 14103	2003	% (m/m)	96.5	-	94.7	-
Density at 15 ℃	DIN EN ISO 12185	1997	kg/m³	860	90 0	859.7	900.3
Sulphur Content (UV)	DIN EN ISO 20846	2004	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[3]	mg/kg	-	24	-	32
Oxidation Stability at 110 ℃	DIN EN 14112	2003	h	6 .0	-	4.9	-
Acid Number	DIN EN 14104	2003	mg KOH/g	-	0.50	-	0.54
Iodine Number	DIN EN 14214 Annex	2010	g Iodine/100g	-	120	-	124
Iodine Number	DIN EN 14111	2003	g Iodine/100g	-	120	-	123
Linolenic Acid Content	DIN EN 14103	2003	% (m/m)	-	12.0	-	14.2
Content of free glycerol	DIN EN 14105	2003	% (m/m)	-	0.02	-	0.032
Monoglyceride Content		2003	% (m/m)	-	0.80	-	0.94
Diglyceride Content		2003	% (m/m)	-	0.20	-	0.24
Triglyceride Content		2003	% (m/m)	-	0.20	-	0.26
Total Glyceride Content		2003	% (m/m)	-	0.25	-	0.31
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	℃	from 15.4 to 30.9 from 1.10 to 15.11 from 16.11 to 28/29.2 from 1.3 to 14.4	0 -10 -20 -10	- - - -	1.5 -7.9 -17.3 -7.9
Cloudpoint	DIN EN 23015	1994	℃	-	-	--	-

[3] 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.

Arbeitsgemeinschaft
Qualitaetsmanagement Biodiesel e.V.
Claire-Waldoff-Strasse 7
D-10117 Berlin

Table 2: Limits and Test Methods for the Parameters Tested of the Samples of Campaign 6 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 at 15 °C	DIN EN ISO 12185	1997	kg/m³	860	90 0	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[4]	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 15.4 to 30.9 from 1.10 to 15.11 from 16.11 to 28/29.2 from 1.3 to 14.4	0 -10 -20 -10	- - - -	1.5 -7.9 -17.3 -7.9
Cloudpoint	DIN EN 23015	1994	°C	from 15.4 to 30.9 from 1.10 to 15.11 from 16.11. to 28/29.2 from 1.3 to 14.4	5 0 -3 0	- - - -	7.4 2.4 -0.6 2.4

[4] 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.