Biodiesel Quality in Germany

Results of the Sampling Campaigns
at AGQM Production Plants
and Warehouse Operators

2015

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1 Introduction

Arbeitsgemeinschaft Qualitaetsmanagement Biodiesel e.V. (AGQM) is the alliance of Biodiesel producers and traders who joined forces to market a product which safely complies with the requirements of the standard (DIN EN 14214). AGQM represents about two thirds of the Biodiesel producers in Germany.

In Germany, Biodiesel – also known as FAME (Fatty Acid Methyl Ester) – is still the most important fuel based on renewable raw materials. However, nowadays it is marketed almost exclusively as B7 by admixture to Diesel fuel. Rapeseed oil is still the raw material which is used mainly for the German Biodiesel production. In addition, more and more used cooking oils and fats (Used Cooking Oil Methyl Ester / UCOME) are used as well as soybean and palm oils.

Within the EU there are two decisive directives for the use of Biofuels. According to the ‚Renewable Energy Directive‘ (RED), by 2020 in the transport sector the proportion of renewable energy must be at least 10 %. For 2015 the ‘Fuel Quality Directive’ (FQD) stipulates a reduction of 3 % of greenhouse gas (GHG) for emissions from fuels by using biofuels; the percentage shall rise in two steps to 6 % in the year 2020. In Germany both directives are put into practice as part of the ‘Bundes-Immisionsschutzgesetz’ (BISchG).

Since 2015, in Germany the Biofuel quota is no longer determined on the basis of the energy contents but with regard to the greenhouse gas saved, thus GHG based. This means that mineral oil companies are no longer given a set energetic proportion of Biofuels but the percentage of greenhouse gases which must be saved with the use of Biofuels. For that there is a specific default value for every type of Biodiesel, which can be used if the balance cannot be self-determined. However, this default value is not very attractive. For example, the default value for Biodiesel produced from rapeseed oil is 38 %.

Thus Biodiesel producers observe and add up the GHG emissions along the production chain step by step, starting with the agricultural cultivation. With the implementation of appropriate measures and thus lowering the emissions, the Biofuel industry succeeded in raising the GHG savings to about 60 %. However, on the downside the effect is that the fuel amount necessary to fulfill the quota is reduced correspondingly. Consequently, the Biodiesel sales volume in Germany has decreased considerably since the introduction of the GHG quota.

Until the end of 2014 the use of used cooking oils and fats was double counted for the quota. Since 2015 such raw materials can be used as waste and residual materials with a GHG preload of 0 g CO_{2eq}/MJ so that UCOME achieves a GHG savings potential of 80 to 85 %.

AGQM's quality management system (QM system), which has been successfully implemented by AGQM members for many years, is the basis for any quality assurance measures. It is continually revised by AGQM's Quality Assurance Committee (QA Committee) so that it continues to meet the increasing requirements with regard to the Biodiesel quality. The QA Committee members are experts in the field of quality management and come from AGQM member companies as well as non-AGQM companies like commercial laboratories.

While in some other European countries there are massive quality problems with regard to blending Biodiesel, in Germany the operation with Biodiesel has been smooth for many years. Certainly AGQM's QM system has contributed significantly to this development. In 2010 the results of the unannounced sampling of AGQM member companies was published in a quality report[1] for the first time. The extremely positive quality development of AGQM members' products which was decisively due to AGQM's quality assurance measures, can thus be observed and clearly tracked.

[1] http://www.agqm-biodiesel.de/en/downloads/reports/

2 Description of Sampling

In 2015 there were four sampling campaigns. Member sampling is one of the most important AGQM quality assurance measures. Sampling without prior announcement is of crucial importance since it assures that the results reflect the actual Biodiesel production and handling by our members. Sampling is not carried out by AGQM itself but there is an annual call for tenders which leads to the assignment to an independent laboratory accredited for Biodiesel analytics. The laboratory must have successfully participated in AGQM's annual Round Robin Test for fatty acid methyl ester (FAME), jointly carried out by AGQM and *Fachausschuss für Mineralöl- und Brennstoffnormung (FAM) im DIN*.

The Biodiesel parameters to be tested are determined by the QA Committee in the QM system. All parameters essential for the verification of the fulfilment of the standard according to the legal stipulations of the 36th BImSchV are included.

The relevant current version of the standard always forms the basis of AGQM's quality check, i.e. the required standard limits as well as their related acceptance limits comply with DIN EN 14214:2014. In addition, more stringent requirements, so-called 'AGQM limits', were determined for some parameters which documents AGQM's particular quality commitment. In December 2014, the German 10th BImSchV – which stipulates the properties of Biodiesel – was adapted to the latest version of the standard. Thus the problem arising in 2014 of some Biodiesel fulfilling the legal requirements for admixture of the German 10th BImSchV on the one hand, but not meeting the quality requirements of AGQM on the other hand, is thus no longer existent.

The tested parameters and their limits according to DIN EN 14214:2014 are listed in table 1 of the attachment. Table 2 subsequently shows the parameters of the AGQM requirements which are higher than those of the current standard. For parameters 'water content', 'total contamination', and 'Cold Filter Plugging Point (CFPP)' AGQM's requirements for the Biodiesel quality of its members are more stringent than those demanded by legal stipulations.

AGQM also supports special needs of its members. In 2013 a special regulation was established for Biodiesel made from used cooking oil and fats. Biodiesel produced thereof is exempted from the determination of the 'sulphur content', 'CFPP' and 'Cloud Point' and is not sanctioned if the limits of those parameters are exceeded. However, such Biodiesel must not be marketed directly but only as blend component for Biodiesel.

In 2015 sixteen Biodiesel producers and one trader participated in AGQM quality assurance measures; beside the production plants two fuel depots of the trader were sampled. Spread throughout the year four sampling campaigns took place in different seasons; 65 Biodiesel samples were taken, analysed and then evaluated. Compared to the previous year the number of tested samples dropped by 8. The reason for the reduced number of samples is that in 2015 two AGQM member companies quit or were excluded.

The sampling dates were selected so that the AGQM member companies were sampled in summer, in winter and during the intermediate period because for summer, winter and intermediate grades there are different limits for parameters 'Cold Filter Plugging Point (CFPP)' and 'Cloud Point' which are stipulated in the national Attachment NB of the standard and differ from country to country since their climatic conditions are also different. The individual campaigns are named K1 to K4. The sampling periods are listed below:

K1:	26 January to 6 February	Winter grade
K2:	17 April to 30 April	Summer grade
K3:	6 July to 17 July	Summer grade
K4:	5 October to 16 October	Intermediate grade

3 Individual Results and Evaluation

In the following section the test method used, the limit, the acceptance limit and a brief description can be found for every parameter followed by a graphical illustration of the evaluated measuring values. In case of questions regarding the individual parameters please consult the Quality Report of 2014 which is very detailed and contains additional information.

The results given in this report were made anonymous and do not reveal the origin of the sample. Internally AGQM numbered all samples. However, this individual number is only given in the report so that conspicuous features of individual samples can be pointed out, should an acceptance limit have been exceeded.

The measuring values for every sampling campaign are given in ascending order to illustrate the spread. The axis 'Number of Samples' illustrates how many samples were taken in the relevant campaign; the internally assigned numbers are not given. In the diagrams the limits are marked by a black line, the acceptance limits – determined considering the precision of the method – by a red line. The acceptance limits are the decisive factor when it comes to Customs matters or the assignment of sanction points according to AGQM's QM system. In the diagrams of parameters 'total contamination', 'water content', and 'CFPP' both AGQM limit and AGQM acceptance limit are given additionally.

As mentioned in the section before, there is an exception for parameters ‚sulfur content', ‚CFPP', and ‚Cloud Point'. AGQM members producing Biodiesel from used cooking oils and fats may exceed the quality parameters of the standard if they apply for exemption at the AGQM office in advance. The measuring values referring to such an exception are marked accordingly in the diagrams.

In the following section the sampling results are graphically illustrated and discussed individually for every parameter.

3.1 Fatty Acid Methyl Ester Content

Test method: *DIN EN 14103:2011*
Limit of DIN EN 14214:2014: ≥ 96,5 % (w/w)
Minimum acceptance limit: 94,0 % (w/w)

The content of fatty acid methyl esters, briefly ester content, is a measure for the purity of Biodiesel. The ester content is determined by gas chromatography and given as sum of all fatty methyl esters from C6:0 to C24:1 in weight by weight [% (w/w)]. DIN EN 14214 demands a minimum Fatty Acid Methyl Ester Content of 96,5 % (w/w).

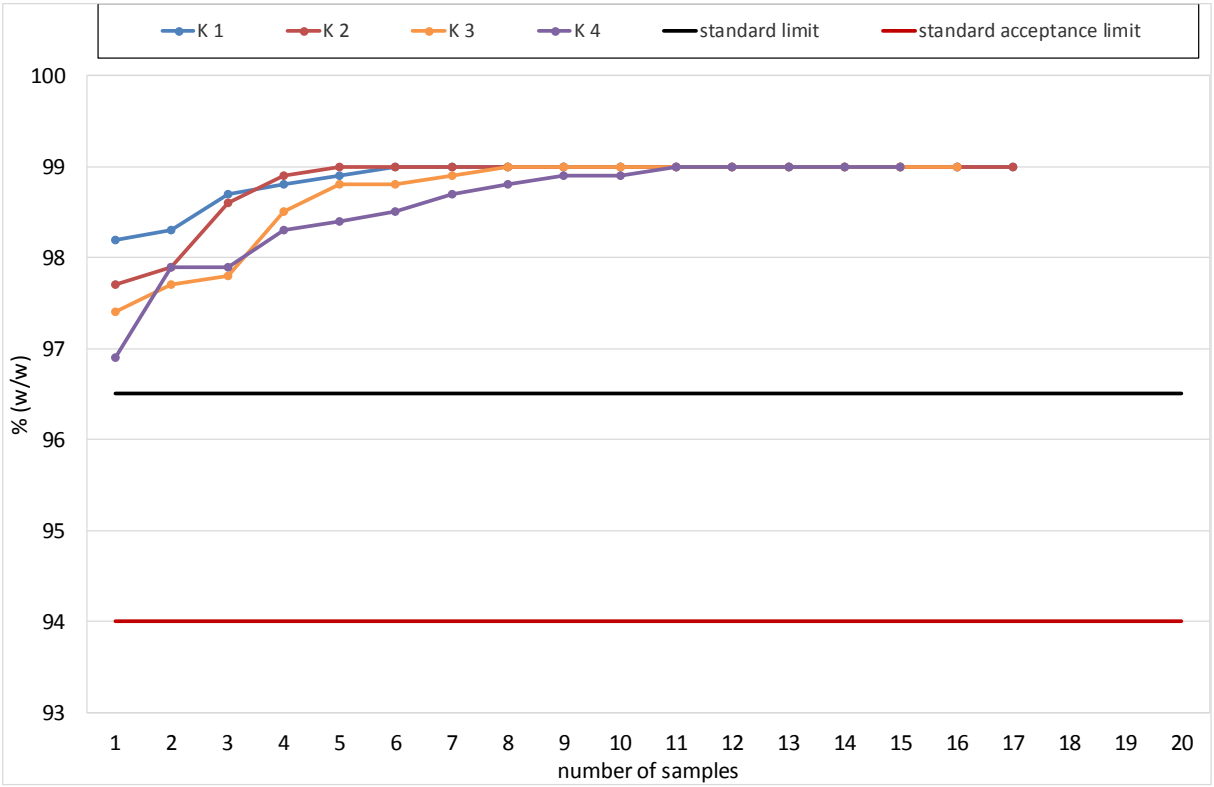

Diagram 1: Fatty Acid Methyl Ester Content according to DIN EN 14103.

Diagram 1 shows the values of the Fatty Acid Methyl Ester Content of the tested sample. The evaluation of the results reveals that all samples fulfill the requirements of the standard.

3.2 Density at 15 °C

Test method: DIN EN ISO 12185:1997

Limit of DIN EN 14214:2014: between 860 and 900 kg/m³

Minimum acceptance limit: 859,7 kg/m³; maximum acceptance limit: 900,3 kg/m³

The density of a substance is the quotient of its mass and volume at a stipulated temperature. It is a substance-specific property and is determined by means of an oscillating U-tube density meter. According to DIN EN 14214 the density of Biodiesel must be between 860-900 kg/m³ at 15 °C.

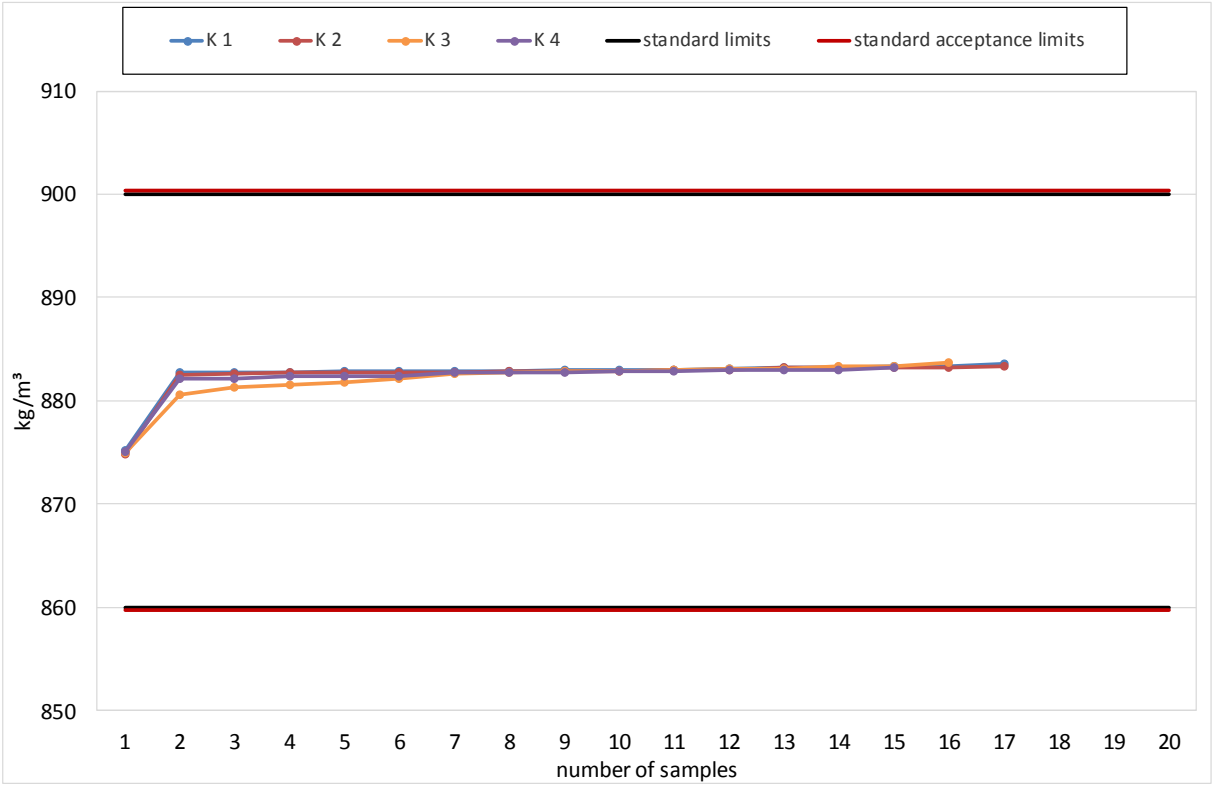

Diagram 2: Density at 15 °C DIN EN ISO 12185.

Diagram 2 shows the measured values for parameter ‘density’. It is clearly visible that all analysed samples meet the density range demanded by the standard. Almost all analysed samples range very closely between 881 and 883 kg/m^3. The predominant majority even meets the exact value of 883 kg/m for Biodiesel from rapeseed oil.

3.3 Sulfur Content

Test method: DIN EN ISO 20846:2011
Limit of DIN EN 14214:2014: ≤ 10 mg/kg
Maximum acceptance limit: 11,3 mg/kg

Biodiesel can contain sulfur compounds from different sources: on the one hand from the use of a sulfurous catalyst for the production and on the other hand from the raw materials used. If vehicles are operated with fuel rich in sulfur more sulfur dioxide and particulates are thereby emitted. Furthermore, sulfur is a catalyst poison which reduces the operating time of the catalytic converters in the vehicles. For that reason, the sulfur content in Biodiesel is limited to 10 ppm.

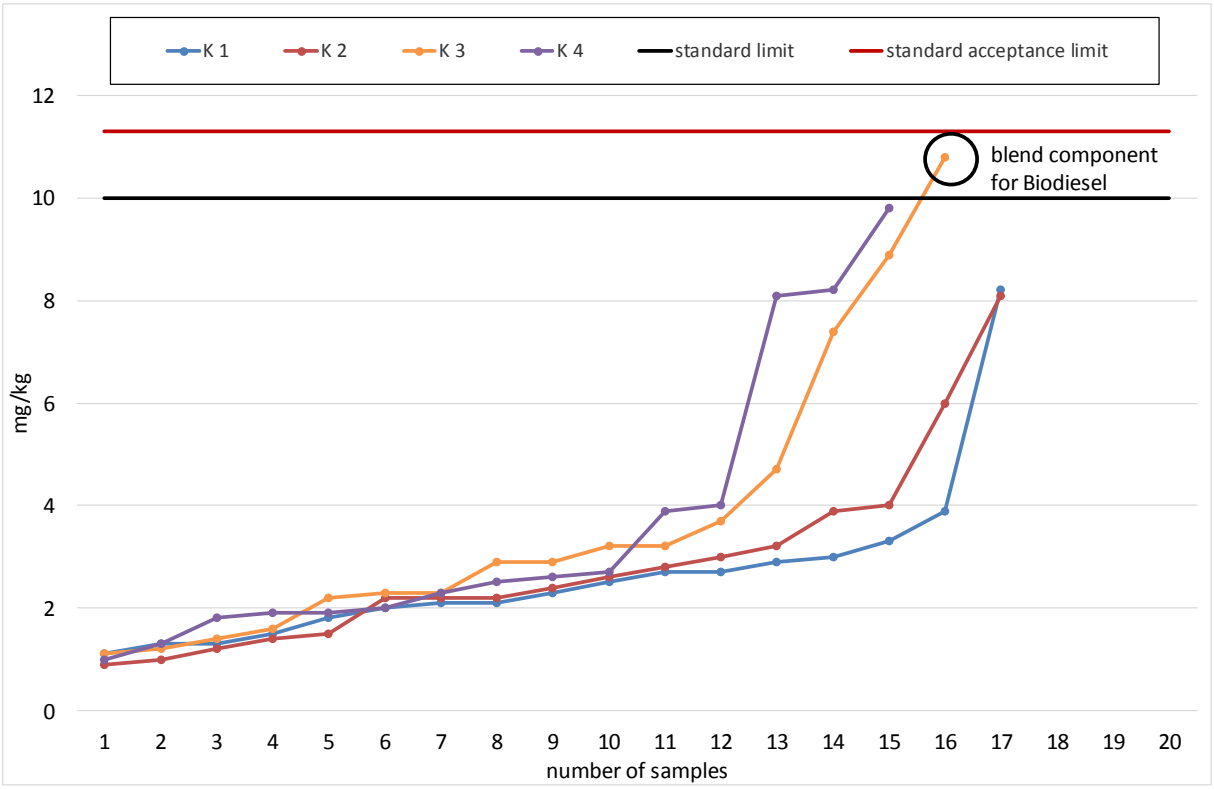

Diagram 3: Sulfur Content according to DIN EN ISO 20846.

Diagram 3 shows the measuring values of the sulfur content of the tested samples. With the exception of the sample in the black circle, which is a blend component for Biodiesel, all samples fall below the limit.

3.4 Water Content

Test method: DIN EN ISO 12937:2000

Limit of DIN EN 14214:2014: ≤ 500 mg/kg

Maximum acceptance limit: 591 mg/kg

AGQM limit: ≤ 220 mg/kg for producers

Acceptance limit: 280 mg/kg

AGQM limit: ≤ 300 mg/kg for warehouse operators

Acceptance limit: 370 mg/kg

Due to its polar properties Biodiesel can physically dissolve large amounts of water as opposed to carbon-based fuels. Since almost all Biodiesel production processes use water to remove free glycerol, soaps and other contamination as last refining step the product must be dried subsequently. Due to high air humidity water can ingress into Biodiesel so its storage conditions must be selected accordingly. In order to compensate any possible water ingress AGQM introduced more stringent limits for this parameter.

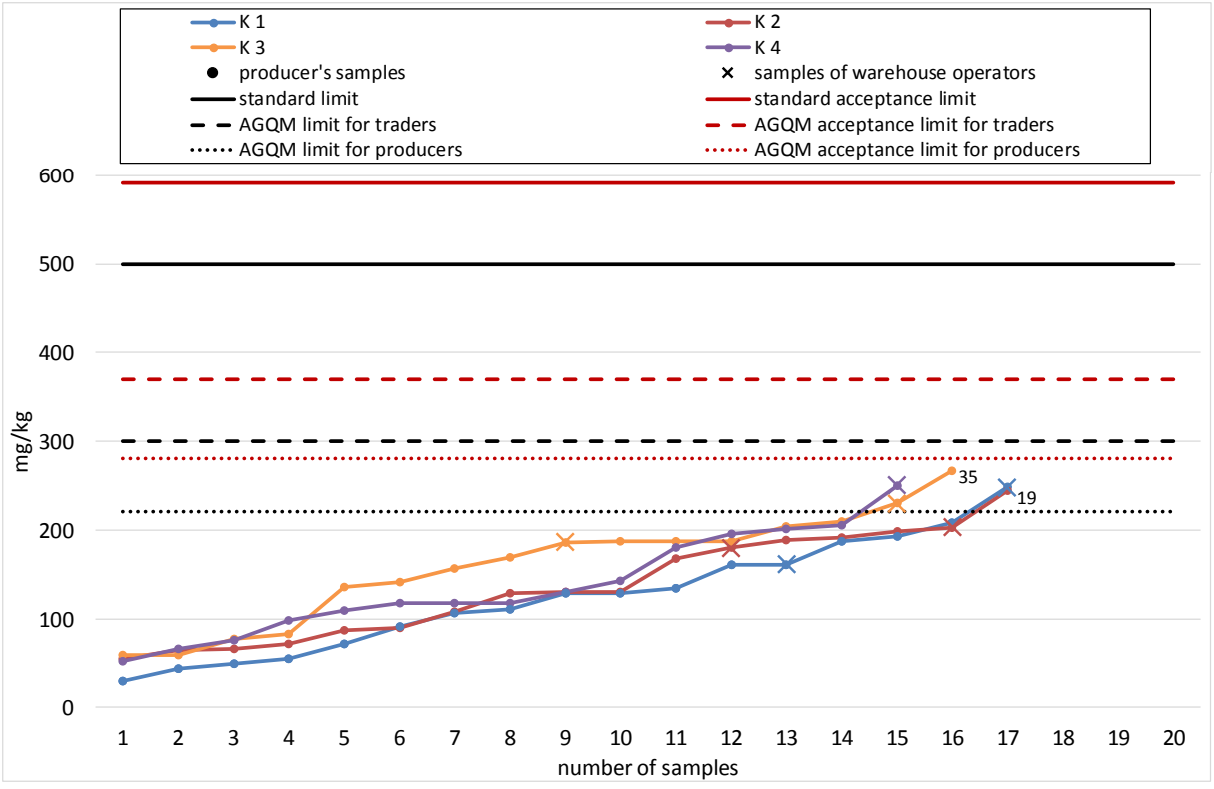

Diagram 4: Water Content according to DIN EN ISO 12937.

Diagram 4 illustrates that all tested samples fall significantly below the standard limit. However, it also shows that two production samples in K2 and K3 (19 and 35, illustrated as dot) exceeded the AGQM limit for producers (220 mg/kg) with values of 244 and 266 mg/kg. Yet those two values still range within AGQM's acceptance value for producers (280 mg/kg) and the products may be marketed as AGQM conform.

Samples marked 'X' in diagram 4 are samples of warehouse operators. The AGQM limit of 300 mg/kg as well as an acceptance limit of 370 mg/kg applies for such samples. All samples of warehouse operators fulfil AGQM's requirements.

3.5 Total Contamination

Test method: DIN EN 12662:1998
Limit of DIN EN 14214:2014: ≤ 24 mg/kg
Maximum acceptance limit: 32 mg/kg
AGQM limit: ≤ 20 mg/kg
(AGQM's limit for parameter 'total contamination' is also AGQM's acceptance limit.)

Due to the fact that the current version of DIN EN 12662 is unsuitable for FAME 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.

'Total contamination' is a measure for the content of insoluble particles which are obtained by filtration of a heated sample. It is determined gravimetrically by weighing the filters. Biodiesel is usually not distilled which is why 'total contamination' is an important quality feature. Rust, dust but also organic solid substances like sterylglycosides, polymer particles or soaps may be found in Biodiesel. AGQM set its own more stringent limit of 20 mg/kg to improve the application security of Biodiesel and to account for the imprecision of the method.

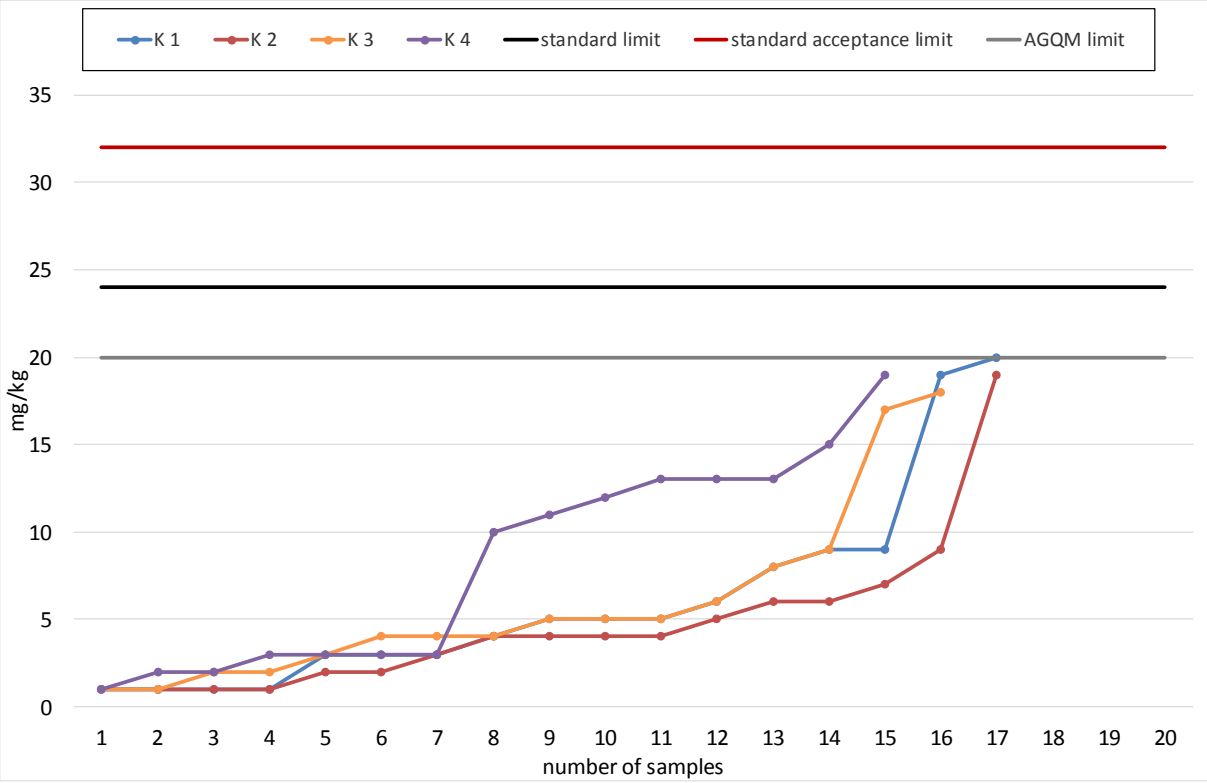

Diagram 1: Total contamination according to DIN EN 12662.

As can be seen from diagram 5 all values range within the AGQM limit for total contamination.

3.6 Oxidation Stability

Test method: DIN EN 14112:2003
Limit of DIN EN 14214:2014: ≥ 8 h
Minimum acceptance limit: 6.6 h

The oxidation stability of Biodiesel is the measure for the resilience against oxidative processes. Test method is EN 14112, the so-called 'Rancimat test'. The limit for the oxidation stability is 8 hours.

Vegetable oils contain natural antioxidants like tocopherols which slow down the ageing process. Synthetic stabilizers are used in addition. Upon request of interested additive producers AGQM annually tests products which can be used to enhance the Biodiesel oxidation stability. Additives passing the test are published in the so-called 'No-Harm List' on AGQM's homepage.

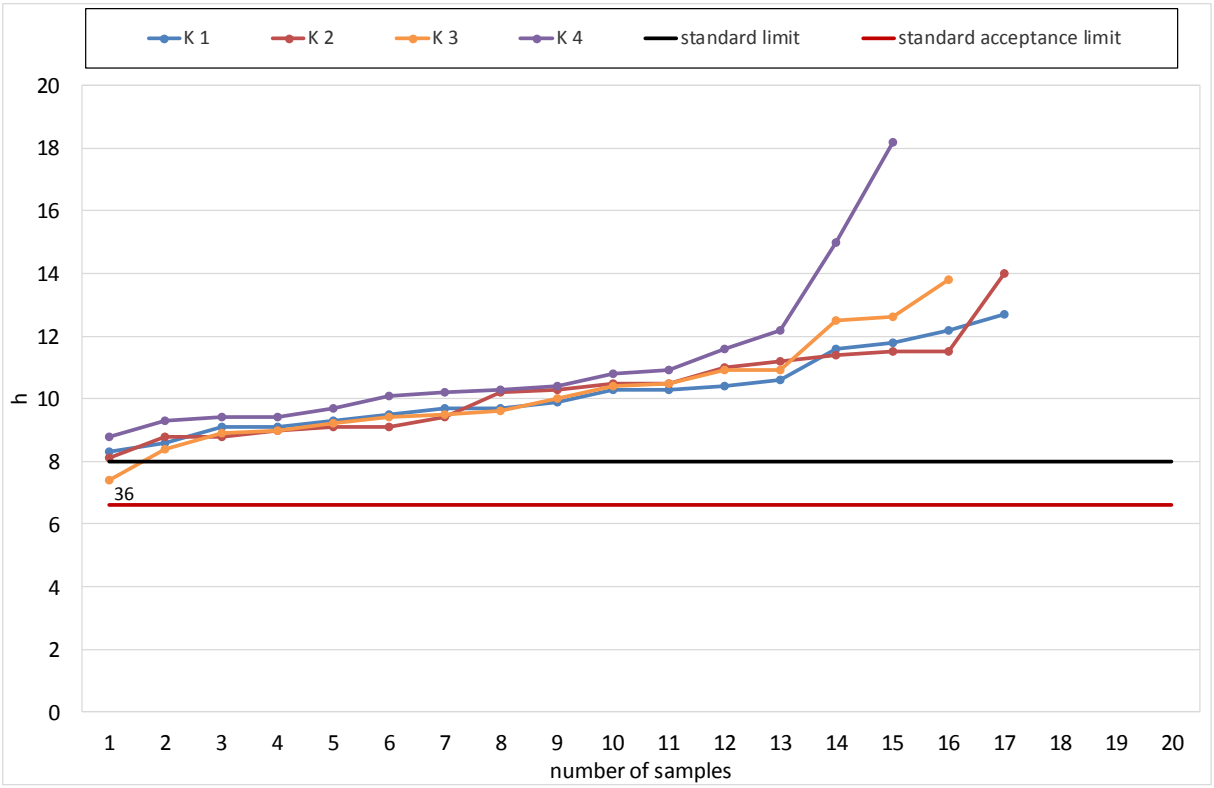

Diagram 6: Oxidation Stability according to DN EN 14112.

The measuring results in diagram 6 show that all but one samples achieve an oxidation stability of more than the limit. With 7,4 h sample 36 falls below the limit but it is still within the acceptance limit (6,6 h).

3.7 Acid Number

Test method: DIN EN 14104:2003

Limit of DIN EN 14214:2014: ≤ 0,5 mg KOH/g

Maximum acceptance limit: 0,54 mg KOH/g

The acid number is the measure for free acids (especially fatty acids) in Biodiesel and thus indirectly for its corrosive properties. However, fatty acids are weak acids and thus only little corrosive. During the storage of FAME the acid number can rise when ageing processes (primarily oxidation) cause ester cleavage and the formation of short-chain carbon acids. However, this effect can hardly be observed under normal storage conditions. Also inorganic acids used for washing, which have a stronger corrosive effect, may contribute to the acid number.

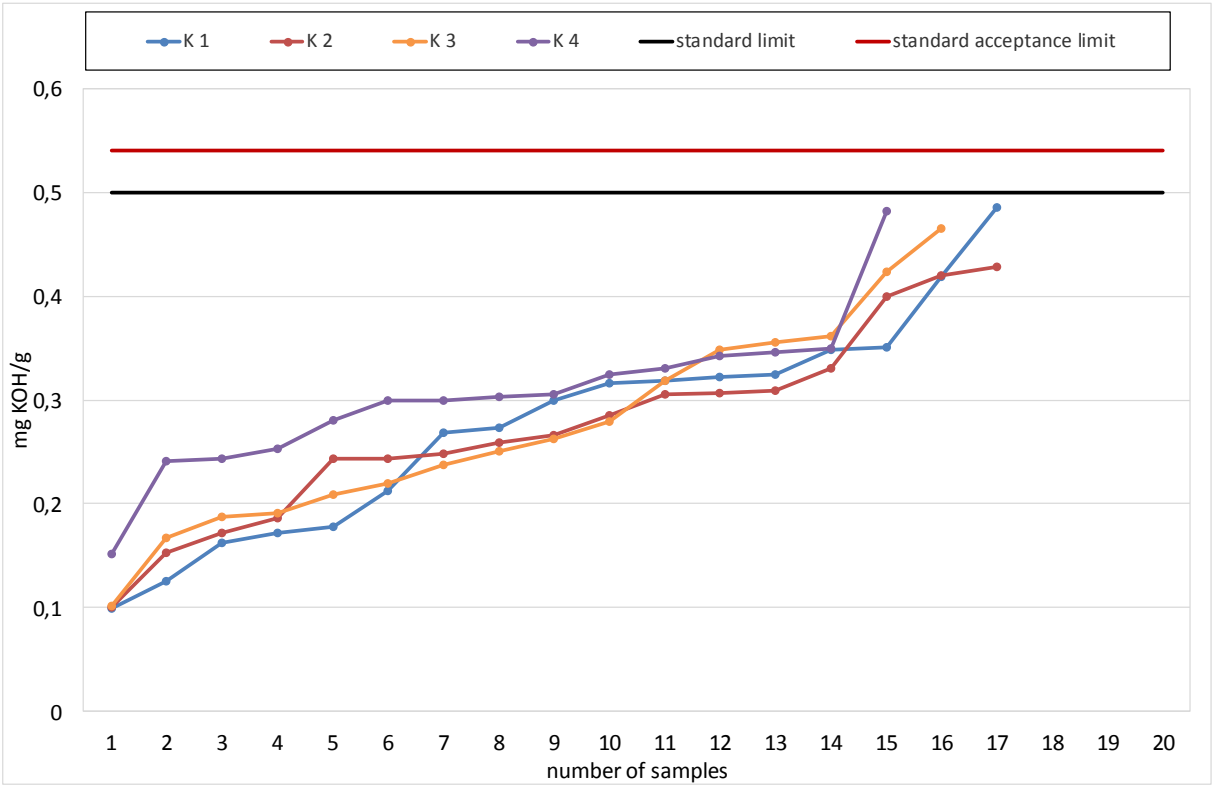

Diagram 7: Acid Number according to DIN EN 14104.

Diagram 7 shows the measurements for the acid number. All samples are below the standard limit (0,5 mg KOH/g).

3.8 Iodine Number

Test method: DIN EN 14111:2003
Limit of DIN EN 14214:2014: 120 g iodine/100g
Maximum acceptance limit: 123 g iodine/100g

Test method: DIN EN 16300:2012
Limit of DIN EN 14214:2014: 120 g iodine /100g
Maximum acceptance limit: 124 g iodine /100g

The iodine number is a measure for the proportion of double bonds in the fatty acids found in fats and oils as well as in Biodiesel. It varies dependent on the raw material used. There are two different methods for its determination: on the one hand arithmetical determination based on the fatty acid profile measured by gas chromatography according to DIN EN 16300; on the other hand, wet chemical determination according to DIN 14111. The result is given in g iodine/100 g of Biodiesel.

Since unsaturated fatty acids are more prone to oxidative reactions, it applies that the Biodiesel stability decreases with the rising number of double bonds thus also rising iodine consumption. Therefore, apart from the oxidation stability, the iodine number is an indication for the stability of Biodiesel.

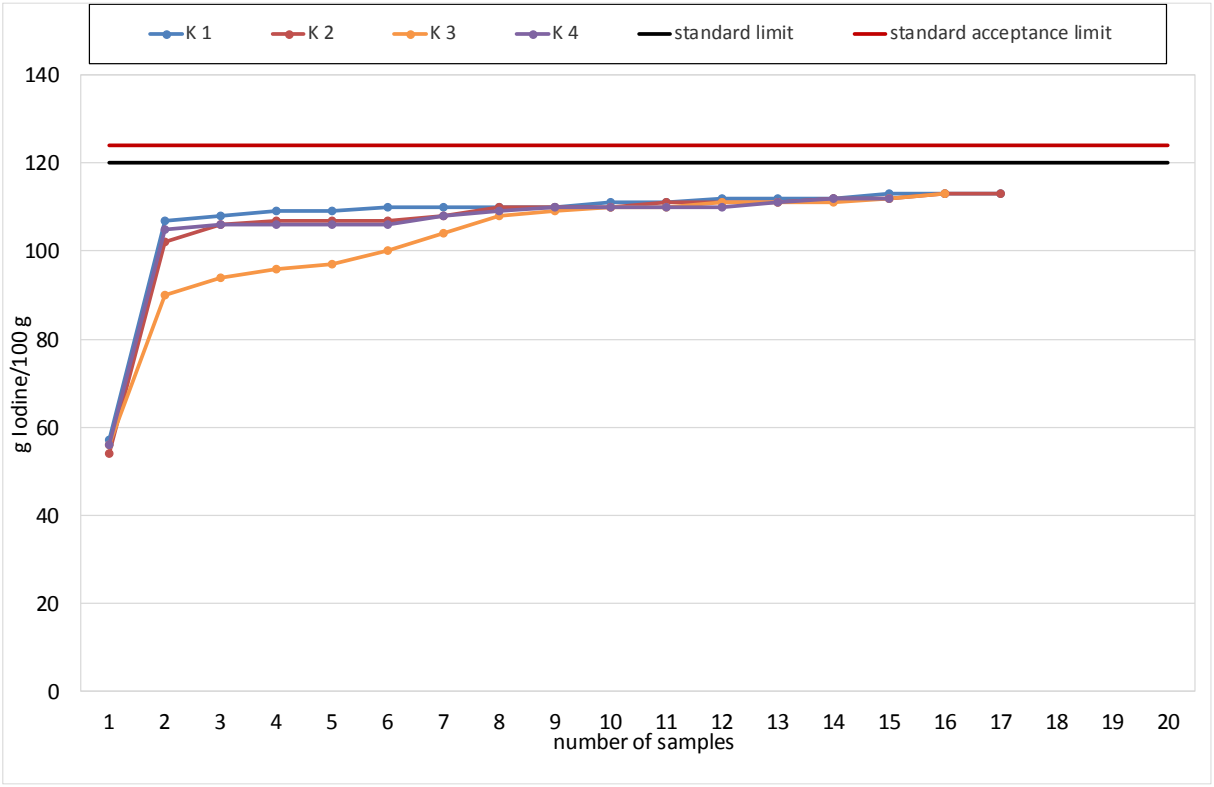

Diagram 8: Iodine Number according to DIN EN 16300 (determined from the methyl ester profile).

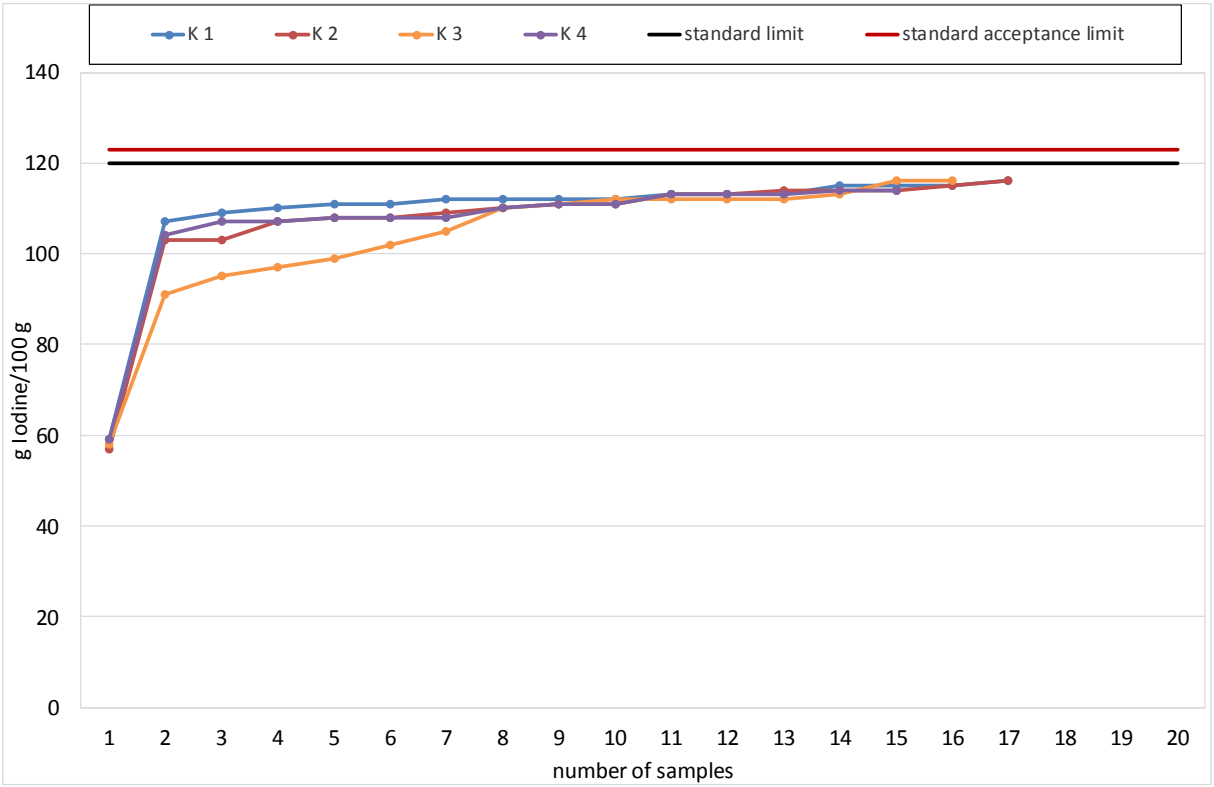

Diagram 9: Iodine Number according to DIN EN 14111 (titrated).

The results of the two test methods for the iodine number (arithmetical determination from the methyl ester composition and titrated) illustrated in diagrams 8 and 9 do not show any difference worth mentioning. All tested samples fall below the limit of the standard. It is remarkable that in K3 some lower iodine numbers were measured which results from the use of raw materials with a higher saturation degree. A high saturation degree causes bad cold properties (with regard to CFPP and Cloud Point) which is of little importance during the summer months.

3.9 Glycerides / Free Glycerol

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

Dependent on the type of reaction control during the transesterification of vegetable oils with methanol, apart from the main product 'fatty acid methyl ester', intermediate products (monoglycerides and diglycerides) and unprocessed vegetable oil (triglycerides) can be found. Therefore, the contents of mono, di-, and triglycerides are a measure for the completeness of the transesterification reaction. In general, the concentration increases in the order 'triglyceride < diglycerides < monoglycerides' since the cleavage of the last fatty acid residue is the slowest step of the reaction. With reasonable effort the glyceride content can only be reduced to a certain degree, since chemical equilibrium between products and educts adjusts in any case. Glycerides can only be completely removed by distillation.

3.9.1 Monoglycerides

Limit of DIN EN 14214:2014: ≤ 0,70 % (w/w)

Maximum acceptance limit: 0,82 % (w/w)

With 0,7 % (w/w) a significantly higher value was selected as limit for monoglycerides compared to those for di- and triglycerides. The reason is that the cleavage of the last fatty acid residue is the slowest step of the transesterification reaction. Here, it is most difficult to achieve a complete reaction.

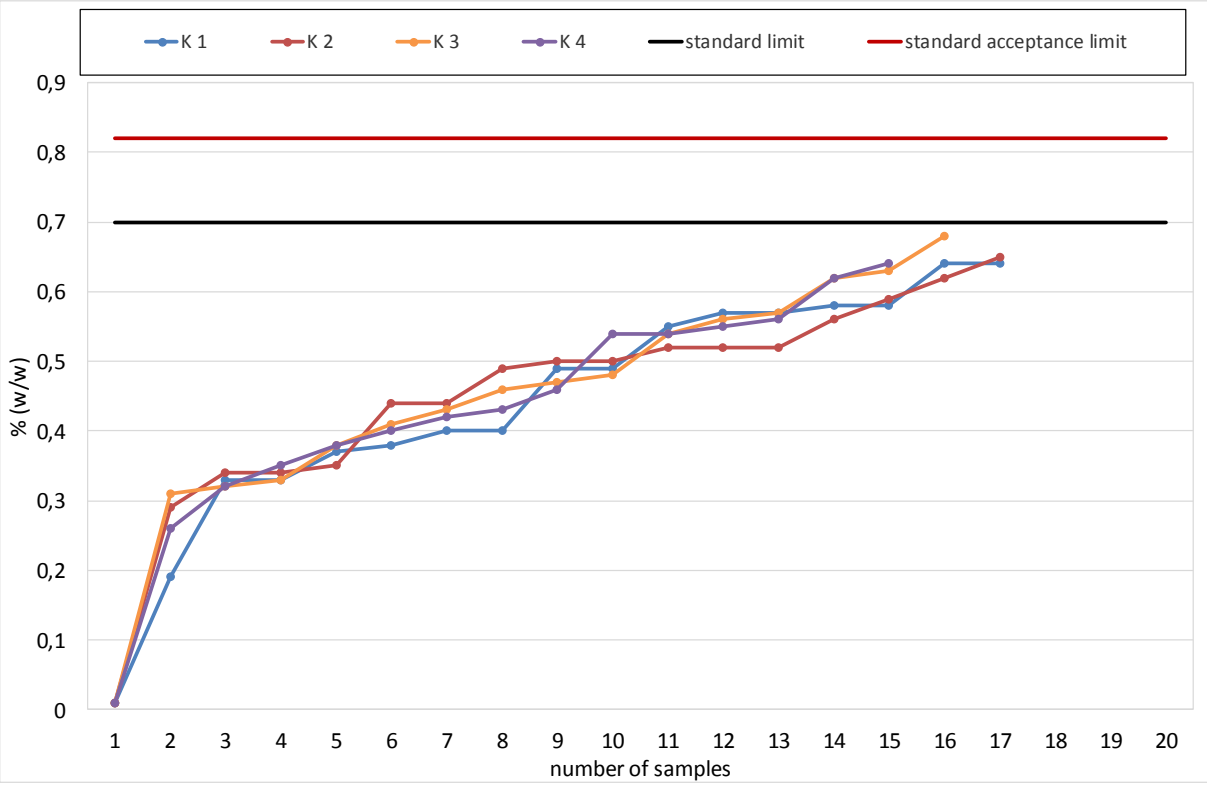

Diagram 20: Monoglycerides according to DIN EN 14105.

Diagram 10 shows the measurements for monoglycerides. All tested samples fulfil the requirements of the standard and fall below the limit of 0,7 % (w/w).

3.9.2 Diglycerides

Limit of DIN EN 14214:2014: ≤ 0,2 % (w/w)

Maximum acceptance limit: 0,24 % (w/w)

Due to their high boiling points, diglycerides are not fully combusted. Thus coking in injector system and cylinder can be caused. The limit for the content of diglycerides is 0,2 % (w/w).

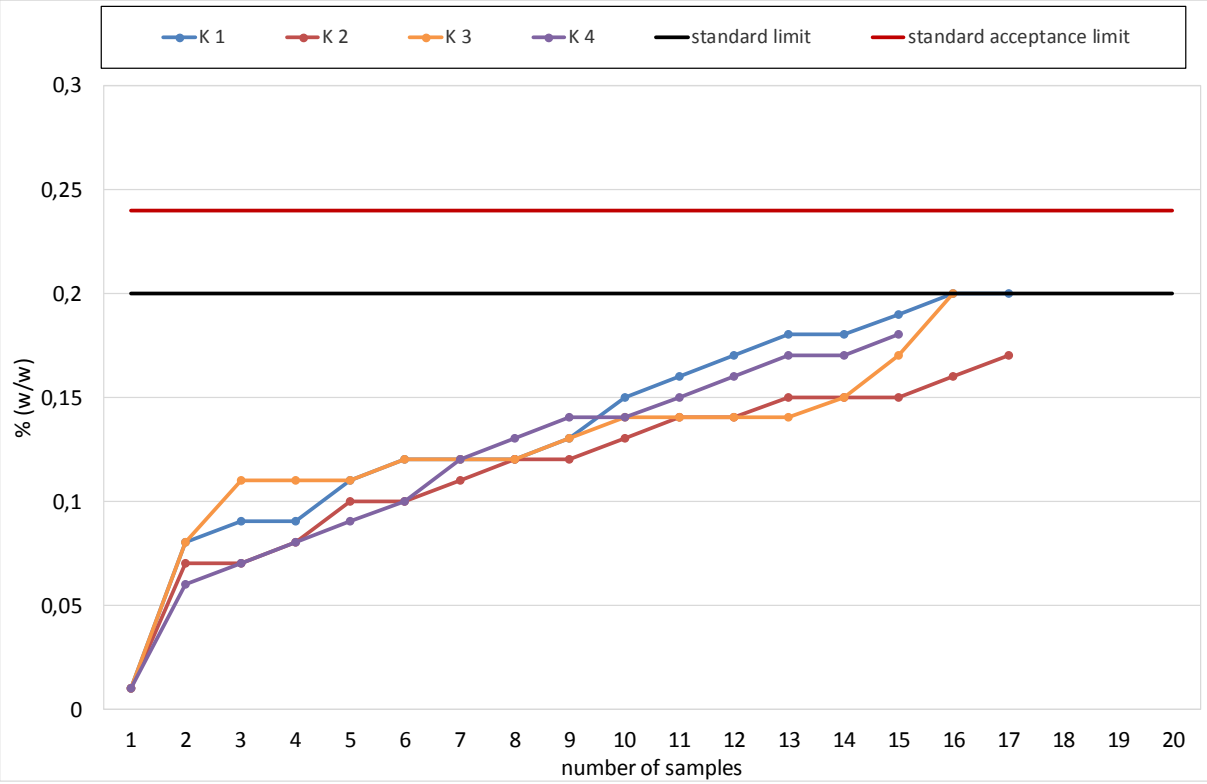

Diagram 11: Diglycerides according to DIN EN 14105

Diagram 11 shows the measurements for the content of diglycerides. All values meet the requirements of the standard.

3.9.3 Triglycerides

Limit of DIN EN 14214:2014: ≤ 0,2 % (w/w)

Maximum acceptance limit.: 0,27 % (w/w)

High contents of triglycerides in combination with low contents of mono and diglycerides are an indication for mixtures of Biodiesel with oils or fats (e.g. in the logistics chain).

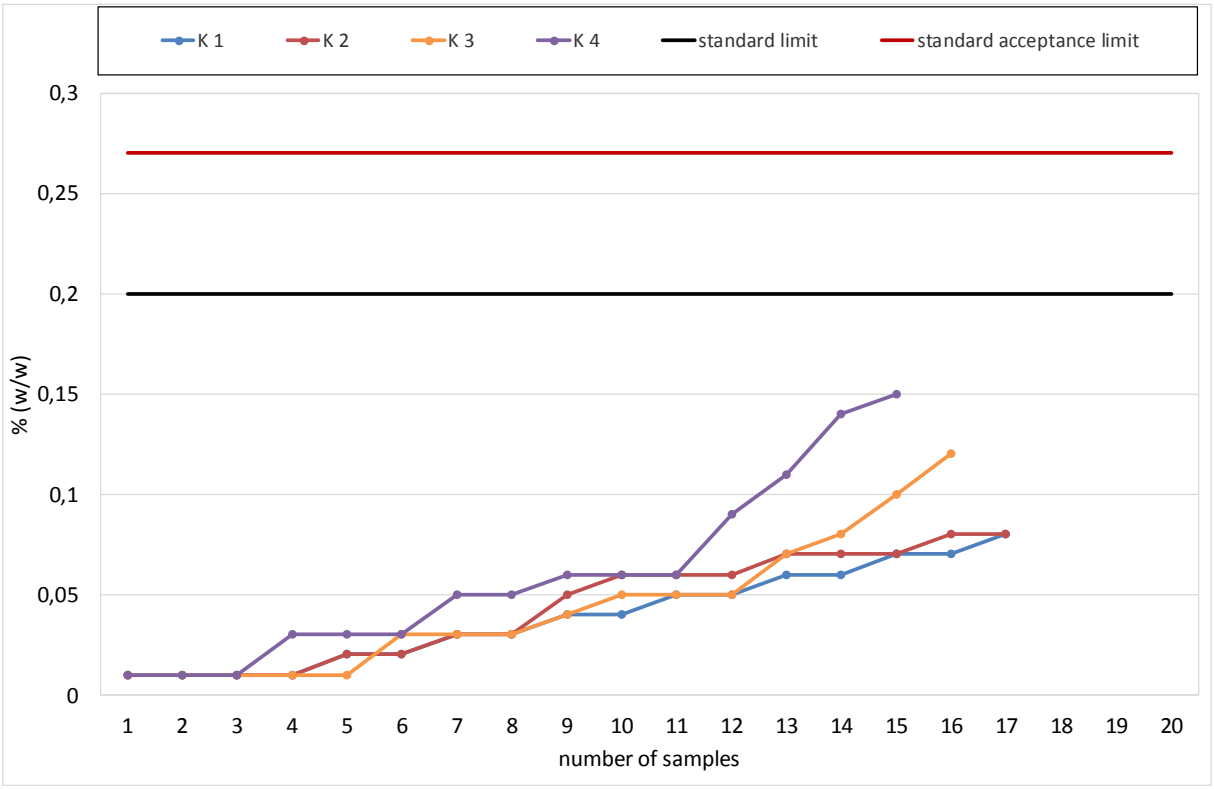

Diagram 12: Triglycerides according to DIN EN 14105

Diagram 12 shows the measuring results of the triglyceride content. All tested samples fall below the limit.

3.9.4 Free Glycerol

Limit of DIN EN 14214:2014: ≤ 0,02 % (w/w)

Maximum acceptance limit: 0,026 % (w/w)

Glycerol is generated during the transesterification of fats and oils to fatty acid methyl esters. Since glycerol is practically insoluble in Biodiesel it can be separated almost completely by decantation and subsequent water wash.

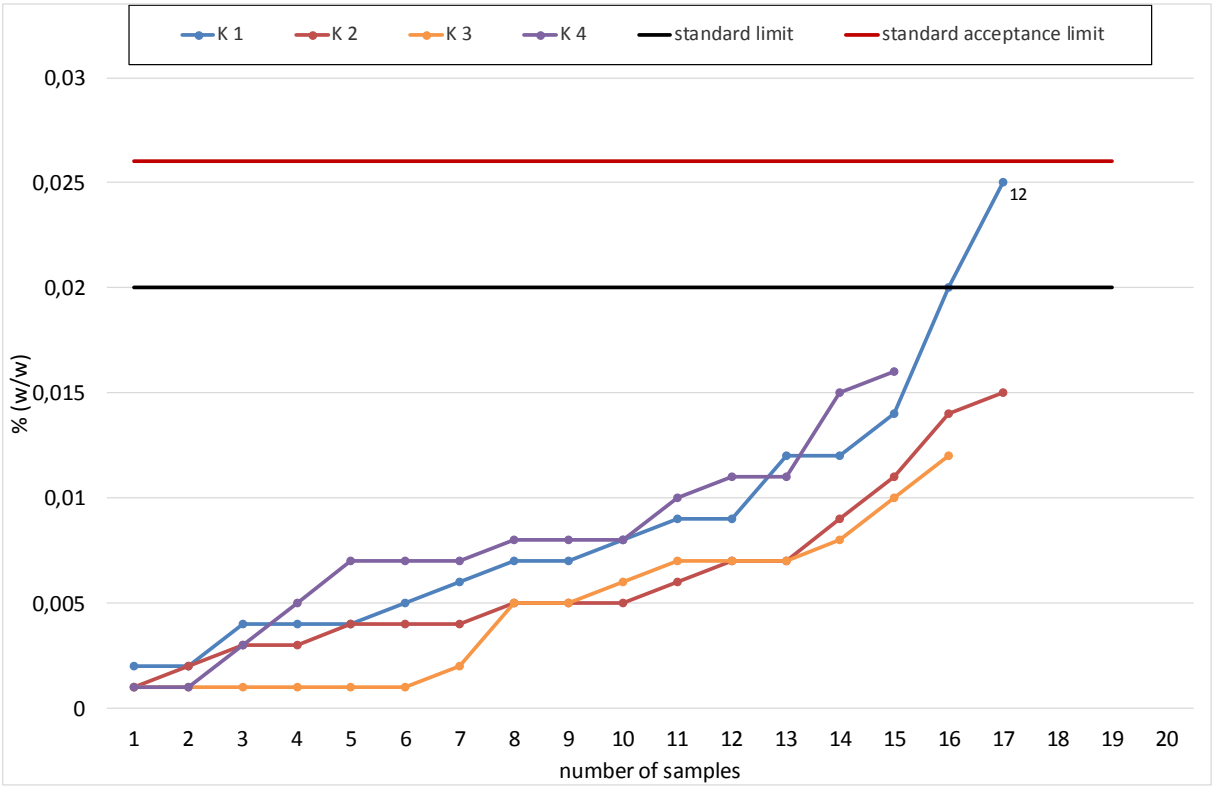

Diagram 13: Free Glycerol according to DIN EN 14105

The content of free glycerol falls below the limit for all samples but sample 12 (see diagram 13.) Member sample 12 falls just under the acceptance limit of 0,026 %. This value is the result of an arbitration sample. Originally this sample had clearly exceeded the acceptance limit with a value of 0,03 % (w/w). However, in case of doubt concerning the analysis results of the sampling campaign member companies are entitled to address AGQM to apply for arbitration proceedings. For that, the member assigns an independent laboratory accredited for Biodiesel analytics. The arbitration sample is one of the two retain samples taken during the sampling campaign. The result of the arbitrational analysis is binding for both parties. In this case the

result of the arbitration sample showed a value of 0,025 % (w/w) which is within the acceptance limit, as described above. Therefore, the member company could avert a sanction point. The member informed the AGQM office later on that a washer with a defect (which was repaired in the meantime) had been detected as cause for the bad value.

3.10 Alkali Metals: Sodium and Potassium

Test method: DIN EN 14538:2006

Limit of DIN EN 14214:2014: ≤ 5 mg/kg

Maximum acceptance limit: 6,1 mg/kg

For the production of Biodiesel sodium and potassium hydroxides or methylates are used as catalysts. Their residues in Biodiesel are often present as soaps which were not completely removed during washing.

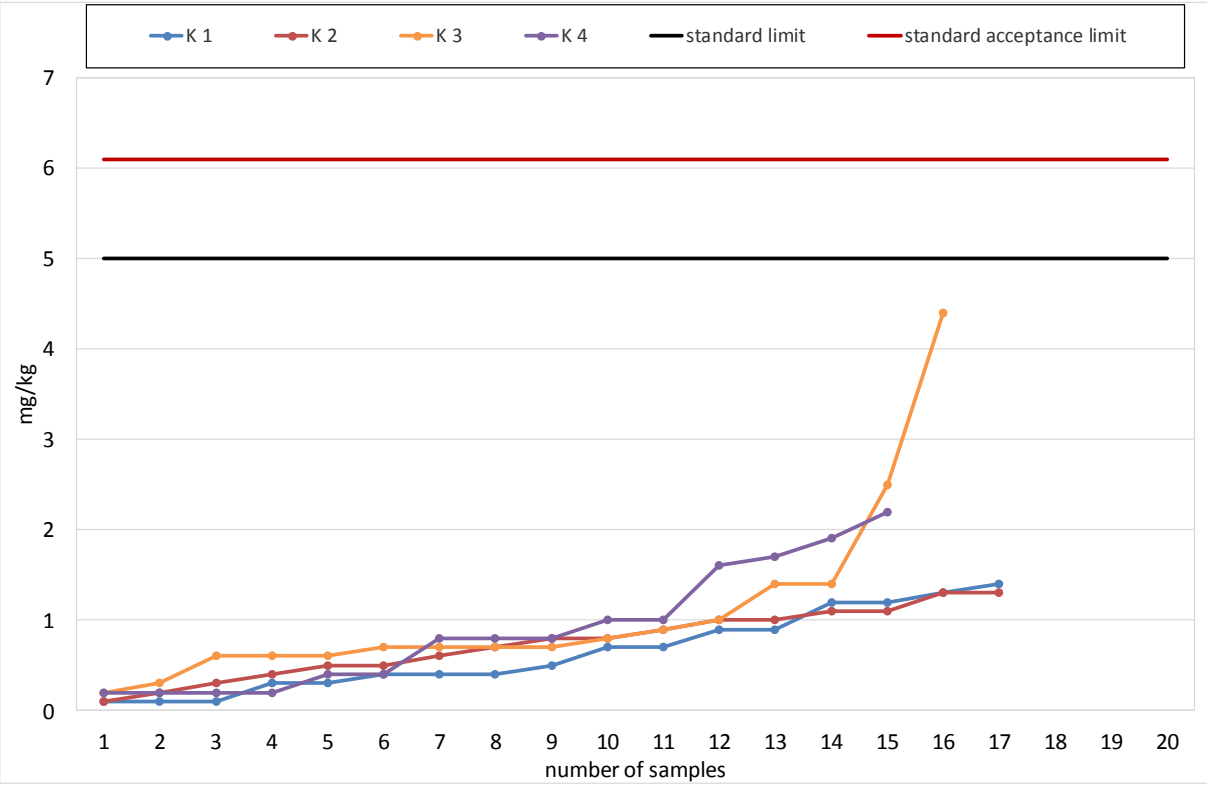

Diagram 14: Sum of Alkali Metals Sodium and Potassium according to DIN EN 14538.

Diagram 14 shows the sum of the alkali metals sodium and potassium. The majority of samples falls way below the limit. Only three measured values are above 2 mg/kg.

3.11 Earth Alkali Metals: Calcium and Magnesium

Test method: DIN EN 14538:2006
Limit of DIN EN 14214:2010/2014: ≤ 5 mg/kg
Maximum acceptance limit: 6,1 mg/kg

Earth alkali metals calcium and magnesium are either introduced into the process by the raw material or they can get into the final product during the production process when tab water is used for washing. Calcium and magnesium soaps, which are more voluminous than alkali metal soaps, form by reacting with free fatty acids. The use of softened water can reduce the ingress of earth alkali metals into Biodiesel.

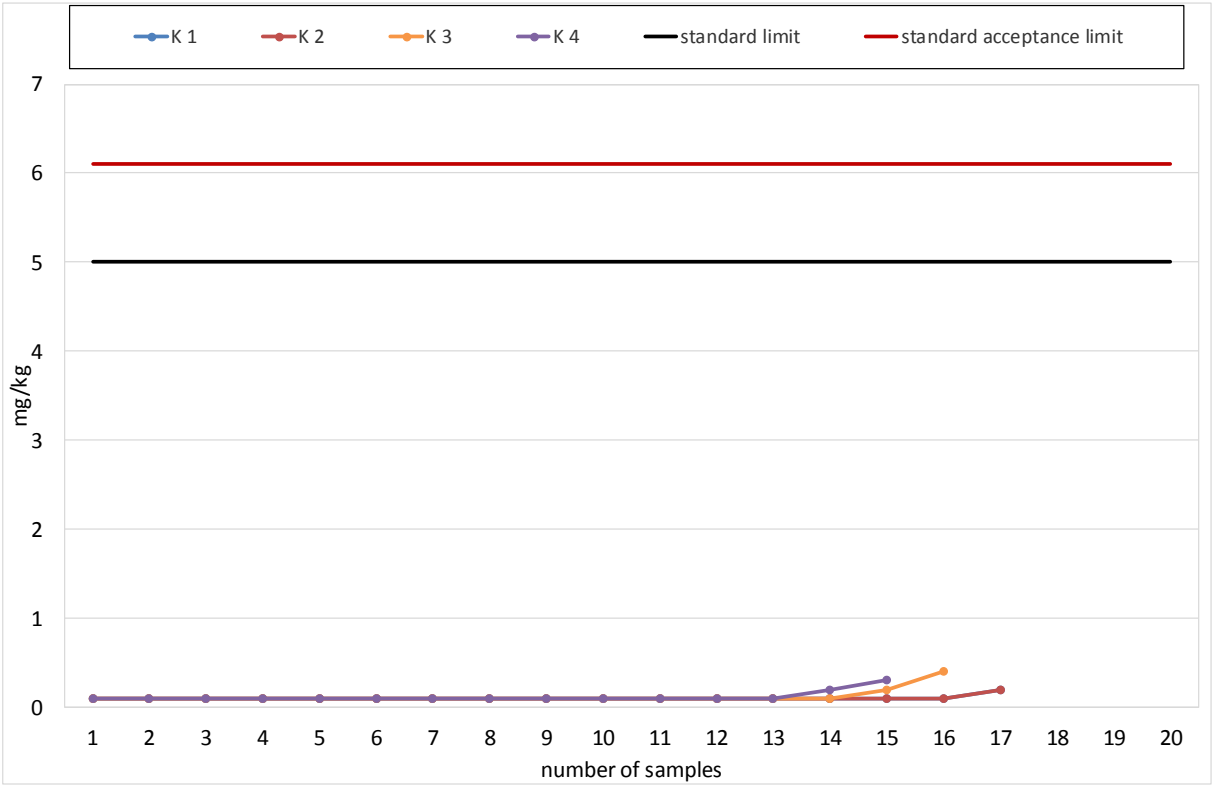

Diagram 15: Sum of Earth Alkali Metals Calcium and Magnesium according to DIN EN 14538.

The sum of the earth alkali content given in diagram 15 falls far below the limit for all samples. All samples have a content of below the determination limit of 1 mg/kg.

3.12 Phosphorus Content

Test method: DIN EN 14107:2003

Limit of DIN EN 14214:2014: ≤ 4 mg/kg

Maximum acceptance limit: 4,5 mg/kg

The phosphorus content must already be considered when the raw material is selected or it must be reduced to minimum residual contents by a refining process before the transesterification. Phosphorus is contained in vegetable oils in form of phospholipids as well as in animal fats. The transesterification process is influenced if the content of phospholipids is too high because phospholipids work as emulsifiers and thus interfere with the phase separation. Phosphorus can also be introduced into Biodiesel during the production process if phosphoric acid is used as catalyst or for the cleavage of acids. In general however, used phosphoric acid can be well removed from Biodiesel with water.

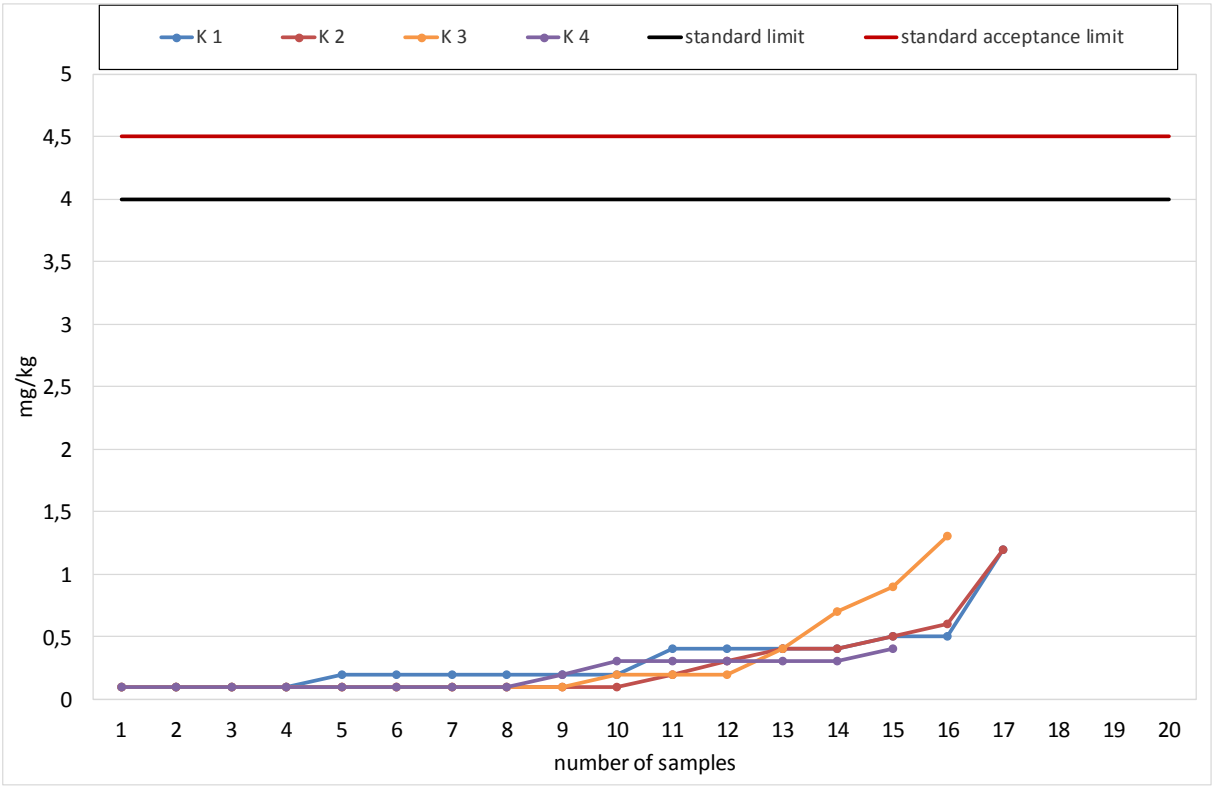

Diagram 16: Phosphorus Content according to DIN EN 14107, K2 to K4.

Diagram 16 shows the results of the phosphorus content. The values fall way below the limit; 95 % of the samples are even less than 1 mg/kg. The maximum limit is 4 mg/kg; however, at present the precision of the method does not allow for any additional tightening of the limit.

3.14 Content of Linolenic Acid Methyl Ester

Test method: DIN EN 14103:2011
Limit of DIN EN 14214:2014: ≤ 12,0 % (w/w)
Maximum acceptance limit: 14,9 % (w/w)

The content of linolenic acid is determined from the fatty acid profile by gas chromatography. 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 in Biodiesel is limited to 12 % (w/w).

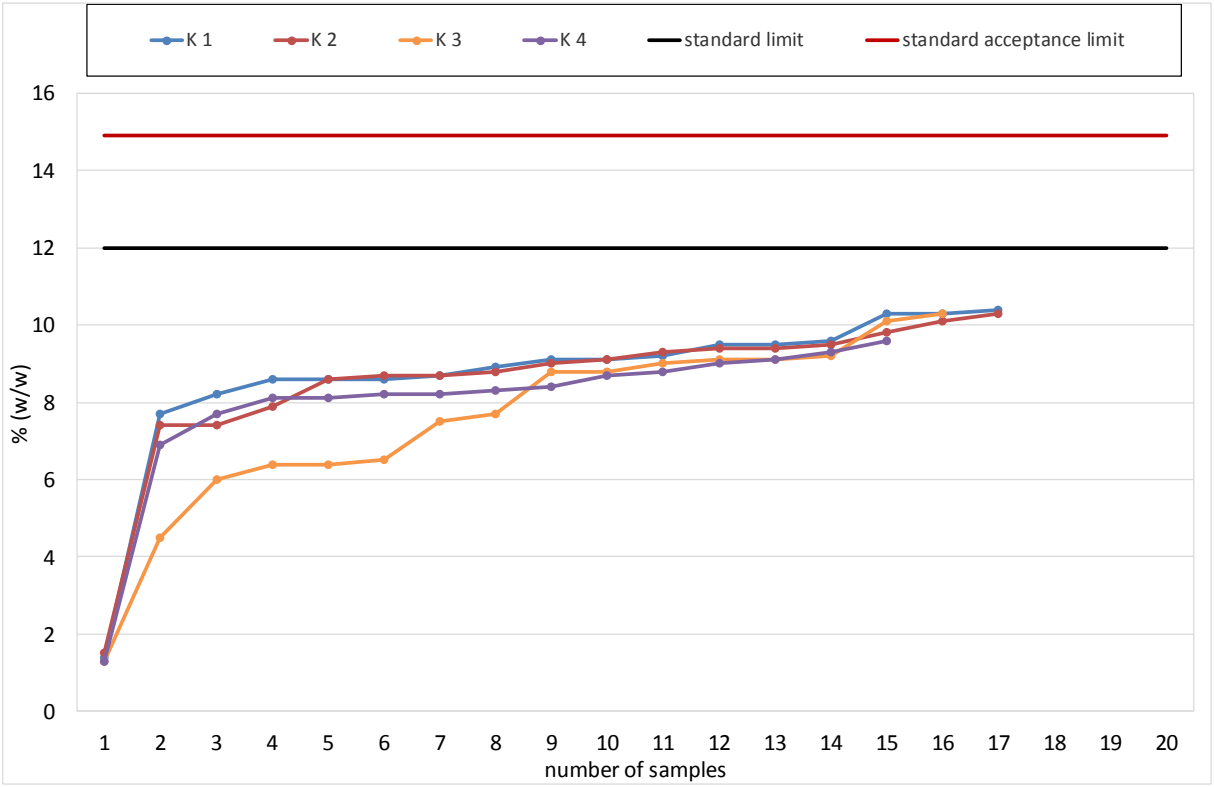

Diagram 17: Content of Linolenic Acid Methyl Ester according to DIN EN 14103.

As shown in diagram 17 all analysed samples fulfil the requirements of the standard. The reduced content of linolenic acid of a large amount of samples during the summer campaign (K3) shows that the raw material 'rapeseed oil' usually used for the production of Biodiesel was at least partly substituted by other oils. The content of linolenic acid of rapeseed oil is between 8 and 10%[2].

3.14 Cold Filter Plugging Point (CFPP)

Test method: DIN EN 116:1997

Limit according to DIN EN 14214:2014

	Limit	*Acceptance Limit*
15 April to 30 September	*0 °C*	*1,5 °C*
1 October to 15 November	*-10 °C*	*-7,9 °C*
16 November to 28/29 February	*-20 °C*	*-17,3 °C*
1 March to 14 April	*-10 °C*	*- 7,9 °C*

AGQM Limit: -20°C max. from 19 October to 28/29 February

The CFPP is the measure for Biodiesel cold properties. As already described at the end of chapter 2, the requirements for 'resistance to cold' are regulated nationally according to the prevailing climatic conditions. As applicable for Diesel fuel, there are differing requirements for summer, intermediate and winter grades.

Since nowadays Biodiesel is almost exclusively used as blend component for Diesel fuel, additivation is often dispensed with. According to the German legal regulations applicable to the cold properties, Biodiesel as blend component only needs to adhere to a CFPP value of -10 °C between 16 November and 28/29 February if the value of -20 °C as demanded by DIN EN 14214 can be achieved by additivation.

The results of the summer and winter campaigns were depicted in two separate diagrams to enable a clearer presentation of the determined data.

[2] M. Mittelbach, C. Remschmidt: Biodiesel The Comprehensive Handbook, 1. Edition, Graz 2004, ISBN 3-200-00249-2, S. 135.

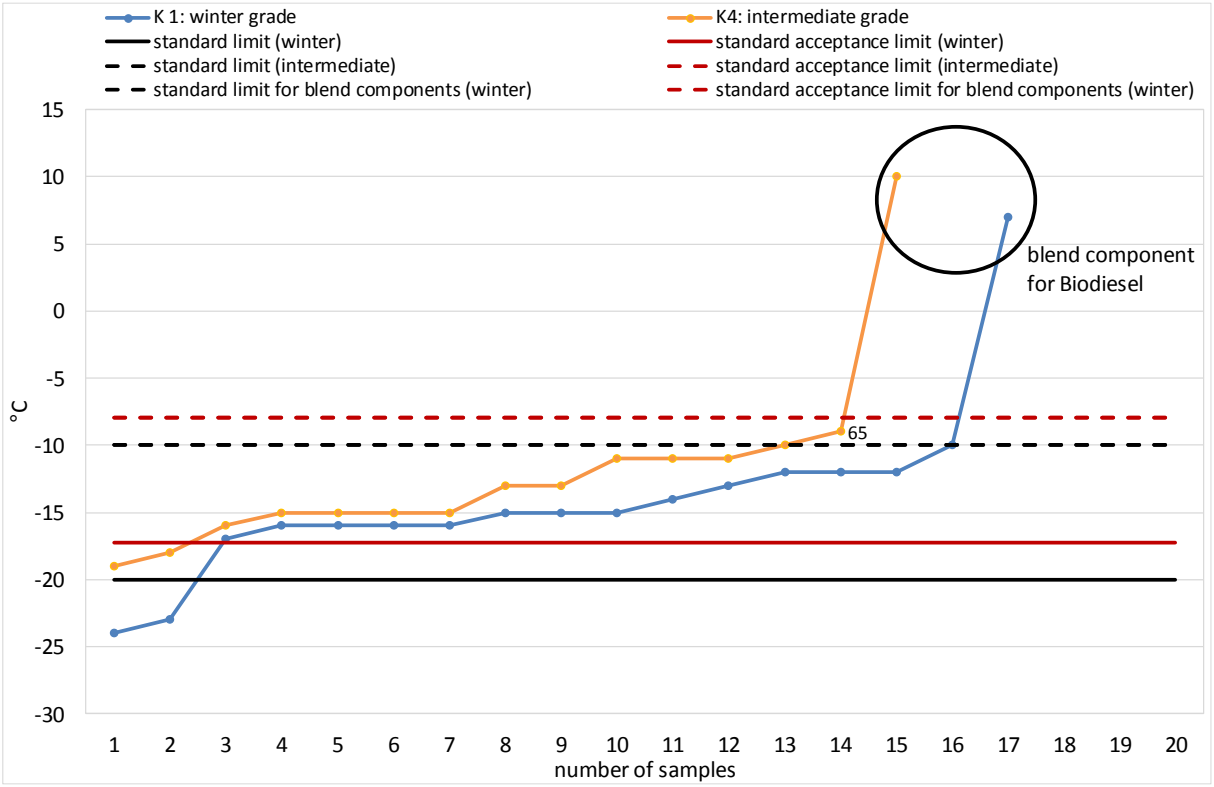

Diagram 18: CFPP (intermediate and winter grades) according to DIN EN 116

In K1 the samples were taken during the period 26 January to 6 February, so the samples are winter grade. K4 took place from 5 to 16 October which makes the samples intermediate grade. The limit for pure Biodiesel in winter is illustrated by an uninterrupted line; the limits for intermediate grade as well as for blend components in winter are given by a dotted line.

Due to the fact that all samples given in diagram 18 are blend components, all but three samples meet the relevant limit. Sample 65 exceeds the limit with -9 °C within the range of the acceptance limit. The other two samples (circled in diagram 18) are blend components for Biodiesel, as noted above in the diagram; they cannot be marketed directly but only if blended to other Biodiesel.

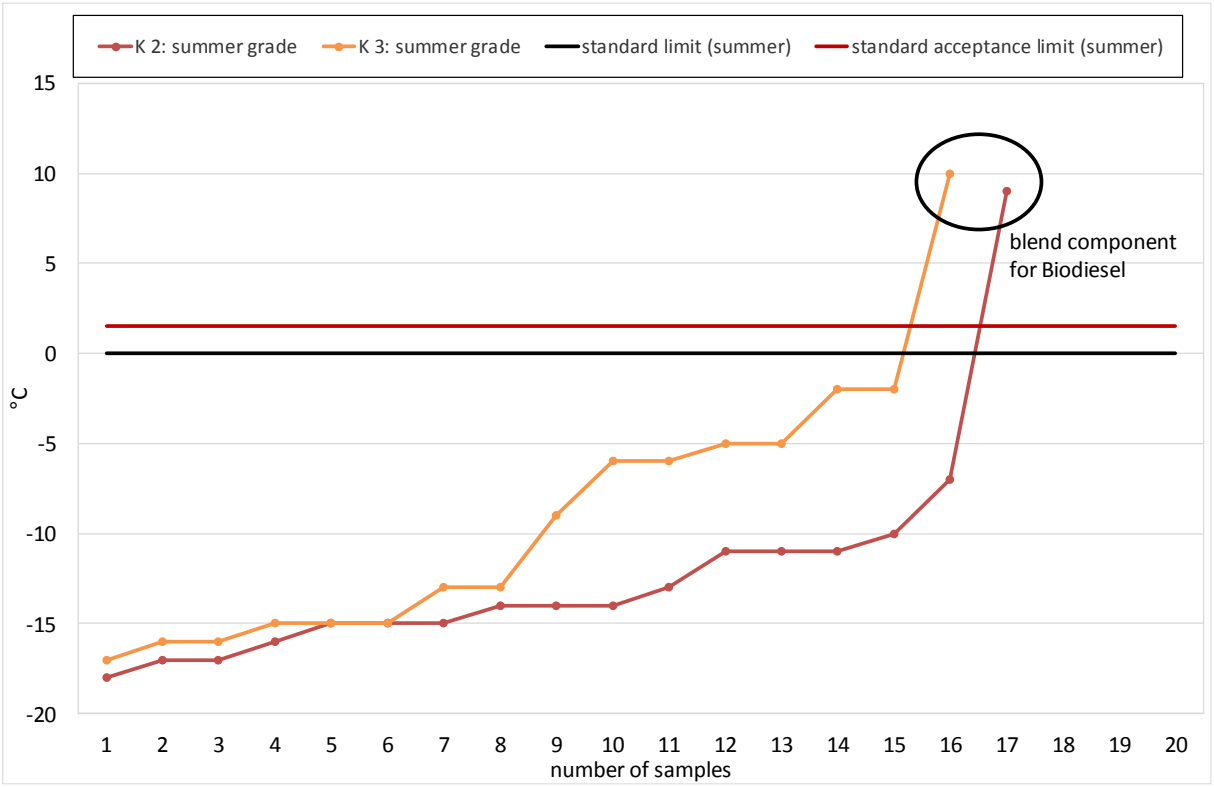

Diagram 19: CFPP (summer grade) according to DIN EN 116

In K2 the samples were taken during the period April 17 to 30; K3 took place from July 6 to 17 which means that the samples were taken in summer. As shown in diagram 19 all but two tested samples fulfil the requirements of the standard.

The two samples circled in diagram 19 are blend components for Biodiesel, as noted above in the diagram, which can only be marketed after adjustment of the quality, e.g. by mixing with other appropriate fuel.

3.16 Cloud Point (CP)

Test method: DIN EN 23015:1994

Limits according to DIN EN 14214:2012:

	Limit	*Acceptance Limit:*
15 April to 30 September	*5° C*	*7,4 °C*
1 October to 15 November	*0 ° C*	*2,4 °C*
16 November to 28/29 February	*-3° C*	*-0,6 °C*
1 March to 14 April	*0° C*	*2,4 °C*

The Cloud Point is the temperature at which temperature-induced clouding sets in when a clear liquid product is cooled down under stipulated test conditions. Upon publication of DIN EN 14214:2012 in November 2012 the Cloud Point has since been part of the requirements for Biodiesel as blend component. So far this parameter is not required for Biodiesel used as pure fuel.

As for the CFPP, the results of the summer and winter campaigns were depicted in two separate diagrams to enable a more clearly presentation.

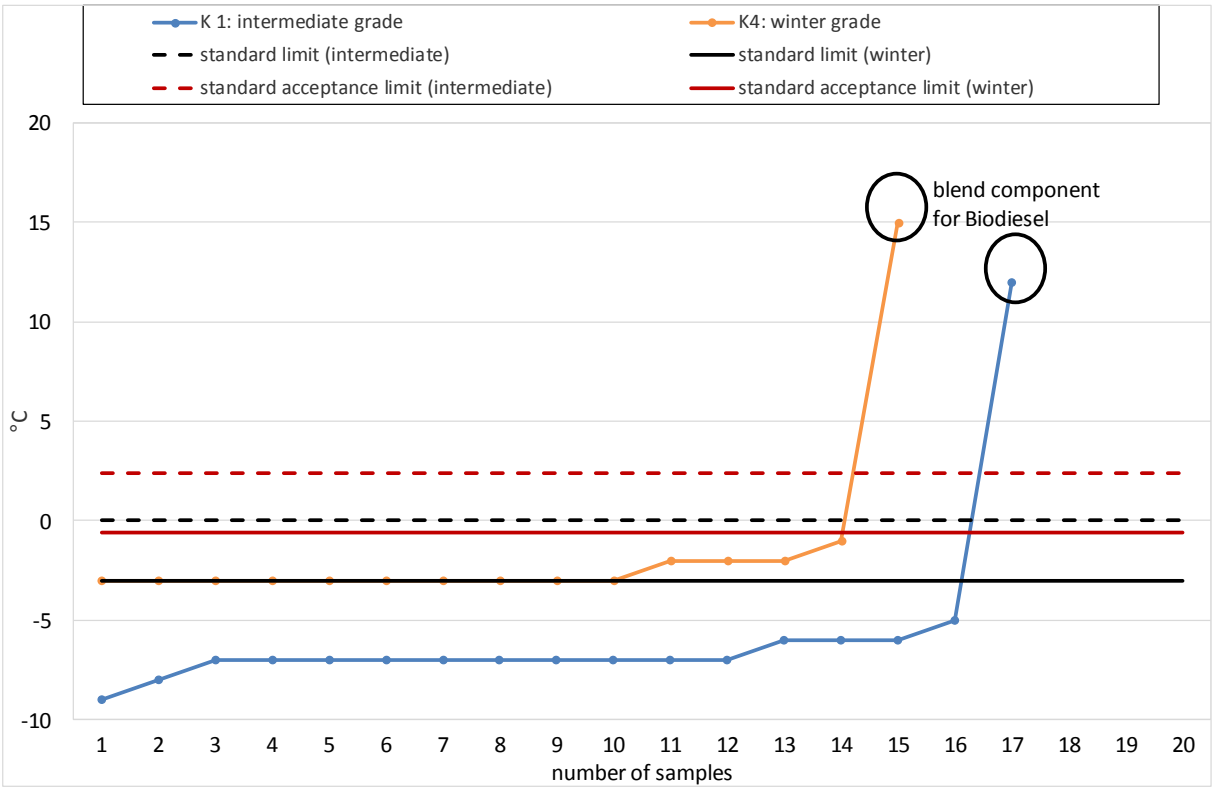

Diagram 20: Cloud Point (intermediate and winter grades) according to DIN EN 23015.

There was one limit violation in each the winter and intermediate period. As described above, these samples are blend components for Biodiesel which are only marketed after admixture to appropriate fuel.

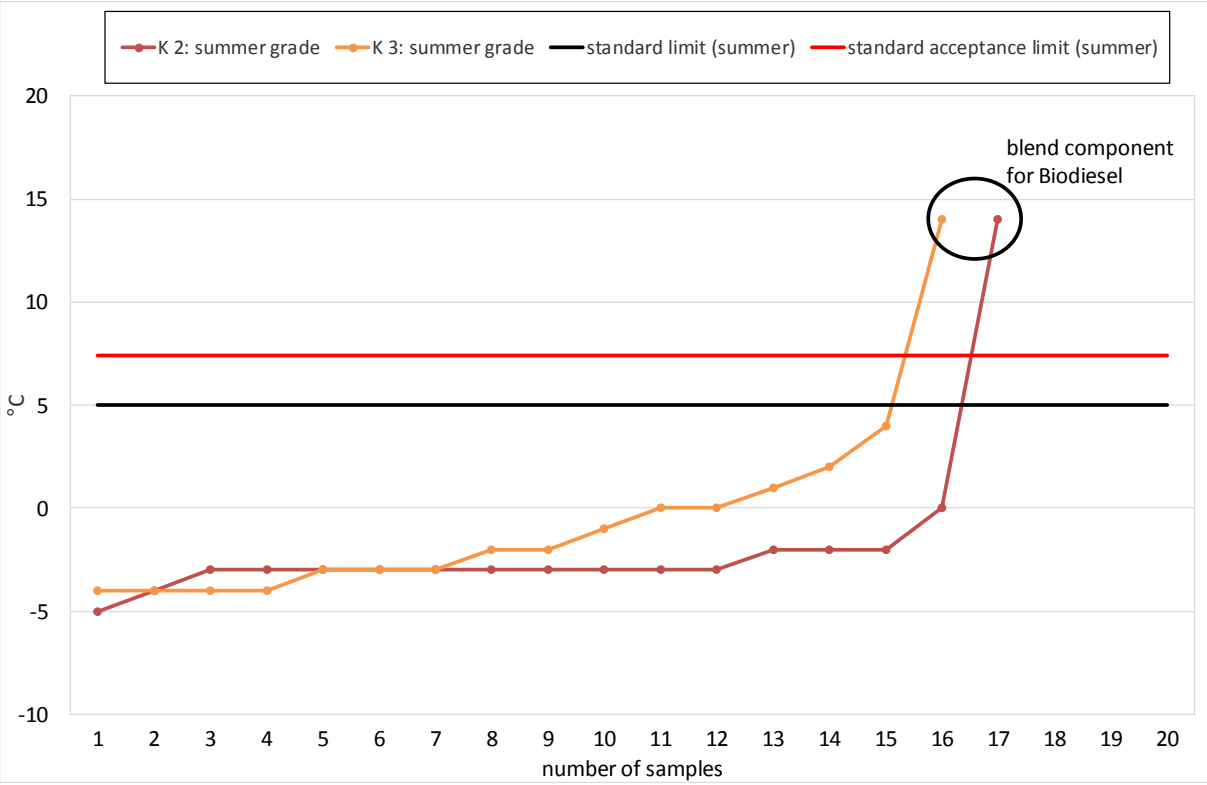

Diagramm 21: Cloud Point (summer grade) according to DEIN EN 23015.

As described for parameter CFPP, sampling of K2 and K3 was arranged in the summer time. There are also two limit violations. Again these samples are blend components for Biodiesel which will not be marketed directly.

4 Summary

Despite some changes of the legal requirements for the Biofuel quota, the result of all four sampling campaigns in 2015 is a very positive one. While in 2014 the overall result still showed five limit violations, in 2015 for the first time all tested Biodiesel samples (65 samples in total) fulfill the requirements of DIN EN 14214 considering the relevant precision of the test method.

Though five samples do not meet a limit, their values exceed or fall below the relevant acceptance limits. For parameter 'water content' two samples exceed AGQM's more stringent limit. Three samples exceed or fall below the standard limits for parameters 'content of free glycerol', 'CFPP', and 'oxidation stability'. Since all of those results are still within the acceptance limits, the products are eligible for the market and sanctions points were not assigned.

For five samples the exception for Biodiesel produced from used cooking oils and fats was applied for. As described before Biodiesel produced thereof is exempted from the determination of parameters 'sulfur content', 'CFPP', and 'Cloud Point'; and limit violations are not sanctioned for these parameters. However, it must not be marketed directly but only as blend component for Biodiesel.

The purpose of unannounced sampling – firmly anchored in AGQM's QM system – is to check whether the limits of the parameters specified in the 36th BImSchV and stipulated by 10th BImSchV are met. In addition, the member companies' self-control is thus supported.

Since 2010 AGQM has published an annual report[3] on the Biodiesel quality in Germany. With the data obtained, the AGQM member companies' positive development of optimizing production processes and quality assurance measures can be observed and clearly tracked.

In 2015 for the first time 100 % of the samples fulfilled the requirements of DIN EN 14214. Once again, this excellent result substantiates and proves the high quality level of Biodiesel produced by AGQM member companies.

[3] http://www.agqm-biodiesel.de/en/downloads/reports/

5 Annex

5.1 Limits and Test Methods

Table 1: Limits and Test Methods for the Parameters tested according to DIN EN 14214:2014

Test Parameter	Method	Year of Publication	Measuring Unit	Standard Limit		Acceptance Limits	
				min.	max.	min.	max.
Content of Fatty Acid Methyl Ester	DIN EN 14103	2011	% (m/m)	96,5	-	94,0	-
Density at 15 °C	DIN EN ISO 12185	1996	kg/m³	860	900	859,7	900,3
Sulfur 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
Content of Linolenic Acid Methyl Ester	DIN EN 14103	2011	% (m/m)	-	12,0	-	14,9
Content of free Glycerol	DIN EN 14105	2011	% (m/m)	-	0,02	-	0,026
Content of Monoglycerides		2011	% (m/m)	-	0,70	-	0,82
Content of Diglycerides		2011	% (m/m)	-	0,20	-	0,24
Content of Triglycerides		2011	% (m/m)	-	0,20	-	0,27
Overall Glyceride Content		2011	% (m/m)	-	0,25	-	0,28
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	-		-	
Content of Earth Alkali Metals (Ca + Mg)		2006	mg/kg	-	5,0	-	6,1
Calcium Content		2006	mg/kg	-	5,0	-	6,1

[4] Due to the fact that the current version of DIN EN 12662 is not suitable for the determination of parameter ‚total contamination' in FAME, DIN EN 12662:1998 applies until further notice.

Magnesium Content		2006	mg/kg	-		-	
Phosphorus Content	DIN EN 14107	2003	mg/kg	-	4,0	-	4,5
CFPP	DIN EN 116	1997	°C	15 April to 30 September 1 October to 15 November 16 November to 28/29 February 1 March to 14 April	0 -10 -20 -10	- - - -	1,5 -7,9 -17,3 -7,9
Cloudpoint	DIN EN 23015	1994	°C	15 April to 30 September 1 October to 15 November 16 November to 28/29 February 1 March to 14 April	5 0 -3 0	- - - -	7,4 2,4 -0,6 2,4

Table 1: Limits and Determination Methods for the Parameters Tested according to AGQM's QM System

Test Parameter	**Method**	**Year of Publication**	**Measuring Unit**	**Standard Limit**		**Acceptance Limits**	
				min.	**max.**	**min.**	**max.**
Water Content K.-F. (for Producers)	DIN EN ISO 12937	2000	mg/kg	-	220	-	280
Water Content K.-F. (for Warehouse Operators)	DIN EN ISO 12937	2000	mg/kg	-	300	-	370
Total Contamination	DIN EN 12662	1998[5]	mg/kg	-	20	-	20
CFPP	DIN EN 116	1997	°C	19 October to 28/29 February	-20 (applicable for use of Biodiesel as pure fuel (B100))	- - - -	-17,3

[5] Due to the fact that the current version of DIN EN 12662 is not suitable for the determination of parameter ‚total contamination' in FAME, DIN EN 12662:1998 applies until further notice.

5.2 Abbreviations

AGQM	Arbeitsgemeinschaft Qualitätsmanagement Biodiesel e.V.
B7	Short for blended fuel permissible according to DIN EN 590 with a proportion of 7 % Biodiesel
BImSchV	Bundes-Immissionsschutzverordnung (German Federal Emission Protection Directive)
ca.	Circa
CEN	Comité Européen de Normalisation (European Standardization Committee)
CFPP	Cold Filter Plugging Point
DIN	Deutsches Institut für Normung (German Institute for Standardization)
FAM	Fachausschuss für Mineralöl- und Brennstoffnormung (FAM) im DIN
FAME	Fatty Acid Methyl Ester
FQD	Fuel Quality Directive (2009/30 EG)
GHG	Green House Gas
JWG	Joint working group
K.F.	Karl Fischer
K1	Campaign 1
K2	Campaign 2
K3	Campaign 3
K4	Campaign 4
QA Committee	Committee for Quality Assurance
QM System	Quality Management System
RED	Renewable Energy Directive (2009//28/EG)
RME	Rapeseed Oil Methyl Ester
TC	Technical Committee
UCOME	used cooking oil methyl ester (fatty acid methyl ester from used cooking oils and fats)