A short study to assess the metal, phosphorus and sulfur content in biodiesel

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0. Summary and overview

More than 7900 individual data of the element contents (Na, K, Ca, Mg, P, S) of market-relevant biodiesel samples from the years 2000 to 2010 were evaluated in the present short study. The data bases of the biodiesel quality management working group (AGQM) and the Analytik-Service GmbH (ASG) were used as database.

The values of the actual dominant concentrations were approximated by three different calculations. Neither the alkali nor the alkaline earth element contents have exceeded the value of 2 mg/kg over the past 10 years. A similar result is shown for the phosphorus and sulfur content. Essentially values of below 2 mg/kg can be determined for the phosphorus content and/or below 3 mg/kg for the sulfur content.

The comparison of the mean values calculated for the alkaline earth, alkali and phosphorus content with field test data regarding the contamination of the Deutz AG tractor and/or bus engines SCR catalysts demonstrated a very good correlation. At the same time it could also be demonstrated that the elements contained in the lubricating oil exert a substantially greater influence on the contamination of a catalyst than the biodiesel proportion contained in the diesel fuel. Actual values show that the filter blockage and catalyst deactivation problem is probably not severe in the field.

Based on the data presented here, engine and exhaust after treatment systems developers will be able to make more realistic calculations concerning the dangers of catalysts contamination and/or the blockage of soot particle filters when using biodiesel according to DIN EN 14214 and/or biodiesel blends (e.g. B30). In view of the study results it appears erroneous to use the limit values provided in DIN EN 14214 as the computing basis.

Additionally, the short study emphasises the quality assurance measures implemented by the biodiesel manufacturers. In a super ordinate trend and taking into account the respective measurement conditions, the data demonstrate a trend toward lower element contents.

1. Introduction and purpose of the data analyses

The trace element content in fuels plays an important role in the release of exhaust after treatment systems. All metals which are emitted with the engine combustion can increase the exhaust gas back pressure in the after treatment system to inadmissible values due to the formation of ashes. Additionally, exhaust after treatment systems metal coatings and catalysts are contaminated and/or deactivated by phosphorus and alkali element.

Consequently, in the project promoted by the Union zur Förderung von Oel- und Proteinpflanzen e.V. (UFOP) and the Verband der Deutschen Biokraftstoffindustrie e.V. (VDB) "Determination of the emissions and the particle size distribution (fine dust) in the exhaust gas of a modern Euro-4-commercial vehicle engine with SCR emission control using biodiesel"1 (project manager Prof. Dr. Munack/Prof. Dr. Krahl, vTI Braunschweig), it was established that the ageing of the SCR catalyst, caused by the use of biodiesel with a content of 10 mg/kg phosphorus, was substantially accelerated with continuous 1000 operating hours. The content in RME corresponded to the maximum permissible limit value according to standard DIN EN 14214 (2003).

One result of this project was the lowering to the currently valid limit value of max. 4 mg/kg of phosphorus in the further standardisation project of DIN EN 14214.

Asides from the phosphorus limit value, the currently valid limit values of the alkali and alkaline earth elements are also critically assessed by the vehicle and motor industry for future releases of biodiesels as pure biodiesel (B100) as well as for higher bio-fuel blend ratios of biodiesel in diesel fuel.

Alkali metals (Na + K)max. 5 mg/kgAlkaline earth metal (Ca + Mg)max. 5 mg/kgCurrently a limit value of 10 mg/kg is regarded as sufficient for sulfur.

Over the past ten years the AGQM has collected approximately 8000 analytical data of phosphorus, sulfur and metal contents in biodiesels by the unannounced sampling of biodiesel producers. AGQM therefore has the world's largest interrelated data base in which biodiesel analysis data are systematically gathered on the basis of the national and European specification standard. There are, therefore, data which on the one hand reflect the development of quality in the production of biodiesel and, on the other, the advancement of the analysis methods.

¹ Munack A, Ruschel Y, Schwarz S, Bünger J, Krahl J (2006) Bestimmung der Emissionen und der Partikelgrößenverteilung (Feinstaub) im Abgas eines modernen Euro-4-Nutzfahrzeugmotors mit SCR-Abgasreinigung im Betrieb mit Biodiesel, Abschlussbericht zum Forschungsvorhaben, Bundesforschungsanstalt für Landwirtschaft, Institut für Technologie und Biosystemtechnik, Braunschweig, http://www.ufop.de/downloads/Abschlussbericht_SCR-final.pdf

The goal of the project is the evaluation of the element concentrations of sodium, potassium, calcium, magnesium, phosphorus and sulfur in biodiesel for the period between 2000 and 2010. The short study is supplemented in chapter 5 by the extrapolation results of the Deutz company for the catalyst contamination by the elements discussed herein.

2. Introduction in the ICP OES measurement

The biodiesel standard DIN EN 14214:2003 provides different measuring techniques for the detection of element contents in sodium, potassium, calcium, magnesium, phosphorus and sulfur. The alkali contents (Na, K) are to be determined by means of atomic absorption spectrometry (AAS) in accordance with DIN EN 14108 and/or DIN EN 14109. Two standards exist for the sulfur (S) content. On the one hand DIN EN ISO 20846, in which the sulfur content is measured by burning the sample in a high temperature combustion tube and detection of UV fluorescence. On the other hand DIN EN ISO 20884, which is based on the measurement of x-ray fluorescence.

In the case of the alkaline earth elements (Ca, Mg) and phosphorus (P), an inductively coupled plasma and subsequent optical emissions spectroscopy (ICP OES) is used. The specific standards for this are DIN EN 14538 together with DIN EN 14107. With the revision of DIN EN 14214 in April 2010, the measurement of sodium and potassium contents by means of DIN EN 14538 was introduced. Since then the official possibility of measuring all of the elements discussed here exists (up to sulfur) using ICP OES. The ICP OES measuring technique will be demonstrated in more detail in the phosphorus content example. A detailed explanation of the measuring technique is, however, not provided. The aim is instead to focus on the analytic possibilities.

DIN EN 14107 describes the execution of a phosphorus measurement for biodiesel and provides an application range of 4 to 20 mg/kg. This means that the limit of determination is actually 4 mg/kg for analytic laboratories which work to this standard. However the measuring technique allows to measure precisely below 4 mg/kg. Figure 1 visually clarifies this statement with a calibration curve which begins at 4.0 mg/kg and ends at 0.5 mg/kg.

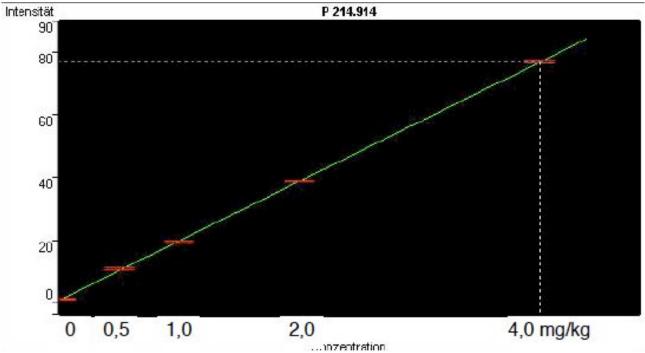


Figure1: Phosphorus calibration curve between 0.5 mg/kg and 4.0 mg/kg

The following illustrations are for further clarification. In Figure 2 the OES signal for a phosphorus concentration of 0.5 mg/kg is shown. This clearly stands out from the background lines (dashed) and can be evaluated without any great difficulty.

Figure 2: OES signal for a phosphorus content of 0.5 mg/kg

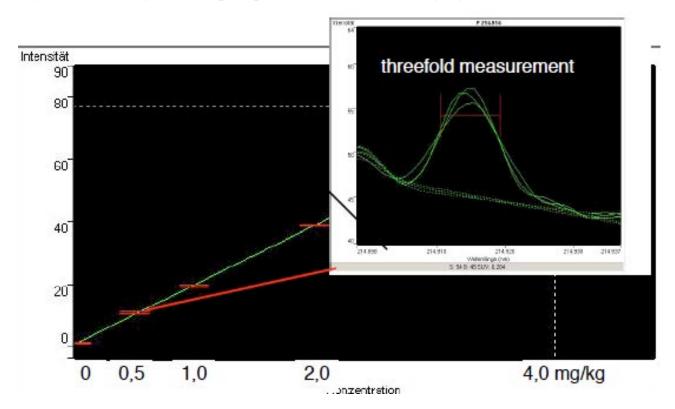


Figure 3 shows the OES signals for more phosphorus contents between 0.5mg/kg and 4.0 mg/kg. The dashed background lines are shown in the figure.

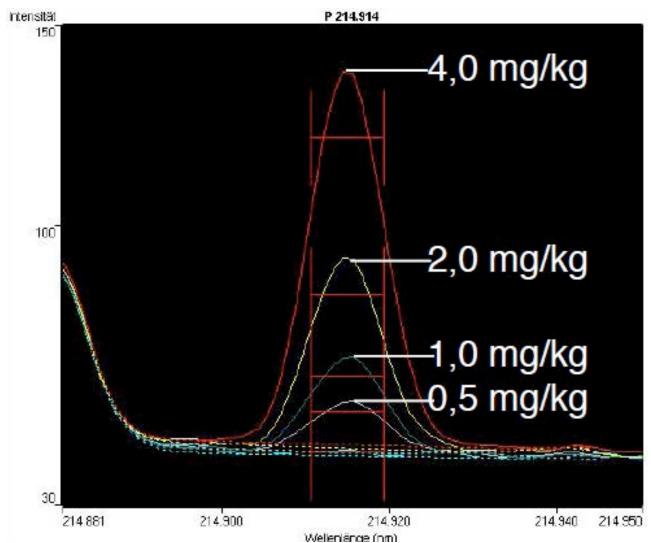


Figure 3: OES signal for a phosphorus content between 0.5 mg/kg and 4.0 mg/kg

From a purely measurement technique viewpoint, the evaluation of phosphorus contents below 4.0 mg/kg can be considered as unproblematic. Such low contents are also easily manageable in the working day practise. Figure 4 shows the representation of the results of a quality assurance sample for the period of 18 days from November 2008. Asides from phosphorus, the measurement results for the sodium, potassium, calcium and magnesium elements are also specified. Phosphorus exhibits the largest fluctuation margin. In accordance with DIN EN 14107 the repeatability (in the range of application) lies at \pm 0.6 mg/kg. All repetition measurements shown lie within this value. Despite the large - at least purely optical - fluctuation margin, the margin between the highest and lowest measurement result amounts to only 0.14 mg/kg.



Figure 4: Result representation QA-sample November 2008

With regard to the following data evaluations, the above remarks are only partially supported. The AGQM data base includes measurement results of different laboratories, so that it was not always possible to evaluate values below 4.0 mg/kg for phosphorus and/or 1.0 mg/kg for the alkali and alkaline earth elements. If measurement results beneath these limits of determination were available, they were consistently processed as the previous trials clearly attest to the reliability of such data. The type of the data evaluation is more clearly described in chapter 3 followed by the results presented in chapter 4.

3. Data evaluation principles

The study took a total of 7900 measurement results into account. The data of the Biodiesel quality management working group (AGQM) and the Analytik-Service GmbH (ASG) served as database. Whilst the AGQM data were to be regarded throughout as market relevant since they stemmed from routine, unannounced manufacturer samples, the measurement results of the ASG had to be filtered beforehand in order to avoid, for example, that experimental data from transesterification attempts etc. led to misinterpretations.

The values deposited in the AGQM database came from different laboratories which submitted the measurement results with different limits of determination (LoD: limit of Determination) so the basic conditions of the evaluation change from year to year and from element to element. These circumstances are referred to in both of the following explanation text, as well as in the result representations.

The characteristics for the different elements are treated separately and chronologically in the following.

Sodium:

In the year 2000 the sodium element was determined in accordance with DIN 51 797-3. For this purpose the biodiesel sample had to be ashed in a first step and subsequently solubilised. The limit of determination of the laboratory was not reached at any time, whereby the lowest documented value was analysed with 0.05 mg/kg. From 2001 to 2003 sodium was directly measured by ICP and a limit of determination of < 1 mg/kg was estimated. Likewise, between 2004 and 2007 direct measurement took place by means of ICP with, however, a lowered LoD of < 0.5 mg/kg. In 2007 the data record for sodium was reduced to 27 as the sodium contents in the AGQM database were not determined by ICP but rather by atomic absorption spectroscopy (AAS) and could, therefore, not be consulted for the evaluation.

For the year 2008 the limit of determination was indicated as < 1 mg/kg, in the year 2009 as < 0.5 mg/kg and for 2010 as < 1.0 mg/kg by the assigned laboratory. In all three years the direct measurement by means of ICP as measuring procedure was used.

Potassium:

The information and documented limits of determination expressed for sodium are similarly valid for potassium.

Calcium:

Like the Sodium and Potassium elements, Calcium was measured according to DIN 51 797-3. The limit of determination was not fallen short of and the lowest documented value was about 0.05 mg/kg. Starting from the year 2001 the measurement of the calcium content came directly from the untreated biodiesel sample. The limits of determination indicated by the laboratories were thze following: 2002 up to 2006 as well as 2009 < 0.5 mg/kg, 2007, 2008 and 2010 < 1.0 mg/kg.

<u>Magnesium:</u>

Magnesium was not included in the examinations in the year 2000. Starting from the year 2001 the assertions made for calcium also apply to magnesium.

Phosphorus:

Phosphorus was also not included in the biodiesel analysis in 2000. In the years 2001 and 2002 its measurement took place from the untreated biodiesel sample with a limit of determination of < 1.0 mg/kg. Between 2003 and 2006 and/or for the year 2009 the limit of determination lay at < 0.5 mg/kg. For the years 2007 and 2008 the limit was at < 4.0 mg/kg and for the year 2010 at < 2.0 mg/kg.

Sulfur:

In many regards the sulfur content represents an anomaly in this investigation. On the one hand the underlying testing method is not an ICP measuring procedure, and on the other hand only data from the years 2007, 2008 and 2009 exist which could be consulted for the evaluation. Contrary to all other elements, no specific evaluation was made for sulfur as the testing methods were inadequate. For the completeness of this report, the sulfur content has more or less been integrated particularly since this parameter – in so far as a maximum value of 10 mg/kg is not exceeded – is regarded as less critical by the vehicle and catalyst manufacturers

For 2007 the limits of determination lay at <3.0 mg/kg, for 2008 at <5.0 mg/kg und for 2009 at <1.0 mg/kg.

The preceding listing of the limits of determination has significant meaning for the results reported in the study. For example, for the year 2010 only values below the 1.0 mg/kg limit of determination were determined for the alkali elements. A mathematical evaluation of these data is not possible from a statistics point of view. Only discrete values can be consulted for this. One of the goals of the study should, however, also be to consider the worst case so that a risk assessment for the amount of ashforming elements can be provided. Consequently, data which laid below the respective limit of determination have also been included in the evaluation. The values which have been applied for the calculation are shown in table 1.

Limit of determina-	"worst case" content	"worst case" con-	"worst case" phos-
tion		tent of Alkali or	phorus and/or –
		alkaline earth ele-	sulfur content
		ments	
<0.5 mg/kg	0.44 mg/kg	0.88 mg/kg	0.44 mg/kg
<1.0 mg/kg	0.94 mg/kg	1.88 mg/kg	0.94 mg/kg
<2.0 mg/kg	1.94 mg/kg	-	1.94 mg/kg
<3.0 mg/kg	2.94 mg/kg	-	2.94 mg/kg
<4.0 mg/kg	3.94 mg/kg	-	3.94 mg/kg
<5.0 mg/kg	4.94 mg/kg	-	4.94 mg/kg

Table 1: Listing of the limits of determination and the value used for the "worst case"

The non-relevance of the values to the calculation is shown in table 1 by a line. The "Sum content of alkali or alkaline earth element" and "phosphorus and/or sulfur content" columns make it clear which values have been incorporated into the calculations for "worst case" views. With the sum parameters it concerns double the "worst case" content.

As given in chapter 2, ICP measurement procedures provide the possibility of evaluating measurement results below the respective limit of determination. This is also valid if the limits lie at 0.5 mg/kg. In order to further specify the "worst case", 24 randomly selected samples with a value below 0.5 mg/kg were consulted for each element. The 24 samples are from the years 2007 to 2009. The "specific" determination results in a mean value for biodiesel contaminated with Na, K, Ca, Mg and P that is closer to the real values.

Figure 5 demonstrates the evaluation for sodium as an example.

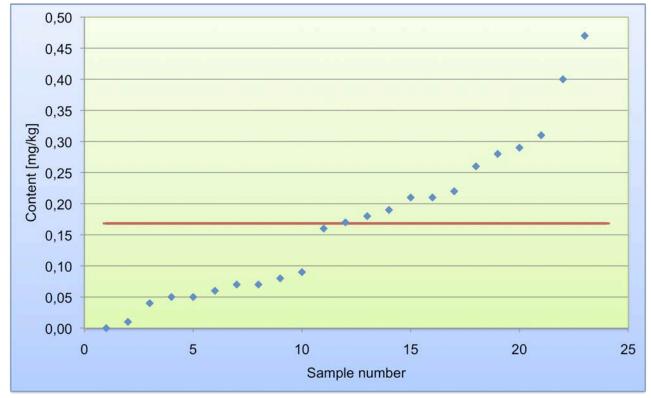


Figure 5: "Specific" evaluation of sodium content beneath the 0.5 mg/kg limit of determination

The red line in figure 5 marks the new mean value for the sodium element at 0.17 mg/kg. The data for potassium, calcium, magnesium and phosphorus were evaluated in the same way. Table 2 summarises the "specific" mean values, on the basis of which a more precise evaluation of all data found below the limit of determination took place.

Element and/or element group	"specific" mean value (mg/kg)
Sodium	0.17
Potassium	0.15
Alkali metals (Na + K)	0.32
Calcium	0.04
Magnesium	0.01
Alkaline earth metal (Ca + Mg)	0.05
Phosphorus	0.11

Finally a "specifically weighted" calculation was implemented for the year 2009 which considered the respective production capacities of the biodiesel manufacturers. The purpose of this calculation was to allow the market share of the data to be incorporated into the evaluation. The maximum production capacity of the respective biodiesel manufacturer for the year was converted into a priority factor in relation to the overall capacity of all tested producers. "Specifically weighted" mean values could be determined therewith. Table 3 shows the results.

Table 3: "Specifically weighted" mean values for the element group and/or the phosphorus individual element

Element and/or element group	"Specifically weighted" mean value		
	(mg/kg)		
Alkali metals (Na + K)	0.64		
Alkaline earth metal (Ca + Mg)	0.14		
Phosphorus	0.30		

4. Results

The results for elements and/or element groups are provided below.

4.1. Results for alkali metal content (Na + K)

Figure 6 shows the percentage on all measurement results for the alkali metal contents which were above the limit of determination (LoD) used in each case.

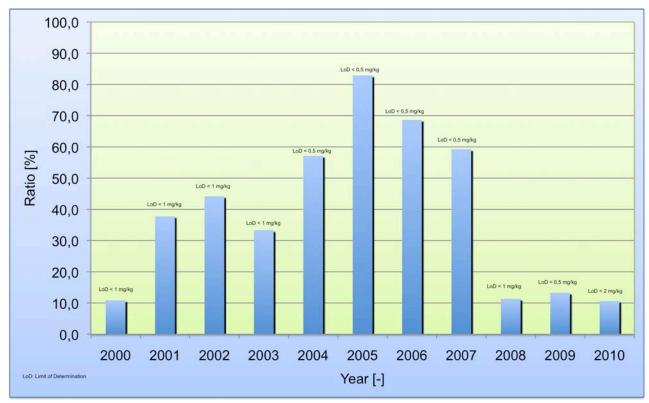
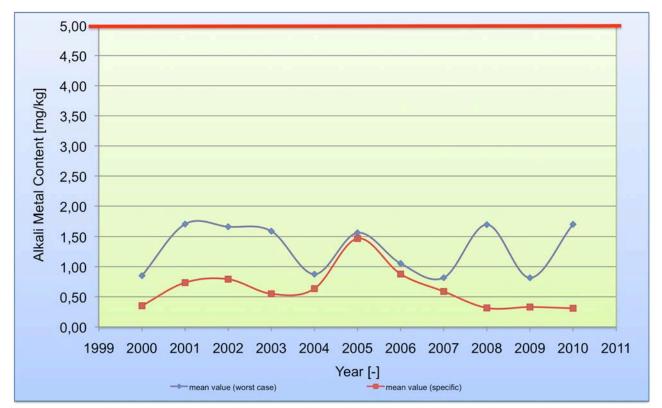


Figure 6: Percentage of the alkali content above the relative limits of determination

Figure 6 shows an increase of the percentage of values above the limits of determination for the years 2000 to 2005 for all variations which are due to the different limits of determination. This trend was only reversed and moved in the opposite direction in 2006. Essentially there are two reasons for this development. The standardised measuring procedure DIN EN 14538 has only existed in draft form since 2003 and as an adopted standard since 2006. The standardisation of the results has meant that measurements by different laboratories could be compared to one another. This contributed to a learning process within the biodiesel industry to implement the appropriate process optimisations regarding the alkali element contents.

On the other hand, between the years 2000 and 2005 a kind of "biodiesel boom" took place which led to a massive extension in the production capacity. The increase in

production capacity during this period was achieved at the expense of the percentage of alkaline contaminants above the limits of determination.



In figure 7 the progress of the annual mean value is reported under "worst case" and "specific" requirements.

Figure 7: Annual mean value of alkali metal content for two different calculation scenarios

The values for the alkali metal content under "worst case" requirements fluctuated over the years between 1 mg/kg and 2 mg/kg. The results for the "specific" evaluation continue to lie under these values and fluctuate between 0.5 mg/kg and 1.5 mg/kg. The additional red line at 5 mg/kg marks the limits of EN 14214 (2003 and/or 2010 version) for alkali element content. It is clear that the mean value - regardless of the type of calculation – was not close to the limits at any time.

4.2. Results for alkaline earth metal content (Ca + Mg)

Comparable to figure 6, figure 8 also shows the percentage of all measurement results for the earth alkaline contents which lay above the relative LoD (Limit of Determination).

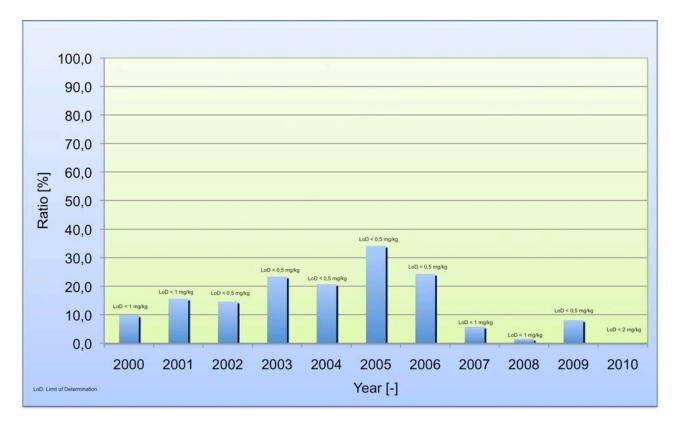


Figure 8: Percentage of the alkali content above the relative limits of determination

Contrary to the alkali elements discussed under 4.1., the increase in the proportion of the measurement results above the limits of determination is considerably weaker. Nevertheless, the two trends can also be recognised here and can be traced back to the causes discussed above. In connection to this it should, however, be noted that the contamination of the biodiesel with alkaline earth metals, contrary to the alkali metals (process catalysts), does not occur through this process but rather through the raw material used for the transesterification and/or also by the residual contents of the process water necessary for the biodiesel washing steps.

Figure 9 shows the calculated mean values for both the "worst case" and "specific" scenarios.

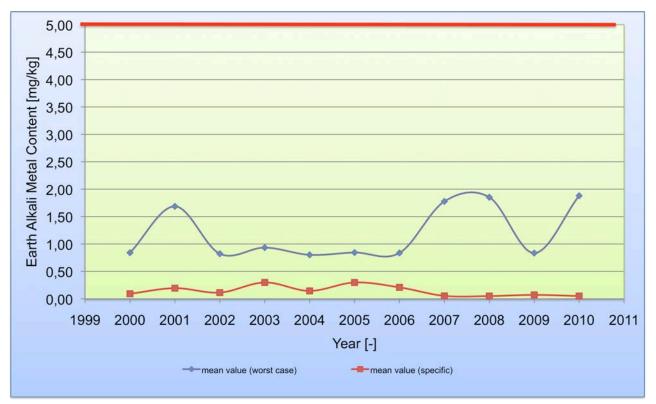


Figure 9: Annual mean value of the alkaline earth metals for two different calculation scenarios

Similar to the alkaline metal content, the mean value for the "worst case" calculations of the alkaline earth element content fluctuated between 1 mg/kg and 2 mg/kg. However, lower mean values resulted for the "specific" analysis lying between 0 mg/kg and 1 mg/kg.

The additional red line at 5 mg/kg again marks the limit value provided in EN 14214 (2003 and 2010 version). The alkaline earth content mean values also lie far beneath this.

4.3. Results for phosphorus content (P)

Due to the fact of insufficient data for phosphorus content between 2000 and 2002, these were only analysed as of 2003. Figure 10 provides the percentage of measurement results above the relative limits of determination.

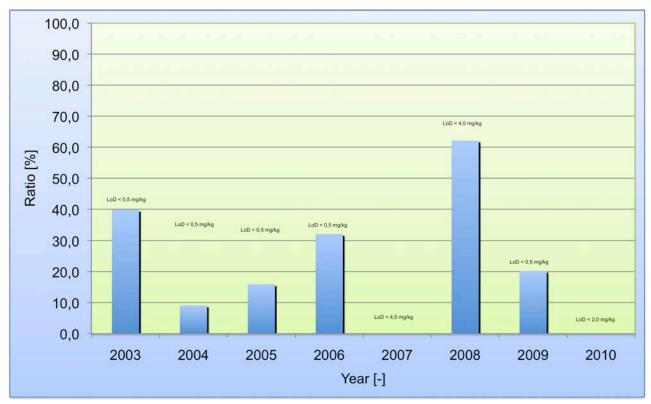


Figure 10: Percentage of phosphorus above the relative limit of determination

Over the years the portion of phosphorus content above the relative limit of determination has slightly decreased and lies at 40 % (or below), clearly beneath the trends encountered for the alkali and alkaline earth elements. Nevertheless, 2008 is significant. The sudden rise is not easily explained. Possibly only a particularly phosphorus rich raw material for biodiesel production was available to the producers during this year so that, despite the high limit of determination of 4.0 mg/kg, round about 65% of the measurement results lay above the limit of determination.

Figure 11 shows the annual mean values of both calculation variants for phosphorus content.

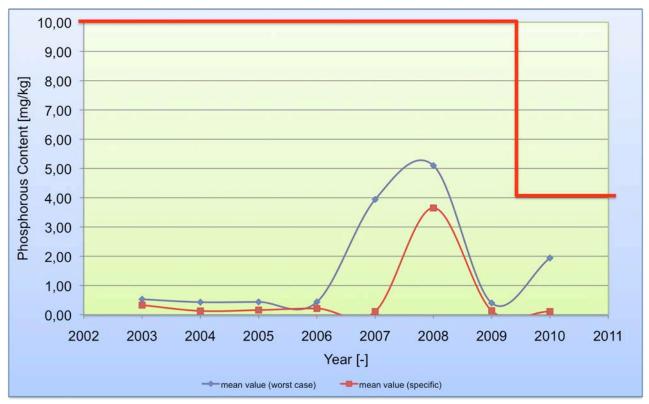


Figure 11: Annual mean values of phosphorus content for two different calculation scenarios

As expected, the year 2008 also stands out in the above representation. Furthermore, at this stage it is important to note that due to the lowering of the limit value for the phosphorus content in the 2010 version of DIN EN 14214, the red line has experienced a kink. In each case, therefore, the calculated annual mean values clearly lie below the limits for all the years considered.

In figures 7 and 9 the DIN EN 14214 version is not significant since the limit values for the alkali and alkaline earth metal contents were not changed.

The values for the "worst case"- calculations have a tendency to lie between 1 mg/kg and 4 mg/kg. With one "specific" evaluation the mean values sink to beneath 1 mg/kg.

4.4. Results from the "specifically weighted" evaluations

In table 3 the results for the "specifically weighted" mean values of the element groups and/or for phosphorus are presented. In table 4 the remaining calculated mean values are also shown. Due to the particularly time-intensive and complex "specifically weighted" evaluations, this was implemented for the year 2009 as an example. All remaining values documented in table 4 are likewise related to this year.

Element group and/or element	"worst case" mean value ² [mg/kg]	"specific" mean value ³ [mg/kg]	"specifically weighted" mean value ⁴ [mg/kg]
Alkali elements (Na + K)	0.82	0.33	0.64
Earth alkali element (Ca + Mg)	0.83	0.07	0.14
Phosphorus (P)	0.40	0.14	0.30

Table 4: Mean values for the groups of elements and/or the individual component phosphorus of 2009

The "specifically weighted" mean values are always higher than the "specific" mean values. With respect to the results that are presented in chapter 5. it seems that the "specifically weighted" mean values are even closer to the "reality" than the "specific" mean values. Overall it has to be pointed out that all mean values – even the "worst case" mean values - of all groups of elements and/or of phosphorus are clearly under the limit values provided in DIN EN 14214.

The footnotes 2 to 4 explain the way how the different mean values are calculated for a better understanding.

Table 5 in the annex additionally provides a summary overview of each calculated mean value.

² <u>"worst case" mean value</u>: Measurements result beneath the limit of determination were excluded from the calculation. <u>Example</u>: Limit of determination of 1.0 mg/kg, from which a value of 0.94 mg/kg resulted which could be consulted for the calculation – see Chapter 3, Table 1

³ <u>"specific" mean value:</u> Measurement results below the limits of determination were not generally considered, but on the basis of a specially calculated mean value which was determined by targeted later analysis by 24 randomly selected measurement results from the years 2007 to 2009. – see Chapter 3, Table 2

⁴ <u>"specifically weighted" mean value:</u> Introduction of a priority factor for each biodiesel manufacturer, that considers the quantity brought in comparison to the entire tested production capacity. Example: the overall capacity amounted to 3.928.000 tons per year. A biodiesel factory with a yearly capacity of 100.000 t thus had a priority factor of 0.025 – see Chapter 3, Table 3

4.5. Results for sulfur content (S)

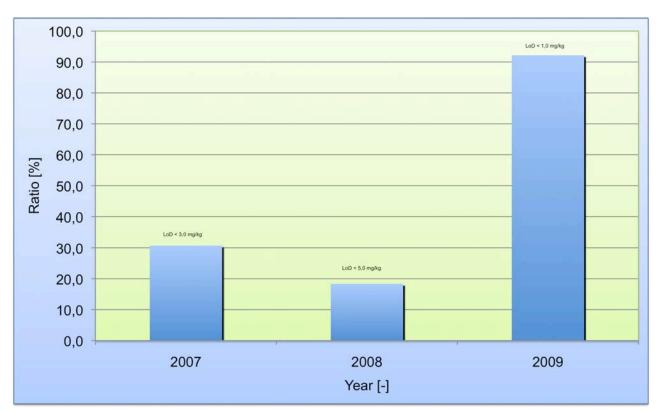


Figure 12 shows the percentage of the measurement results above the limit determination for the years 2007 to 2009

Figure 12: Percentage of sulfur content above the limits of protection

Depending on the respective limit of determination, many different sulfur contents were found to be above it. Figure 13 helps to better interpret the bar chart shown above.

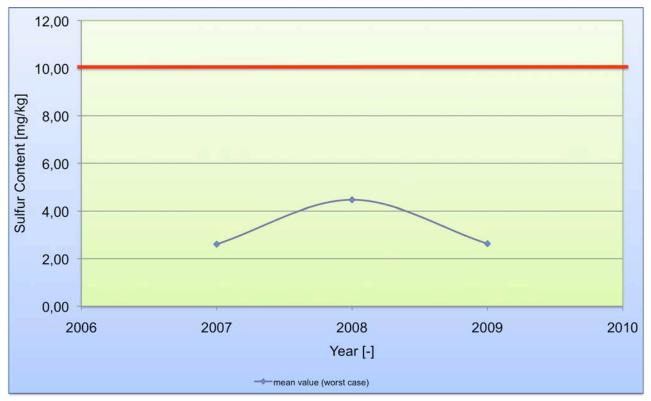


Figure 13: Annual mean value of the sulfur content for the "worst case"-calculation scenario

Even the inclusion of the measurement value which lies below the current limit of determination as "worst case" value - clearly demonstrates that the limit value of maximum 10 mg/kg provided in the DIN EN 14214 standard, which is also valid for the current diesel fuel standard DIN EN 590, is never exceeded.

The representation of the sulfur contents rounds up the overall view of the element content examinations and confirms that, based on the limit values provided in the standard, "worst case" calculations are far removed from the actual situation in the biodiesel market.

5. SCR contamination model of Deutz AG

In the years 2008/2009 and 2010/2011 the Deutz AG, in the context of the UFOP, promoted research projects 540/0805 and 540/1036, carried out field tests with tractor and city bus engines and modelled the contamination of the "Washcoat" of SCR-catalysts (selective catalytic reduction for nitrogen oxide) with Ca, Mg, Na, K and P. The influence of lubricants and fuel based elements was differentiated between. The following figures 14 to 17 demonstrate the results of these field tests. The results obtained here are also incorporated in the representation. The "worst case" mean values and the specific mean values (see table 5 in annex) calculated for 2010 were integrated in the underlying calculation model together with the limit values provided in the DIN EN 14214 standard. The four representations show especially good correlation between the field test results and the specific mean values on "worst case" or limit values assumptions lie high above the current catalyst contamination found. The portion of lubricant in the contamination of the "washcoat" is considerably greater than the one of the biodiesel.

⁵ Knuth H-W, Winkler M (2009) implementation of a test stand-endurance test over 500 hrs as well as field tests for the release of DEUTZ-Common-Rail-Motors in u EURO IV in Biodiesel. Final report of UFOP-Projekt 540/080, DEUTZ AG, Cologne, http://www.ufop.de/downcontamination s/Abschlussbericht_deutsch_Sep09.pdf

⁶ Knuth H-W, Winkler M (2010) Durability test on DEUTZ Agripower-Motors for the emission level IIIB with SCR-Systems for the release of Biodiesel. Final report to UFOP-Project 540/103, DEUTZ AG, Cologne,

Calculated element accumulation in the SCR catalyst of a tractor engine -TCD 6.1 L6 stage IIIB after 3000 h

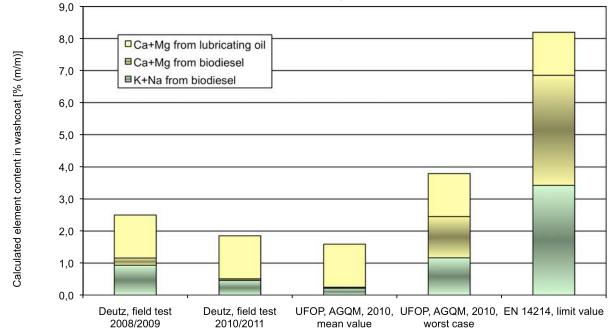
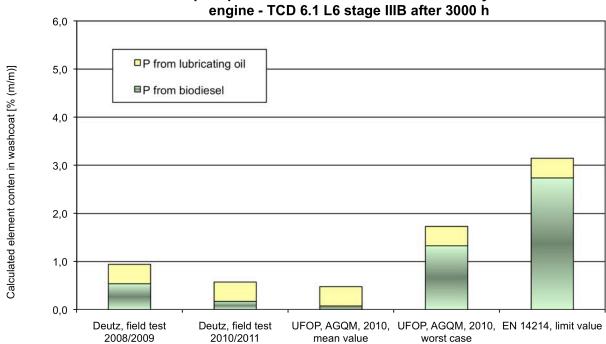
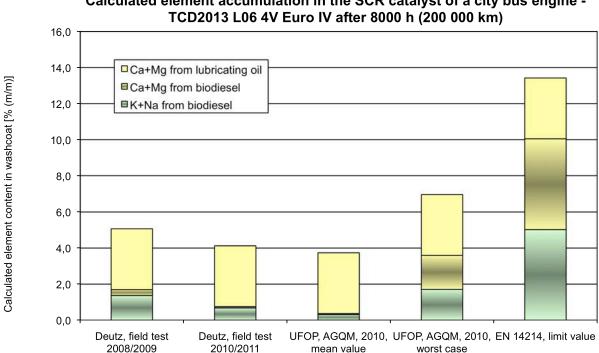


Figure 14: Metal element content in SCR-catalyst of a tractor motor



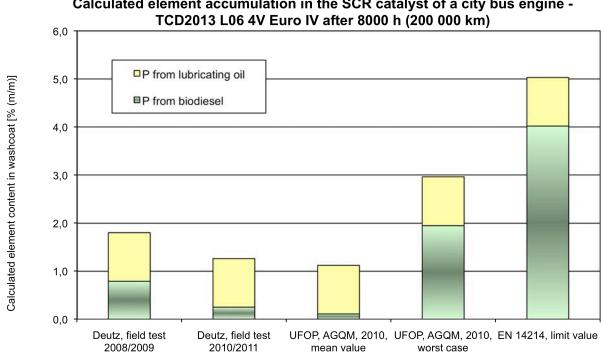
Calculated phosphorus accumulation in the SCR catalyst of a tractor

Figure 15: Phosphorus content in SCR-catalyst of a tractor motor



Calculated element accumulation in the SCR catalyst of a city bus engine -

Figure 16: Metal element content in SCR-catalyst of a city bus motor



Calculated element accumulation in the SCR catalyst of a city bus engine -

Figure 17: Phosphorus content in SCR-catalyst of a city bus motor

6. List of abbreviations

AAS	Atomic absorption spectroscopy
Ca	Calcium
ICP OES	Inductively coupled plasma with optical emission spectroscopy
Κ	Potassium
LoD	limit of determination
Na	Sodium
Mg	Magnesium
Р	Phosphorus
S	Sulfur
UFOP	Union zur Förderung von Oel- und Proteinpflanzen e.V.
VDB	Verband der Deutschen Biokraftstoffindustrie e.V.

7. Annex

	Alkali content	t mean value	Alkaline earth content mean value		Phosphorus content mean value		Sulfur content mean value	
Year	worst case	specific	worst case	specific	worst case	specific	worst case	specific
2000	0,85	0,35	0,84	0,09	-	-	-	-
2001	1,71	0,74	1,69	0,19	-	-	-	-
2002	1,66	0,79	0,82	0,11	-	-	-	-
2003	1,59	0,55	0,93	0,30	0,53	0,33	-	-
2004	0,88	0,64	0,80	0,14	0,43	0,13	-	-
2005	1,56	1,47	0,84	0,30	0,44	0,16	-	-
2006	1,05	0,88	0,84	0,21	0,44	0,21	-	-
2007	0,82	0,59	1,78	0,05	3,94	0,11	2,61	-
2008	1,70	0,32	1,85	0,05	5,10	3,65	4,46	-
2009	0,82	0,33	0,83	0,07	0,40	0,14	2,63	-
2010	1,70	0,31	1,88	0,05	1,94	0,11	-	-

Table 5: Overview table of the calculated averages