Oil Condition Monitoring (OCM) with FTIR spectroscopy – Comparison, challenges and solutions

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Summary

FTIR spectroscopy is offered as a powerful tool to get insights into the oil condition of a lubricant and is therefore essential in predictive and proactive maintenance strategies. The applicability and challenges of the method are investigated on various oil samples in fresh and artificially aged states as well as in-service oils from the field.
Overall, these results underline the robustness and applicability of the applied methods and provide valuable insights into the characterization and assessment of aging processes. This study provides an important contribution to the use of easily accessible analytical techniques in this field and shows practical solutions for monitoring and maintaining oil and, subsequently, machine condition.

1. Introduction

Oil condition monitoring is essential in predictive and proactive maintenance strategies, enabling valuable insights into both equipment health and lubricant performance. By utilizing oil condition data, maintenance schedules can be fine-tuned to optimize equipment uptime, prevent unplanned downtime, and extend the life of machinery as well as lubricants in use [1], [2].

Numerous studies have been carried out on the effects of oil degradation on physical and chemical properties or tribological performance. In most cases, oil samples are artificially aged under laboratory conditions using various methods. It has been found that degradation is responsible for higher acidity, higher oxidation and viscosity, lower additive activity, and the formation of by-products [3], [4], [5], [6]. Contaminations with water or metallic particles are also known to have harmful effects and increase oil degradation [3]. During combustion, fuel is oxidized, forming water and carbon dioxide at elevated temperatures. Incomplete combustion, especially during the warm-up of the engine, however, can lead to a series of by-products. Partial oxidation of fuel may lead to esters, ketones, carboxylic acids and other substances dissolving in the lube oil. In particular, carboxylic acids increase the acidification of the oil and are typically neutralized by the base reserve of the oil. Reactions with nitrogen stemming from the combustion air can lead to nitration forming mainly nitrogen oxides. Sulfur containing heavy fuels may lead to the formation of SO_2 and SO_3 resulting in sulfation of the lubricating oil [1], [7].

Mid-infrared spectroscopy or Fourier transform infrared (FTIR) spectroscopy is becoming an important analytical tool in the field of condition monitoring of lubricants, offering the ability to monitor a wide range of functional groups associated with chemical changes in lubricants. With a single measurement and within seconds multiple degradation processes like oxidation or additive depletion can be determined simultaneously. This allows the overall quality of lubricants to be assessed and informed decisions made as to whether a lubricant needs to be replaced or not. Among others, FTIR tools provide trending information on soot, moisture, glycol, oxidative status, anti-wear additives, and nitration, amongst other measures [8]. A very useful guideline is given according to ASTM E2412 [9].

Additionally, properties like Total Acid Number (TAN) or Total Base Number (TBN) can be predicted from the recorded infrared spectrum [8], [10]. TAN measures the acidity level of oil resulting from the oxidation process. As oil ages and undergoes thermal

stress, its molecular structure begins to break down, leading to the formation of acidic byproducts. These acids can accelerate corrosion, degrade lubricant performance, and compromise equipment integrity. Consequently, monitoring TAN levels provides valuable insights into the extent of oil degradation, enabling proactive measures to mitigate potential damage and prevent costly equipment failures. In contrast, Total Base Number (TBN) measures the alkalinity and reserve alkalinity of a lubricant to neutralize acids and maintain chemical stability. As oil ages and reacts with contaminants and combustion byproducts, its alkalinity gradually diminishes, reducing its ability to counteract acidic compounds. Monitoring TBN levels allows maintenance professionals to assess the remaining alkaline reserve of the oil and predict its remaining useful life. Declining TBN values signal the depletion of additives and the onset of lubricant degradation, highlighting the need for proactive maintenance interventions, such as replenishing additives or replacing the oil, to prevent accelerated wear, corrosion, and equipment damage.
TAN and TBN are both measured classically by titration. However, Fourier Transform Infrared Spectroscopy (FTIR) – a powerful analytical technique used to assess the chemical composition and condition of lubricating oils – offers a very interesting alternative. Chemometric analysis with FTIR involves statistical methods for analyzing spectra and predicting parameters such as the concentration or composition of substances. By correlating spectral patterns with known data, chemometric models can accurately predict various properties. This technique can be easily and quickly applied to estimate TAN and TBN and can be determined simultaneously with the additives and the chemical condition. [2]
In modern laboratories FTIR spectrometers are often in use because they bring clear advantages due to high signal-to-noise ratios. There are several brands and types of so-called "laboratory spectrometers" available. These analyzers are typically a general approach and not specified for any application. The operator often needs to calibrate and define required parameters. The era**spec oil** device, on the other hand, is a specifically defined portable FTIR spectrometer for the purpose of measuring fresh and in-service lubricants with all relevant standards pre-programmed.
In this study, a conventional laboratory FTIR (Fourier-transform infrared) spectrometer and the portable "Oil Condition Monitoring-FTIR" era**spec oil** are compared based on selected industrial oils in fresh, used and aged conditions. A critical view of the available standards and their applicability to the lubricants are discussed as well as the determination of TAN and TBN using chemometric analysis with FTIR involving statistical methods.

2. Approach and methods

2.1. Oil samples

For these investigations, nine industrial lubricants were selected which were extended by three to four additional samples for each lubricant which were artificially aged using corresponding methods for the type of lubricant (see 2.2). Finally, samples of in-service lubricants of the same type were added to the sample matrix resulting in a total of 67 samples. Table 1 provides an overview of the number of samples in each category and oil type.
Table 2 lists the investigated used or in-service oils including the category, base oil type as well as appearance, color, and odor. The engine oils are partly characterized by strong soot contamination. Some gear oils showed sedimentation of contaminations which were due to particle contamination from wear.

Table 1: Overview of samples

Oil Type	Engine Oil	Gear Oil	Hydraulic oil
Fresh oil	2	3	4
In service oils	6	12	8
Artificially aged oils	8	12	12
	Total: 67 samples		

2.2. Artificial oil alteration

All considered fresh oils were artificially aged to exploit the lifetime span of the lubricant. The applied degradation was focused on thermal-oxidative ageing. Several preliminary tests were carried out to determine the correct duration and parameters. A modified method according to CEC-L-48-A-00 Method B [11] was used for the gear oils and engine oils. For the hydraulic oils, a test setup according to ASTM D2272 [12], the RPVOT test, was used. In order to simulate the actual conditions in real operation, a certain amount of water was added depending on the sample and the temperature was adjusted accordingly.

Table 2: List of in-service oils including appearance, color and odor

Oil type	Sample code	Viscosity Class	Base oil	Appearance	Color	Odor
Engine oils	EO-A-U1	SAE 10W40	mineral	strong soot	black	typical
	EO-A-U2	SAE 10W40	mineral	strong soot	black	typical
	EO-A-U3	SAE 10W40	mineral	strong soot	black	typical
	EO-B-U1	SAE 10W40	mineral	strong sediment	brown	typical
	EO-B-U2	SAE 10W40	mineral	light sediment	brown	typical
	EO-B-U3	SAE 10W40	mineral	light sediment	brown	typical
Gear oils	GO-A-U1	ISO VG 150	mineral	light sediment	brown	typical
	GO-A-U2	ISO VG 150	mineral	light sediment	red-brown	typical
	GO-A-U3	ISO VG 150	mineral	strong sediment	dark brown	typical
	GO-B-U1	ISO VG 220	mineral	sediment	brown	typical
	GO-B-U2	ISO VG 220	mineral	sediment	red-brown	typical
	GO-B-U3	ISO VG 220	mineral	strong sediment	red-brown	typical
	GO-B-U4	ISO VG 220	mineral	clear	dark brown	typical
	GO-C-U1	ISO VG 460	mineral	clear	red-brown	typical
	GO-C-U2	ISO VG 460	mineral	strong sediment	dark brown	typical
	GO-C-U3	ISO VG 460	mineral	strong sediment	dark brown	typical
	GO-C-U4	ISO VG 460	mineral	strong sediment	dark brown	typical
	GO-C-U5	ISO VG 460	mineral	strong sediment	dark brown	typical
Hydraulic oils	HO-A-U1	ISO VG 46	mineral	clear	brown	typical
	HO-A-U2	ISO VG 46	mineral	clear	light brown	typical
	HO-A-U3	ISO VG 46	mineral	clear	light brown	typical
	HO-A-U4	ISO VG 46	mineral	clear	light brown	typical
	HO-B-U1	ISO VG 46	mineral	clear	green	typical
	HO-B-U2	ISO VG 46	mineral	clear	green	typical
	HO-C-U1	ISO VG 46	ester	clear	yellow	typical
	HO-D-U1	ISO VG 46	ester	clear	yellow	typical

Table 3 summarizes the condition of the artificial alteration applied for engine oils and gear oils. Engine oils were degraded at a temperature of 160 °C for a duration up to 16 days (384 h) and gear oils were aged at 121 °C for up to 20 days (480 h).

Table 3 Parameters for "CEC" for gear oils and engine oils:

Parameter	Engine oil	Gear oil
Oil quantity	100 mL	
Air flow rate	10 L/h	
Test temperature	160°C	121°C
Duration	4, 8, 12, 16 days	7, 13, 16, 20 days

Table 4 summarizes the conditions of the applied artificial alteration for the hydraulic oils. The experiments were performed at a temperature of 95 °C in a pressure vessel with additional amounts of added water depending on the type of base oil.

Table 4: Parameters for "RPVOT" for hydraulic oils:

Parameter	Mineral oil	Ester
Oil quantity	100 g + water	
Added water	0.10%	0.50%
Initial pressure	6.2 bar	
Test temperature	95°C	
Duration	4, 8 days	

2.3. FTIR spectrometer and oil analysis

All obtained oil samples were analyzed by FTIR spectroscopy using a conventional laboratory FTIR Brucker Tensor 27 (Bruker, Ettlingen, Germany) and the portable "Oil Condition Monitoring-FTIR" era**spec oil** (eralytics, Vienna, Austria). The comparison was done by several standards and guidelines like ASTM E2412 [9] or DIN 51453 [13] for oxidation, nitration or sulfation.

In addition, Total Acid Number (TAN) and Total Base Number (TBN) according to ASTM D664 [14] and ASTM D2896 [15], respectively, were analyzed for the chemometric verification of the determination of these by FTIR.

3. Results

3.1. Results of artificial alteration

In the following the results of the artificial alteration are discussed based on the FTIR spectra of the lubricant samples.

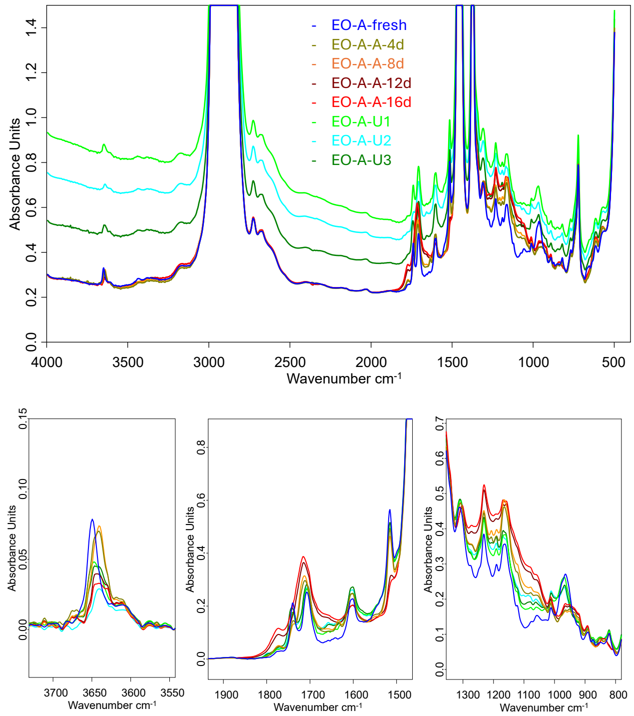

Figure 1: FTIR spectra of engine oil EO-A in fresh, used and aged condition; TOP: complete spectra; BOTTOM: details of spectra after baseline correction

Figure 1 shows the FTIR spectra of the lubricant samples from engine oil EO-A. The shift of the spectra of the used oils can be clearly seen. This is due to contamination with soot. The details are focused on specific areas of the spectrum to highlight the changes in the oil condition which can be evaluated by specific methods. In the range of 3650 cm^{-1} the phenolic antioxidant can be evaluated, which is present in this lubricant. The lowering at this band can be due to the consumption of the antioxidant until the end of the oil lifetime. A second type of antioxidant, the aminic antioxidant, can be identified in the range of 1515 cm^{-1}. Also, with this type of antioxidant a significant lowering of the band due to the aging can be observed. In the area from 1800 cm^{-1} to 1600 cm^{-1} oxidation can be determined. An increase in this range can be due to the formation of e.g. carboxylic acid due to reactions with oxygen. Using specific methods, the oxidation of the oil can be determined. However, as can be seen in the spectra, also the fresh oil has already specific bands in this range which are due to additives of specific compounds in the lubricant. The band at around 1700 cm^{-1} identifies dispersant additives whereas at 1740 cm^{-1} ester compounds are absorbing. For the evaluation of the oxidation a consumption of these compounds could falsify the determination which could lead to misinterpretation of the oil condition. Further considerations are given in section 3.4.

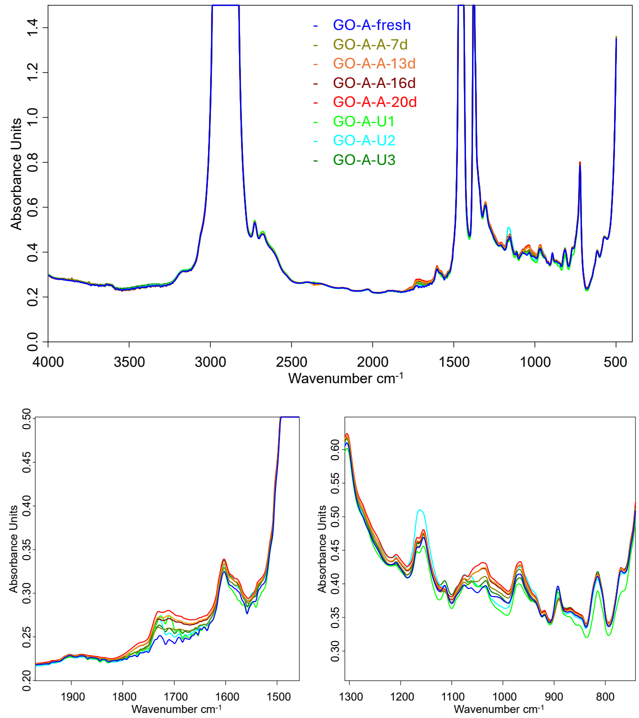

Figure 2: FTIR spectra of gear oil GO-A in fresh, used and aged condition

Figure 2 shows the FTIR spectra of the oil samples from the gear oil GO-A. In comparison to the engine oil, it is clear that lower amounts of components absorbing in the infrared range are present in this type of lubricant. The main differences can be seen in the range from 1800 cm^{-1} to 1600 cm^{-1} which is due to oxidation of the oil.

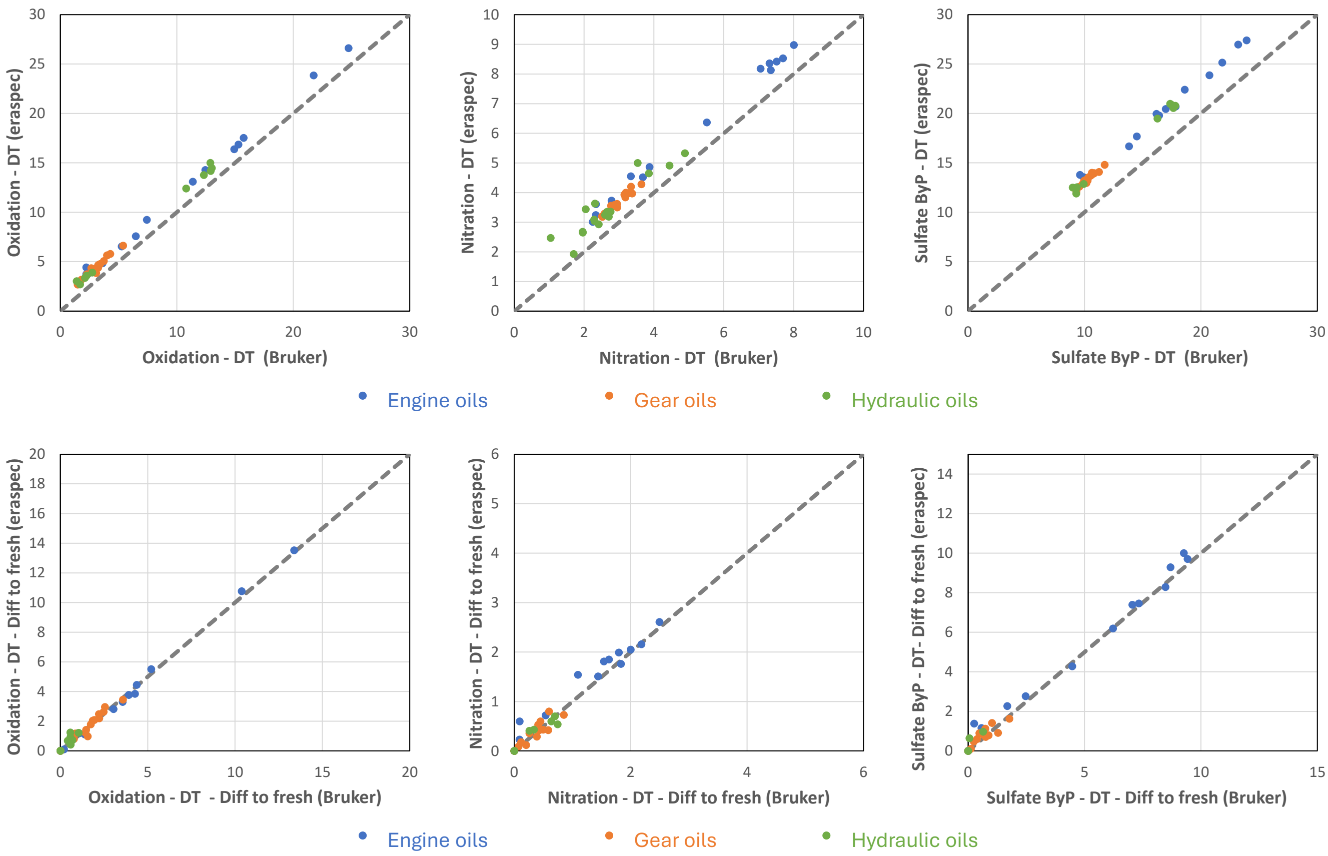

Figure 3: Correlation of FTIR evaluation between the laboratory FTIR spectrometer (Bruker) and portable "Oil Condition Monitoring-FTIR" eraspec oil (eraspec) by means of oxidation, nitration and sulfate by-products (ByP) with direct trending evaluation according to ASTM E2412; TOP: Results as obtained from each spectrum; BOTTOM: Difference to the corresponding fresh oil

3.2. Evaluation and comparison of FTIR spectra

For the evaluation of specific parameters different methods exist. A particularly useful guideline is given according to ASTM E2412 which was applied to the FTIR spectra of the investigated oil samples measured with two different devices. Figure 3 shows the correlation of the results for the evaluation of oxidation, nitration and sulfate by-products (ByP) evaluated using the direct trending methods. This means that the evaluation was done on the FTIR spectra as obtained from the device without subtraction of the spectra of the fresh oil. In the TOP diagrams of Figure 3 the results as obtained are correlated between the devices. Additionally, the 1:1 correlation is indicated by the dashed line. As can be seen, a device-specific offset is noticed. However, the slope can be seen as equal as the points are just parallel shifted to the 1:1 correlation line. Observing only the changes in comparison to the corresponding fresh oil, which is drawn at the bottom diagrams in Figure 3 by subtraction of the values of the fresh oil, all points are lying on the 1:1 correlation line with only a minor error. Consequently, both devices deliver the same results when considering the changes in comparison to the fresh oil.

Please note that it is important to obtain the results of the oil samples with the same device. However, the results showed that when considering the changes in comparison to the fresh oil, also results from different devices can be compared as long as both values are evaluated using the same method.

A direct comparison between the FTIR spectra is shown in Figure 4. For better visualization, the spectra were normalized at a wavenumber of 2000 cm^{-1}. Generally, the spectra look very similar, and nearly all peaks are identically available. However, some differences in the general height are visible which can be due to device-specific circumstances in obtaining and calculation of the FTIR spectra. Besides, some differences occurred at the FTIR spectra in the range below 650 cm^{-1}. In particular the spectra from the device era**spec oil** show at some samples a different behavior as shown as an example in Figure 4 in the top and bottom diagram. As indicated, this different behavior could lead to a different setting of the base line for the evaluation of the FTIR spectra and consequently to an additional error. This different behavior between the two devices can be due to different considerations of the background. With the spectra obtained with the device from Bruker, empty space (without cuvette and oil sample) was used as the background. The device era**spec oil** uses a factory set background spectra with an empty cell. As in this wavenumber range the window material of the cuvettes, which are made from ZnSe, starts to absorb much of the infrared light itself, even small errors in obtaining the FTIR spectra could lead to these circumstances. Consequently, the setting of the baseline could have an influence on the results as the baseline is defined in areas with less practical information but having device-specific deviations. Therefore, a rework of the baseline setting could deliver a remedy. However, as shown in Figure 3, these errors in the measurement of the FTIR spectra are only on a minor level and do not significantly influence the overall evaluation of the oil condition.

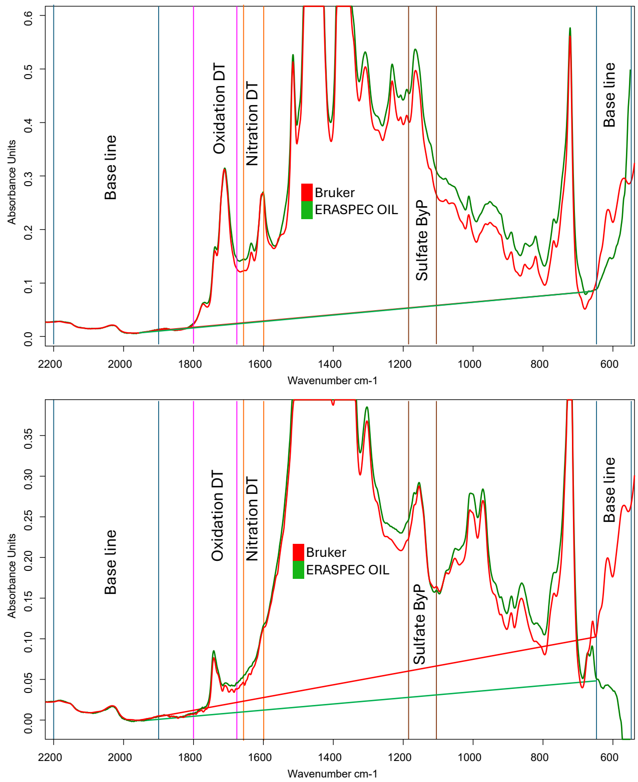

Figure 4: Influence of spectra trend on base line definition and spectra evaluation; TOP: good reference example; BOTTOM: example with higher deviation

3.3. Total Acid Number (TAN) and Total Base Number (TBN)

A further advantage of FTIR evaluations is the determination of parameters which are not directly accessible, e.g. Total Acid Number (TAN) and Total Base Number (TBN). In the course of this case study, models for both parameters were calculated using era**spec oil** and analyzed in terms of accuracy. This Oil Condition Monitoring-FTIR era**spec oil** includes a chemometric model according to the multi-linear regression method (MLR) for TAN and TBN as standard. The advantage of the MLR method is the robustness of the model and the relatively small number of calibration samples required.

For total acid number (TAN), the reference of choice was a potentiometric titration according to ASTM D664, which was used to build the model by adding reference values to the measured FTIR spectra directly on the instrument. The good correlation between the FTIR evaluated values to the reference titration is shown in Figure 5. A useful parameter to describe the accuracy of a chemometric model is “SEC”, the standard error of calibration. For a total of 112 samples, including some duplicate determinations, a SEC of 0.27 mg KOH/g could be achieved. This is clearly a proof of concept if it is compared to a reproducibility (R) of 0.56 mg KOH/g at 3 mg KOH/g according to ASTM D664 although different lubricants were used in this to build the model.

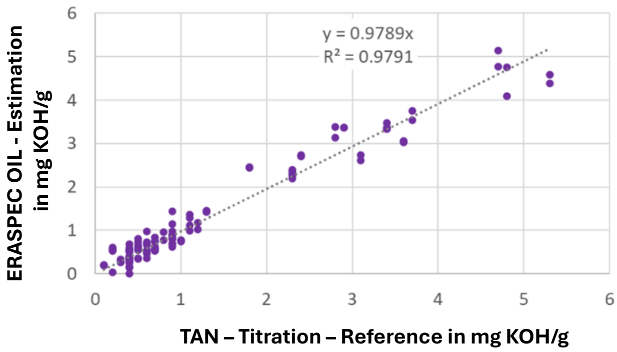

Figure 5: Chemometric FTIR model of TAN in relation to conventionally obtained TAN by ASTM D664

The same conclusion could be drawn for the total base number (TBN) (see Figure 6) , where a SEC of 0.10 mg KOH/g could be compared with a reproducibility (R) of 0.6 mg KOH/g at 8 mg KOH/g according to ASTM D2896 for 32 samples.

3.4. Challenges in oil condition monitoring using FTIR

The investigation has shown that condition monitoring using FTIR spectroscopy is a powerful tool that provides essential information about the degradation state of the lubricant and therefore also the condition of the machinery itself, while being cost efficient, easy to handle and fast. Furthermore, the need for specific chemicals or solvents can be minimized as in most cases only solvent for cleaning the infrared cell for the next measurement is needed. This gives environmental advantages.

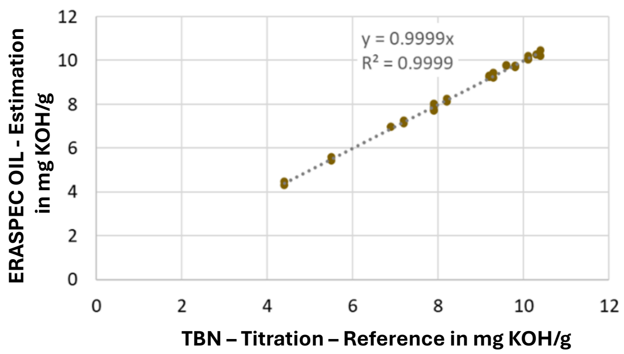

Figure 6: Chemometric FTIR model of TBN in relation to conventionally obtained TBN by ASTM D2896

However, it is essential to carefully select the right evaluation method for the desired application. If in condition monitoring the changes of the lubricant in comparison to the fresh are considered, the availability of the correct spectra of the fresh oil is of crucial importance. For methods evaluating the difference spectra, mostly dedicated to spectral subtraction methods, the entire fresh oil spectrum must be available and has to be selected prior to the measurement of the used oil in most cases. Using direct trending methods each spectrum is evaluated itself and the results are subtracted in a second step. Using this only the evaluated or reference values of the fresh oil are required which could be easily stored in a database or directly in the FTIR spectrometer.

Apart from having the corresponding fresh oil or fresh oil spectrum, it has to be considered that all spectra are obtained with the same device. As discussed above using Figure 3, device-specific differences can occur when comparing different FTIR spectrometers. As the results obtained here show a direct correlation of the evaluated values, fully evaluated parameters can also be analyzed by different devices, i.e. the comparison of values between different laboratories should be possible. However, when comparing results from different laboratories the use of the same evaluation method is a prerequisite.

Taking oxidation as an example, several methods and standards exist for evaluating the oxidative degradation of a lubricant. Besides DIN 51453[13] or ASTM E2412 [9] also several in-house methods from component manufacturers or spectrometer manufacturers exist. Typically, the methods evaluate the changes in the FTIR spectrum in the range of 1800 cm^{-1} to 1600 cm^{-1} using the absorbance at a specific wavenumber – e.g. 1710 cm^{-1} at DIN 51453 – in comparison to a reference point or baseline. The choice of the correct evaluation method is primarily dependent on the composition and type of oil being treated. As several additives like ester compounds or dispersant additives have absorption bands in this wavenumber range, degradation or consumption of these compounds could falsify the assessment of the oxidative degradation of the lubricant. Also, contamination of these compounds due to top-up with a different oil type could falsify the results and lead to misinterpretations. Consequently, the chosen method has to be adapted to the composition and type of lubricant. This is demonstrated in Figure 7. The top diagram shows difference spectra of the engine oil EO-A with the highlights of the wavenumber for evaluating the oxidation according to ASTM E2412 or DIN 51453. As already mentioned above and shown in Figure 1, the presence of dispersant additives can falsify the evaluation of the oxidation. When evaluating according to DIN, clearly lower values are obtained in comparison to ASTM E2412 which could lead to misinterpretation of the oil condition.

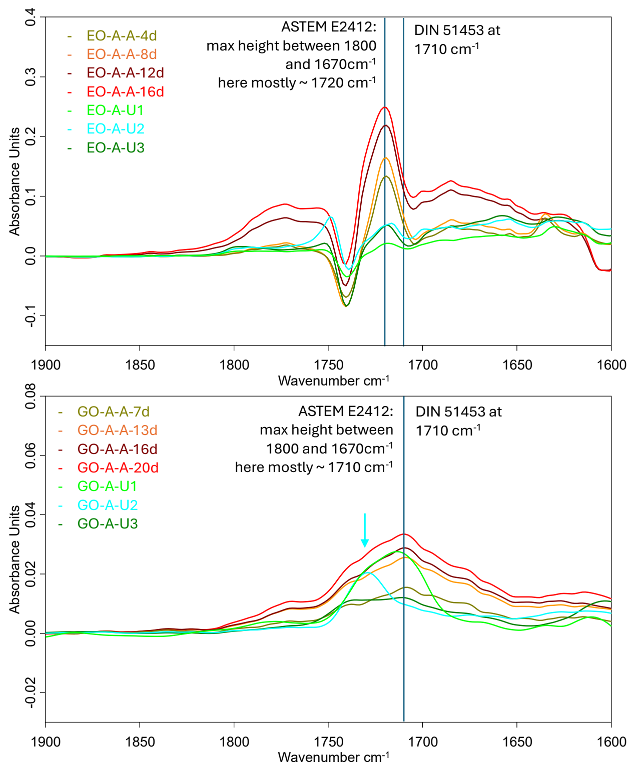

Figure 7: Difference spectra of oil samples with indication of oxidation evaluation according to ASTM E2412 and DIN 51453; TOP: samples from engine oil EO-A; BOTTOM: samples from gear oil GO-A

In the bottom diagram of Figure 7, an example of the gear oil GO-A is shown. In this case, mainly similar results are obtained at both evaluation methods. However, in one sample a contamination of an ester compound was observed which would falsify the oxidation evaluation using the method according to ASTM.

Summarizing, FTIR spectroscopy is nowadays one of the most important tools when talking about condition monitoring. Considering the key facts, like the corresponding fresh oil and the right evaluation methods, a good comprehensive picture of the oil condition is obtained for decision-making on maintenance issues.

4. Summary and conclusion

This comprehensive case study in the field of oil condition monitoring successfully demonstrated the comparability of the methods used. The clarification of differences in FTIR measurements improved the understanding of the analysis results. In addition, the chemometric FTIR model proved its usefulness by estimating TAN and TBN results that agreed well with the reference values.

Overall, these results underlined the robustness and applicability of the applied methods and provided valuable insights into the characterization and assessment of aging processes. The investigation demonstrated the power of FTIR spectroscopy for condition monitoring of lubricants as it is cost-efficient, easy to handle and fast. The study also considered the challenges for application when comparing results.

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