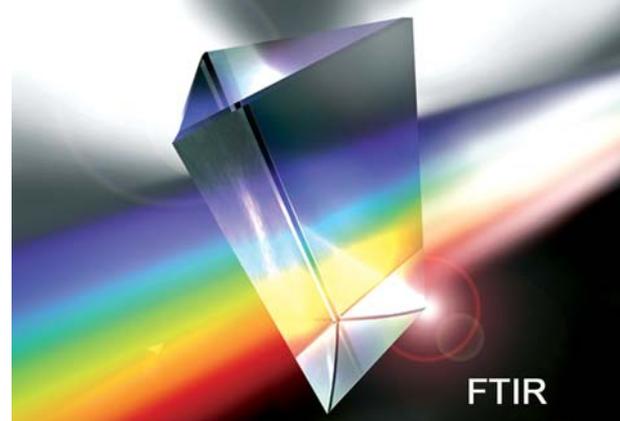


Application Note

Saponification number – determination of fats in rolling oil using FTIR spectroscopy and single reflection accessory



In a production plant everything is fine when the belts transport correctly and rolls are doing a fine job. Rolls can work only properly when they are for example with rolling oil. The rolling oil must have good anti-friction quality. This quality is depending on the contents of fats in the rolling oil. The control of the fat content gives an indicator if it is aged or must be replaced by better quality.

The fat contents in a mineral oil can be determined. This is done by the determination of the saponification number. The saponification number in mineral oils is regulated world wide in national and international norms:

Germany	DIN-51559-1
	DIN-51559-2
Great Britain	BS-2000-136.1
	BS-2000-136.2
ISO	ISO-6293
ASTM	ASTM D0094
Japan	JIS-K2503

- (1) colour titration indicator
- (2) potentiometer

The norms are related to titration method or potentiometer method.

The amount of fatty material in oil is done using a chemical test named Saponification and this is so called one index of anti-friction. Saponification is a chemical process of converting fats to soap. A lot of lubricants like worm gear oils or pneumatic tool oils, contain fatty type additives to improve anti-friction properties. Saponification number is performed according to for example ASTM D 94. The saponification number indicates the amount of fatty substances in the oil. Saponification number is the number of milligrams of KOH that combines with the fat in 1 gram of oil to form the soap. Therefore, it

can be concluded the higher the number, the higher the amount of fatty material is in the oil.

This determination following the norms is in comparison to a FTIR measurement a time consuming work. Within this application will be shown that the FTIR technique is an easy, speedy and precise alternative in doing this style of analysis. In one step it can be done identification of the oil and determination of the saponification number

To keep the system as simple as possible a single reflection ATR accessory with diamond was used. The diamond is the most robust material and a recommendation for simple cleaning and long time stability. Using this accessory the time which is needed for a measurement can be calculated with 2min. It is 1min for measurement, result and another one for the cleaning procedures.

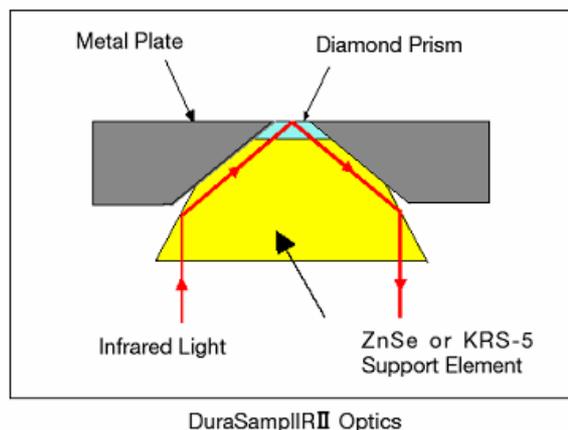


Fig. 1: Light path through the accessory DuraSampIR II, a single reflection ATR measurement technique

A drop of the oil is simply placed on the top of the diamond prism. After measurement it is easy to clean with solvent on a tissue and a blank tissue.

The measurement is done with FTIR instrument which can establish measurement within one minute. Result will be a spectrum and the calculation of the saponification number. Saponification number presented here is based on a calibration of rolling oils which were classified following the norms.

SAP Unit	
1593.202 - 1851.663	
Concentration	Peak area
128.000	5.460
147.000	6.355
137.000	5.909
179.000	7.562
195.000	7.935
163.000	6.984
153.000	6.647
145.000	6.242
138.000	5.950
170.000	7.223
160.000	6.944
170.000	7.128

Fig. 2: List of 12 standards for the calibration model with known saponification number (SAP)

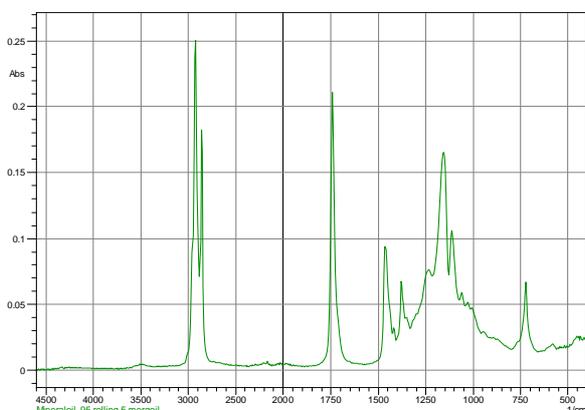


Fig. 3: typical FTIR mid infrared spectrum of rolling oil

It is possible to use the classical least square quantitation because FTIR is following the Lambert-Beer-Law which explains the linear relation of extinction to concentration using a fixed sample thickness. In the oil system is the relation of the ester signal to the saponification value in same relation. So, it is possible to calibrate the saponification number via absorbance of a mid infrared spectrum. Table 2 represents the 12 Standards with the known SAP number. In this calibration model the area under signal of the ester group at 1740cm⁻¹ was analyzed.

The result is shown in Fig. 4. A correlation coefficient $r=0.991605$ was easily reached.

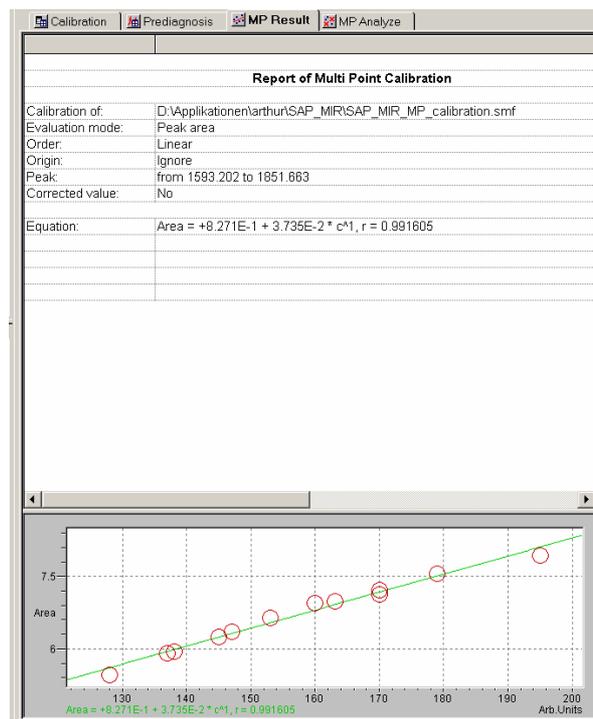


Fig. 4: calibration model for the saponification number of rolling oil using the area under the -CO- signal at approx. 1740cm⁻¹.

To proof the quality of the calibration a test was done with standard 4 and 9 (see fig. 2).

Methode	Saponification number	
	ASTM D0034	FTIR
Sample 9	160	160.114
Sample 4	153	153.702

This seems to be a good result in comparison to other techniques. Naturally it is better to expand the calibration set to improve the quality of calibration curve.

Instrumentation

- IRPrestige-21
- DurasamplerII single reflection ATR system with diamond sample plate and KRS-5 support element
- IRsolution software and Multi Point quantitation module

The given specifications serve purely as technical information for the user. No guarantee is given on technical specification of the described product and/or procedures.