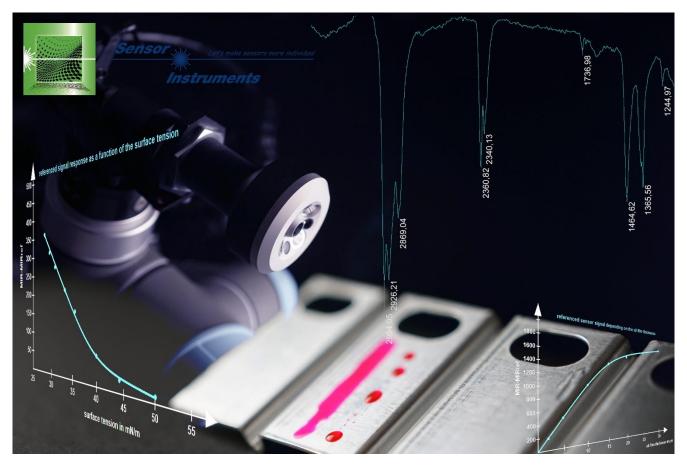


SPECTRO-M-10-MIR Inline control of thin oil layers on metal



The MIR measurement procedure deployed in the SPECTRO-M sensors was developed by Sensor Instruments in order to measure very thin organic layers on metal surfaces. As such, SPECTRO-M sensors are highly suited to the detection and inline control of thin oil films on metal.

In practice, it is necessary to control the de-oiling process of parts and monitor the homogeneity of an oil application. Within of the course of tests accompanying the development, we ascertained that the system can be used to measure the evaporation characteristics and oil residues of cutting oils on metal surfaces performed under realistic application conditions (thin layers).

To this end, we have also performed tests on special punching oils from the CLF series from Raziol; the results are summarized in section 6 of this document.

This document described the sensor principle of the SPECTRO-M-10-MIR/(MIR1+MIR2) inline sensor from Sensor Instruments. Further SPECTRO-M sensors of the type with other measurement geometries are currently in development. As the developer of special sensors, we are also able to adapt the technology to match special applications and customer requirements.



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1 Introduction

The SPECTRO-M series is established on a measuring principle known as oscillation or absorption spectroscopy.

Oscillation spectroscopy is the analysis of materials through radiation with various light wavelengths (energy). Light shone into the material is partially translated (absorbed) into molecule oscillations and the exiting radiation in the form of spectra is recorded. Information about the molecular structure of organic materials can be gained from the oscillation spectra. This means that it can be used to ascertain and control chemical and physical characteristics.

IR spectroscopy uses infrared light to stimulate the degrees of oscillation freedom in the molecules known from quantum physics. We then observe the absorption bands in the IR spectrum. The ranges of the basic oscillations lie in the mid infrared range, (MIR, ~ 2500nm - 16000nm); that of the combination or harmonic oscillations lie in the near infrared (NIR, ~ 800nm - 2500nm) range. The NIR absorption bands are usually broad, overlapping structures, whilst MIR absorption operates are mapped in narrow band ranges.

The absorption of the NIR bands is many magnitudes lower than the basic oscillation, but provides sufficient information with which to classify polymers. NIR absorption procedures are used in plastics recycling. MIR absorption is suitable for characterizing thin organic layers on metals.

The SPECTRO-M family from Sensor Instruments are inline sensors which evaluate various absorption behaviors of organic materials in MIR, in order to obtain qualitative and quantitative statements about organic layers on metals in the production process.

Oil layer control and measurement is an interesting area for testing organic layers on metals. Special oils are used to optimize the punching or deep drawing processes, but must be completely removed after completion of the processing.

The inline testing of the oiling of metal parts or the removal of this oiling is a measurement challenge which has become ever more complex with the increasingly thin layers and specific oil recipes and functions.

The evaluation of surfaces is often performed via the testing of the surface of interfacial tension. Two common procedures for measuring surface tension include the test ink method and the contact angle measurement on which this document focuses.

We have summarized our investigations of punching and drawing oils with the SPECTRO-M-10-MIR and compared the results with the red test ink method. This involves the detection of the smallest oil residues or measurement of various layer thicknesses. There are also oils which evaporate in a time window defined for the purpose but which leave residue on the surface. The reproducible measurement of the evaporation characteristics could be interesting for the manufacturers of such oil products.

2 The functional principle of the SPECTRO-M-10-MIR

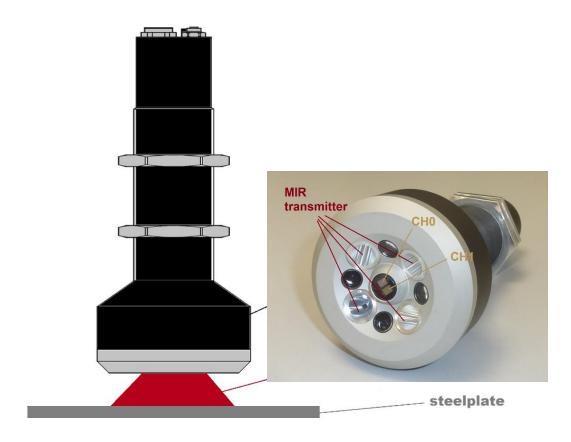
An inline measurement system depends primarily on the capability of the contact-free measurement. The measuring time required must be able to keep pace with the component feed rate. The measurement system should be compact and robust and be equipped with multiple interfaces to enable feed of the measurement data to the superordinate control system or a PLC, preferably in real time.

The SPECTRO-M-10-MIR/(MIR1+MIR2) sensor is fitted with relatively wide band illumination for the MIR range. On the receiver side, there are two measuring windows (wavelength ranges) which can be compared in a standardized fashion. One of these windows serves as the reference and in the first view acts neutrally towards oils, whilst in the second window, absorption caused by the oil can be determined. The Windows®-software SPECTRO MIR Scope V1.0 can be used to parametrize the sensor, perform test measurements and save measured values. A monitoring software is available for inline operation which displays the current measured values on a screen or records measurement data in ASCII files.



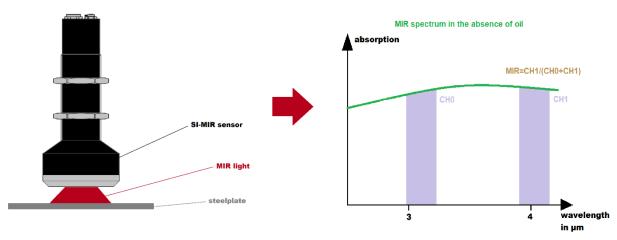
During the development of the SPECTRO-M-MIR, we tested a series of commercially-available punching and drawing oils with our optical sensors. We also analyzed and compared the measurement effects in the UVC range (255nm, SPECTRO-1-UVC) and UVA fluorescence (SPECTRO-3-UV) and Absorption in the MIR range (5µm).

All types of punching and drawing oil previously tested could be measured with the MIR procedure. A measuring window of between 3µm and 4µm proved to the best working spectral range for the SPECTRO-M-10-MIR. The comparative procedure for two spectral ranges provides standardized MIR measurement results, which enable compensation of the greatest proportion of the reflection characteristics of the contact surface. As the various oils present a strong MIR absorption, calibration to the oil-free metal surface is necessary (MIRreff). If you wish to convert the measured values into the units of a reference system, for example if surface tension values are to be issued as a measured value in mN/m incorporating the test ink method, a one-off reference calibration (CONVERSION) is to be produced. The following sketches show the principal mode of operation of the MIR sensors:



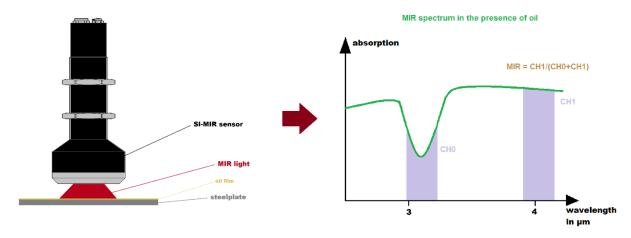
Wide band MIR transmitters cover the requisite spectral range required for the two measuring windows CH0 and CH1. A part of the light reflected by the metal surface impinges on the two measuring windows; one measuring window (CH0) reacts to the presence of an oil film, whilst the other measuring window (CH1) remains almost unreactive to the presence of an oil film (reference window). A standardized evaluation produces an intensity-independent measured value:



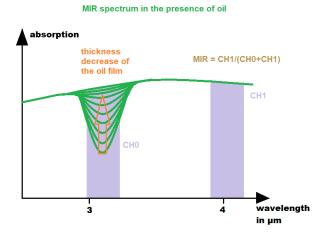


MIR = CH1/(CH0+CH1)

The sketch above does not contain an oil film on the metal surface. Both measuring windows give almost the identical signal level (standardized value = MIRref). In the following sketch on the other hand, the collapse of the signal level from measuring window CH0 results in an offset of the standard value MIR.



The thicker the oil layer, the more severe the signal dip in the measuring window CH0:

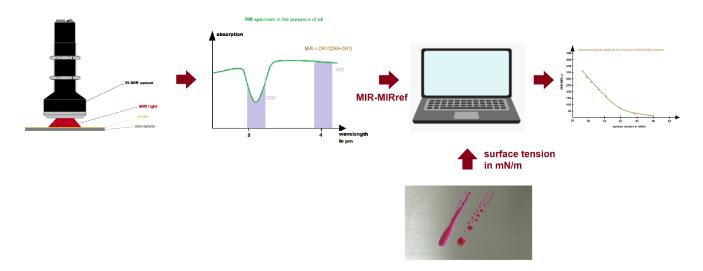


A standardized value, MIR = CH1/(CH0+CH1) can be calculated from the two signals, which represents the two channels. This standard value is a proprietary value determined by the sensor parameters, the oil and the metal surface. The sensor software can be used to correlate the standard value with a reference measurement procedure and to output it in a reference format as a "calibrated measured value".

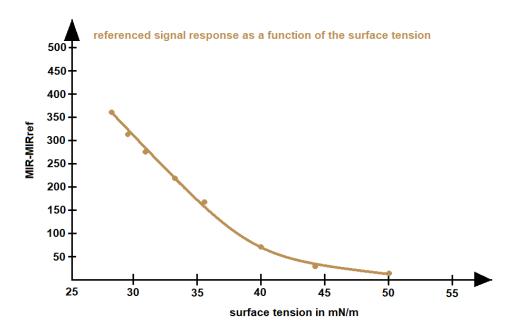


Example: Red ink method (mN/m) as an example for a reference system calibration:

To ensure that the surface tension value of the metal oil layer surface and not the standardized value (MIR) can be transferred to the sensor interface, the relation to the test ink method must first be established (calibration).



To this end, one of the metal parts to be tested is first degreased and then the standardized reference value (MIRref) is ascertained using the MIR sensor. The corresponding surface tension value is then determined in mN/m using the test ink method. As a next step, a degreased metal part is subject to the same procedure with the difference that in each procedure (measured value with oil present: MIR) an increasingly thicker oil layer is applied to the degreased component in an homogeneous fashion (experience shows that the thickness of the oil layer moves between 100 nm and 5µm). The measured values (MIR-MIRref) are then recorded in a table and this is used as a conversion table in a non-volatile sensor memory. Graphical depiction shows the following relationship:



A separate spacer is available to perform offline measurement in production or in a laboratory. The measurement is performed continually. The working distance of the sensors to the surface to be measured amounts to 10mm.

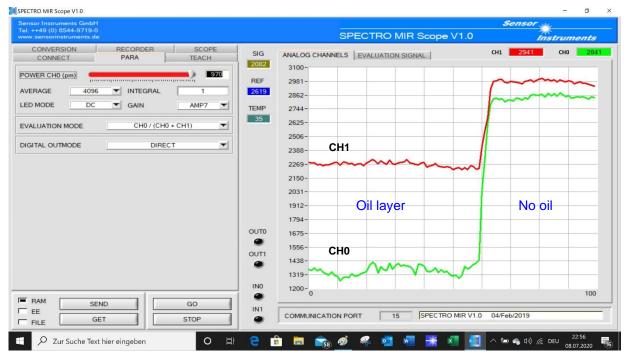






SPECTRO-M-10-MIR/(MIR1+MIR2) - sensor head

Offline attachment for laboratory measurements



SPECTRO MIR Scope - Windows®Software for configuration of the sensor, test measurements and sensor calibration to a reference measurement procedure.



3 Reference methods for determining the surface tension

3.1 Surface tension and interfacial tension

Why is it so important to know about the surface tension of metal surfaces?

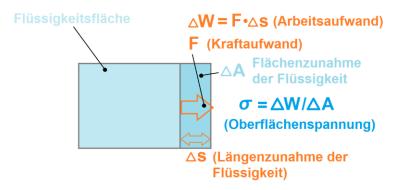
An example: Before applying color paints to a metal part, it is necessary to check the surface tension value of the metal surface. If this lies under the surface tension value of the paint, the paint would peel from the metal surface.

The same applies to the application of adhesives or other types of coating. Sufficient adhesion is only generated if the surface tension of the metal surface is greater than that of the glue or the coating material.

How do we define surface tension?

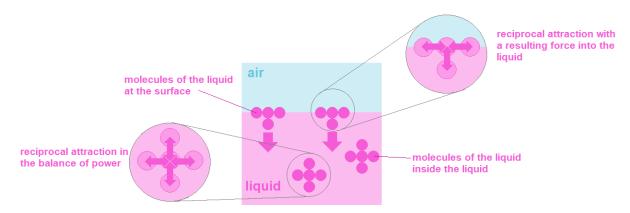
A certain amount of force must be expended to increase the surface area of a liquid. This requires a certain input. The surface tension is the ratio resulting from the output required to increase the surface area (increase of surface area of the liquid):

 $\sigma = \Delta W/\Delta A$ (SI unit: N/m)



The surface tension means that liquids always attempt to reduce their surface area, which results in a spherical surface (a sphere has the lowest surface of all forms with the same volume).

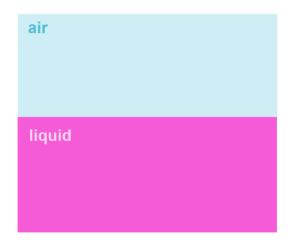
What is interfacial tension?



All the molecules inside a liquid are equally surrounded by their neighboring molecules, thereby maintaining balance between the reciprocal attractive forces. A force develops on the edge of the liquid (boundary surface), which due to the absence of a symmetrical division of the reciprocal attractive forces, points to the inside of the liquid.



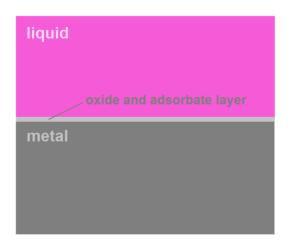
The surface tension is therefore referred to as the interfacial tension. The term interfacial tension is used for the transition from a liquid to a substrate, for example a metal surface, whilst the transition from the liquid to the air is referred to as the surface tension.



The term surface tension is used for the transition from air to water.



The processes occurring at the transition from liquid to metal is termed the interfacial tension.



Strictly speaking, this transition (liquid / metal) is a transition from a metal oxide layer / an adsorbate layer (carbon dioxide and hydrocarbon on the metal surface) to a liquid.

What is the wetting of metal surfaces?



A unit for the wetting of a metal surface by the application of a liquid (e.g. as droplets or brushing) is the wetting angle Θ . The wetting of the metal surface increases with a reducing wetting angle Θ .

The wetting process is described by Young's equation:

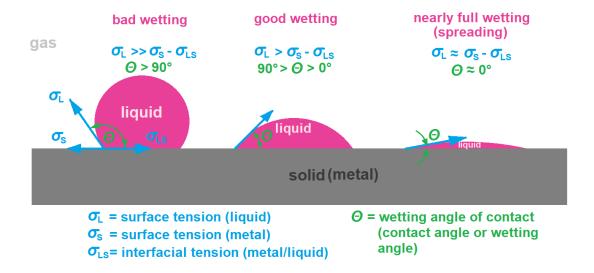
$$\cos \Theta = (\sigma_s - \sigma_{LS})/\sigma_L$$

with

 σ_L = surface tension of the liquid

 σ_S = surface tension of the metal

 σ_{LS} = interfacial tension between metal and liquid



 Θ = wetting angle (contact or wetting angle) of the liquid on the metal.

The independent distribution of the liquid on the metal surface (spreading of the brush stroke or the droplets, also referred to as spreading) is achieved with a wetting angle of $\Theta = 0^{\circ}$ (accords to $\cos \Theta = 1$). In this case, Young's equation produces $\sigma_L = \sigma_S - \sigma_L s$. If $\sigma_L s << \sigma_S$, the following applies for the liquid which is to wetten a metal surface: $\sigma_L < \sigma_S$. This produces the following situation: if the surface tension of the liquid is lower than the surface tension of the metal, the metal is wetted by the liquid. If on the other hand, the surface tension of the liquid is greater than the surface tension of the metal, no wetting is performed.



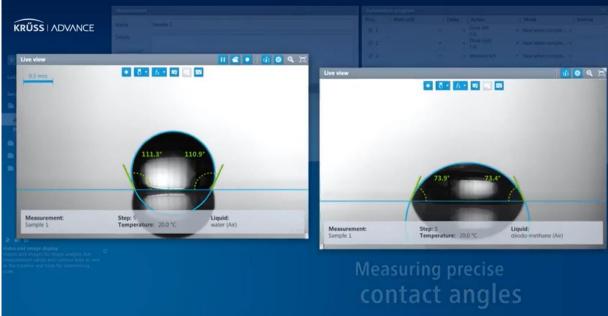
3.2 Determining the surface tension by measuring the contact angle

The surface tension values stated in the specialist literature relate primarily to liquid metals close to their melting temperature. To ascertain the wetting, the surface tension of the metal surfaces covered with oxide and adsorbate layers at room temperature is significant.

A measurement method for the local determination of the surface tension of metal surfaces is contact angle measurement. A droplet of a liquid with a defined surface tension is applied to the metal surface and the wetting angle of the droplets are subject to optical evaluation.

Various measuring devices are available for the measurement tasks. The surface tension value (free surface energy) could for example, be ascertained using the Mobile Surface Analyzer- MSA from KRÜSS GmbH www.kruss-scientific.com; two drops of the test liquids with different surface tension are applied to the metal to be tested and the surface tension of the metal is ascertained by measuring the wetting angle.

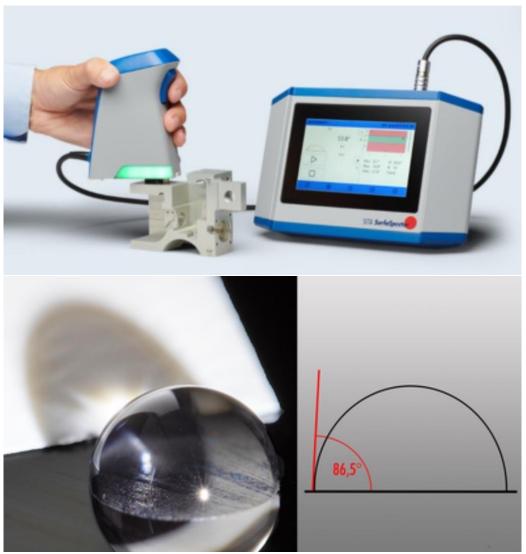




Copyright: Krüss GmbH

Another provider is SITA Messtechnik GmbH www.sita-process.com. SITA provides a measurement system for the "fast ascertainment of the surface wetting capability in the manufacturing process by measuring the contact angle". The SITA SurfaSpector is a hand-held measuring device for testing the wettability e.g. of metal surfaces before and after surface treatment or cleaning. This method applies a droplet (1µI) of ultrapure water to the surface to be measured via a metering unit integrated in the sensor head. A high-resolution camera in the front end of the sensor is used to ascertain the droplet contour from which the contact angle can be ascertained.



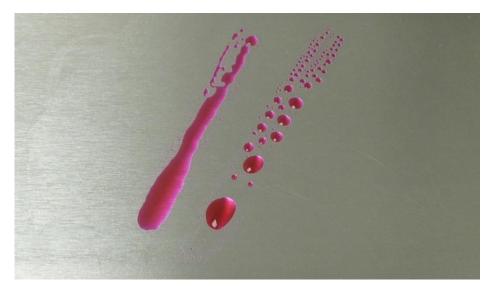


Source: SITA Messtechnik GmbH



3.3 Determining the surface tension using the test ink method

A popular method with which to ascertain the surface tension of metals at room temperature is the test ink method. Test ink with a defined surface tension is applied to the metal surface to be investigated using e.g. a brush. If the test ink wettens the metal surface, the surface tension (interfacial tension) of the metal surface corresponds to or even lies over the value of the test ink. If on the other hand, the test ink rolls off the metal surface, the surface tension (interfacial tension) of the metal surface is lower than the surface tension of the test ink.

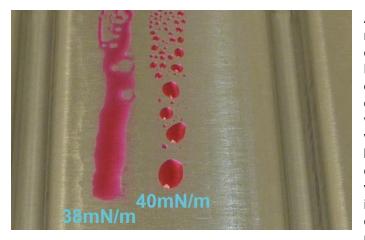


Test inks are available e.g. from arcotest GmbH <u>www.arcotest.info</u>. PINK test inks cover a surface tension value of 22mN/m to 60mN/m in 2mN/m steps.



As already mentioned, a defined surface tension of the substrate is a very important factor in the coating process. What is the cause of too low a surface tension value for a metal surface?





As already mentioned, the metal surface is really a metal oxide and adsorbate layer. Oxygen atoms and carbon atoms are integrated in the metal surface. Measurements have shown that a chemically degreased metal surface (more precisely, a metal oxide and adsorbate surface) has a surface tension value > 60mN/m. If we consider e.g. blanked parts, wettened with oil as part of the blanking and bending procedure and then cleaned in a conventional cleaning system, a surface tension value of the blanked parts is c. 38mN/m; in individual cases it can reach 44mN/m. Before the cleaning process, the surface tension value on the metal surface of the blanked parts usually lies below

30 mN/m, depending on the punching and drawing oil used.

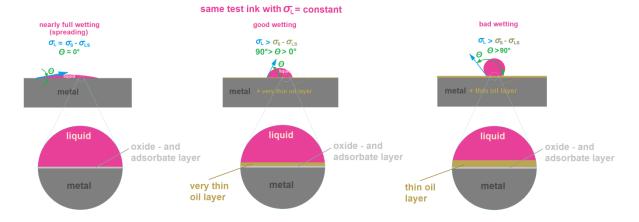
Blanked parts and more importantly, plug contacts require a high surface tension value (> 38mN/m), as this value points to the proportion of residual oil on the metal surface. For example, the residual oil increases the contact resistance, which can reduce performance with the plug contact transition.

Using two test inks (38mN/m and 40mN/m) it was possible to demonstrate that the surface tension value of this contact surface lay between 38mN/m and 40mN/m.

Clean wetting is no longer possible with a test ink of 40mN/m; the wetting angle Θ deviates correspondingly strongly from 0°, whilst close to a complete spread is achieved at 38mN/m.

The following diagram provides a schematic view of how the surface tension of the contact surface reduces with an increasing oil layer thickness (controls with the same test ink are performed):

The wetting of the contact surface reduces with an increasing thickness of the oil layer.



Test inks with various surface tensions are used in sequence to ascertain the surface tension of a contact surface. Should the surface tension of the contact part amount to a minimum of 38mN/m, you can start with a test ink of an identical surface tension value: 38mN/m test ink wetted, 40mN/m test ink not completely wetted, 42mN/m test ink rolls off the contact surface.



same metall surface with same thin oil layer

(constant surface tension of the metal surface and the thin oil layer)



The test ink method is a simple and quick to implement procedure for the random testing of components. It would be preferable to obtain direct information about the residual oil layer thickness, which in the past has proven difficult to perform.

The test ink method has established itself as a reliable method for controlling for the presence of thin oil layers in an industrial context.

This explains why the following test measurements also use the test ink method as a reference method.

3.4 Reference procedure and inline measurement

The reference procedure for ascertaining the surface tension presented here is a test procedure in which individual random samples are taken from the production process and which are used to make representative statements about the surface quality of the entire batch. This test method is often used in the scope of laboratory or At-line quality control.

A 100% control of the produced components is becoming increasingly desirable, which requires an inline measurement procedure.

Ideally, the measured values would be outputted by an inline procedure comparable with an established reference procedure e.g. the test ink method. For example, the inline measurement system would display values for the surface tension of the component in mN/m.

The inline sensors of the SPECTRO-M sensors provide a calibration function (CONVERSION) with which the internal MIR values can be converted into the values of the offline system (test ink method) without problem.

4 Areas of application for SPECTRO-M sensors

In principle, all organic bonds, e.g. polymers, oils, ..., oscillation absorption in the MIR range. Nevertheless, every MIR procedure reaches its limitations when the organic layer is so thick that it absorbs all the stimulus energy and reflects almost no wavelets.

However, the system works very well if thin organic layers are applied to non-organic substrates, i.e. substrates which do not present any MIR absorption. The detection of thin oil layers on metals (in metalworking, especially in punching) is extremely well-suited for the use of the MIR measurement procedure.

The sensors of the SPECTRO-M family are especially suited to the following applications:



- Inline control of the surface tension of metal surfaces after cleaning.
- Inline control of the layer thickness of thin oil layers applied to metal surfaces.
- Laboratory analysis of the evaporation of oils on metals and the ascertainment of the remaining application.

The following sections describe our test measurements of various oil products and the method used.

5 Test measurements on various oils

5.1 The motivation for measuring oil layers on metal surfaces

The use of oils is imperative in the forming process of metals. For example, the application of cutting oils to metal bands ensures low wear on the punching tools. Drilling oils make an essential contribution to the protection of the drilling and milling tools during the chipping processes. Oils also serve to protect semi-products such as sheets and metal foils against corrosion. After further processing, it is important to remove the oil residues from the finished products as completely as possible. This task is performed by special cleaning systems, in which the metal parts are washed and blown dry.



In order to ensure that environmental specifications are maintained during the application of the oils, and to ensure that the process is economical, it is advisable to ascertain the quantity of the oil applied. Oil quantity measurement can be performed inline, in a running process. UVA fluorescence measurement procedures are often used for this purpose. Not all oils exhibit the requisite fluorescence characteristics, and additional UV additives cause higher costs.

The quality control of the cleaning process can also be performed using sensors. The challenge is to determine even small quantities of oil residue accurately. Oil residue reduces the surface tension and interfere with the subsequent coating process.

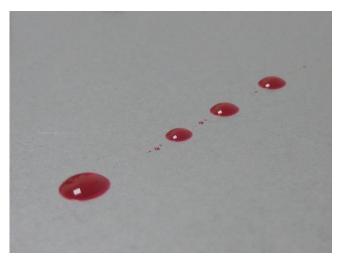
Electrically conducting components such as copper busbars or power current lines require the lowest possible transfer resistance. A residual oil layer would pose a problem as the conductive efficiency is restricted to a considerable degree.



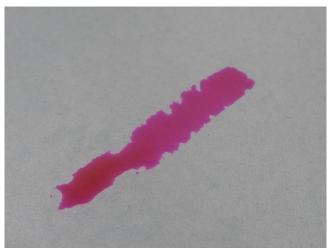
5.2 Test ink method – a common reference measurement procedure for controlling the residual oil

When measuring oil layers, we usually first think of a certain layer thickness, for example measured in µm. One possibility would be to ascertain the grammage of the oil film. This requires knowledge of the weight of the entire component. Precise scales and an identical non-oiled component are required to ascertain the difference in weight. To determine the layer thickness from the grammage requires information about the concentration of the respective oil and the surface of the respective component. The relatively large difference in weight between the actual oil layer and the weight of the component is not conducive to exact determination of the oil layer thickness. A further source of error is the difference in weight between the two unoiled components.

The lack of effective and economical methods for measuring the layer thickness of the residual oil could be the increasing popularity of a simple test method for determining the surface tension of metal parts. The presence of a thin oil layer on a metal surface reduces the surface tension from > 50mN/m in non-covered state to under 40mN/m with the oil layer (depending on the layer thickness and the oil type). Test inks covering a range of 30mN/m to 50mN/m in intermediate steps (30mN/m, 32mN/m, 34mN/m, ...) serve to prove the respective surface tension. The test ink is brushed onto the area to be investigated using the brush included in the scope of delivery of the bottles. If the test ink rolls off of the surface, the next higher test ink will be applied in the same fashion until the test ink adheres to the surface over a longer period and does not run off. The surface tension lies between the two test inks last used. The principle is that reduction in the surface tension is associated with increase of the thickness of the oil layer.



The test ink rolls from the oil-covered metal surface, which indicates a surface tension which is smaller than the value specified on the test ink.



In this diagram, the test ink adheres to the oilcovered or degreased metal surface. The surface tension of the metal surface is higher than the value specified for the test ink.



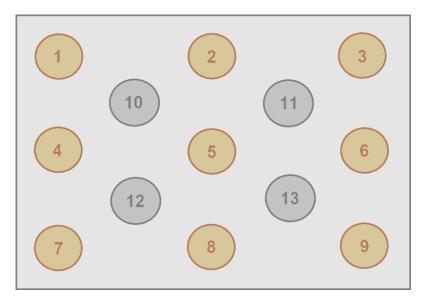
5.3 Test structure and method

The SPECTRO-M-10-MIR/(MIR1+MIR2) was upgraded with a spacer or an offline attachment to perform laboratory measurements (see right).

Using the SPECTRO MIR Scope v1.0 PC software, the sensor was configured for the tests; the RECORDER function enables the recording and export of the measured values in Excel® that was used to prepare the data.

A degreased stainless steel sheet was used as a measurement object, onto the surface of which oil layers of various thicknesses (but of the same type of oil) were applied. The oil layers can range from <1µm and c. 20µm. Oil quantities of various size were tested (in the sectors 1,2,3: medium oil quantity, in the sectors 4,5,6: low oil quantity and in the sectors 7,8,9: large oil quantities). Then the oil was spread homogeneously over a diameter of c. 60 mm using a single-use cloth. This procedure was performed in all the specified sectors. Then the oil quantity already used was spread in sectors 2,3,5,6,8 and 9 with a single-use cloth, without changing the diameter of the respective sector. This procedure was repeated in sectors 3,6 and 9.





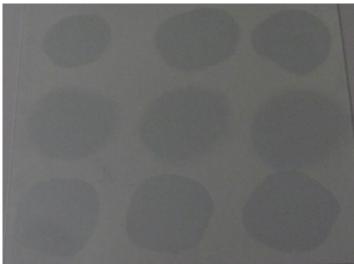


1,2 and 3: medium layer thicknesses (from 1 to 3 descending)

4,5 and 6: low layer thicknesses (from 4 to 6 descending)

7,8 and 9: large layer thicknesses (from 7 to 9 descending)

10,11,12 und 13: Sectors which serve reference measurement (sectors without oil application).



Stainless steel sheet with the 9 various sectors. After the respective tests with a certain oil type, the metal sheet was cleaned from oil almost completely (degreased). Measurements were performed between the individual oil applications (sectors 1-9) to enable us to test whether the oil from previous tests were removed from the metal surface These areas are called sectors 10-13.

As already mentioned, the surface tension test in the respective sectors (determined by the test ink method)



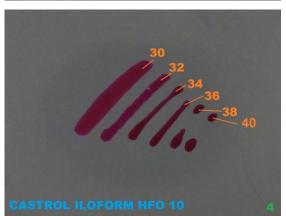
serves as the reference for the respective measurements. This was ascertained for every oil type using the available test inks (30mN/m to 50mN/m). This was performed after the measurement with the three different SI sensor systems, as the test ink applied would influence the optical measurement results.

30 32 34 36 36 CASTROL Hoform HFO 10

Oil sample: CASTROL Iloform HFO 10

Test inks: 30mN/m, 32mN/m, 34mN/m and 36mN/m

Sector: 1

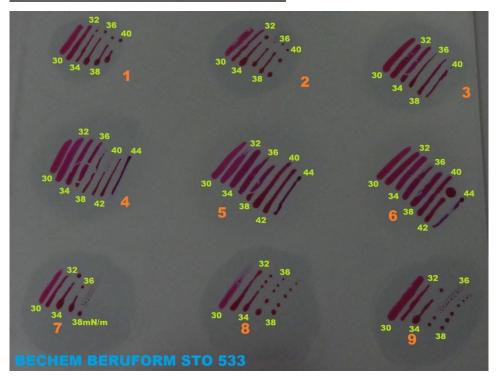


Oil sample: CASTROL lloform HFO 10

Test inks: 30mN/m, 32mN/m, 34mN/m, 36mN/m, 38mN/m

and 40mN/m

Sector: 4



Oil sample: BECHEM BERUFORM STO 533, test inks used: 30mN/m - 44mN/m in the 9 various sectors



5.4 List of the tested oil types

The following oils were available for the tests:

- CASTROL HFO10
- BECHEM STO533
- Z+G KTL N16
- BECHEM KFP95
- BECHEM MF85
- WILKE 628
- WISURA AK3614SF
- RAZIOL CLF11S
- RAZIOL CLF11L
- TRUMPF C462
- TRUMPF B30
- TRUMPF SRH/5
- TRUMPF AKAMIN
- OW 30 NEUTRAL
- OW 30 0.01% GREEN
- OW 30 0.01% UV-COLOUR

Comparison of the surface tension of the various oils with an approximately identical oil layer thickness.



Investigation of the various oils has shown that the surface tension in similar oil layer thickness have different values depending on the respective oil.

We compared the surface tension values in sector 6, the smallest oil quantities applied (layer thickness <1µm). Inferences from the surface tension to the oil layer thickness is not possible without knowledge of the oil.

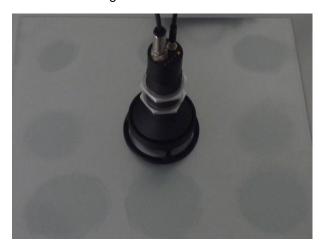


5.5 Oil residue control with the SPECTRO-M-10-MIR/(MIR1+MIR2)

5.5.1 MIR sensor and application

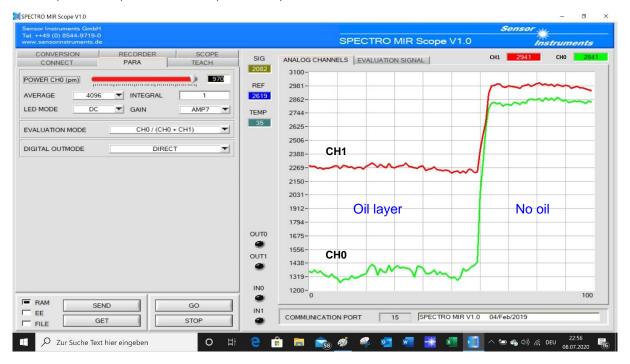
The **SPECTRO-M-10-MIR/(MIR/MIR1)** was used to measure measuring points 1-9 one after another in a working clearance of 10mm, whilst positions 10-13 serve as reference points. The standardized measured value is the result of the two ascertained measured values of the two measuring windows in MIR.





During measurements, a connection to the PC was established using a serial interface. The parametrization of the sensors and the numerical and graphical display of the measurement data is performed via the Windows® software **SPECTRO MIR Scope V1.0**.

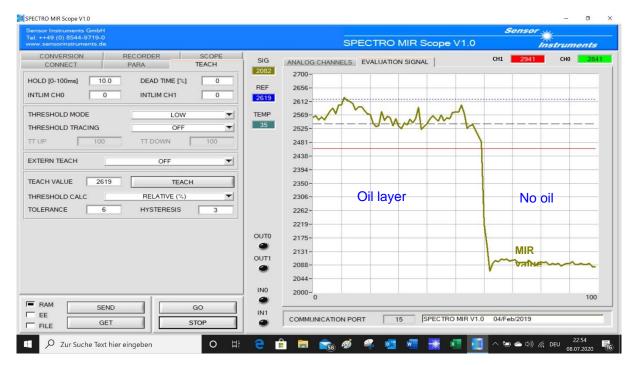
Working in the software via EVALUATION MODE, you can choose how the standard signal is constituted: MIR = CH0/(CH=+CH1) or MIR = CH1/(CH0+CH1)



The optical characteristics of the metal surface are changed by the oil layer; the surface becomes darker, thereby reducing the optical reflection on both channels.

In addition to the display of the two measured values CH1 (reference value with $4\mu m$) and CH0 (measured value at $3\mu m$) the standardized SIG = MIR value = CH1/(CH0+CH1) x 4095 is shown.

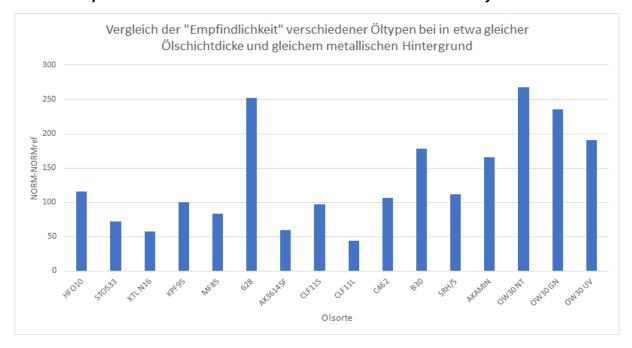




Display of the standardized MIR value = SIG = CH1/(CH0+CH1)

The PC software "MIR monitoring" can be used for inline measurement. The system operator can use it to display the current quality situation and the trend on a PC monitor during production via a column diagram. The measurement data can also be recorded and saved in ASCII format, so as to enable evaluation with a spreadsheet program such as Excel®.

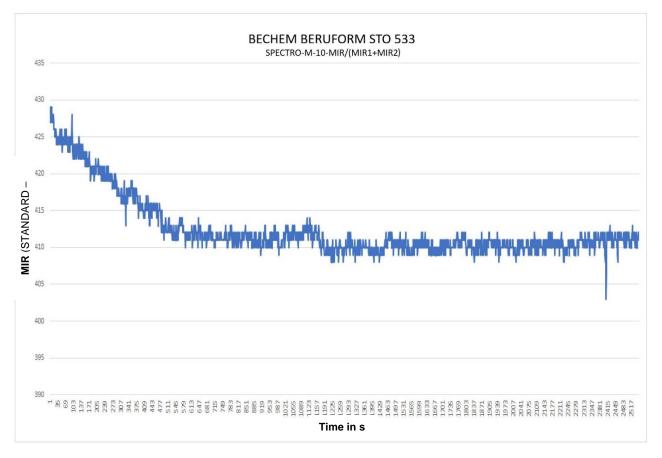
5.5.2 Comparison of the sensitivities of the various oils with the same layer thickness.



The MIR measurement method was also able to ascertain differences in sensitivity between the various oil types (with comparable layer thickness). In comparison to the two UV measurement methods, it was able to detect all oil types and the difference between the most sensitive and non-sensitive samples amount to a difference of only a factor of 7.



5.5.3 Dependence of the MIR measurement signal on time



Some of the oils subject to investigation displayed evaporation characteristics. This means that the signal became weaker over a long period until a certain level was reached. We have conducted test measurements on these oils over a long period of time to investigate this behavior. The sensors were placed on a measuring point to which an oil film of a certain oil type had been applied and measurement data recording was performed over a longer period (c. 43 min).

A 5% decrease of the initial signal in the standardized range was detected for the oil BECHEM BERUFORM STO 533. This corresponds to a factor of 1.05. The observable effect can probably be explained by the evaporation of part of the oil which then resulted in a balance.

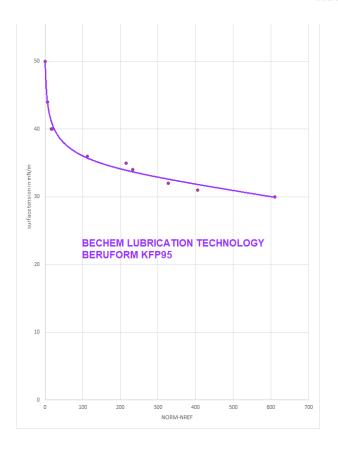
As the stimulus energy in the MIR range is lower than the procedures with UVA and UVC by a factor of 10, the influence of the MIR stimulus on the polymer bonds and thus on the measurement should be significantly lower than the comparably high-energy UV stimulus.

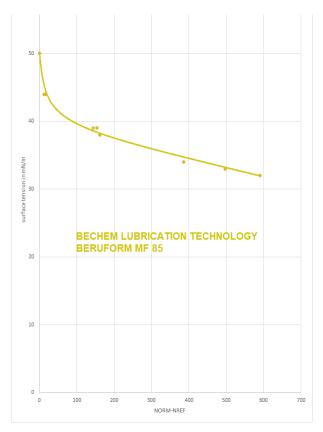
5.5.4 Correlation between the MIR signal and the measured surface tension per oil type

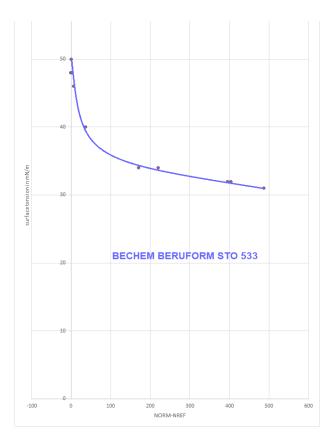
The oil types were measured on positions 1 - 9 (**STANDARD**) after an average reference value was first reached (STANDARDref) on the four reference positions (position 10, 11, 12, 13). The difference between the two values **STANDARD - STANDARDref** serves as the dimension of the surface tension.

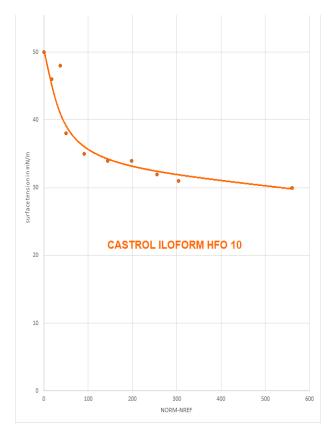
The following diagrams document the measuring results. The MIR values = **STANDARD - STANDARDref** measured by the SPECTRO-M-10-MIR were applied on the ordinate (x axis); the values of the surface tension in **mN/m** were applied to the coordinate (y axis) which were determined with the red test ink method.



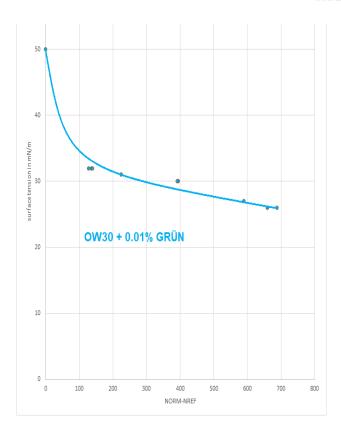


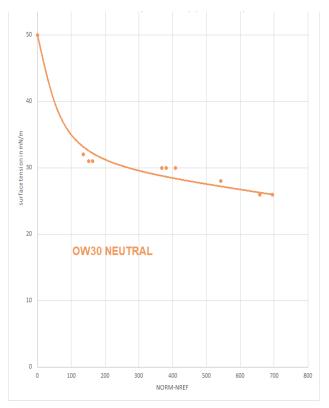


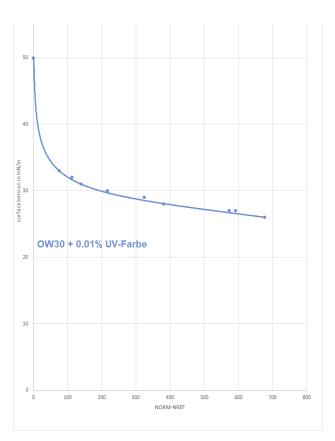


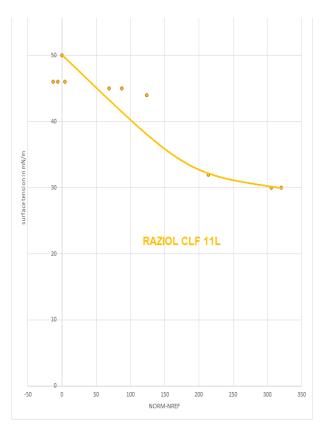




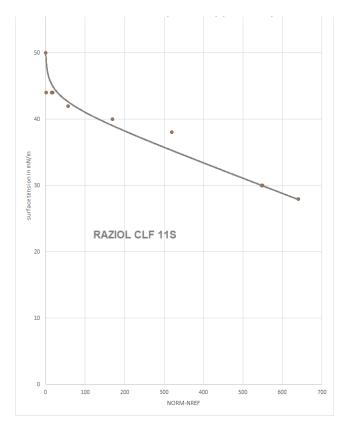


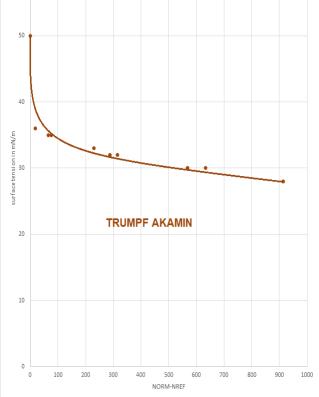


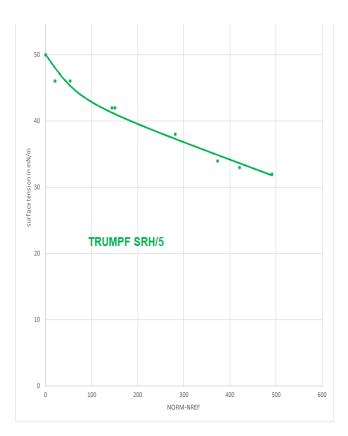


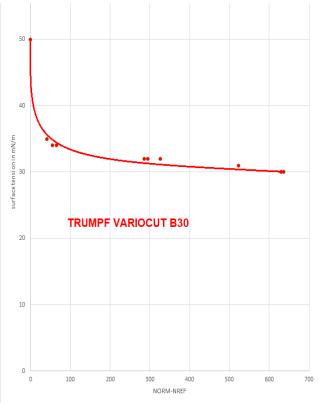




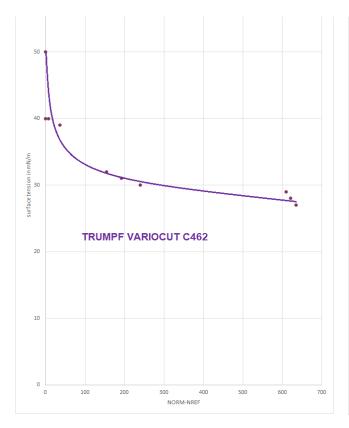


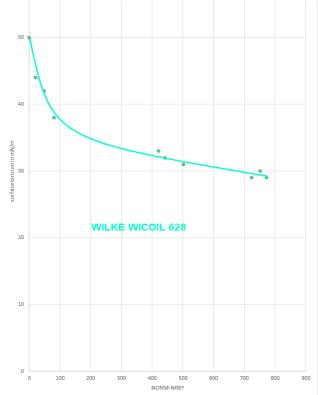


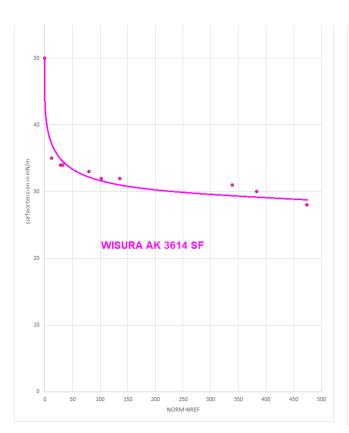


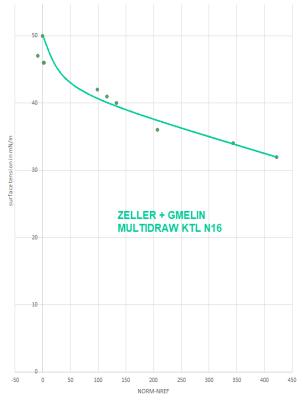






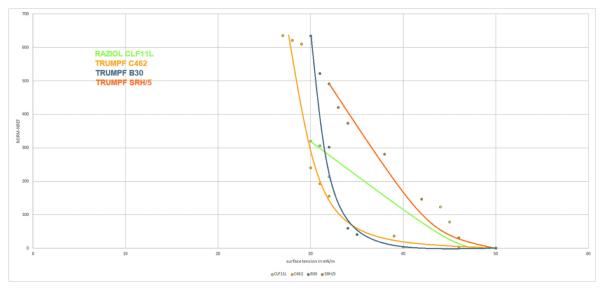


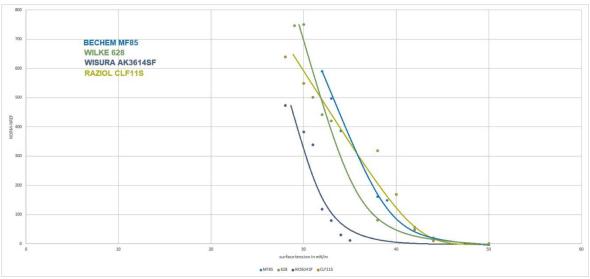


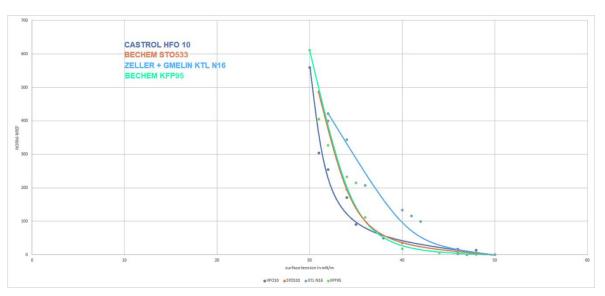




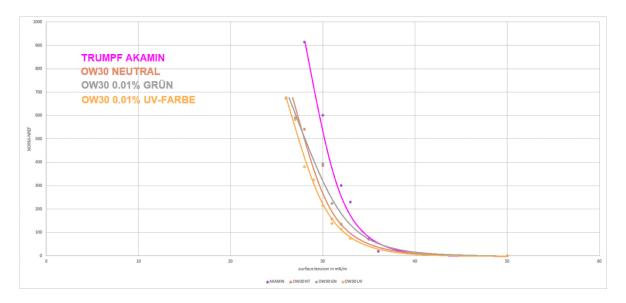
5.5.5 Comparison of the MIR measured values in dependence on the surface tension











The gradients show that with MIR measurement method, background calibration (standardref) to the basal metal surface is also to be recommended. Selected oil layers should be measured using both methods and the values should be entered in the CONVERSION tool of the SPECTRO MIR Scope software to enable calibration of the reference measurement procedure.

The user can perform both calibrations using the Windows® software **SPECTRO MIR Scope V1.0**. The value of the surface tension can also be displayed in addition to the respective STANDARD value.

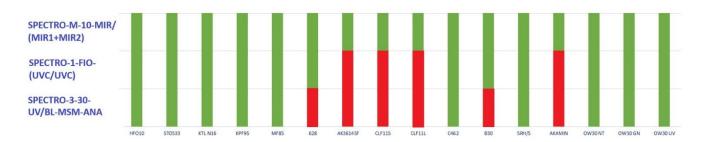
5.5.6 Comparison of the MIR method with the UVA and UVC sensors

Parallel to the measurements with the MIR system, the same oils were tested with UV sensors (UVA und UVC) from Sensor Instruments.

The SPECTRO-1-FIO-(UVC/UVC) is a fiber optic sensor with UVC stimulus (265nm) which records the reflection/absorption in UVC, whilst the SPECTRO-3-30-UV/BL-MSM-ANA is a fluorescence sensor with UVA stimulus (high-performance LED, 365nm) which evaluates emissions in the visible range (type 450nm – 700nm).

The characteristics of the tested oil types mean that the two UV sensors cannot register all oils, as some showed little or no analyzable UVA fluorescence or UVC absorption. The following table compares the MIR measurement procedures with the two UV measurement procedures in terms of their suitability for measuring the tested oil types.

The oils suited to the respective measurement procedures (green: suitable, red: unsuitable):





Further important characteristics of the measurement procedures used in comparison:

Messverfahren	UVC	UVA	MIR
Empfindlichkeitsunterschiedsfaktor der verschiedenen Ölsorten	22	61	7
Signalabschwächungsfaktor bei Langzeitbelichtung	2	3	1.05
Fremdlichempfindlichkeit gegenüber Kunstlicht (LED - Weißlicht)	mittel	stark	gering
Messabstand in mm	5	15 (11)*	10
Detektionsbereich in mm	5	12 (1)*	10

^{*} für das UVA - Messverfahren gibt es auch eine Lichtleiterversion, womit entsprechend kleine Detektionsbereiche erreicht werden können.

In comparison to the UV measurement procedures with fiber optics (small measurement area) the SPECTRO-M-10-MIR/(MIR1+MIR2) requires a larger, plane test surface. When tested for all other criteria, this procedure performs better than the UV procedure. One of the important advantages of the MIR measurement procedure is the standardized evaluation. These cannot replace the individual calibration per oil type and metal surface, but variations of the metal surface and any intensity drift of the light source used can be compensated for the most part. No influence from artificial light (white-light LEDs) for room illumination (ambient light) on the measuring result can be observed.

5.5.7 Summary of the oil residue control

In practice, the presence of oil films on metal surfaces is very often checked with offline procedures such as test inks using random sampling. This is a contact method, as test ink must be applied to the respective surface. This test ink method for determining the surface tension is also dependent on the visual approach of the observer (droplet formation yes/no and in which time) and the fact that the only relatively coarse graduation of test inks in relation to the surface tension (in 2mN/m steps) makes an exact analysis of little value. This method requires a certain size of test surface (width of the brush stroke x c. 15mm in length). Measurement procedures 1 and 2 permit very small measurement surfaces, but not all oils react to the respective physical effect, in effect remaining neutral.

The SPECTRO-M series provides inline sensors which following reference balancing (with a cleaned metal surface) can detect very thin oil applications (< 1µm) contact-free on metal surfaces.

The CONVERSION function of the sensor enables the standardized MIR values of the sensor to be assigned reference values from other procedures for the measurement of the surface tension. After the reference system has been calibrated, the sensor calculates the measured values automatically into the values of the reference system before they are displayed or outputted. The measured values can be outputted to a PLC via a data interface or an analog output (0...10V, or 4...20mA).

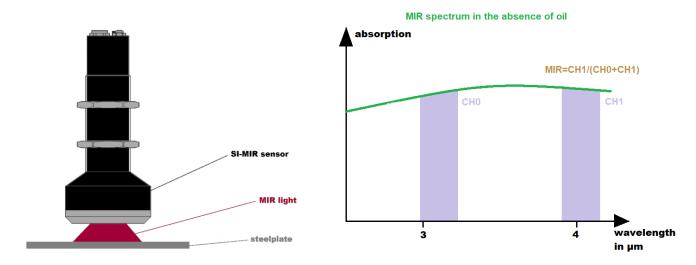


5.6 Inline control of the thickness of the oil layer

Those wishing to measure the layer thickness of a paper coating e.g. of a print color, can adopt the grammage method as an adequate approach. The grammage of a paper coating is not all that different from that of the paper with a thickness of between 0.05 mm and 0.2 mm,. Correspondingly precise scales would probably produce a reliable result. What happens when we replace printing ink with oil and paper with a 1 mm steel sheet? We would probably come to the limits of the grammage method.

We still need a reliable, low-outlay and contact-free method of determining the thicknesses of oil layers. One such method is the fluorescence method. UVA light is used to stimulate the fluorescence. Secondary emissions are emitted in the visible wavelength range. The intensity of the fluorescence signal establishes a measure for the thickness of the respective oil layer. There are also oils with a fluorescence effect which is almost impossible to measure or is missing entirely, and for which a layer thicknesses measurement cannot be performed in this way. The fluorescence signal is not very strong with very thin layers.

If we consider the mid-wavelength infrared light range (MIR) we see that the organic coatings, e.g. an oil layer, display significant absorption at CH0, whilst no absorption occurs in the reference wavelength window (CH1). The MIR signal of the SPECTRO-M-10-MIR/(MIR1+MIR2) displays a (upon first inspection) directly proportional relationship between thickness of the oil layer and standardized signal.

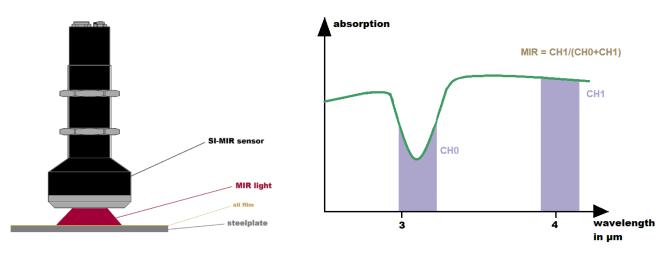


The SPECTRO-M-10-MIR/(MIR1+MIR2) sensor is fitted with two wavelength windows for evaluating the reflected signal. Without an oil layer, the absorption in both wavelength windows is comparable. This value can be used as a reference value for the unoiled metal surface: MIRref = CH1/(CH0+CH1) corresponds to the standardized reference value of the metal surface without oil application and CH0 and CH1 are the determined raw signals from the two wavelength windows.

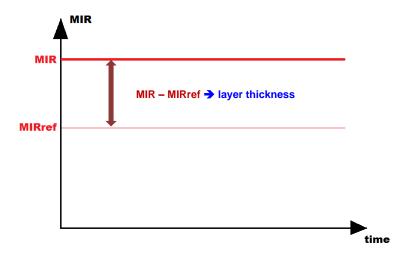
Covering the steel surface with a homogeneous oil layer effects the following increase of MIR signal in the MIR spectrum:



MIR spectrum in the presence of oil



The left-hand measuring channel CHO detects the extra absorption caused by the oil layer, whilst the right-hand measuring window CH1 remains largely unaffected by this.



The measured value MIR=CH1/(CH0+CH1) moves upwards in the CH0 window through the additional absorption. The more intensive the absorption, i.e. the thicker e.g. the oil layer, the further removed is the MIR value from the reference value MIRref (without oil layer).

Investigations of various oils have shown that the MIR absorption is dependent on both the thickness of the oil layer and the oil type. The reflective metal background also influences the measuring result a little; this requires

a calibration for oil type and background before measuring the actual oil layer thickness.

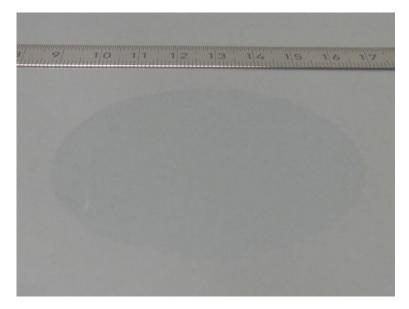
<u>Procedure:</u> Oil droplets with a volume of 20µl were applied to a degreased metal surface of the same type in order. The oil droplets were then spread homogeneously across a 70 mm diameter surface using a spatula. It is now possible to determine the layer thickness: an oil volume of 20μ l across a diameter of 70mm produces a layer thickness of c. 5μ m, with two droplets (40μ l) correspond to 10μ m, with 3 droplets (60μ l) 15μ m etc.

Once samples with the respective layer thickness have been prepared, you can start with the calibration procedure. First, the averaged background value MIRref (on oil-free sections) was determined between the oil samples. After calibration,



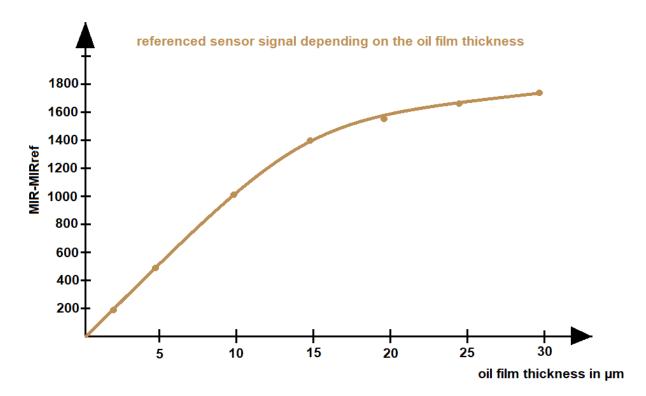
the **SPECTRO-M-10-MIR/(MIR1+MIR2)** sensor of the series was positioned on the various test surfaces and the measured values were recorded for the individual layer thickness.







Upon completion of the process we have a table of values shown here as a diagram:



The diagram shows that the resolution of the measurement procedure lies at c. 10nm (5000nm/500 digits) and the measuring accuracy moves in the range of c. 50nm. The MIR signal runs almost linear to the layer thickness up to one layer thickness. The upper threshold of the MIR layer thicknesses measurement for the measured oil lies at $c.15\mu m$.

To perform inline measurement, all you need to do is to remove the spacer, then you can start! The sensors are fitted with digital and analog outputs and in future can optionally also be fitted with a Feldbus. The system can be parametrized and monitored easily via the Windows® MIR Scope V1.0 software. The clearance from the sensor to the surface of the object typically amounts to 10mm, the detection range has an approx. 10mm diameter. Fixture covers are used for small measurement objects which restrict the measurement range accordingly. As the measurement system usually works in DC mode, measurement cycles of up to 10 kHz can be achieved.



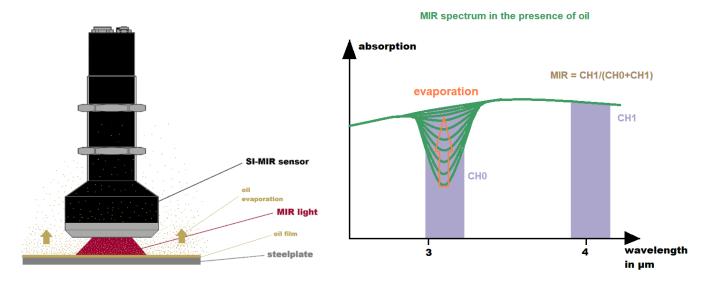
5.7 Measuring the evaporation of oil applications

The production of blanked and flexible parts is increasingly using punching and drawing oils which are supposed to evaporate after a certain time. Such processes need an outcome which minimizes the oil residue on the blanked or formed metal parts in order to obviate the need to subject the products to a cleaning process during further processing. The question is: how much of the applied oil remains on the component and how long does the evaporation process take?

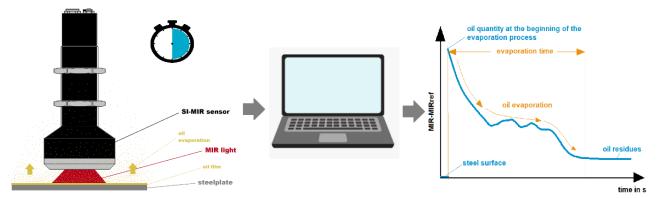
To measure the evaporation we applied five drops of oil (5x20µl) to a degreased steel sheet and spread it over a 70mm diameter surface.

The oil layer thickness at the start of the measuring procedure amounted to some 25µm. The **SPECTRO-M-10-MIR/(MIR1+MIR2)** sensor was then placed on the center of the oil and the measurement was started.

The MIR spectrum immediately after application of the 25µm thick oil layer. The wavelength range CH0 is influenced by the presence of oil, whilst no signal reduction can be observed in the wavelength range CH1.



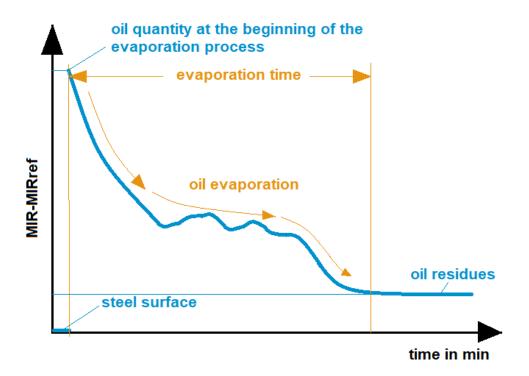
MIR signal in time lapse: the signal reduction in the CHO caused by the oil declines with increasing oil evaporation.



The standardized MIR value minus the standardized MIRref reference signal (MIR: MIRref) is recorded over a period of a number of hours using the Windows® MIR scope V1.0 software:

A similar progression was presented as the following diagram by all the oils tested:





The duration of evaporation lay between 60 - 90 minutes. The oil residue of the sample which evaporated the strongest lay at 1% of the original layer thickness and at 50% of the original layer thickness for the sample that evaporated the most weakly.

The measurement procedure enables quick and uncomplicated testing of the evaporation duration of oils working on the basis of defined layer thicknesses. The residual signal that remains measurable after the MIR signal has stabilized has organic remains on the surface which remain after the evaporation process.

The recorder function of the SPECTRO MIR scope software of the SPECTRO-M sensor permits the continual measurement and recording of the measured values as a ASCII file, which can then be exported for evaluation in a spreadsheet program.



6 Test measurements on the CLF 11 series

A separate series of tests investigated punching oils from the CLF 11 series from Raziol Zibulla & Sohn GmbH. The results of the test series are summarized on the following pages.

6.1 Test methodology and reference method

The "red ink" method was used as a reference method with test inks from arcotest (www.arcotest.de).

The oils to be investigated were applied to a de-greased metal sheet (e.g. GARDOBOND from Chemetall) and the test ink with a certain surface tension (specifications on the bottles, e.g. 38mN/m) is then applied to the oil surface using a brush included in the bottle.

If the test ink rolls off, continue with the next lowest surface tension value until the test ink covers the oil surface. The surface tension value specified on the test ink bottles corresponds approximately to the surface tension value of the metal surface inc. the oil layer.

The thinner the oil layer, the higher the surface tension of the oil sorts used.

One test series required that the oil used was applied to the degreased metal surface as thinly as possible and was then rubbed intensively onto the metal surface using a non-fuzzing cloth. The oil was distributed homogeneously over a c. 70 mm diameter.





This usually achieves oil layers with a thickness of between 0.1µm and 3µm, depending on the method of distribution: 1x rubbing, 2x rubbing, 3x rubbing with an oil volume of 20µl (1 drop), 40µl (2 drops), 60µl 3 drops. The oil is applied with a pipette, then the oil is rubbed depending on the requisite layer thickness (for layer thicknesses between 0.1µm and 3µm) as described, painted on (for layers from 1µm to 10µm) or allowed to run (for layers between 5µm and 50µm). The last two procedures distribute the oil over a diameter of c. 70 mm. A degreased steel sheet and (depending on the procedure) a new non-fuzzing cloth and a fresh plastic spatula are required for every measurement. A degreased GARDOBOND test sheet from Chemetall is used for every sample. The layer thickness is ascertained using the diameter and the oil volume (c. 20µl per droplet). The calculation is an initial estimate, as neither the diameter not the quantity of oil can be determined exactly. The measurement samples are evaporating punching and drawing oils, which means that a certain loss begins from the start of application. Despite the use of the plastic spatula during application, it is not possible to prevent some of the residual oil adhering to the spatula and not the steel surface. The running of the oil means that it was necessary to accept higher evaporation quantities, as the running procedure took more time than brushing. When rubbing the oil with a non-fuzzing cloth, the largest part of the oil remained on the cloth. This means that calculation of the layer thickness is no longer possible.







We used degreased steel sheets and aluminum sheets.

When ascertained with the test ink method, the surface tension of the oil on an unused GARDOBOND® test surface should lie beyond 50mN/m, as no ink could be observed rolling from the steel surface even when using the highest ranked test ink (50mN/m).





6.2 Manufacturers specifications to the CLF 11 series

Raziol Zibulla & Sohn GmbH specify that the CLF 11 series is quickly-evaporating punching and drawing oils:

"all Raziol CLF 11 ... W products contain additional temporary corrosion protection for indoor storage. The specified products evaporate in a relatively short time, depending on the additive content. The additives remain on the surface of the workpiece."



Raziol Zibulla & Sohn GmbH recommend the use of the CLF 11 series in the following applications (excerpt from: www.raziol.com)

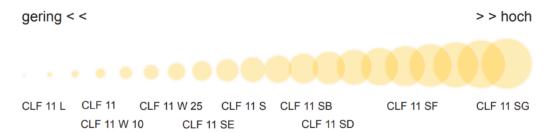
Anwendung der Raziol CLF 11 Reihe

- z.B bei der Herstellung von Elektrokontakten, von Stanz- und Biegeteilen, Federn, Drähten, Uhren, Metallwaren, von Dichtungselementen sowie von weißer Ware und Elementen der Kommunikationstechnik
- für Fertigungsteile geeignet auf denen möglichst wenig Rückstand verbleiben soll
- oftmals können Nachfolgeprozesse wie Kleben und Etikettieren ohne Waschen erfolgen
- Abdeckung eines breiten Leistungsspektrums durch die Zugabe unterschiedlicher leistungssteigernder Zusätze
- in Verbindung mit den Raziol Auftragssystemen wie Rollenbandölern oder Sprühsystemen ist problemlos ein sparsames, definiertes Beölen mit den Produkten der Raziol CLF 11 Reihe möglich



Read the following about the residue behavior (www.raziol.com):

Rückstandsverhalten



The following oils were tested

- CLF 11 L
- CLF 11
- CLF 11 W 10
- CLF 11 W 25
- CLF 11 SE
- CLF 11 S
- CLF 11 SB
- CLF 11 SD
- CLF 11 SB
- CLF 11 SD
- CLF 11 SF
- CLF 11 SG

The investigation criteria were:

- Evaporation time of the volatile components of the oil.
- Correlation between the thickness of the oil layer and the measured standardized signal.
- Standardized sensor signal in dependence on the surface tension (reference system).



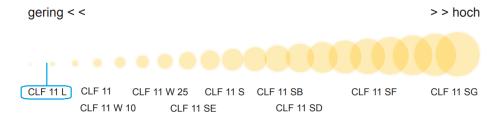
6.3 Raziol CLF 11 L

6.3.1 Evaporation time of the volatile components of the oil.

To this end, five drops of oil (c. 100Ml) were applied to the degreased steel surface and then the oil quantity was allowed to run across a diameter of c. 70 mm. This procedure lasted c. 5 minutes. Irrespective of any deviation from the 100µl oil volume, the deviation from the 70mm diameter and the oil quantity which has volatized within the five minutes of application, calculations come to an oil thickness of c. 25µm. The evaporation time Tv calculated by the sensors, i.e. the time in which the volatile components have left the oil, lay at 72 minutes. The oil residue quantity, i.e. the quantity of oil remaining on the steel surface after elapse of this time lay at 1% of the originally applied oil quantity:

Evaporation time: 72 minOil residue quantity: 1%

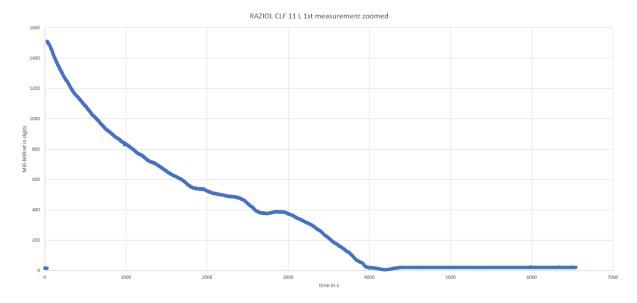
CLF 11 L





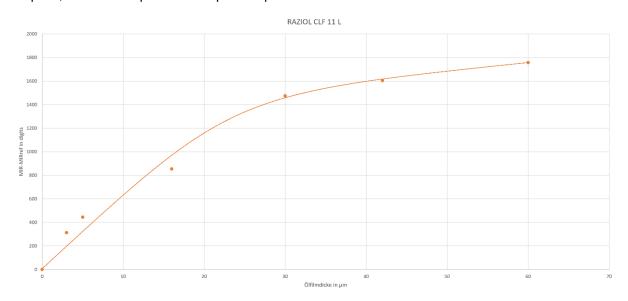


Schichtdicke	MIR	MIR-MIRref	ROTE TINTE	Anzahl	Ölfleck	Schichtdicke	Ölmenge
	NORM	deltaNORM		Tropfen	Durchmesser		
in µm	in digits	in digits	in mN/m		in mm	in µm	in μl
3	2518	312		0,5	70	3	10
5	2650	444		1	75	5	20
16	3060	854		3	70	16	60
30	3680	1474		5	65	30	100
42	3810	1604		7	65	42	140
60	3963	1757		10	65	60	200
0	2206	0		0		0	0



6.3.2 Correlation between the thickness of the oil layer and the measured standardized signal.

Oil droplets were applied to a degreased steel surface and then allowed to run off. With a low number of droplets, the oil was spread with a plastic spatula across a surface with a diameter of c. 70 mm.

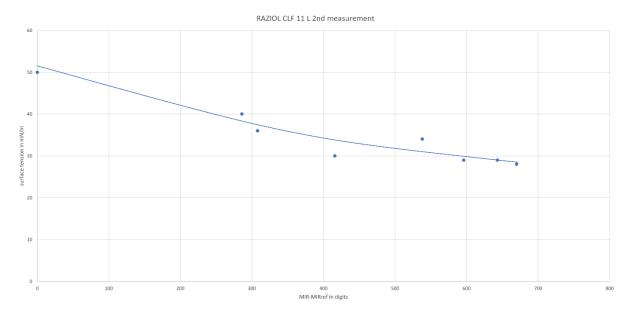


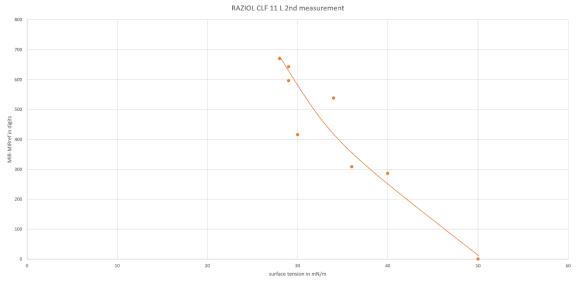


6.3.3 Standardized sensor signal in dependence on the surface tension

A certain number of oil droplets were applied to a degreased steel surface and then rubbed in a different number of times using a non-fuzzing cloth. Measurement using the SPECTRO-M sensor was followed by the ascertainment of the surface tension using the red test ink.

ROTE TINTE	MIR	MIR-MIRref	ROTE TINTE	
in mN/m	NORM	deltaNORM	in mN/m	
	in digits	in digits		
29	2637	643	29	
28	2664	670	28	
29	2590	596	29	
30	2410	416	30	
36	2302	308	36	
40	2280	286	40	
34	2532	538	34	
50	1994	0	50	



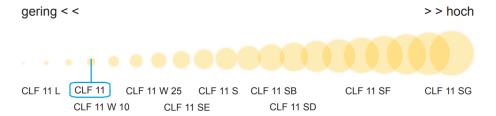




6.4 Raziol CLF 11

CLF 11

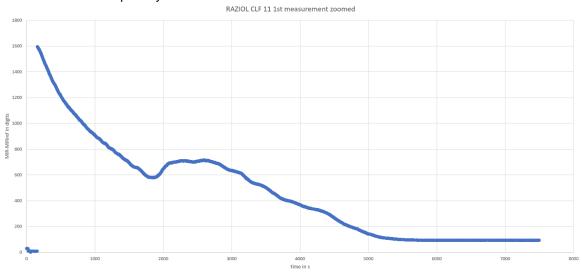
Rückstandsverhalten



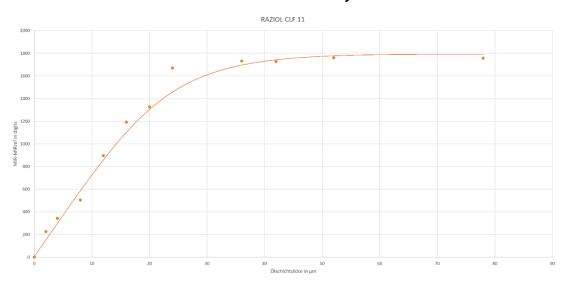
6.4.1 Evaporation time of the volatile components of the oil

Evaporation time: c. 90 min

• Oil residue quantity: c. 6%



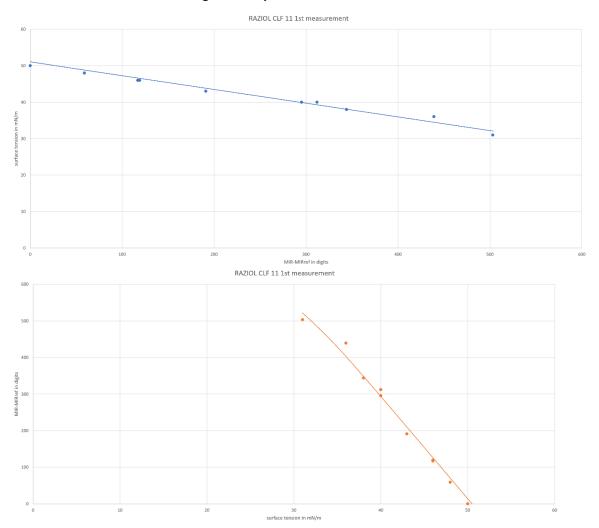
6.4.2 Correlation between the thickness of the oil layer and the measured standardized signal



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6.4.3 Standardized sensor signal in dependence on the surface tension

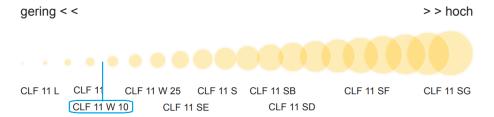




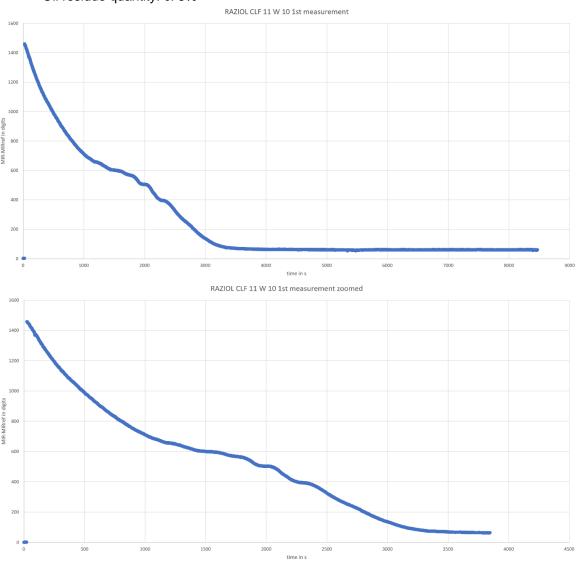
6.5 Raziol CLF 11 W 10

6.5.1 Evaporation time of the volatile components of the oil

CLF 11 W 10

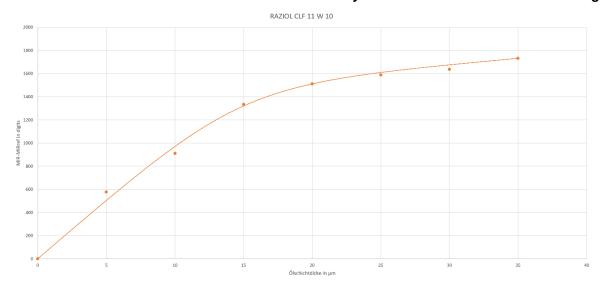


- Evaporation time: c. 62 min
- Oil residue quantity: c. 5%

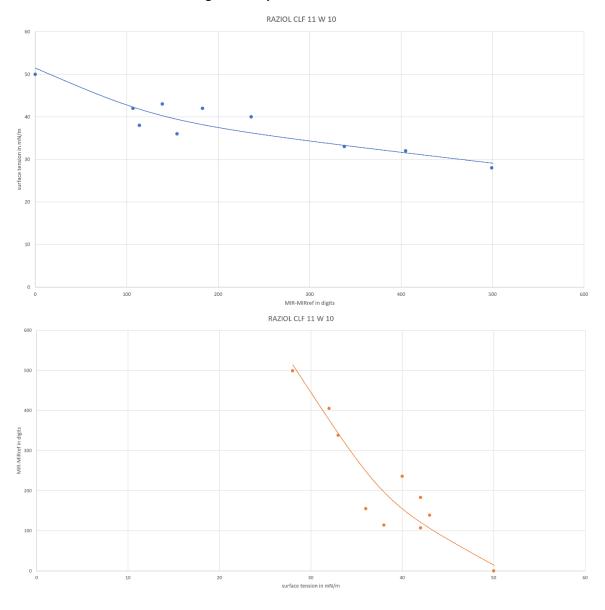




6.5.2 Correlation between the thickness of the oil layer and the measured standardized signal



6.5.3 Standardized sensor signal in dependence on the surface tension



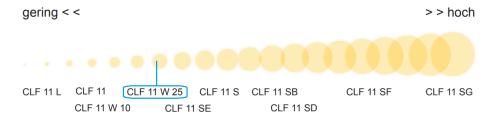


6.6 Raziol CLF 11 W 25

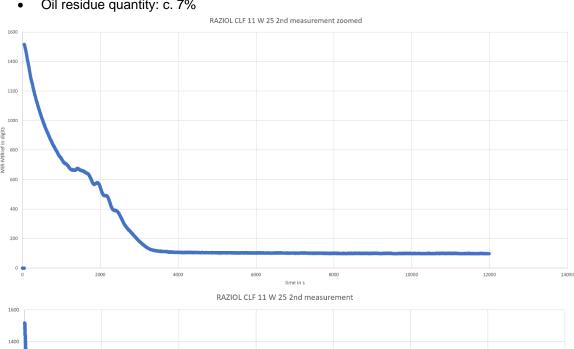
6.6.1 Evaporation time of the volatile components of the oil

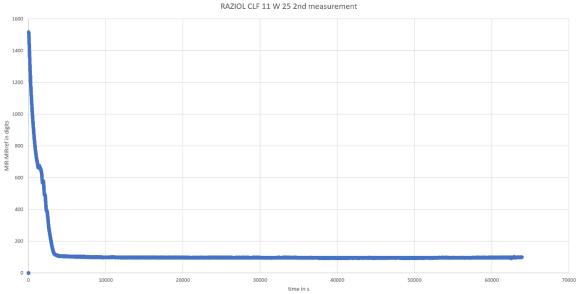
CLF 11 W 25

Rückstandsverhalten



- Evaporation time: c. 58 min
- Oil residue quantity: c. 7%

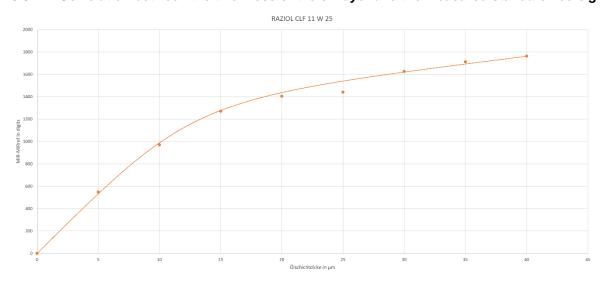




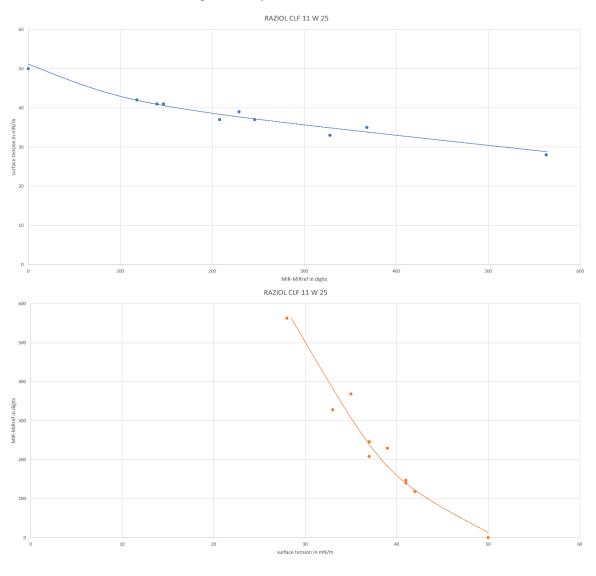
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6.6.2 Correlation between the thickness of the oil layer and the measured standardized signal



6.6.3 Standardized sensor signal in dependence on the surface tension



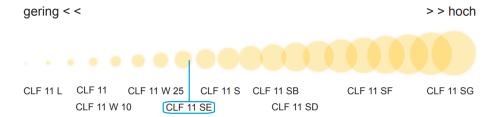


6.7 Raziol CLF 11 SE

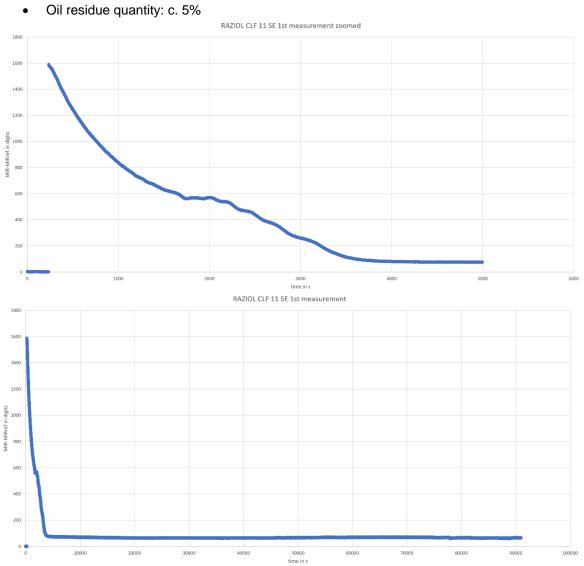
6.7.1 Evaporation time of the volatile components of the oil

CLF 11 SE

Rückstandsverhalten

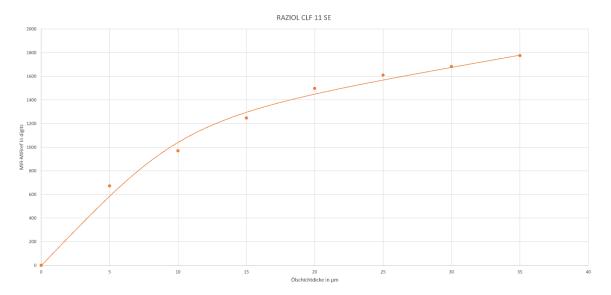


Evaporation time: c. 60 min

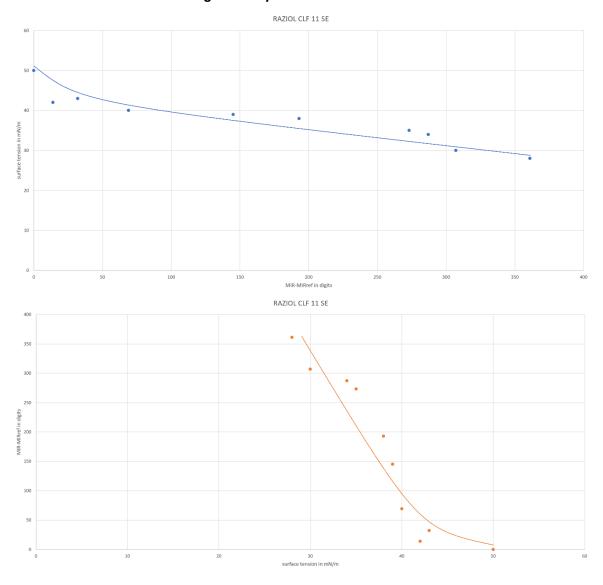




6.7.2 Correlation between the thickness of the oil layer and the measured standardized signal



6.7.3 Standardized sensor signal in dependence on the surface tension



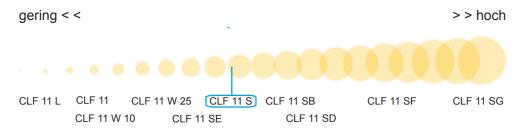


6.8 Raziol CLF 11 S

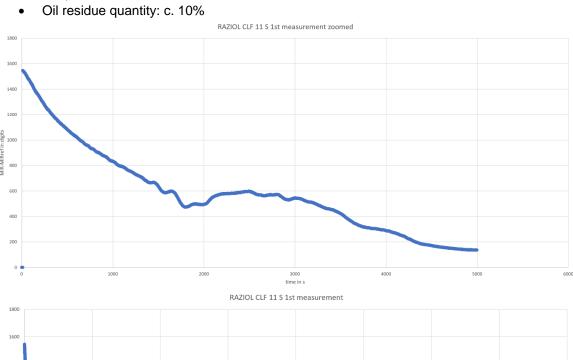
6.8.1 Evaporation time of the volatile components of the oil

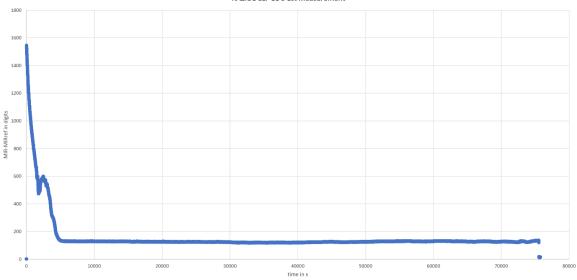
CLF 11 S

Rückstandsverhalten



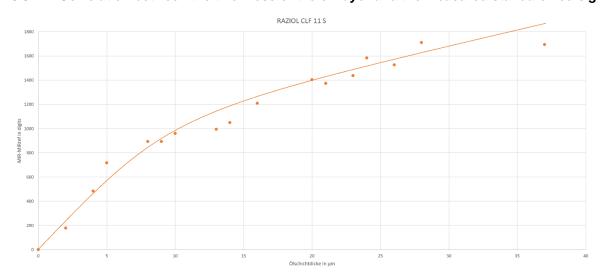
Evaporation time: c. 83 min



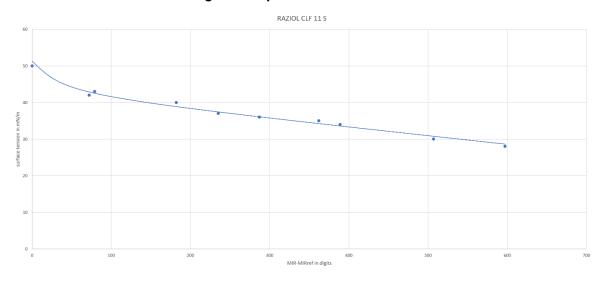


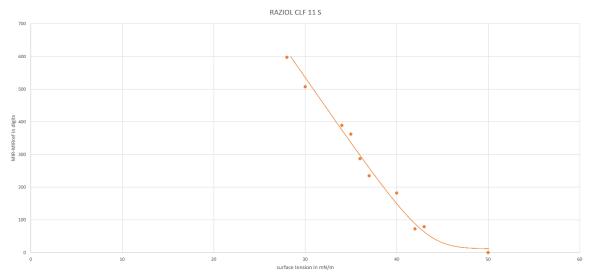


6.8.2 Correlation between the thickness of the oil layer and the measured standardized signal



6.8.3 Standardized sensor signal in dependence on the surface tension



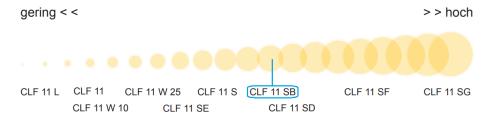




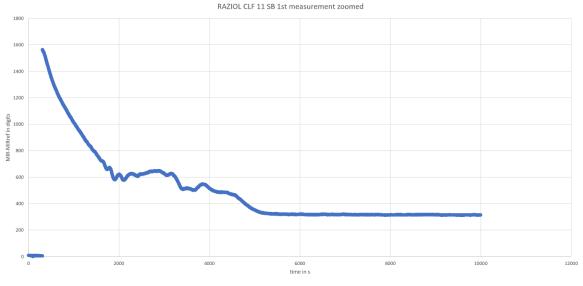
6.9 Raziol CLF 11 SB

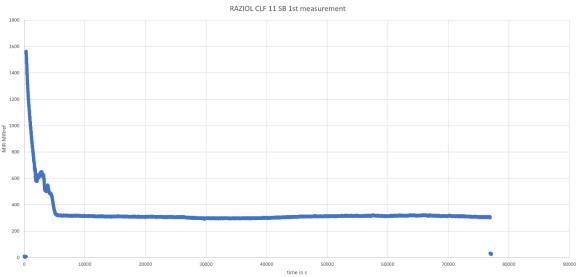
6.9.1 Evaporation time of the volatile components of the oil

CLF 11 SB



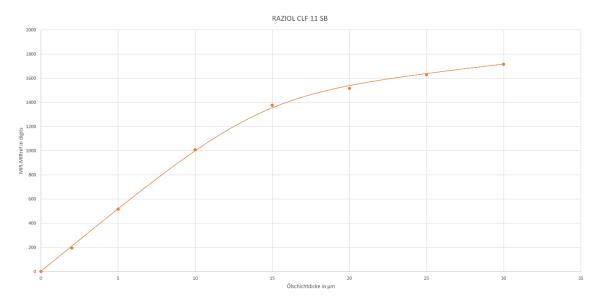
- Evaporation time: c. 72 min
- Oil residue quantity: c. 21%



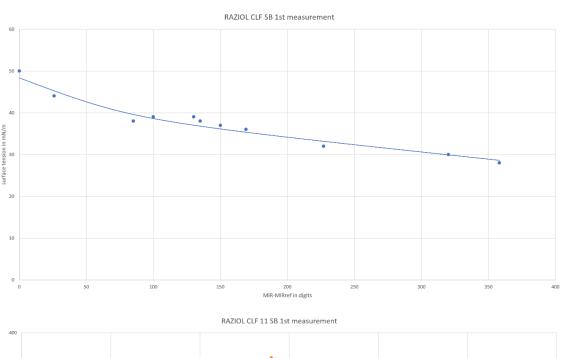


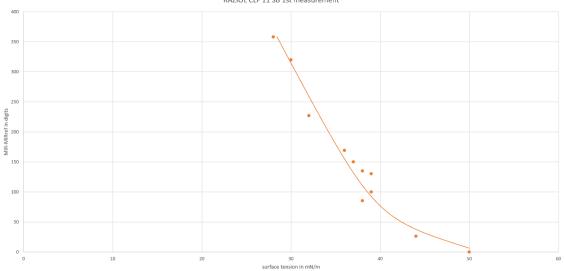


6.9.2 Correlation between the thickness of the oil layer and the measured standardized signal



6.9.3 Standardized sensor signal in dependence on the surface tension



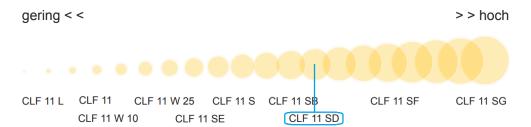




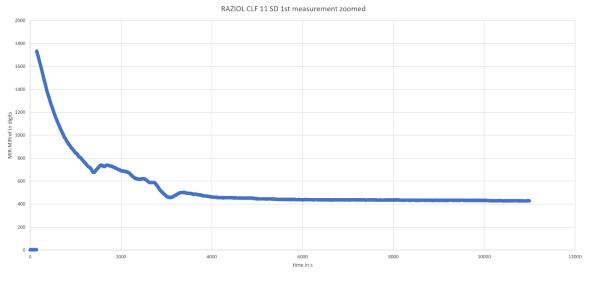
6.10 Raziol CLF 11 SD

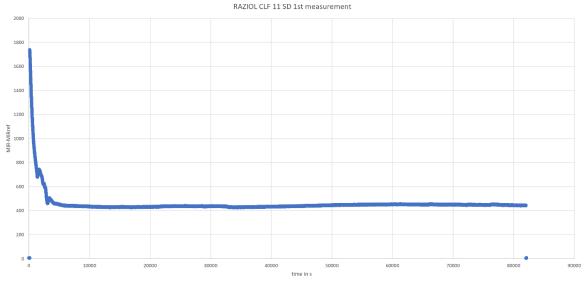
6.10.1 Evaporation time of the volatile components of the oil

CLF 11 SD



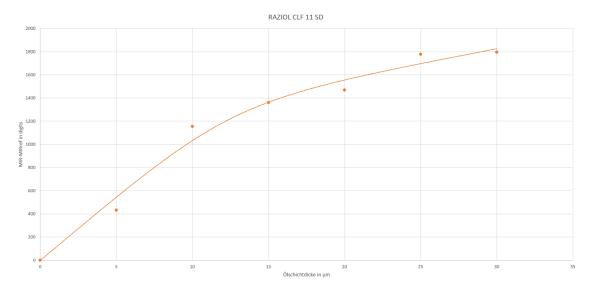
- Evaporation time: c. 76 min
- Oil residue quantity: c. 24%



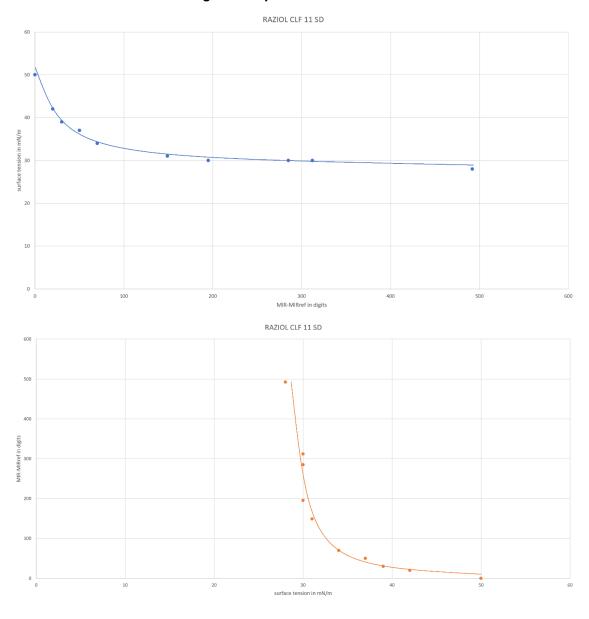




6.10.2 Correlation between the thickness of the oil layer and the measured standardized signal



6.10.3 Standardized sensor signal in dependence on the surface tension

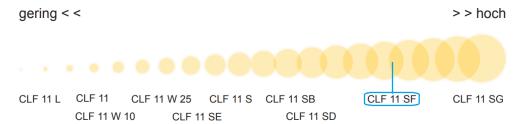




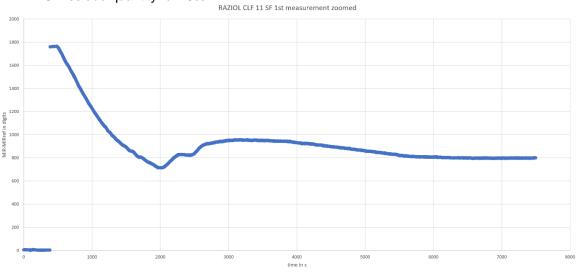
6.11 Raziol CLF 11 SF

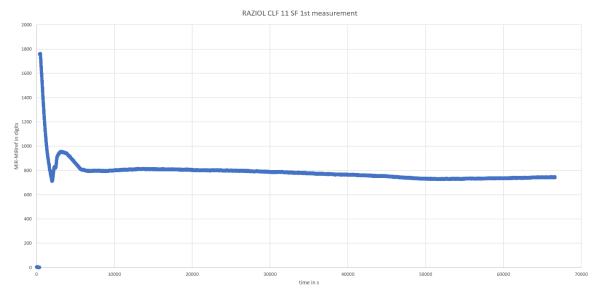
6.11.1 Evaporation time of the volatile components of the oil

CLF 11 SF



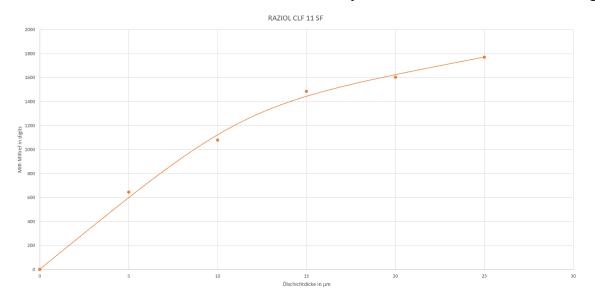
- Evaporation time: c. 83 min
- Oil residue quantity: c. 45%



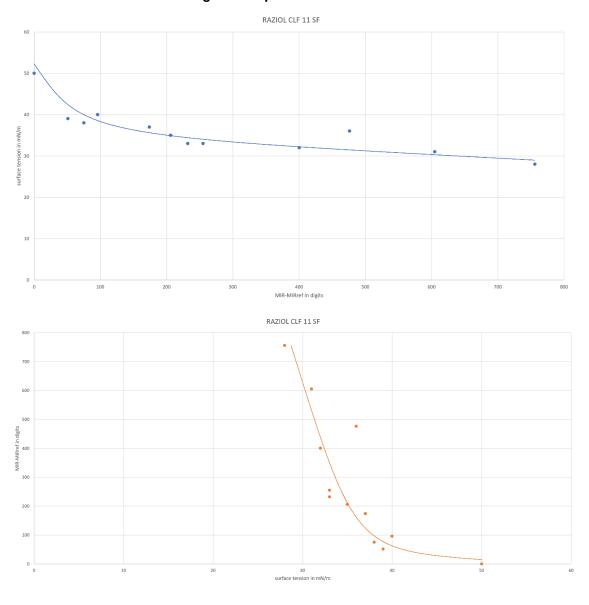




6.11.2 Correlation between the thickness of the oil layer and the measured standardized signal



6.11.3 Standardized sensor signal in dependence on the surface tension



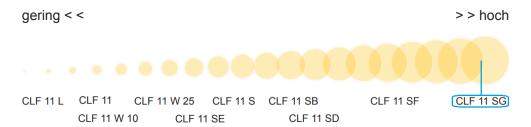


6.12 Raziol CLF 11 SG

6.12.1 Evaporation time of the volatile components of the oil

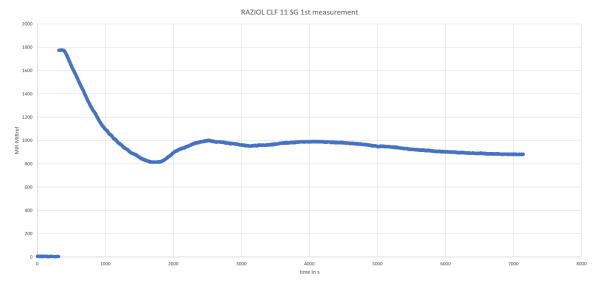
CLF 11 SG

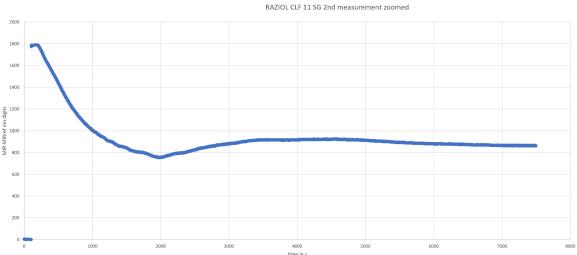
Rückstandsverhalten



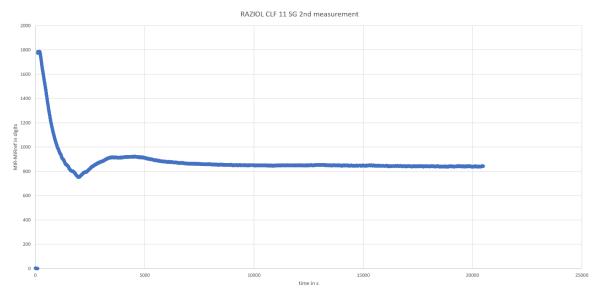
Evaporation time: c. 100 min

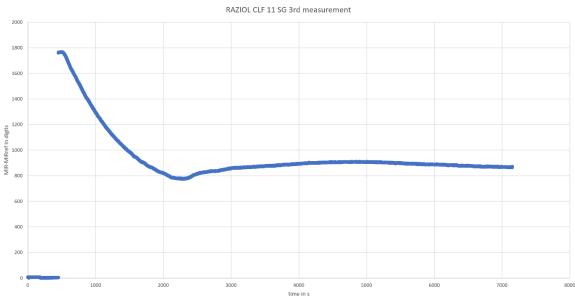
• Oil residue quantity: c. 48%











6.12.2 Standardized sensor signal in dependence on the surface tension

