Press information from Sensor Instruments

January 2021

The detection of oil layers on metal surfaces: comparing the measurement procedures

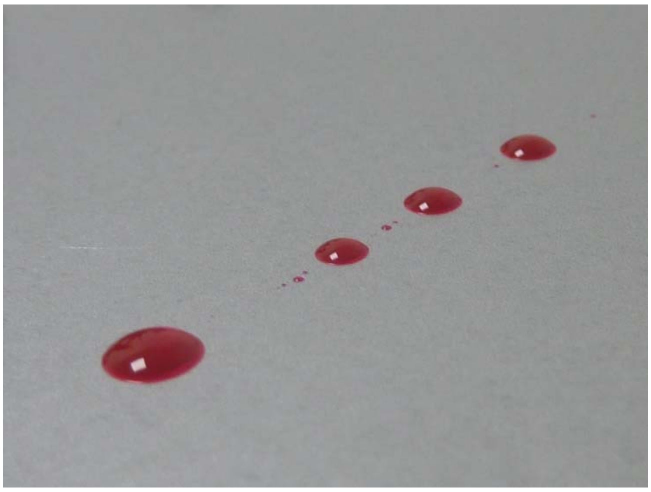
21/01/2021. Sensor Instruments GmbH: The use of oils is imperative in the forming process of metals. For example, the application of cutting oils to metal bands ensures low wear on the punching tools. Drilling oils make an essential contribution to the protection of the drilling and milling tools during 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. A number of measurement procedures are available to this end and which constitute the focus here. The cleaning process can be monitored using the same sensors. The challenge is to determine even small quantities of oil residue accurately, preferably INLINE. 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 this restricts the conductive efficiency to a considerable degree.

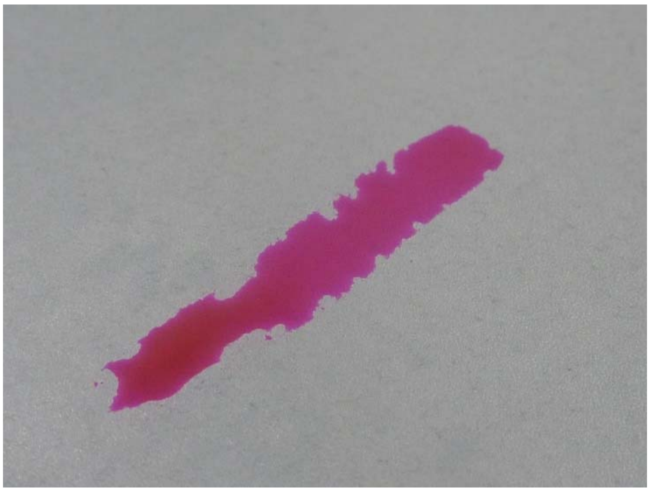
How have we performed monitoring up to now?

When measuring oil layers, we usually first think of a certain layer thickness, for example measured in µm. A common method is 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 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 components in their unoiled state.

The lack of simple methods for measuring the layer thickness directly could explain the increasing popularity of the method for determining the surface tension of parts. The presence of a thin oil layer on a metal surface reduces the surface tension from more than 50mN/m in non-covered state to under 40mN/m in an oiled state (depending on the layer thickness and the oil type). Test inks covering a range from 30mN/m to 50mN/m in two-interval 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 lower 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. It can be observed that reduction in the surface tension is associated with increase in 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.



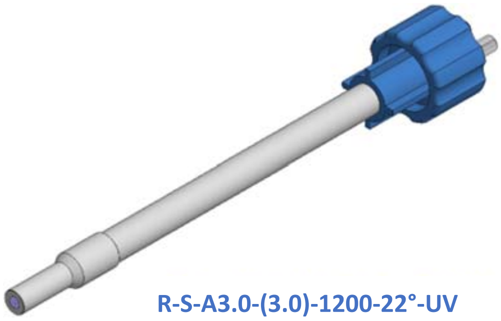
In this diagram, the test ink adheres to the oil-covered or degreased metal surface. The surface tension of the metal surface is higher than the value specified for the test ink.

The three measurement procedures

Measurement procedure 1:
Absorption of UVC radiation (265nm central wavelength range) by the oil layer

A fiber optic sensor (**SPECTRO-1-FIO-UVC/UVC**) is used in reflected light operation. A UV-LED with a central wavelength range of 265nm serves as a light source. A quartz optical fiber **(R-S-A3.0-(3.0)-1200-22°-UV**) directs the UVC light to the section of the metal surface to be measured, which reflects it, partially diffusely and partially directly. A part of the reflected radiation is directed towards the detector integrated in the sensor using the reflex fiber optic bundle. A degreased metal surface to which no oil has been applied (should be the same material and present an identical surface structure as with the subsequent measurements) serves as a reference. If an oil layer is between the surface of the face of the optical fiber and the metal surface, it will absorb a part of the UVC light during both incidence and return. A signal decrease can be measured on the detector side. If the sensors are used for INLINE measurement, we recommend a working distance of c. 5 mm between the surface of the face of the optical fiber and the metal surface. The beam divergence lies at 22° and the light spot assumes a diameter of some 5 mm on the metal surface to be investigated. With OFFLINE measurements, a spacer (**A3.0-OFL**) is also used, which can be attached to the head of the optical fiber.

SPECTRO-1-FIO-(UVC/UVC)



R-S-A3.0-(3.0)-1200-22°-UV

R-S-A3.0-(3.0)-1200-22°-UV + A3.0-OFL

A3.0-OFL

<u>Measurement procedure 2:</u>

Stimulation of an oil layer for fluorescence in the visible wavelength range using UVA light (365nm central wavelength range)

To this end, a color sensor (**SPECTRO-3-30-UV/BL-MSM-ANA**) is used; its transmitter unit consists of UV-LEDs (365nm central wavelength range). When conducting INLINE measurements, we recommend a working distance of 15mm. The spacer (**SPECTRO-3-15-d65-OFL)** can also be used OFFLINE (the distance between the sensor and object also amounts to 15mm). The detection range assumes a diameter of c. 12 mm with this clearance.

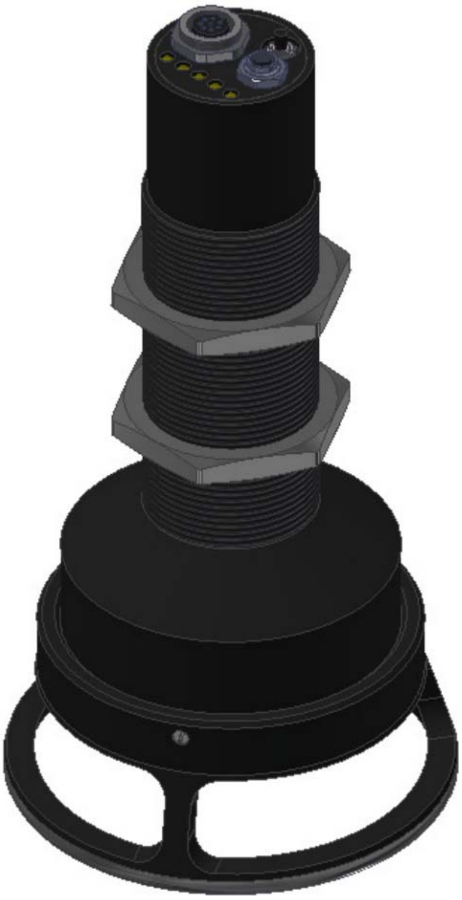
SPECTRO-3-30-UV/BL-MSM-ANA

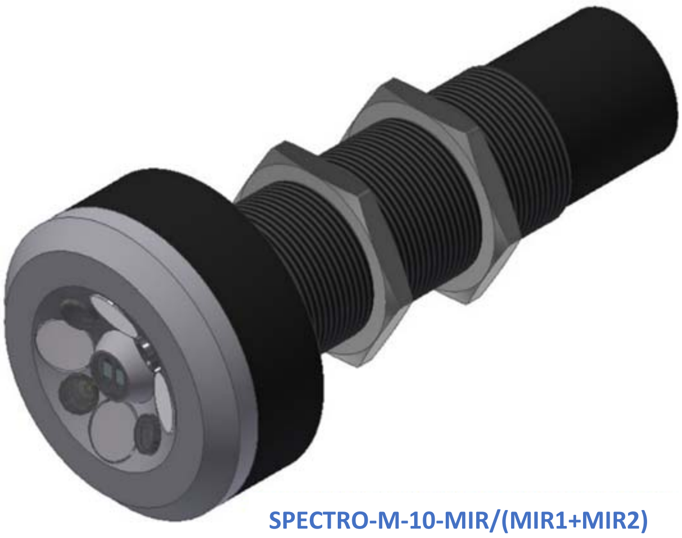
SPECTRO-3-15-d65-OFL

The UV-LEDs are arranged in a ring formation; a receiver part is located in the center of the sensors, which uses upstream optical filters to detect in the visible wavelength range, whilst UV light is blocked. With this measurement method, it should be taken into account that the intensity of the measured fluorescence also depends on the metal surface, as this serves as a reflector. Calibration to the respective surface or the respective object form is mandatory.

Measuring procedure 3:
Absorption of MIR light (3µm central wavelength range) by an oil layer

A wide-band MIR light source covering a wavelength range from c. 2µm to 6µm was used as a light source. A standardized signal evaluation is performed using two receivers fitted with different optical filters. Receiver 1 operates in a narrow wavelength range with a central wavelength range of c. 3µm, whilst receiver 2 also works in a narrow wavelength range and is fitted with a central wavelength range of c. 4µm. Receiver 2 functions as a reference, as its wavelength range is not observably influenced by the oil layer in comparison to the wavelength range of receiver 1. Equal reflection in both wavelength ranges was issued from the metal surface. A standardized evaluation of the two measurement signals provides information about the absorption of the oil layer; the result is largely independent of the metal surface present. The measuring distance between the sensors (**SPECTRO-M-10-MIR/(MIR1+MIR2)**) and the metal surface during INLINE measurement amounts to 10mm; the detection range is 10mm in diameter. Restriction of the measurement resulting from ambient light (white light LED interior lighting) could not be determined and a high measurement frequency (> 1 kHz) can be used. A spacer (**SPECTRO-M-30-OFL**) is available for OFFLINE measurement, which also ensures a measurement clearance to the metal surface of 10mm.



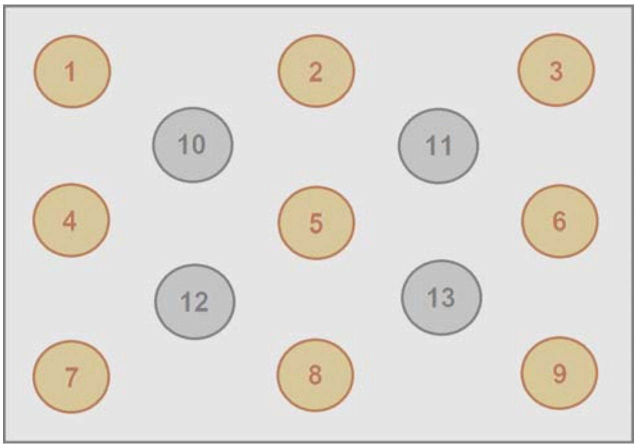


SPECTRO-M-10-MIR/(MIR1+MIR2)

SPECTRO-M-30-OFL

The measurement

A 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 (non-fuzzing) cloth. This procedure was performed in all the specified sectors. Then the oil quantity already used was spread again 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.

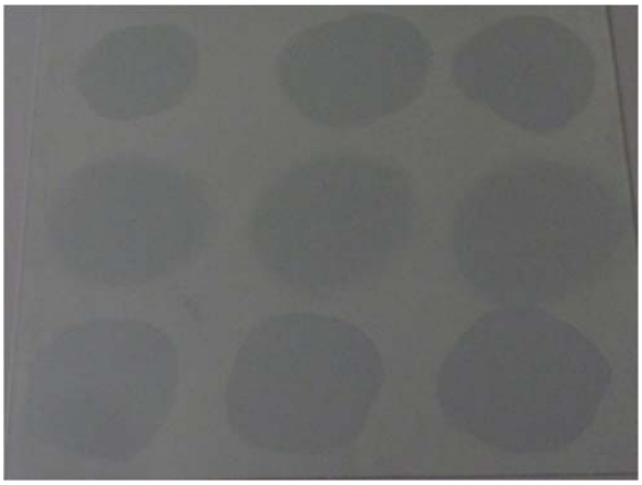


1,2 and 3: medium layer thicknesses (decreasing from 1 to 3)

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

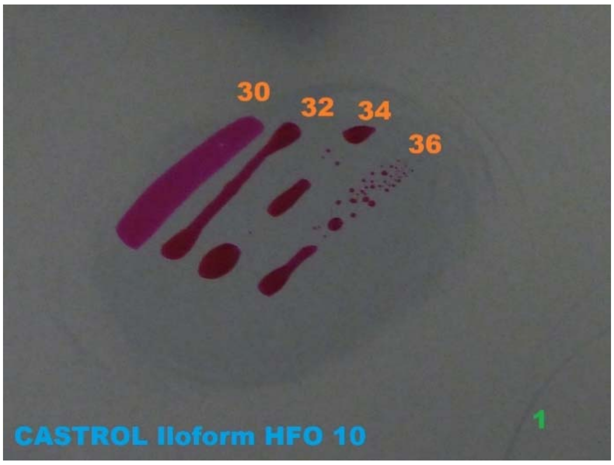
7,8 and 9: large layer thicknesses (decreasing from 7 to 9)

10,11,12 and 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 has been removed from the metal surface These areas are called sectors 10-13

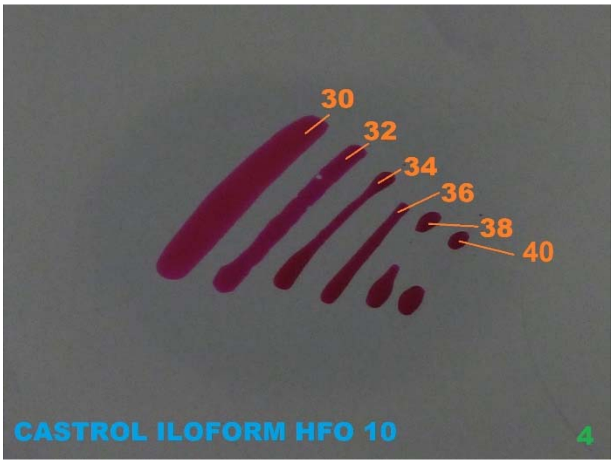
As already mentioned, the surface tension in the respective sectors serves as the reference for the respective measurements. This was ascertained for every oil type using the available test inks (from 30mN/m to 50mN/m) each after the measurements with the 3 different sensors (the test ink was applied to the respective sectors; the measuring result is restricted by the autofluorescence of the test ink, additional absorption or reflection).



Oil sample: CASTROL Iloform HFO 10

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

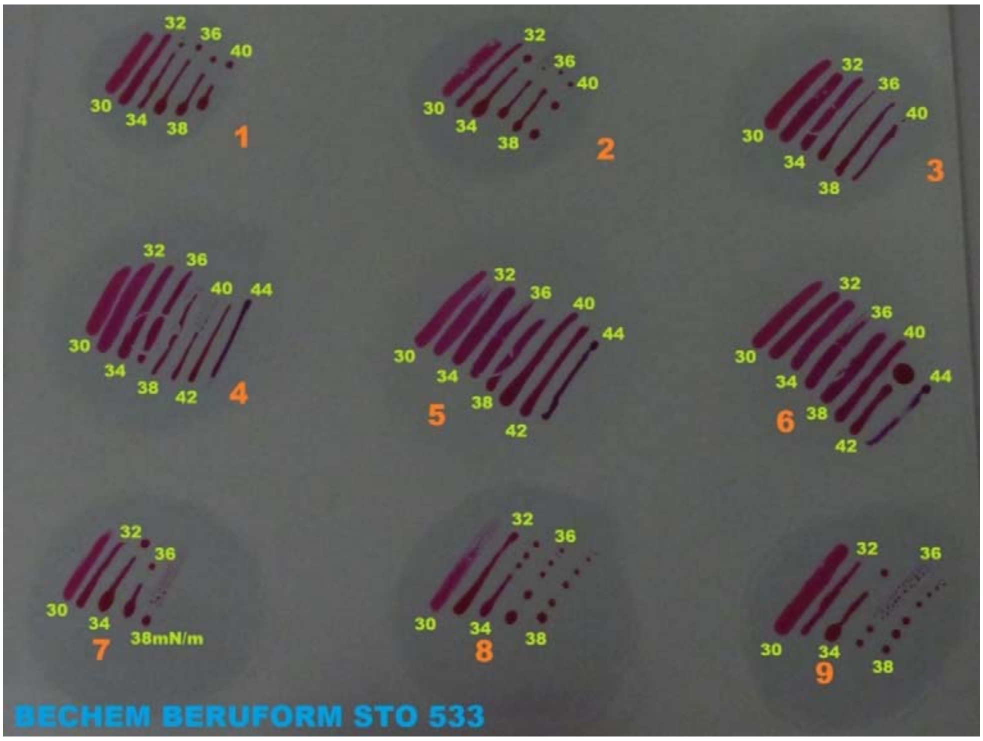
Sector: 1



Oil sample: CASTROL Iloform 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

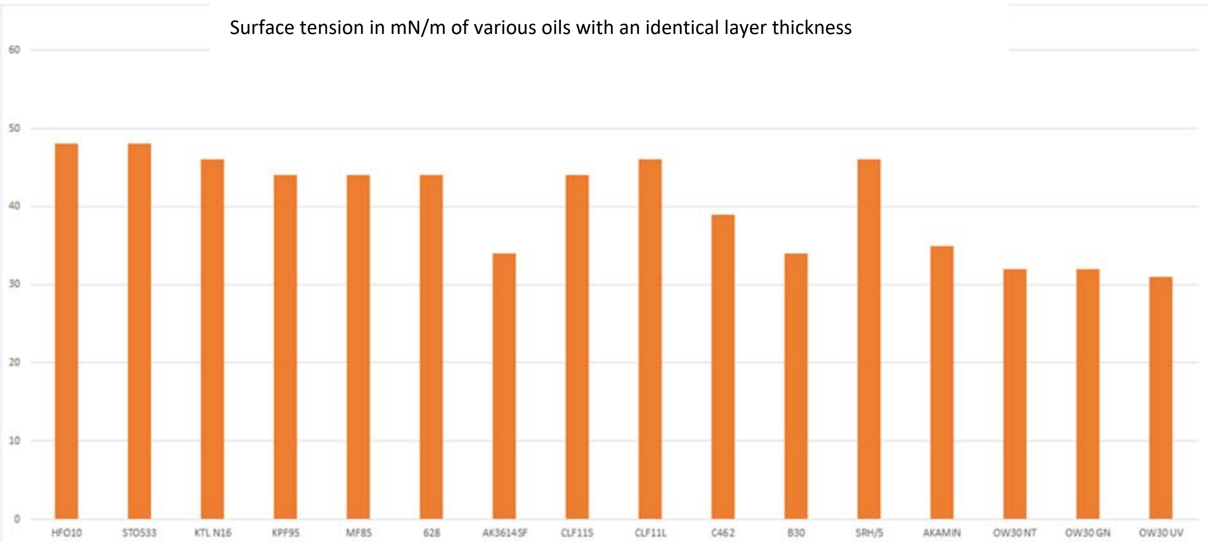
The oil samples

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-COLOR

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). Inference from the surface tension to the oil layer thickness is not possible without knowledge of the oil.

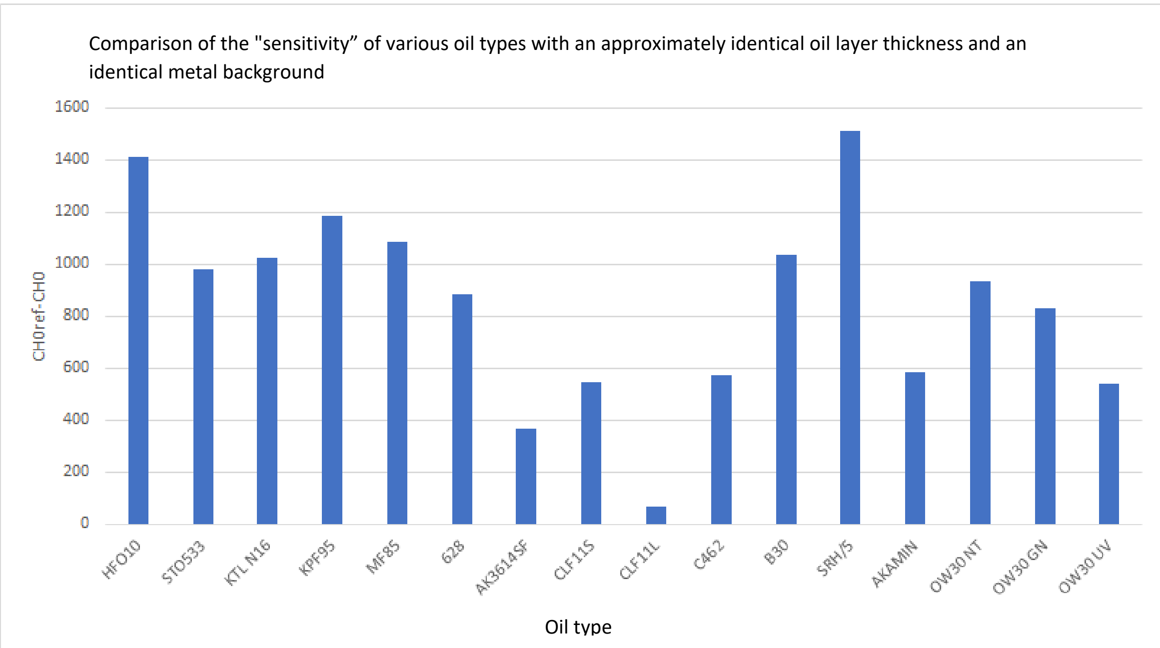
The measurement results

Measurement procedure 1:
Absorption of UVC radiation (265nm central wavelength range) by the oil layer

Measurement was performed with a **SPECTRO-1-FIO-(UVC/UVC)** fiber optic sensor inc. **R-S-A3.0-(3.0)-1200-22°-UV** reflex light guide and an **A3.0-OFL** offline optical fiber attachment, initially per oil type in sectors 10 to 13 (this serves as a reference when ascertaining the measured values and the surface tension of the degreased metal surface) and then in sectors 1 to 9. The optical fiber head inc. offline attachment was placed in the center of the respective sector.

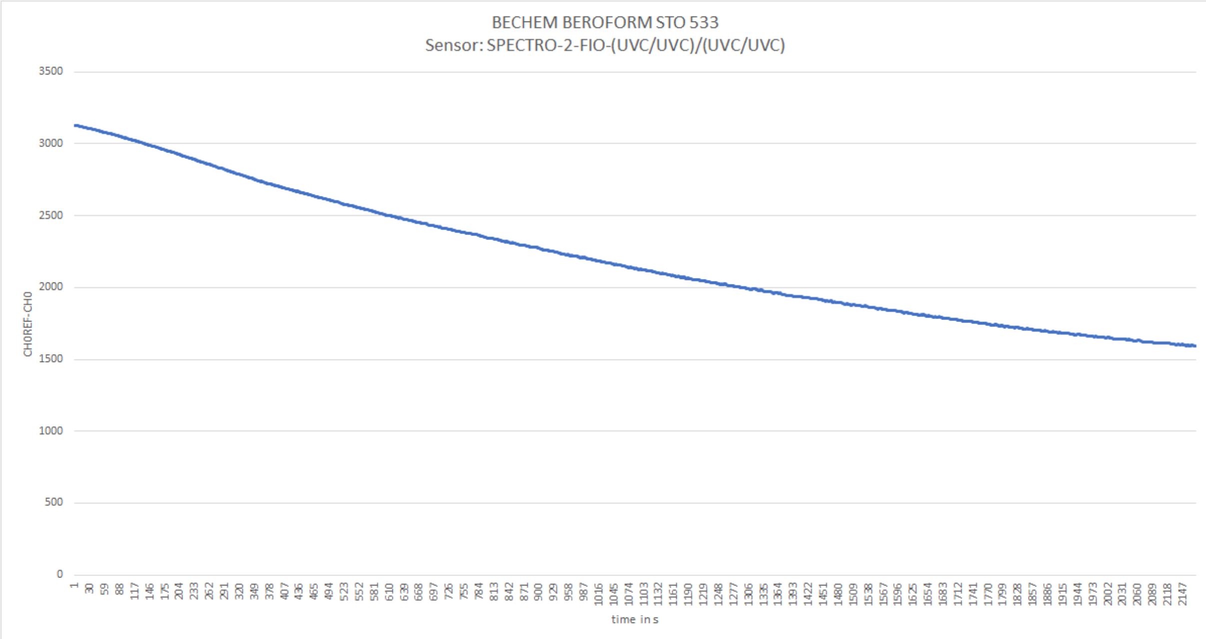
Comparison of the sensitivities of the various oils with the same layer thickness

Investigations of the oil types present using measurement procedure 1 produced significantly different measurement results as shown in the following diagram:



The signal dip of the UVC radiation reflected from the metal surface and registered by the fiber optics*CH0ref* minus the UVC radiation *CH0* reflected from the metal surface and reduced by absorption on both the incidence and return path. The ratio of the difference *CH0ref-CH0* from the oil samples subject to the strongest absorption to the oil sample which behaves almost neutrally amounts to a factor of 22.

Investigation of the UVC absorption of an oil layer in dependence on time



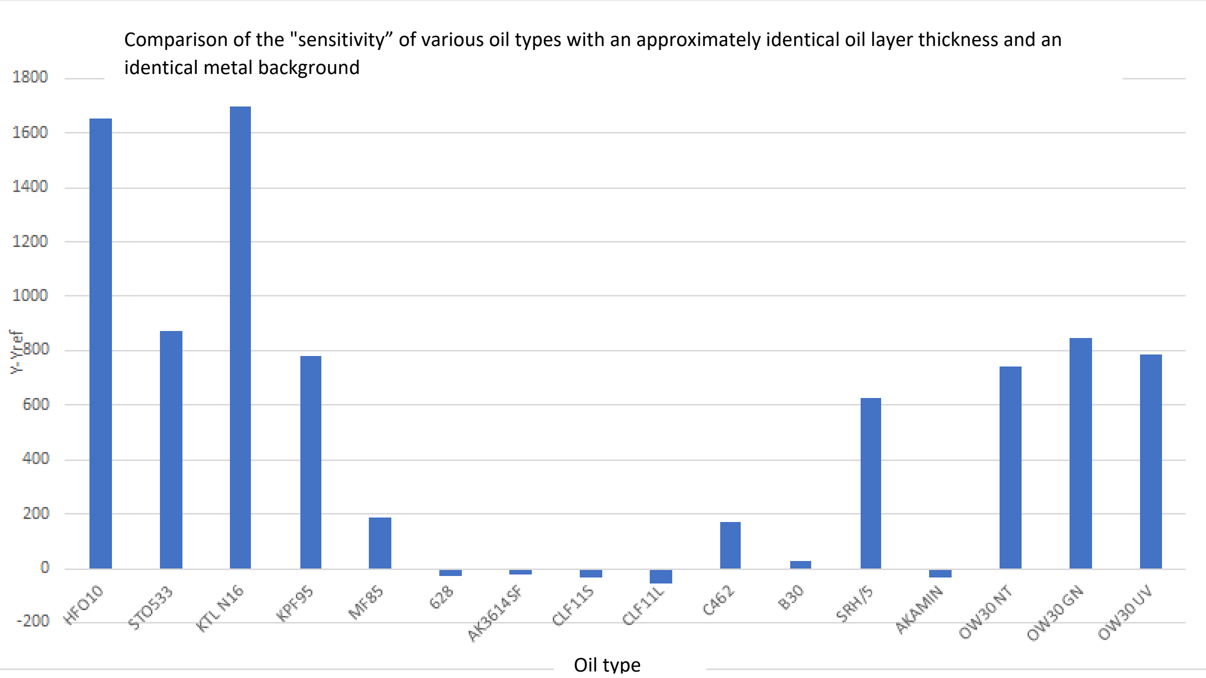
In this measurement method, the fiber optic front end A3.0-OFL is placed on a certain location of the oil film over a long period of time; the signal course CH0 was recorded during this time.

A clear reduction in the absorption (Delta = 1600) of the UVC radiation could be observed over a period of 36 min (CH0ref = 3975), which corresponds to a relative reduction of the absorption effect by a factor of 2. When determining the measured value, the time after the placement of the measuring head on the respective oil sample is decisive.

<u>Measurement procedure 2:</u>
Secondary emission in the visible wavelength range (fluorescence) with stimulation in the UVA range (365nm)

A **SPECTRO-3-30-UV/BL-MSM-ANA** with a UVA ring illumination (365nm) was used as a sensor and a receiver-side detection range of type 450nm to 700nm was used. To enable prone measurement, the INLINE system was fitted with a **SPECTRO-3-15-d65-OFL** spacer at the front. First of all, an average reference value was determined (10-13) on the reference points and then an oil sample was measured for the points 1-9.

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



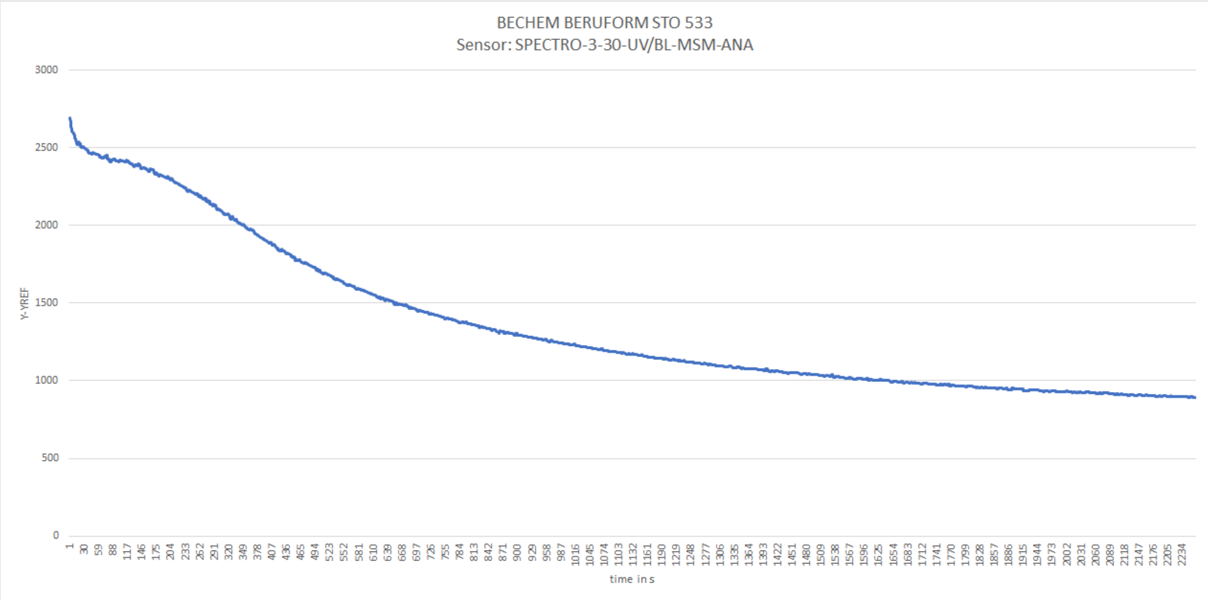
This measurement procedure also produced considerable differences in the *Y-Yref* value with the same oil layer thickness of the individual oil types. If the oil types which do not tend to fluorescence are not taken into account, a difference of c. 61 results between the most sensitive (Z+G KTL N16) and the least sensitive sample (TRUMPF B30).

It was not possible to determine fluorescence with stimulation in the UVA range using the following oil types:

- WILKE 628
- WISURA AK 3614SF
- RAZIOL CLF 11S
- RAZIOL CLF 11L
- TRUMPF AKAMIN

Investigation of the fluorescence in the visible wavelength range with UVA stimulation of an oil layer in dependence on time

The sensors were placed on a certain measuring point over a long period of time (a number of minutes) and the signal strength Y was recorded during this time.

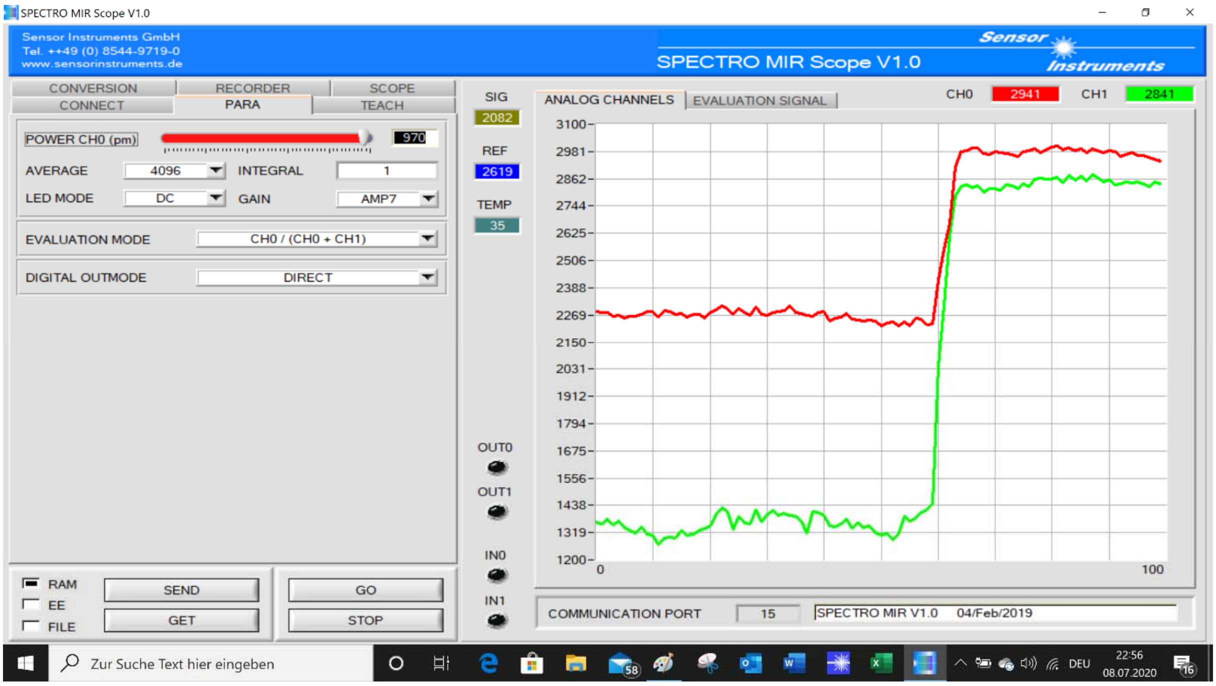


For example, over a period of c. 38 minutes, the oil type BECHEM BERUFORM STO533 presents a reduction in the fluorescence signal *Y-Yref* by a factor of 3. The exact time at which the measured value was recorded after applying the sensors is of decisive importance in this measurement procedure for determining the surface tension at the respective location. This consideration is of less importance during INLINE sensor application, as each measurement sample is subject to the relatively intensive UVA radiation for only a short time and the measurement can be performed at the same time after positioning. The time synchronous sequence is performed between the Programmable Logic Controller (PLC) and the respective actuating elements

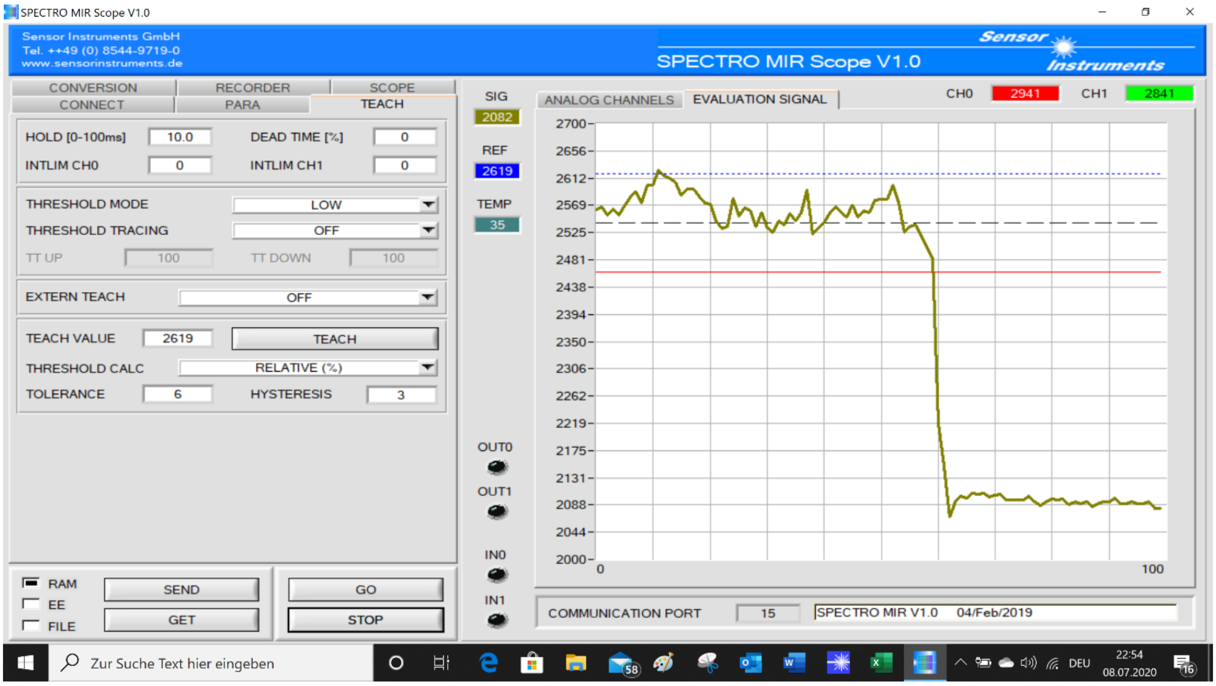
<u>Measurement procedure 3:</u>
Standardized comparison of two wavelength ranges in the mid-wavelength infrared light range (MIR)

A **SPECTRO-M-10-MIR/(MIR1+MIR2)** was used as a sensor; measuring points 1-9 were also taken into account, whilst positions 10-13 served as reference points. The INLINE measurement system has been converted to a hand-held measuring device using the **SPECTRO-M-30-OFL** spacer. The distance to the measuring surface was 10 mm. The standardized measured value is the result of the two ascertained measured values of the two measuring windows in the MIR range with the center wavelengths of 3µm / 4µm. The second serves as a reference window, as prior spectrometric investigations have shown that no noticeable absorption of the MIR radiation occurs in this wavelength range.

During measurements, a connection to the PC was established using a serial interface. 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**.



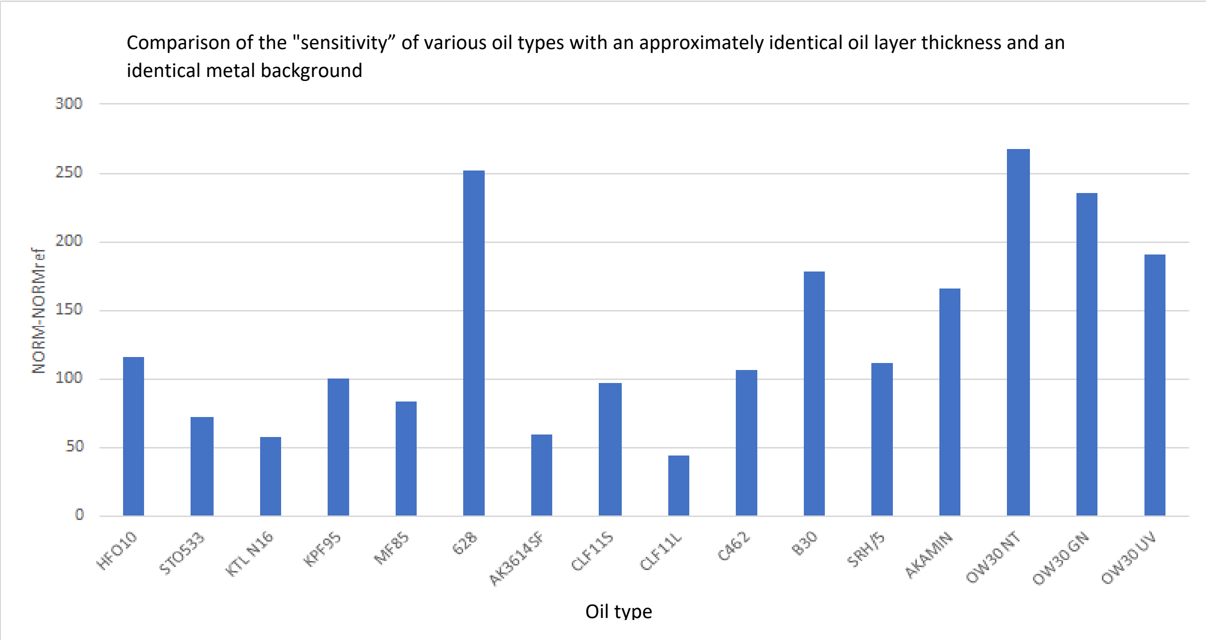
In addition to the display of the two measured values CH0 (reference value with 4µm) and CH1 (measured value at 3µm) the standardized value SIG = 4095xCH0/(CH0+CH1) is also shown.



The **MIR MONITORING** software can be used during INLINE measurement. The system operator is shown the current quality situation and the trend on a PC monitor during production via a column diagram. The measurement data is saved in a suitable format so that it can then be used with Word® or Excel®.

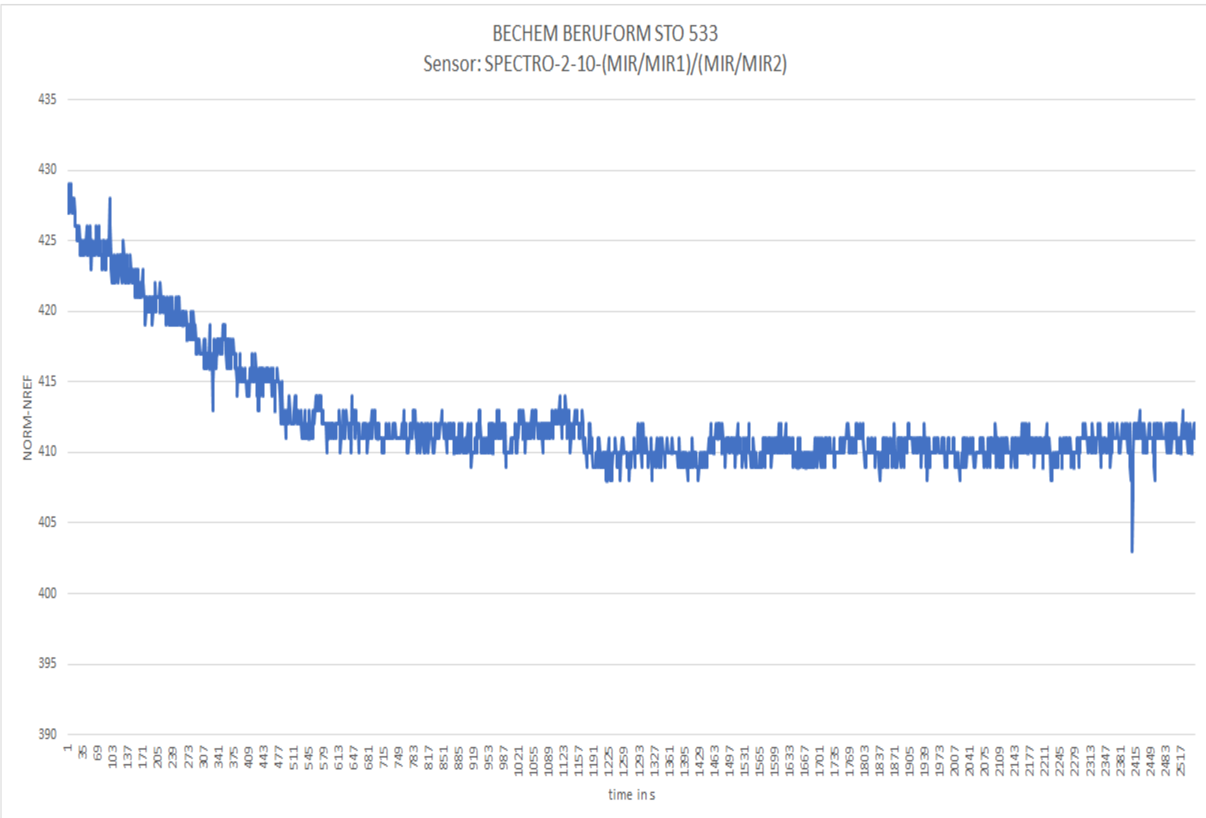
Comparison of the sensitivities of the various oils with the same layer thickness.

Even when using this measurement method, it was still possible to ascertain differences in sensitivity between the various oil types (with comparable layer thickness). In comparison to the two other two measurement methods, it was possible to detect all oil types. The difference between the most sensitive and insensitive sample amounted to a factor of only 7.



Investigation of the standardized absorption of MIR light in the wavelength ranges c. 3µm or 4µm of an oil layer in dependence on time

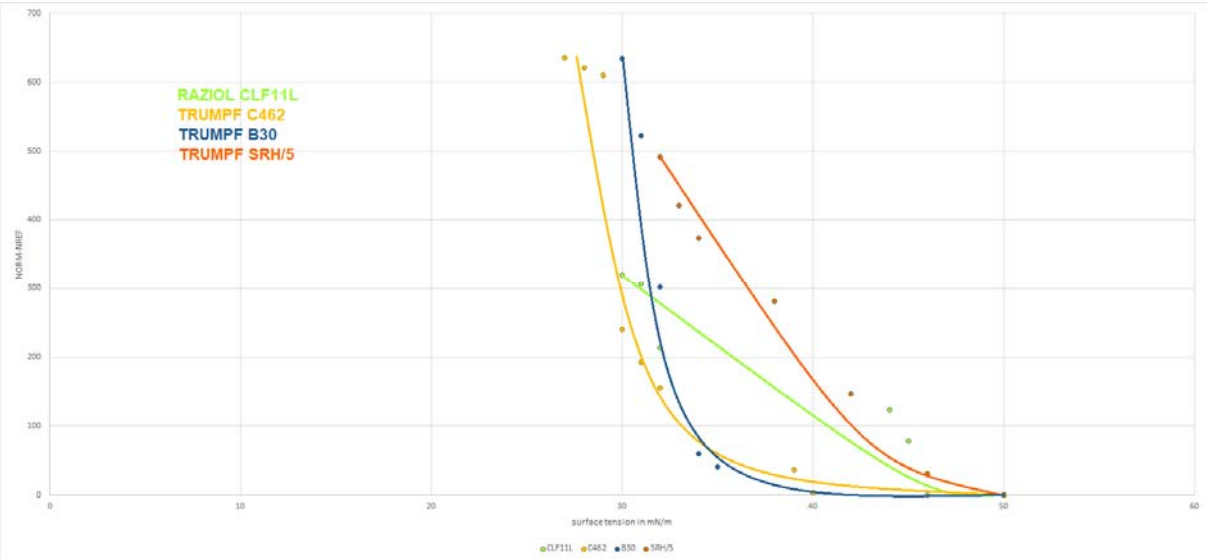
The sensors were placed on a measuring point to which an oil film of a certain oil type had been applied and measurement data recording was performed over a longer period (c. 43 min).

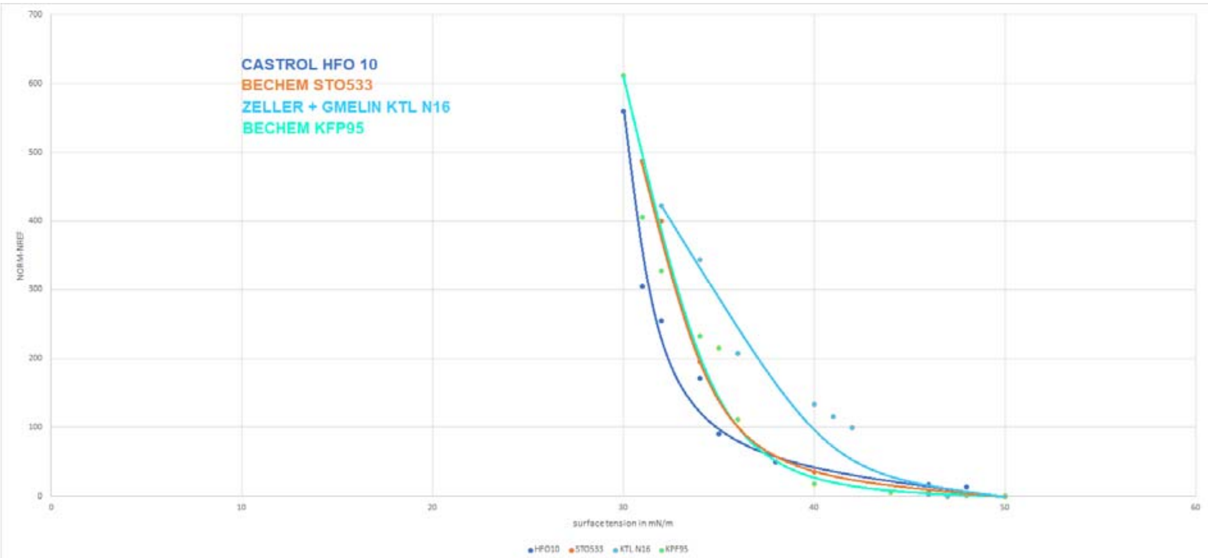


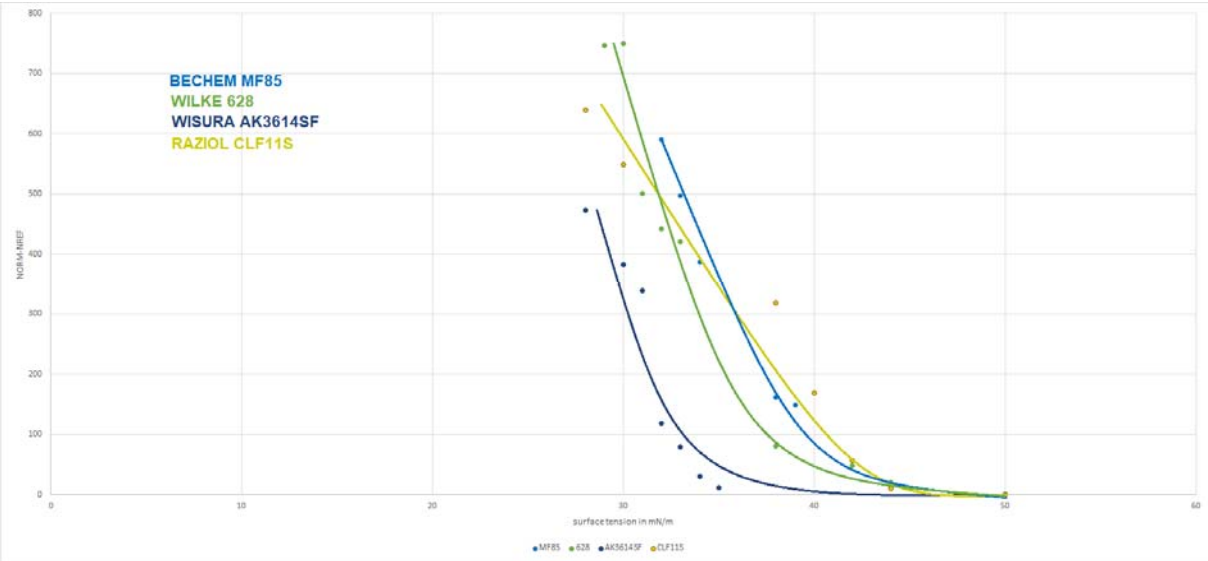
A decrease in the standardized range of only 5% could be determined (corresponds to a factor of c. 1.05). In comparison to the two previous measurement procedures, the signal dip is significantly smaller, which is not just due to the standardized evaluation but the fact that MIR radiation would seem to exercise a much lower influence on a change of the absorption behavior of oils.

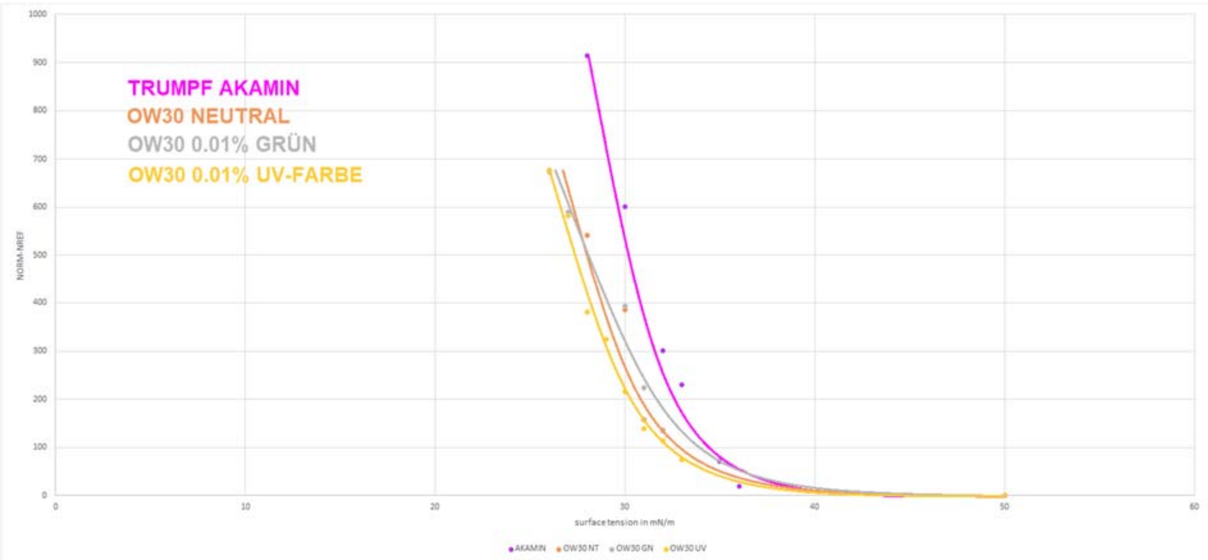
Investigation of the standardized absorption behavior in the MIR range in dependence on the surface tension of the respective oil type

Measurements were conducted at points 1-9 (STANDARD) and an average reference value was established from the four reference positions (*STANDARDref*). The difference between the two values *STANDARD - STANDARDref* serves as the degree of the surface tension. The value of the surface tension was determined using the test ink method.







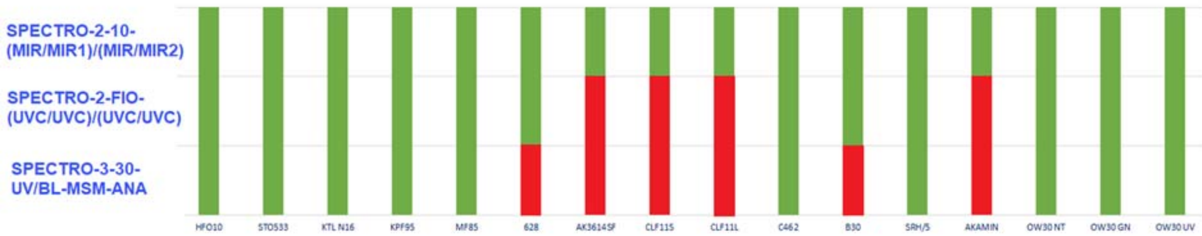


The gradients show that this measurement method also requires the performance of calibration to the oil used in connection with the respective metal surface. The user can perform calibration using the Windows® software **SPECTRO MIR Scope V1.0**. The value of the surface tension can also be displayed in addition to the respective STANDARD value.

Summary

In practice, the presence of oil films on metal surfaces is very often checked using test inks. 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 only a relatively coarse graduation of test inks in relation to the surface tension (in 2mN/m steps) means that an exact analysis is not of especial 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 and remain neutral. Measurement procedure 3 requires a somewhat larger test surface which should also be plane. With all other criteria, this procedure performs better in comparison to measurement procedures 1 and 2. One of the important advantages of measurement procedure 3 is the standardized evaluation. This cannot replace the individual calibration per oil type and metal surface but variations in 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.

Which oils are suitable for the respective measurement procedure? (green: suitable, red: unsuitable)



Further important characteristics of the respective measurement procedures in comparison:

Measurement procedure	**UVC**	**UVA**	**MIR**
Sensitivity difference factor of the various oil types	22	61	7
Signal attenuation factor with long-term illumination	2	3	1.05
Sensitivity to extraneous light in comparison to artificial light (white-light LED)	Medium	Strong	Low
Measurement distance in mm	5	15 (11)*	10
Detection range in mm	5	12 (1)*	10

* There is also a fiber optic system version for the UVA measurement procedure, with which correspondingly small detection ranges can be achieved.

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