AXELSEMRAU

Fully automated workflow for FTIR analysis of used technical oils

with the CHRONECT Workstation FTIR

Application note 1901

CHRONECT Workstation FTIR
Application note 1901

Introduction

The condition of oils in different applications is often an important parameter for optimizing workflows. The replacement of oils in, for example, gearboxes or large machines can be a major cost factor. Therefore, the determination of oil quality is an important process to save time and money. Changes in oil can be easily determined by FTIR analysis. Typically, this involves comparing a known IR spectrum with a measured oil IR spectrum. If, for example, oxygen groups appear, this indicates aging of the oil. With changes in the hydroxyl groups, the % water content in the sample can be determined. In addition, spectrum comparison can identify adulterated oil to ensure that only a specific organic or synthetic oil is used. The same is true for identifying the composition of oil blends.

The demand for this type of oil analysis has increased, so automation of sample feeding is an advantage.

A robot that transports the samples allows the equipment to operate 24/7 and the FTIR to be used at high and efficient capacity.

Device configuration

The basis for fully automated sample processing is provided by a CHRONECT Robotic RTC autosampler with a width between 50 and 200 cm and the CHRONOS control software. The width depends on the desired number of samples to be started simultaneously in one run. In addition, a tray was integrated that stores 98 racks for 1 mL pipette tips, a parking station for the pipette tool and an injection port. To flush the flow cell of the FTIR, a dilutor module with solvent reservoir was also added.

The robot can be configured for standard GC vials (10/20 mL), or for other vials/bottles (e.g. 100 mL plastic bottles).

The general workflow for the method consists of three parts: i) Air bubble free aspiration of the oil sample without droplets ii) Injection into the injection port of the flow cell and subsequent FTIR measurement iii) Washing of the injection port with fresh solvent and generation of a solvent spectrum free of additional peaks. If the solvent spectrum is not free of additional signals, the cell is washed again with solvent. After a defined number of wash cycles, the system reports a contaminated flow cell and advises to clean the cell. CHRONOS can optionally send an e-mail or SMS to the user here, which conveys the information that user intervention is required.

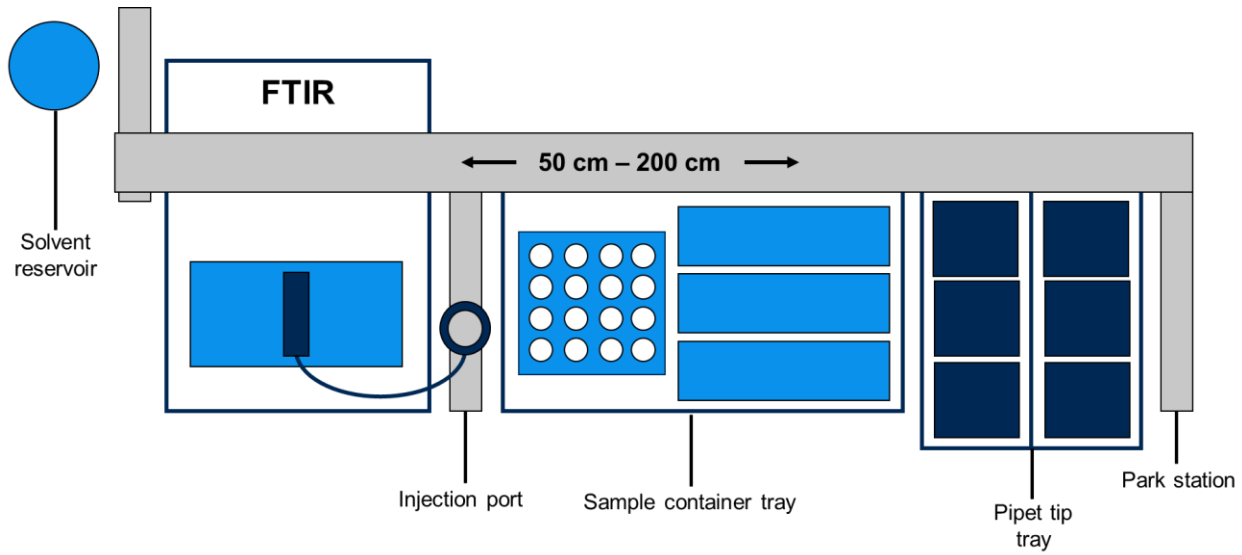


Figure 1: Schematic configuration of the CHRONECT Robotic RTC with all required modules.

The sample area of the instrument is designed for two possible operating modes. One of them is the Day Mode, in which, for example, sample containers can be provided and analyzed in racks of 20. The next rack is then placed by the user. For 160 cm width of the x-axis, the unit could be equipped with six racks of 80 plastic tubes each for the night mode. The higher capacity obtained in this way allows the sample run to be started without supervision for the night.

The autosampler then runs samples automatically during the night. The sample lists can be imported directly from LIMS.

Results and discussion

The fully automated sample processing and measurement was investigated under routine conditions with high sample throughput. Initial tests were performed for general reproducibility of results and showed no carryover. A sample was injected into the system 20 times. A specific band at 1,230 cm^{-1} was identified by the FI-IT software. It showed a reproducibility < 0.23 % over all measurements. Therefore, a robust and carryover-free injection by the autosampler is guaranteed. In addition, a sample was injected several times during routine measurements to check long-term stability. Here, a relative standard deviation (RSD) of less than 0.31 % was found. Both results demonstrate the stability of the system.
Once the system is installed, sample processing can begin to generate a spectral database and eventually measure customer samples. Figure 4 and 5 show sample applications. In Figure 4, customer oil samples were compared to a spectrum of fresh oil and indicated aging. The band around 1,700 cm^{-1} indicates oxidation and the band above 3000 cm^{-1} indicates water in the sample. This comparison can be done automatically if the required comparison oil spectrum is available and the FTIR can handle automatic sample evaluation.

Another example is shown in Figure 5. The same comparison is performed as in Figure 4, but the additional bands indicate an oil mixture with adulteration.

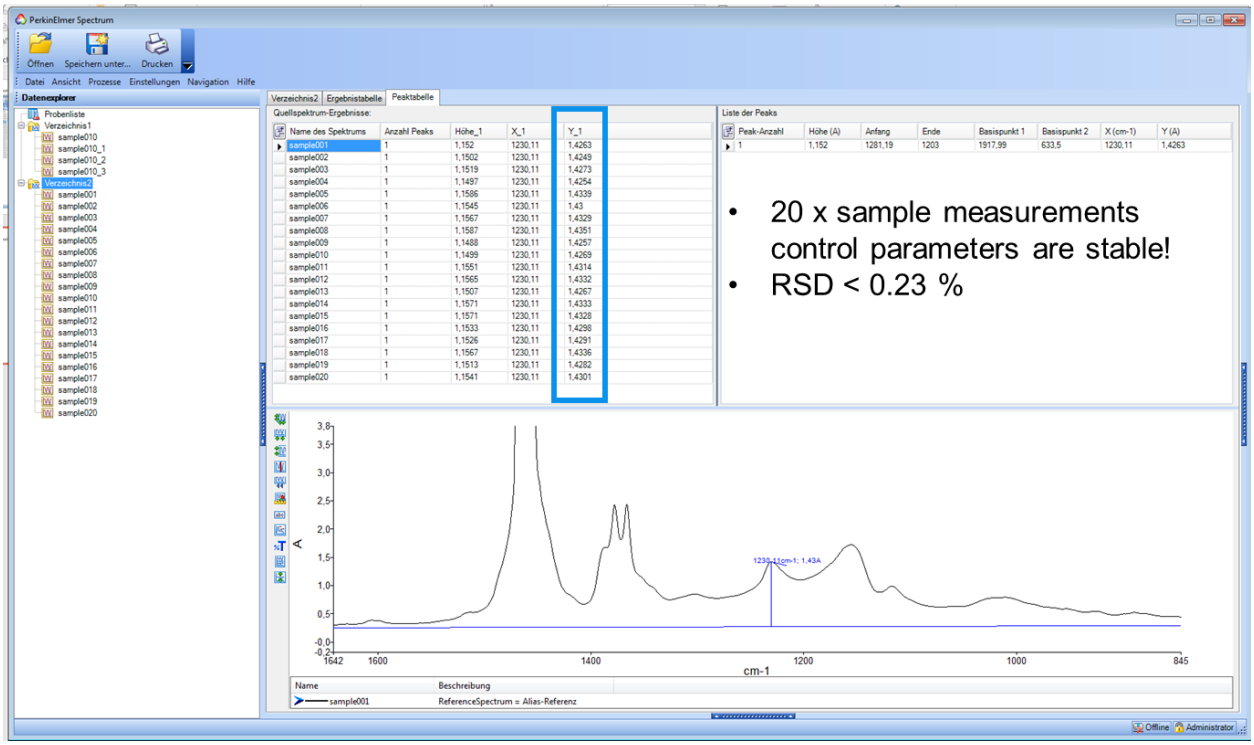
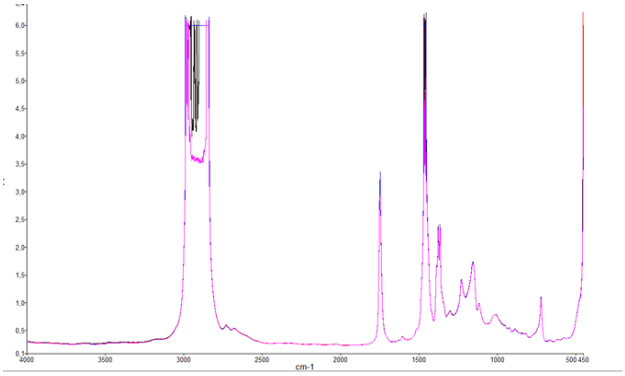


Figure 2: Reproducibility test with a 20-fold injection of a sample. The blue box shows the y-axis control parameters that were measured.



Sample	X	Y
2018-11-16	1.1533	1.4306
2018-12-14	1.1433	1.4234
2019-01-08	1.1436	1.4204
2019-01-18	1.1398	1.416

Figure 3: Long-term stability test of a sample injected several times over a period of two months. The table on the right summarizes the x- and y-axis parameters for reproducibility determined over time..

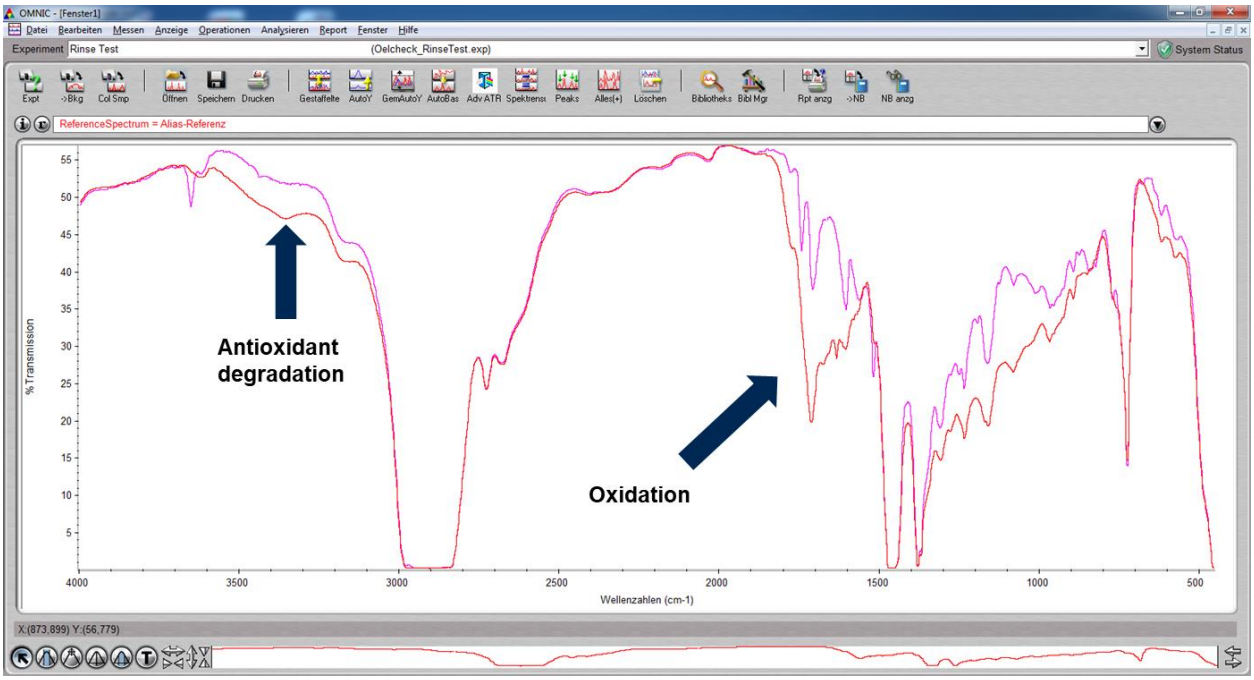


Figure 4: Customer oil samples were compared with a reference spectrum of fresh oil. The additional bands indicate aging.

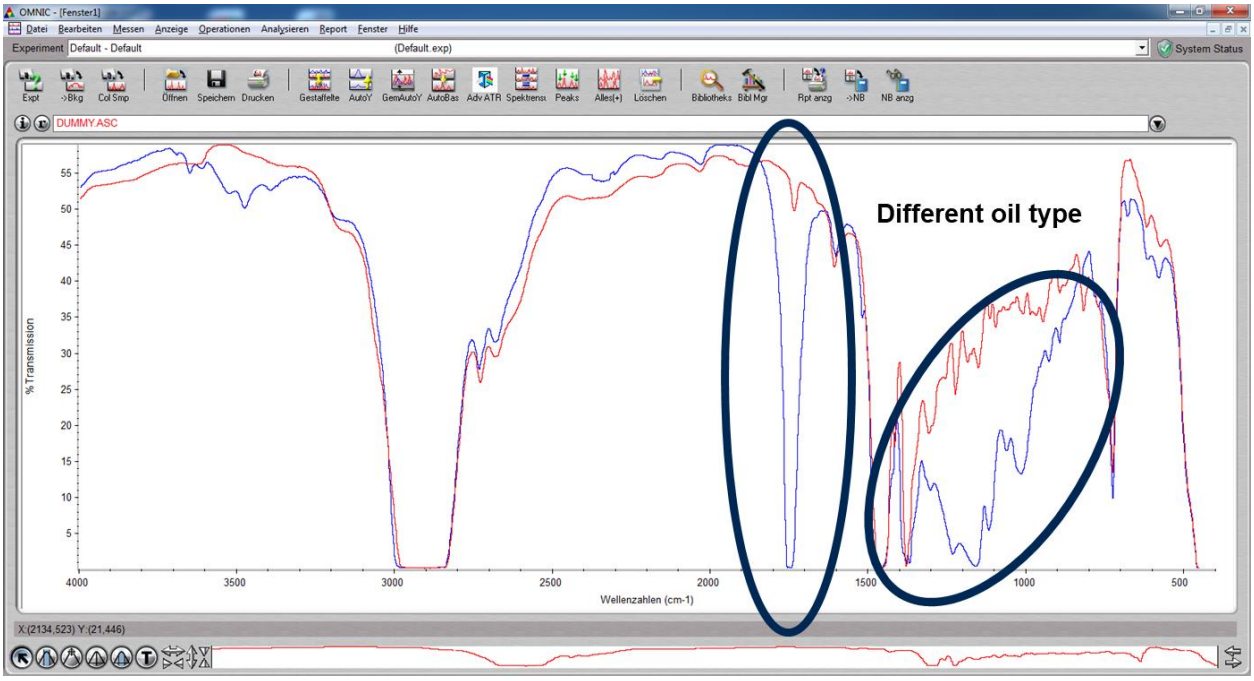


Figure 5: Customer oil samples were compared with a reference spectrum of fresh oil. The additional bands indicate adulterated oil.

Summary

By integrating CHRONECT Robotic, a fully automated workflow was developed: Starting with the sample vial, through the drawing up of the oil sample to the introduction into the FTIR flow cell. Due to the intelligent and efficient integration of washing steps and overlapping of individual steps, a high sample throughput of approx. 650 samples per day can be achieved.

The CHRONECT Workstation FTIR is a development by Axel Semrau.

Subject to technical changes

Axel Semrau GmbH & Co. KG
Stefansbecke 42
45549 Sprockhövel
Germany
Tel.: +49 2339 / 12090
www.axelsemrau.de
info@axelsemrau.de