Application Note AN M102

# FT-IR Microscopic Identification of Fibers

## Introduction

The identification of fibers is of high importance in forensic science as it can yield trace evidence in a criminal case. A fiber found at the crime scene can provide information about the criminal, e.g. about his clothing or specific places he has visited. Furthermore the fiber might be compared to similar items retrieved from suspects and provide the link to the crime.

In textile industry the identification of individual fibers in the textile matrix is interesting as a part of the quality control process, e.g. to track an unwanted polymer fiber used during the spinning process of a yarn.

Also for conservators of textile artifacts the ability to differentiate between fibers originating from different natural materials (e.g. cotton, sisal, bast, silk and wool) is very helpful to choose an appropriate treatment.

FT-IR microscopy is a very powerful technique to characterize the chemical composition of natural and synthetic fibers, both organic and inorganic in nature. Due to its capability to measure with a high lateral resolution usually the availability of a single fiber is sufficient to perform an analysis. As the FT-IR measurement is non-destructive other analytical techniques might be applied afterwards. Furthermore, FT-IR microscopy provides objective results


Figure 1: LUMOS II stand-alone FT-IR microscope

and is in most cases quicker, easier, and sometimes, more selective than classical methods. Due to these multiple benefits the IR-microscopic method is described by ASTM International as standard method for forensic analysis of fibers (ASTM E2224-10) and for identification of fibers in textiles (ASTM D276-12).

Using the ATR-(Attenuated Total Reflectance) technique minimal sample preparation is required to perform an FT-IR-microscopic measurement. Just a fixation of the fiber on a flat substrate like a metal plate is required to avoid its movement during visual inspection and definition of the measurement positions. In this application note measurements of different natural and synthetic fibers using the fully automated FT-IR microscope LUMOS II are presented.

## Instrumentation

The presented fiber measurements were performed using the stand-alone FT-IR microscope LUMOS II (Fig.1). It stands out due its full automation and ease-of-use combined with sample visualization and infrared spectroscopic performance of excellent quality. Its 8x objective provides the measurement modes ATR, transmission and reflection and high quality visual inspection capabilities.

All required changes of hardware settings as well as the complete IR-measurement procedures including background measurements are performed fully automated – even in the ATR-mode which is the typical approach to measure fibers. To provide perfect contact to samples ranging from soft to very hard the ATR-device offers three pressure steps and is equipped with a very precise internal pressure sensor.

Due to a large working distance and an unobstructed access to the planar sample stage the sample positioning is extremely convenient. Additionally for maximum performance and convenience the LUMOS II includes:

- Motorized Germanium ATR crystal with internal pressure control
- Large field of view: 1.5 x 1.2 mm
- Automated change of the numerical aperture between IR and Vis mode to achieve a high depth of field for the visual inspection of a sample, but also highest sensitivity for the IR analysis
- Independent white light LED illumination in transmission and reflection
- Fast CMOS camera with 4x zoom
- Motorized stage (option), position accuracy of 0.1 µm
- Optional macro accessory that allows to use all Quick Snap sampling modules from the compact FT-IR spectrometer ALPHA II

## Work-flow of the fiber analysis

Before measurement the fiber sample is fixed on a metal plate with adhesive tape. For analysis the plate is placed on the sample stage of the IR-microscope. Utilizing full motorization of the LUMOS II, the OPUS software guides the user very efficiently through the process of data acquisition. All required changes of the hardware settings are performed software controlled. The user interface presents the operator always the appropriate functions for the current step.

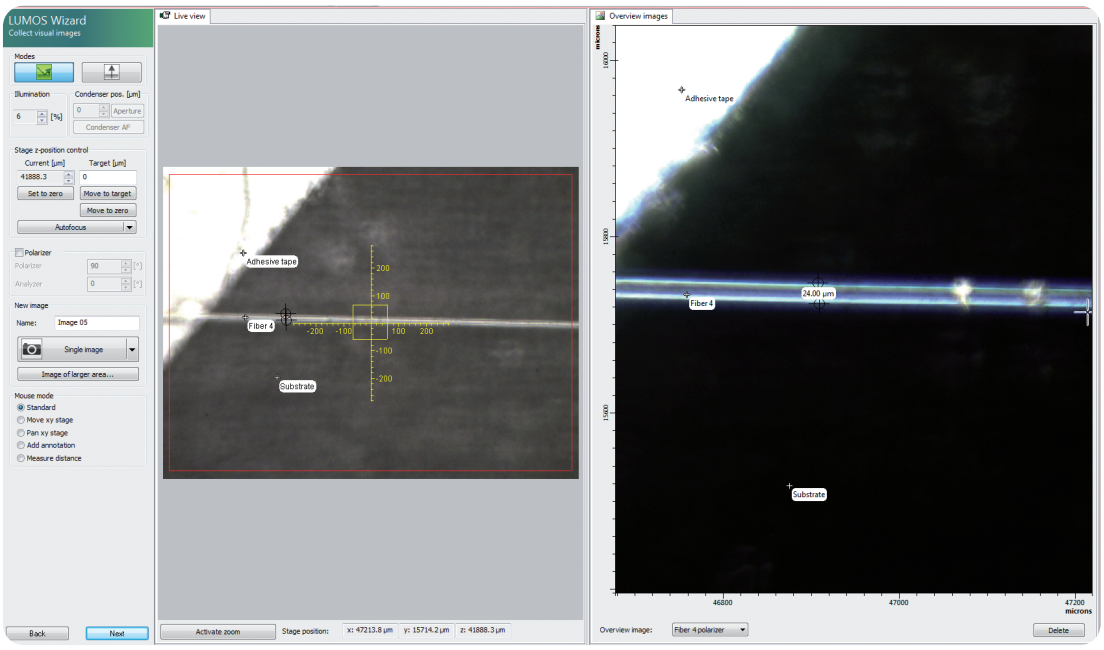


Fig.2: Visual inspection of a fiber sample using the LUMOS II software. The live camera view of the sample is shown in the middle (here without polarizer) and stored images in the overview on the right (here an image taken with crossed polarizers is shown).

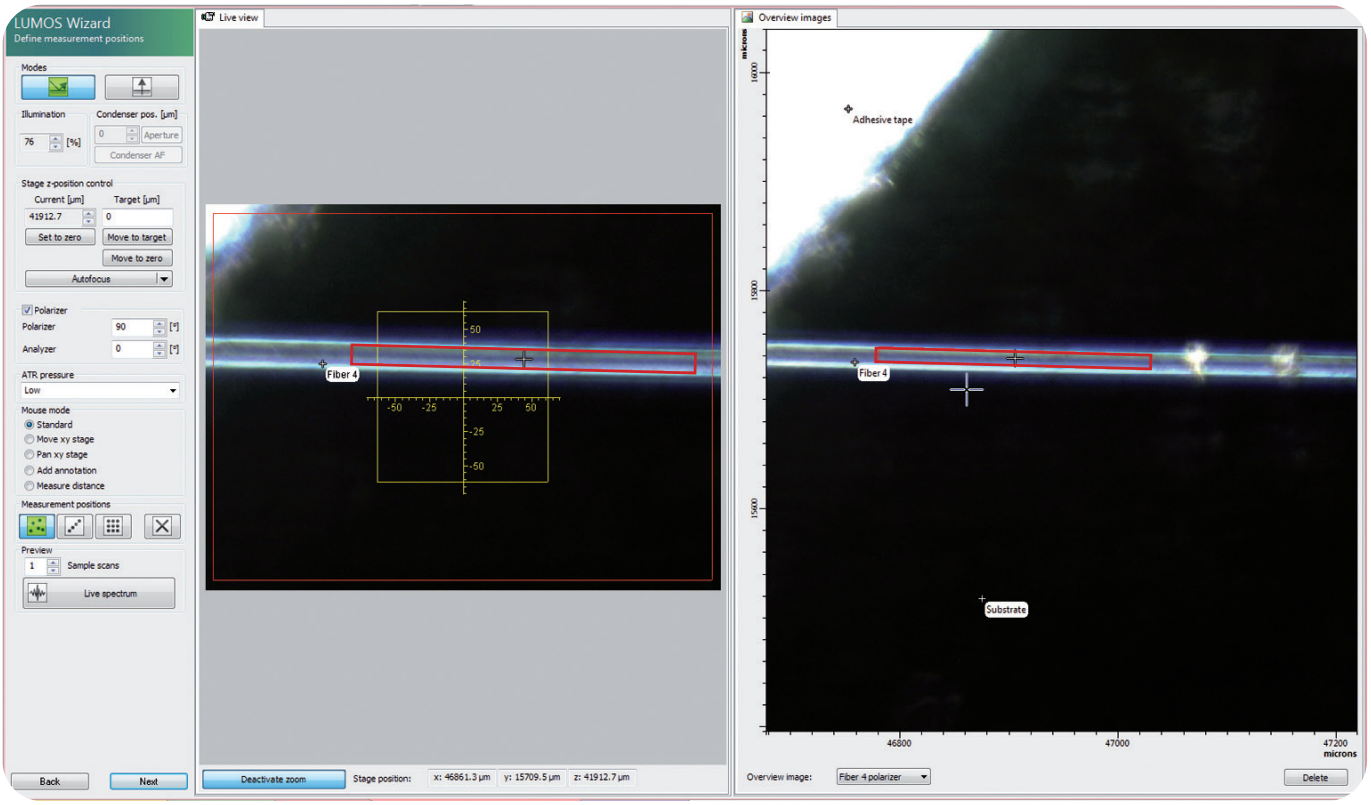


Fig.3: Definition of measurement positions and according knife edge aperture settings (indicated by red rectangle).

Figure 2 shows the user interface during the visual inspection of a fiber sample during which pictures of the sample can be taken using the digital camera. The function bar on the left side provides the functions useful for this step, like the setting of polarizers for contrast enhancement. In the middle window the live view of the camera shows the sample and in the overview on the right side the saved pictures are displayed.

The definition of the measurement positions on the fiber is shown in Fig.3. Resizing and turning of the red rectangles allows setting the automated knife-edge aperture according to the sample dimensions individually at each measurement position. To always provide the optimal background spectrum for sample positions with differing aperture sizes the LUMOS II offers a mode where upon aperture change a new background spectrum is recorded automatically. In the ATR-mode automation of the background measurement is realized by an internal piezo drive moving the crystal into the focus.

## Example: Identification of synthetic fibers

Various synthetic fibers with a diameter in the range of 25 to 40µm were measured using the FT-IR microscope LUMOS II with the automatic knife-edge aperture set accordingly to the fiber dimensions. The measurements were performed using the ATR-mode with an acquisition time of 20seconds/spectrum and at an spectral resolution of 4 $cm^{-1}$.

The picture in figure 4 shows the five fibers which have a quite similar visual appearance prepared on a black sampling plate. Though, despite their visual similarity the IR-microscopic spectra on the right reveal that only for two fibers the same type of polymer was used. By search in dedicated spectral databases (e.g. Bruker Synthetic Fibers Library) the fiber material easily can be determined (figure 5).

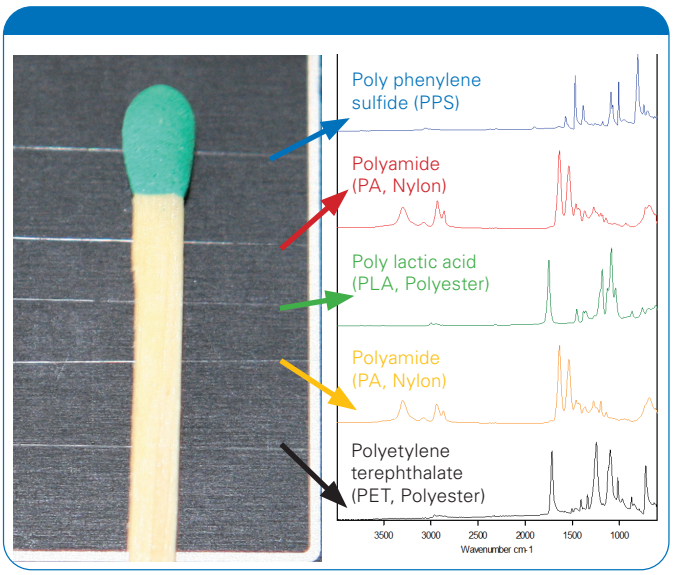


Fig.4: Synthetic fibers prepared on sampling plate (left) and according spectra (right). Library search of the spectra allows easy identification of the fiber's chemical composition. Even though the visual appearance is very similar, only two fibers are made from the same polymer.

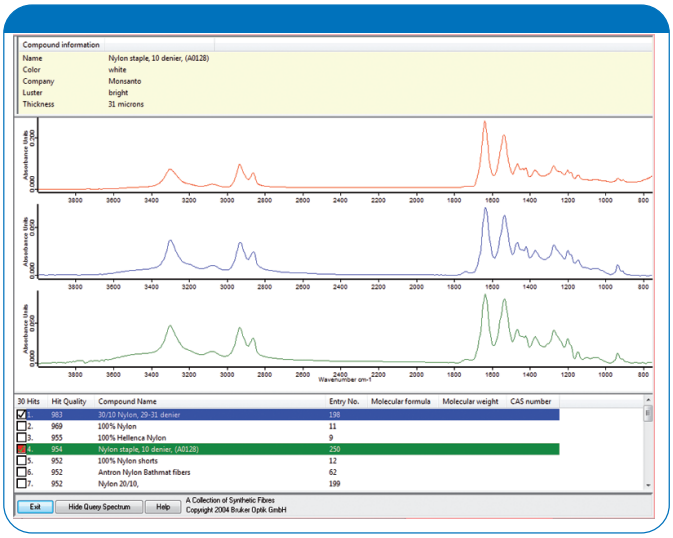


Fig.5: The measured fiber spectrum (red) is unambiguously identified as Nylon by search in the Bruker Synthetic Fibers spectra library: The first seven hits in the list originate from pure Nylon fiber references. The spectra of hit 1 (blue) and hit 4 (green) are shown for comparison.

## Example: Analysis of natural fibers

The result of the measurement of two crossed fibers is shown in figure 6 as displayed in the Chemical Imaging window of the OPUS software. The upper left "Overview" allows selection of available microscopic pictures. Zooming and navigation on the selected picture is performed using the red rectangle. The upper right "Selection view" shows the zoomed area with measurement positions, indications of used apertures and annotations. According to the color of the measurement positions the sample spectra are displayed in the "Spectra view" together with reference

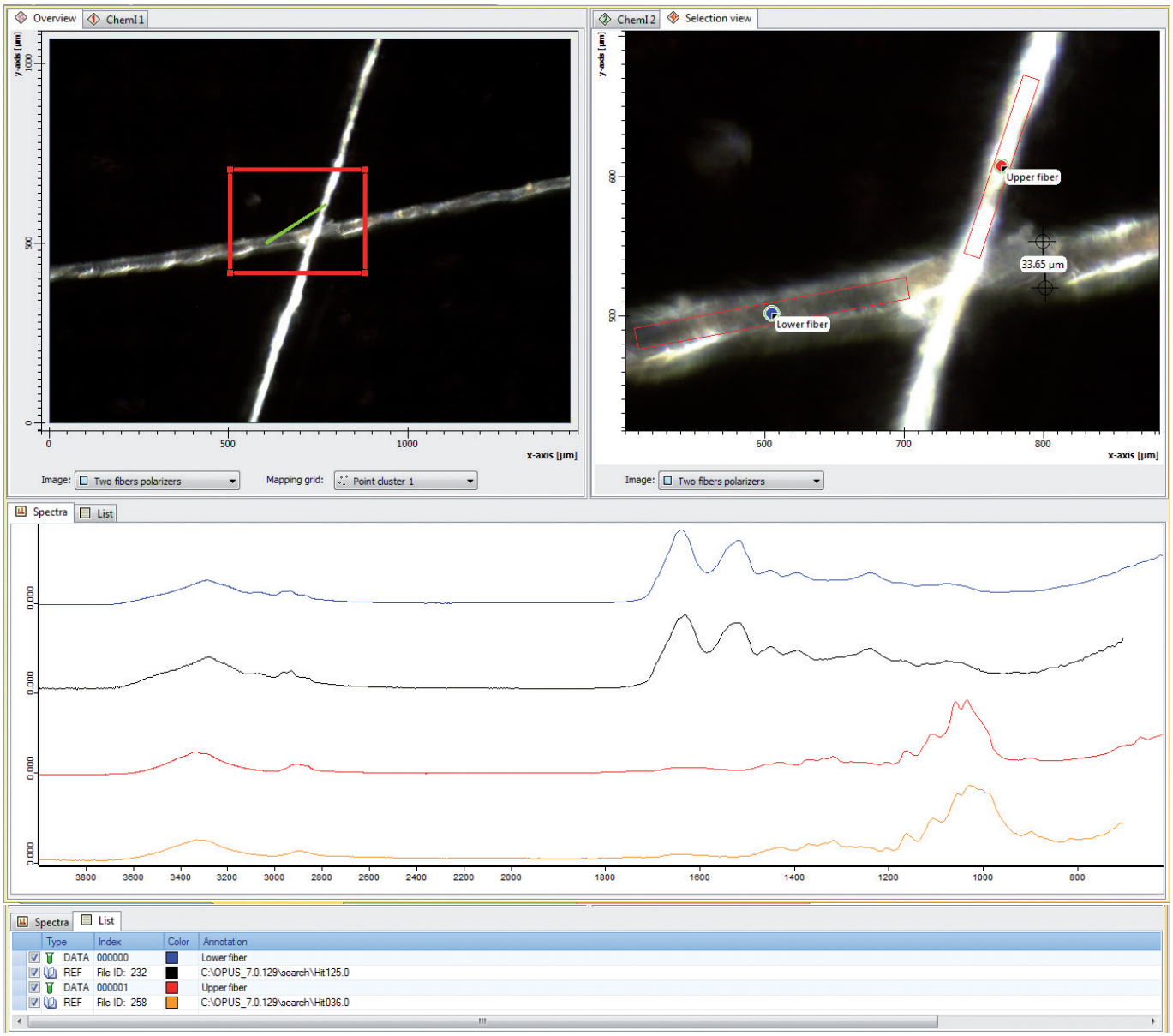


Fig. 6: The blue spectrum originates from the lower fiber, the red one from the upper fiber. For comparison reference spectra are displayed (black: wool, orange: cotton)

spectra, e.g. from spectra libraries. The IR-spectrum clearly shows that the lower fiber (blue spectrum) is made from wool (black reference spectrum) whereas the upper one (red spectrum) is made from cotton (orange reference spectrum).

### Example: Chemical Imaging of a human hair

IR-microscopy even allows detecting and visualizing chemical variances on the surface of a single fiber. This example shows the measurement of a human hair that has been partially bleached. To expand the area on which the mapping measurement can be performed and to prevent sample deformation due to the contact with the ATR-crystal the hair was flattened using a diamond compression cell prior to the measurement.

A line map was performed along the hair covering a bleached and a non-bleached (outgrown) part. The bleaching affects the fingerprinting range of the IR-spectra, e.g. bands at 1180 $cm^{-1}$ and 1040 $cm^{-1}$ increase in intensity. Figure 7 shows the chemical image based on the integration of the 1040 $cm^{-1}$ band superimposed with the visual image of the hair. With the integration intensities at each measurement position being color and size coded the image clearly shows the chemical difference between bleached and unbleached hair.

### Summary

IR-microscopy is an established technique to determine the chemical identity of synthetic and natural fibers. The imaging capabilities of the IR-microscopic method even allow visualizing chemical differences on single fibers with high lateral resolution. With the fully automated stand-alone IR-microscope LUMOS II the analysis of fibers can be performed without specific IR-spectroscopic expertise. The intuitive software guided workflow allows even untrained personal quickly performing the measurement and spectra evaluation.

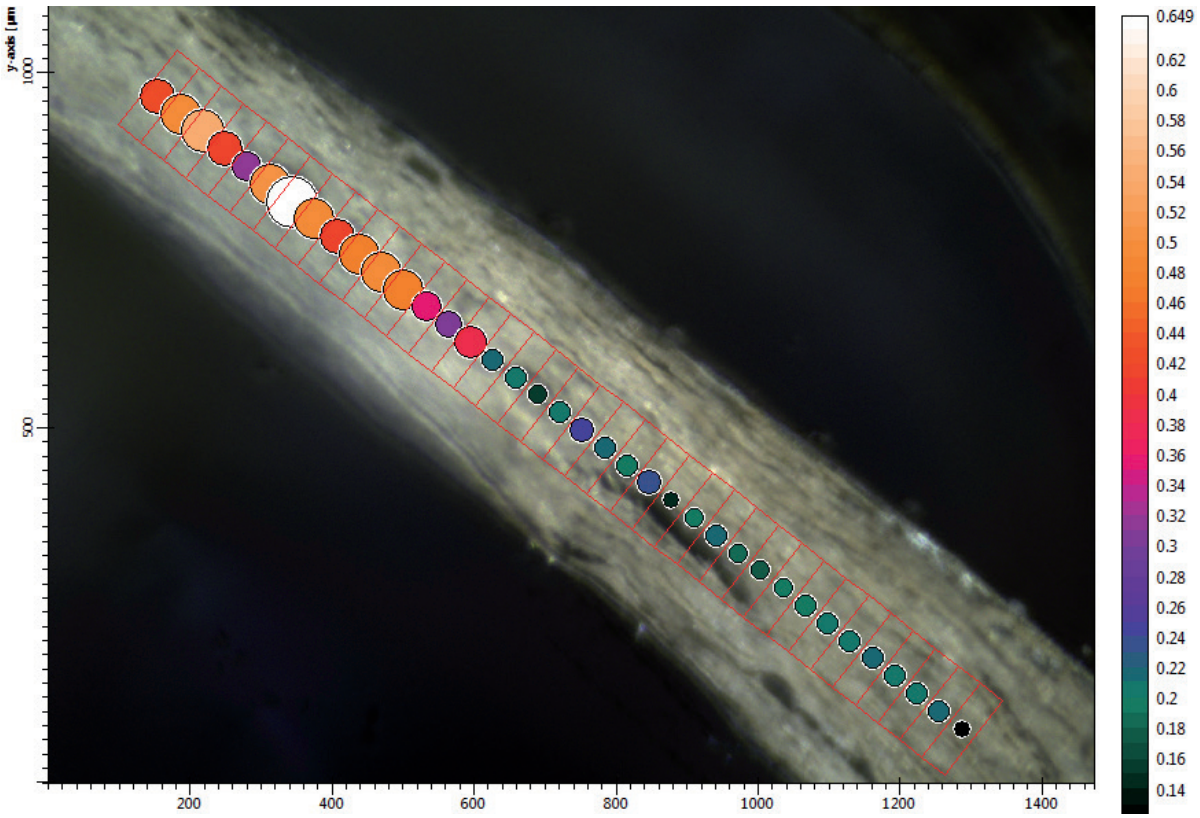


Fig.7: Line map measurement on a human hair covering bleached and non-bleached areas. The red rectangles indicate the applied lateral resolution of the measurement. The superimposed chemical image visualizes the difference between bleached and unbleached hair based on the IR-band at about 1040 $cm^{-1}$.


**Bruker Scientific LLC**
Billerica, MA · USA
Phone +1 (978) 439-9899
info.bopt.us@bruker.com

**Bruker Optics GmbH & Co. KG**
Ettlingen · Germany
Phone +49 (7243) 504-2000
info.bopt.de@bruker.com

**Bruker Shanghai Ltd.**
Shanghai · China
Tel.: +86 21 51720-890
info.bopt.cn@bruker.com

**www.bruker.com/optics**