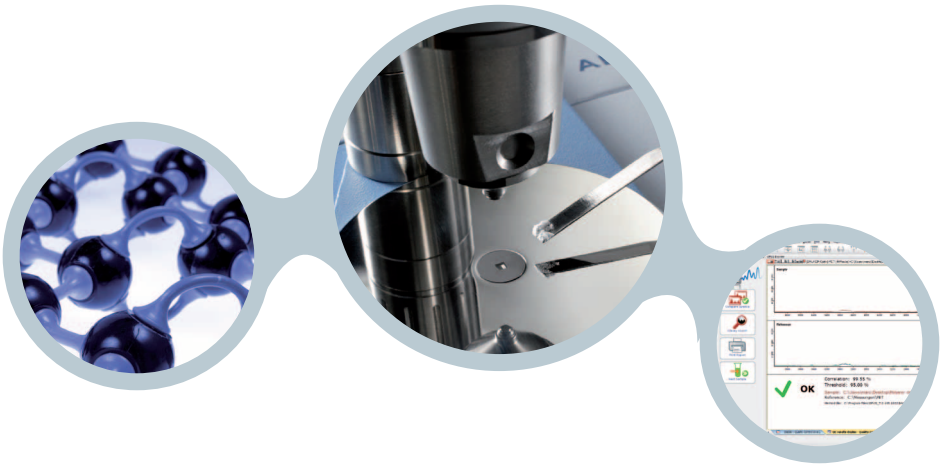

Application Note AN #84

Fast and easy analysis of polymeric samples

Introduction

Modern life would be unthinkable without a large number of polymers suitable for various applications. To obtain the optimal properties for the designated application, thousands of polymeric substances have been developed. With the introduction of copolymers and block-copolymers, the number of possible polymers grew even further and the field of application expanded equally. Therefore, the production of polymers is one of the most important fields in the chemical industry.

In order to analyse a multitude of possible raw materials, additives, flame-retardants and products, innovative measurement techniques are needed. Fourier transform infrared (FT-IR) spectroscopy is one of the fastest and most accurate methods when it comes to incoming goods inspection and quality control of raw materials and polymers respectively. FT-IR spectroscopy provides information about the identity of incoming raw materials, possible contaminations, quality of the products and can even identify unknown polymeric samples. High quality IR spectra can be recorded in a few seconds; usually there is no need for sample preparation or costs for expensive consumables. Within only a minute FT-IR spectroscopy allows to identify an incoming raw material or to verify that a given product is within its specification. The identification of an unknown polymer with the aid of the Bruker polymer library is equally easy; the library search takes just one mouse click.

Instrumentation

Nowadays, incoming goods inspection and quality control using IR spectroscopy is mainly performed applying the ATR (Attenuated Total Reflection) technique, as this is much more comfortable to use than the conventional transmission mode. Hereby the IR radiation penetrates slightly (about one micron) into the sample surface. The IR detector of the FT-IR spectrometer can then measure the absorbance resulting from the sample. All types of samples (e.g. solids, liquids, powders, pastes, pellets, slurries, fibres etc.) are just put on the accessory before the data acquisition is performed. The typical analysis takes just a minute, including sampling, measurement and data evaluation. By using ATR, it is even possible to differentiate between the top layers of a polymer laminate, something that cannot be done when measuring the sample in transmission mode.

The very compact ALPHA FT-IR spectrometer with the Platinum diamond ATR-module is a robust and affordable system that is very easy to operate. Its ergonomic one-finger clamp mechanism allows an extremely easy sampling

of solid samples. To provide the user an unobstructed access to the sampling area, the pressure applicator can be rotated by 360°. Diamond is very robust, chemically inert and therefore an ideal material for the analysis of a wide range of samples. For the measurement of highly absorbing dark samples (e.g. black polymers),the Platinum ATR module can be equipped with a germanium (Ge) crystal plate. Both plates are recognized electronically and parameter sets for most effective measurements are loaded accordingly.

The ALPHA spectrometer produces reliable and reproducible data. A permanent online diagnostics of the spectrometer by the PerformanceGuard™ provides a "real time" display of the instrument status. Instrument validation (OQ/PQ) is performed by fully automated test routines to ensure permanent instrument operation within specifications. Furthermore the OPUS software is fully compliant to cGMP and 21 CFR part 11 when operated in a validated environment.

Figure 1: ALPHA-P with diamond ATR

The measurement process itself is very comfortable by means of a specially designed wizard. The user is guided through the analysis procedure by a dedicated software wizard. Even untrained personal is able to measure and evaluate a sample in less than a minute. During the measurement and evaluation process, the Wizard changes its appearance dynamically presenting the functionality to perform the next step. As an example figure 2 shows the appearance of the user interface after two measurements have been completed and analyzed. The Wizard, located on the left side of the OPUS window, now offers the possibility to measure a new background or to proceed with the measurement of a new sample.

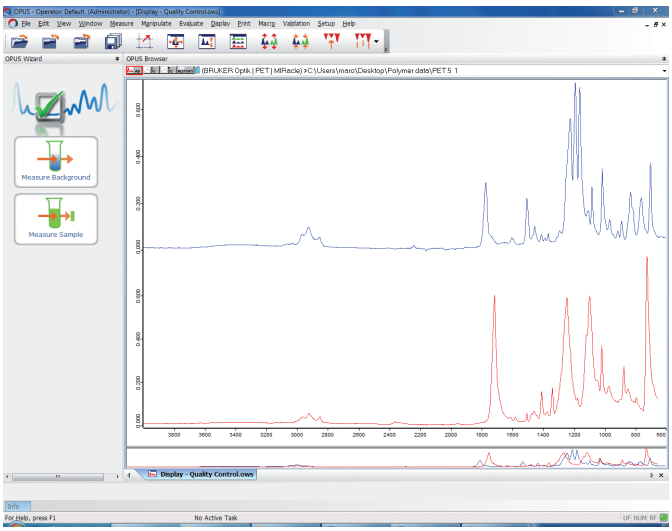

Figure 2: OPUS user interface with the Wizard bar on the left.

1. Polyethylene terephthalate (PET) is a thermoplastic with one of the widest fields of application. It is commonly used as a material for beverage, food and other liquid containers. During incoming goods inspection its identity has to be confirmed and its possible contamination with other polymers has to be excluded.

- First the background spectrum of the clean ATR unit is measured by pressing the "Measure Background" button of the QC-Wizard:

- Then the sample (sheet, foil, powder, liquid etc.) is placed on the ATR crystal. The "one finger" clamp is applied on solid samples to ensure good contact to the ATR crystal.

- Measure the spectrum by a click on the "Measure Sample"-button:

After the measurement, the following buttons are shown on the QC-Wizard bar:

For spectrum evaluation, quick compare or a library search can be selected. Furthermore printing of the spectra or the result pages can be easily done via the "Print Report"- button. The "Next Sample"-button starts the analysis of another sample.

The Quick Compare function compares the sample against references; either one single spectrum, an average of spectra or against many different single spectra. When a comparison against one single spectrum or against an average of spectra is selected, the comparison result will be an "OK" or a "Not OK" of the sample. The high correlation of the sample spectrum with the reference data well above the set threshold confirms the identity of the sample to be PET (Fig. 3).

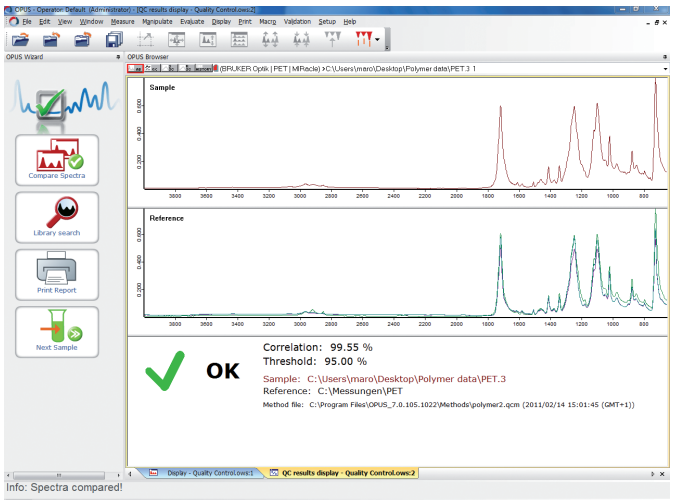

Figure 3: Comparison with an average spectrum of the directory containing previously measured PET-spectra.

2. If the question arises: „Is this sample a polycarbonate / acrylonitrile butadiene styrene-blend (PC-ABS), pure polycarbonate (PC) or cellulose acetate (CA)?" a comparison against a set of polymer spectra that come into consideration is performed. To set up the method just the data path containing the files of the reference spectra has to be selected. The method results in a hit list, indicating those results that are within the desired correlation limit. Results below the threshold are greyed (see Figure 4). In the shown example, the sample clearly is assigned to be PC-ABS.

3. Completely unknown polymers can be identified via a library search. The Bruker ATR-Polymer Library contains many commercially available plastics and blends. Of each compound, the spectrum was recorded with both a Ge- and a diamond-crystal. In the example different entries for the system PC / ABS are on the first six positions of the hit list clearly identifying the sample (Figure 5).

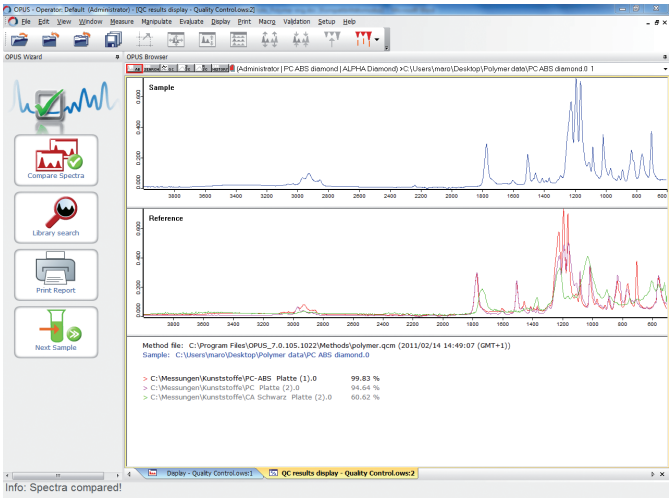

Figure 4: Comparison to several different spectra; hits below threshold (hit #2 and 3) are greyed indicating that only the first hit is correct.

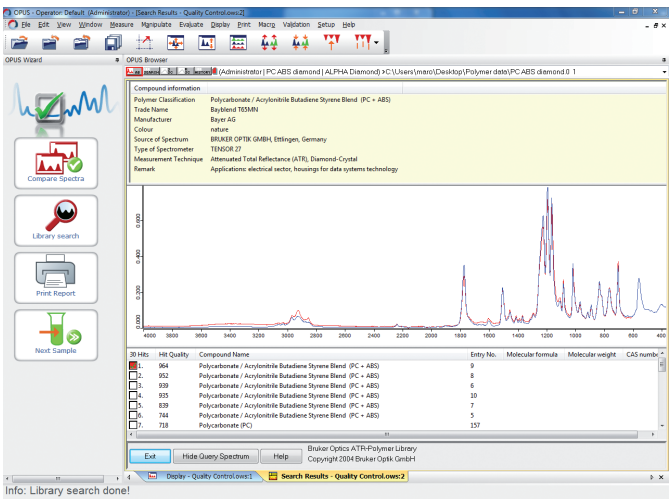

Figure 5: Library search using the Bruker Optics ATR-Polymer Library. The result shows that the sample is a polycarbonate / acrylonitrile butadiene styrene-blend.

A click on the "Next Sample"-button will close the result windows and the QC-Wizard will offer the user to measure a new sample or to measure a new background first.

Measurement of dark colored samples

Highly absorbing substances like carbon black filled rubbers are difficult to measure with a diamond ATR crystal. When using diamond with its rather low refractive index (2.4) the penetration depth into the sample is too high, resulting in spectral artifacts. To overcome this limitation Germanium (Ge) is used as an ATR-crystal, offering a high refractive index (4.01) in combination with a wide spectral range and high chemical resistivity. Therefore it constitutes an ideal material for highly absorbing samples. Figure 6 shows two spectra of a sample measured with a diamond- (top) and a

Ge-crystal (bottom). The spectrum using the Ge-ATR is free from derivative like artifacts that are present when using a diamond crystal and shows much more pronounced single bands.
Due to their low penetration depth, Ge crystals are also very suitable for measurements of thin surface films with high sensitivity.

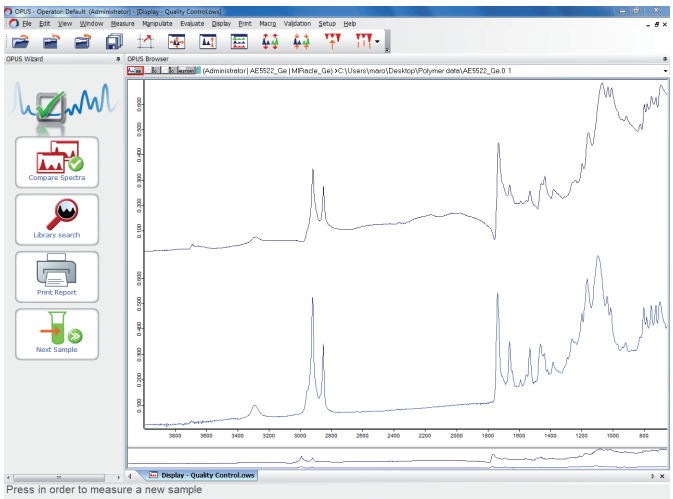

Figure 6: Comparison of a highly absorbing sample measured with a diamond crystal (top) and a germanium crystal (bottom).

Summary

The combination of the compact and robust FT-IR spectrometer ALPHA with a dedicated software wizard, OPUS quick compare and comprehensive spectra libraries results in a reliable and user friendly analysis system for polymers and their raw materials. User specific quick compare methods are set up easily even with just one reference spectrum. Hence, this function is the ideal tool for incoming goods inspection and product quality assurance. Applying a spectrum search on a polymer library, the identification of an unknown polymer sample is a matter of seconds.
Bruker Optics FT-IR-spectrometer ALPHA with a diamond ATR module offers a quick and robust measurement method. Due to the easy handling of the ALPHA instrument and wizard-guided measurement, evaluation and reporting can be performed even by untrained users.

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