Application Note AN M80

Quality Control with Infrared Spectroscopy

When it comes to up-to-date testing of the chemical nature of materials, Fourier Transform Infrared Spectroscopy (FTIR) has established itself as one of the most important analytical methods. Its speed and simplicity make it the method of choice in many applications. The technique is equally suitable for incoming goods inspection, failure analysis, competitive analysis, process- and quality control. The measurement can be performed within a short time and without any sample preparation or the use of consumables.

Stichwörter	Geräteausstattung und Software
Quality Control	ALPHA II
Materials testing	OPUS-TOUCH
Polymer analysis	Platinum ATR
Surface Analysis	Front-Reflection Module

Many industrial sectors now rely on FTIR spectroscopy and over the years it has completely replaced a number of complex physical and wet-chemical methods of analysis. It is widely used in the field of plastics processing, pharmaceuticals, chemical and petrochemical industries, as well as in automotive and electronics sectors. Moreover, new and exciting fields of application are constantly being defined.

The focus of incoming goods inspection is the identity check of materials and to test if they are compliant to reference standards or whether they show deviations (for example due to contaminations). Multi-component products are tested for their composition to ensure that the formulation is within defined limits. All these tasks are conveniently performed by failsafe FTIR spectroscopy in just a few seconds.

Measurement Principle

Infrared spectroscopy makes use of the invisible thermal radiation's property to stimulate molecular or lattice vibrations in the irradiated matter. This vibrational excitation results in the absorption of the incident light at certain wavelengths that are dependent from the absorbing medium. The FTIR spectrometer measures the absorbed energy as a function of the wavelength and provides a spectrum in which the absorption intensities are plotted against the wavelength. Since the number, relative intensity and location of these absorptions (bands) are substance-specific, this spectrum can be used for identification or for answering quantitative questions. Modern IR spectrometers make it possible to record and evaluate a spectrum in a matter of seconds.

Figure 1: ALPHA II FTIR spectrometer with touch panel PC and Platinum ATR-module

Instrumentation

Since it is much more comfortable, the Attenuated Total Reflection (ATR) has almost completely superseded the traditional transmission measurement in incoming goods inspection and quality control. All types of samples (e.g. powders, pastes, fibers, components, liquids, solutions) are just put on the ATR unit and then measured directly. As a chemically inert and above all robust material, the diamond used as an ATR measuring element allows the analysis of a wide range of samples.

Due to the modular principle of the ALPHA II FTIR spectrometer (see Figure 1), only a few steps are needed to replace a measuring module and optimally adapt the spectrometer to almost any requirement. In addition to the aforementioned ATR configuration, for example, modules for the measurement of reflection or transmission are available. These are typically used, when a contactless analysis of surfaces is required or when low-concentration components of a solution have to be quantified.

The ALPHA II is the next generation of its extremely successful predecessor. In addition to a higher measuring sensitivity, a temperature-stabilized detector and the electronically controlled CenterGlow™ source, the ALPHA II is characterized above all by the integrated touch panel. Due to the complete integration of spectrometer and user interface, the ALPHA II is particularly portable and self-explanatory in its operation. Tailored to the integrated touchscreen, the OPUS-TOUCH software guides the user intuitively through the measurement and evaluation process. It allows spectra to be easily measured and subsequently evaluated via a library search, a fast spectra comparison or a quantification method.

The ergonomic one-finger pressure mechanism of the Platinum ATR module simplifies the fixation of solid samples on the crystal. The freely rotatable pressure stamp provides the user unrestricted access to the measurement area, in order to comfortably place samples on the crystal and thoroughly clean it after the measurement.

Incoming goods inspection

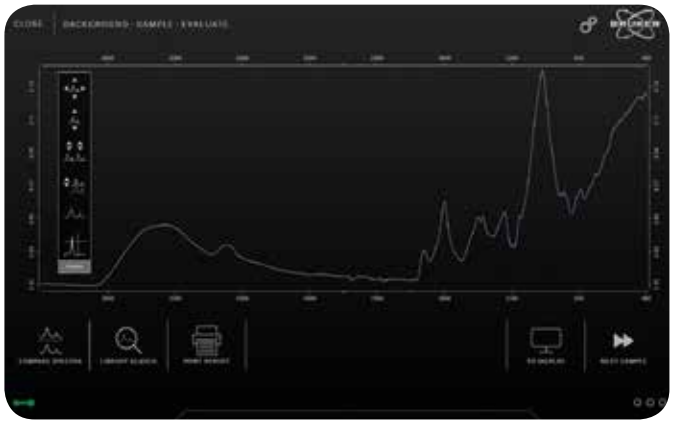
Xanthan is a naturally occurring polysaccharide which is used, for example, as a thickening agent in the food and cosmetics industry. The incoming raw material needs to be thoroughly checked for any mixing or contamination with other white powders, while its identity must be unambiguously confirmed. The central question is whether a sample has the expected identity and composition. The quick spectra comparison of OPUS-TOUCH has two different comparison models available:

- Verification:
  Check whether the sample spectrum corresponds to a selected reference spectrum of the spectral comparison method
- Identification:
  Compare the sample spectrum against all spectra inside the selected method.

For measurement, a background spectrum is first taken with a clean ATR crystal. Then a small amount of the powder is placed on the ATR crystal and pressed with the pressure mechanism onto the ATR crystal to ensure good contact between sample and crystal.

A preview spectrum indicates whether the crystal is clean and the sample is in good contact with the crystal

After the measurement the complete spectrum is shown together with the evaluation possibilities for spectra comparison and identification.



A short touch shows the evaluation result: The Xanthan is well within specifications and can be accepted for further processing.
With „Next Sample“ the measurement for the next sample can be started.

Polymer Analysis

The FTIR spectroscopy is an established method for the analysis of polymers and plastics. It can be used for the identification of different polymer types, additives and fillers and even allows to differentiate between different polymer subtypes (like HD- and LDPE or PA6, PA6.6 and PA12). Furthermore, it is possible to quantify parameters like density, crystallinity or additive- and filler-content. In figure 2 the ATR spectra of three colorless plastic pellets from unlabeled containers are shown. The measuring process is very simple. Although the plastics look very similar from the outside, the different types of polymers are clearly distinguishable by their IR spectra, even for the untrained eye. Thanks to very extensive and dedicated spectral libraries, unknown substances are automatically identified within seconds. (See Figure 3).

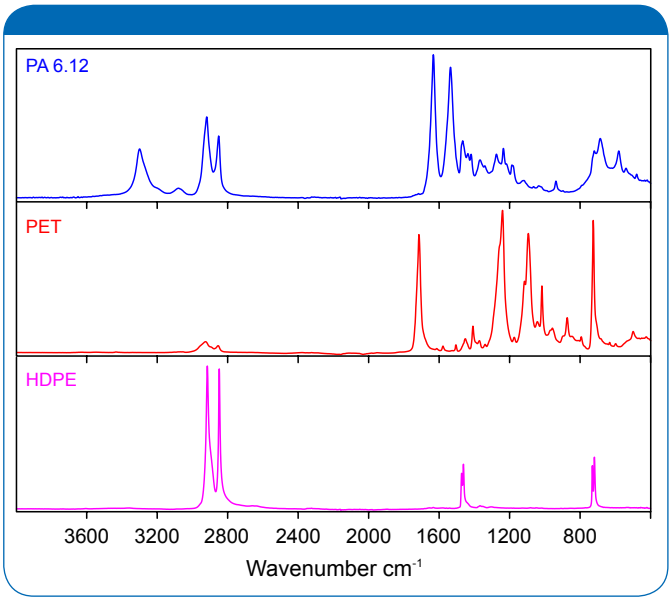


Figure 2: ATR spectra of different polymers

Surface analysis of metals

Surface coatings require absolute clean and dry work pieces devoid of remainders like fat, oil or salts. Already small amounts of these surface impurities will result in a poor adherence of the coating substance with all the resulting negative impacts on the product quality. FTIR spectroscopy using diffuse and specular reflection provides a fast, easy and reliable solution for the detection of surface contaminations on metallic samples like gear wheels or metal sheets. With the aid of spectra libraries and automated search functions, it is also possible to identify the contamination. This information can be very helpful for the determination of suitable cleaning agents or the identification of possible causes of the impurities.

Figure 3: Identification result in OPUS-TOUCH after a search in spectral libraries

The following example is a metal sheet whose surface purity was determined by an ALPHA II measurement with the front reflection unit. Due to the extended focus, this module also allows measurements on very large or difficult to access samples.

Figure 4 shows the spectrum of the contamination (blue) and the reference spectrum found by spectra search (polymethylhydrosiloxane, PMHS). Obviously, the metal surface was contaminated with a very thin layer of silicon oil that was invisible to the naked eye.

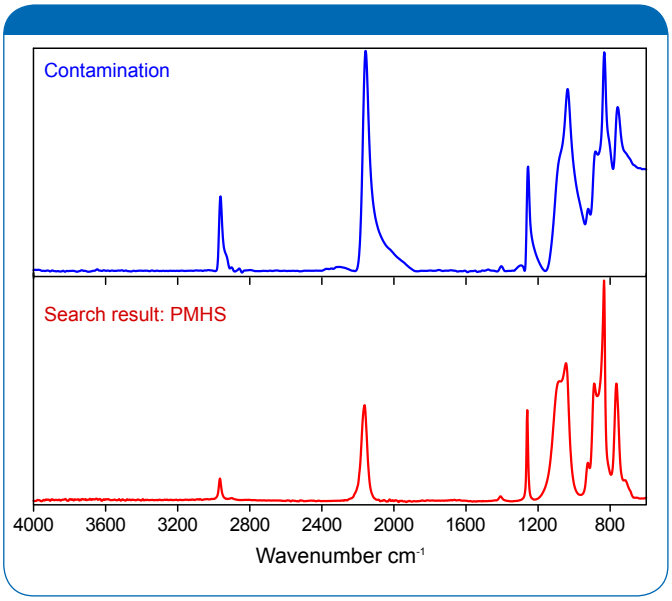


Figure 4: Spectrum of the contamination (blue) measured with the front reflection module and reference spectrum (red) found via a library search.

Summary

FTIR spectroscopy can be used in many ways in the field of materials testing. It quickly and easily provides both qualitative and quantitative results for a wide variety of sample types. Due to user friendly touch operation in the most modern spectrometers it is now possible for untrained users to perform a measurement and evaluation without training.

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