BRUKER

Analysis of Polymers and Plastics

- Quality Control & Failure Analysis

FT-IR

Innovation with Integrity

Reliable quality control is essential to achieve a cost-saving production of high quality plastic products. Bruker's ALPHA II provides a FT-IR spectroscopy-based solution for powerful routine quality control of incoming goods, intermediates, and final products.

Coupling an ALPHA II or the versatile FT-IR platform INVENIO to a thermobalance enables advanced material characterization such as thermodynamic transitions, chemical reactions or thermal stability analysis.

In case of any product defects an effective failure analysis is required to understand the source of error. The FT-IR microscope LUMOS II is a valuable analytical tool for troubleshooting as it allows to determine the chemical composition of smallest structures.

You Analyze:

- Raw materials
- Plastics
- Additives
- Fillers
- Rubbers
- Textiles
- Multilayers
- Pellets
- Monomers
- Auxiliaries
- Composites
- Elastomers
- Fibers
- Films

To Learn About:

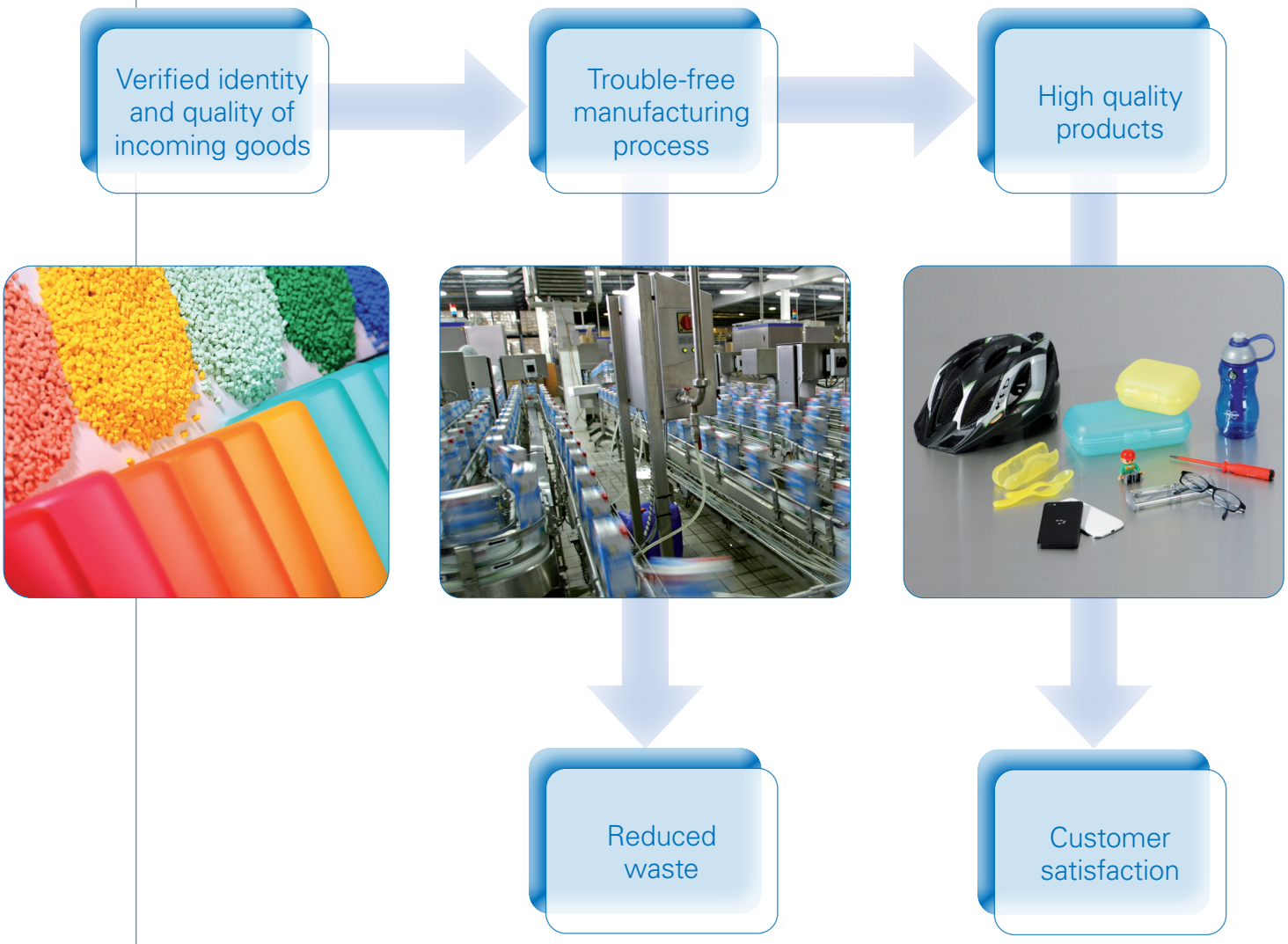
- Quality
- Quantity
- Contaminations
- Root-cause
- Dehydration
- Decomposition
- Solid-gas reactions
- Identity
- Composition
- Particles
- Inclusions
- Outgassing
- Ageing
- Pyrolysis

Quality Control for Cost-Efficiency

Plastics are used in countless products such as automotive parts, packaging materials, home appliance, electric devices, or textiles.

Many of today's plastics are complex multi component systems made from various compounds like different polymers, fillers, and additives. The systematic selection and blending of these constituents in appropriate mixtures results in materials with optimized properties.

As a high product quality at low price is a basic requirement in modern industry, reliable quality control is mandatory. An essential step for a trouble-free manufacturing process without waste is to verify the correct identity of the incoming raw materials. Later, the correct composition in the final plastic product must be checked to ensure the right properties.



Fast and Easy Identification

Chemical Fingerprint of the Sample

Using Bruker's Fourier Transform Infrared (FT-IR) spectrometer ALPHA II (Fig. 1) the identity and correct composition of any plastic product or raw material can be checked in less than one minute.

The IR-spectrum of any sample reflects its molecular composition – just like a chemical fingerprint (Fig. 2). Both organic and inorganic chemical components contribute to the sample spectrum. Therefore, the IR-method is very suitable to identify as well pure compounds as complex materials. Quantification of individual components inside the analyzed material is feasible.

Fig. 1: Measurement of a plastic spoon using the ALPHA II with ATR measurement interface.

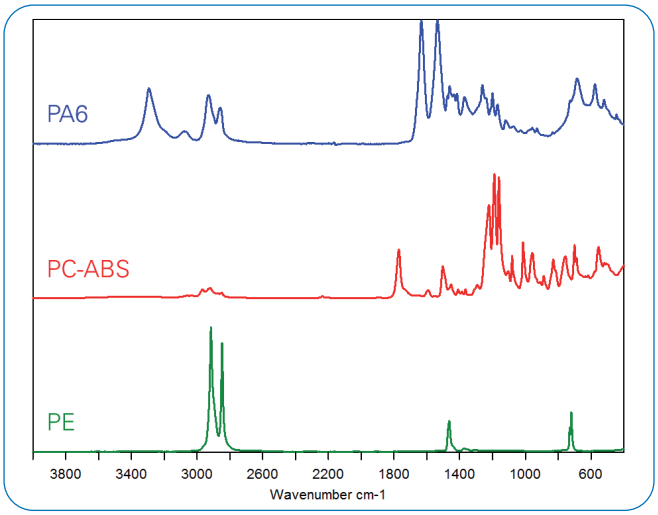


Fig. 2: The IR-spectrum is a chemical fingerprint that allows polymer identification.

Simple and Fast Analysis

The measurement interface of the ALPHA II is a diamond ATR crystal. Its mechanical and chemical robustness allows as well the analysis of reactive liquid samples like monomers as of very hard plastics like polycarbonate. The ATR (Attenuated Total Reflection) technique is very comfortable and fast as there is virtually no sample preparation required. No matter if the analysis is performed on pellets, films, plastic parts, powders, or liquids: For recording the IR spectrum the sample just must be brought into contact with the ATR crystal.

After the measurement the sample is identified by automatic comparison of its spectrum against spectral data from reference materials.

Prepared for Your QC Demands

The ALPHA II is a very compact system with a small footprint (A4 format) and light weight (<7 kg). Being insensitive to vibrations it can be placed almost anywhere and moved easily to relevant places. The option to operate the ALPHA II with an integrated touch panel pc and powered by a battery allow its use even outside the laboratory, e.g. in the warehouse or near production sites.

All hardware components are continually monitored for correct functionality. Fully automated instrument test procedures for Operational and Performance Qualification (OQ/PQ) are performed to ensure permanent instrument operation within specification. Furthermore the OPUS software is fully compliant to cGMP regulation.

IR-Advantages

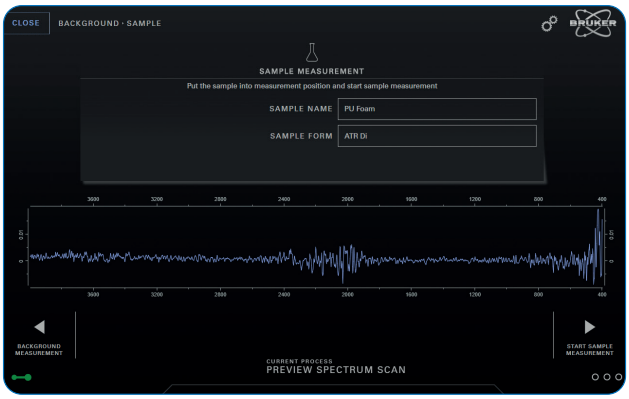
For most samples the FT-IR analysis is performed without sample preparation and without the need of any consumables. Measurement times are typically below 1 minute. Therefore, FT-IR spectroscopy saves your time and cost when being compared to classical wet chemical methods.

The ALPHA II and LUMOS II are products that are designed to be used for many years. Both instruments utilize modern high quality optical components with a guaranteed long lifetime. Adding the low energy consumption these outstanding characteristics result in minimal running costs.

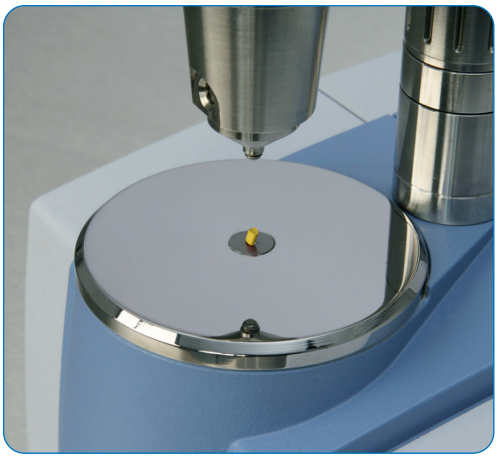
Intuitive Workflow

The ALPHA II user interface enables even untrained operators to verify the identity of a known sample or to identify unknown samples. Guided by the software the user performs measurement, evaluation and reporting in a few steps:

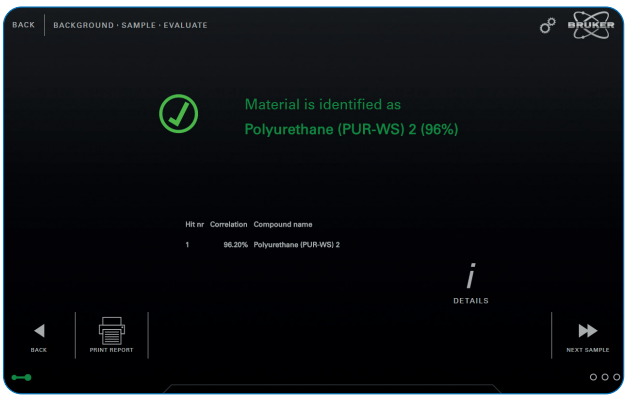
1 Measure background spectrum of the empty ATR measurement interface. Enter sample information.



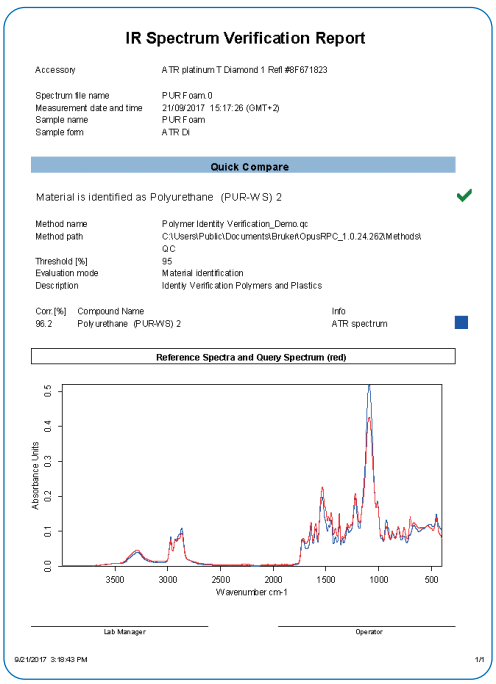
2 Place sample on ATR measurement interface and apply pressure clamp. Start measurement with a single click.



3 Verify the identity of the measured sample by automatic comparison of the sample spectrum against references.



4 Generate a meaningful analysis report by a single click before proceeding with the next sample.



Failure Analysis

The failure of polymer and plastic materials often is caused by the inhomogeneous distribution of the used components inside the polymeric material. Also contaminations like particles, fibers or inclusions may be the reason for its failure. In case of composite materials defect layers or a layer made from the wrong material will have a negative impact on the product properties.

As such defects are often extremely small. They are hard or even impossible to analyze by a macroscopic measurement. However, a successful failure analysis includes the chemical analysis of the faulty region in the sample.

The FT-IR microscope LUMOS II (Fig. 3) is a powerful tool for failure analysis: It allows to obtain IR-spectra anywhere on the sample with high lateral resolution and thereby to reveal the chemical composition of this particular area of the sample.

Due to its outstanding ATR performance the LUMOS II is capable to analyze the vast majority of samples without any sample preparation.

Fig. 3: FT-IR microscope LUMOS II

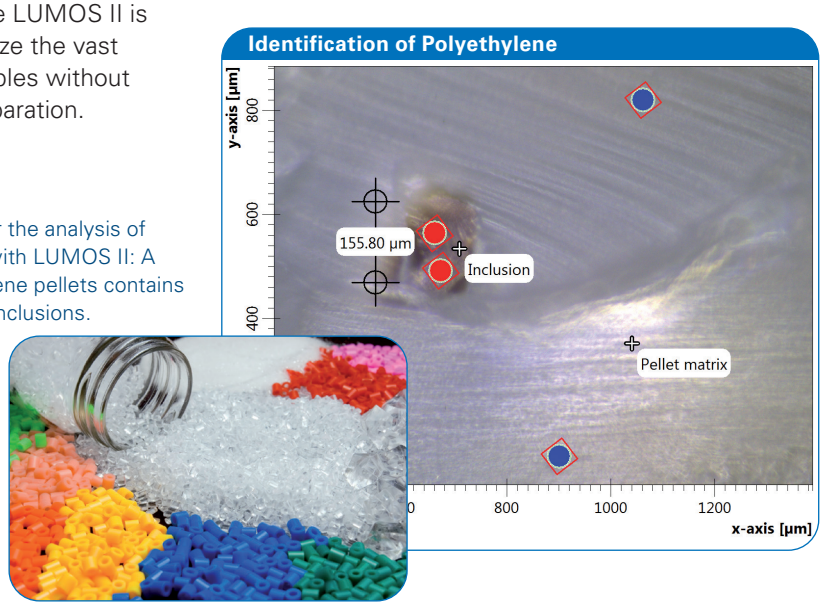


Fig. 4: Example for the analysis of a product defect with LUMOS II: A batch of polyethylene pellets contains unwanted brown inclusions.

Fig. 5: The microscopic visual image of a polyethylene pellet with brown inclusion is shown together with the positions of the IR-analysis.

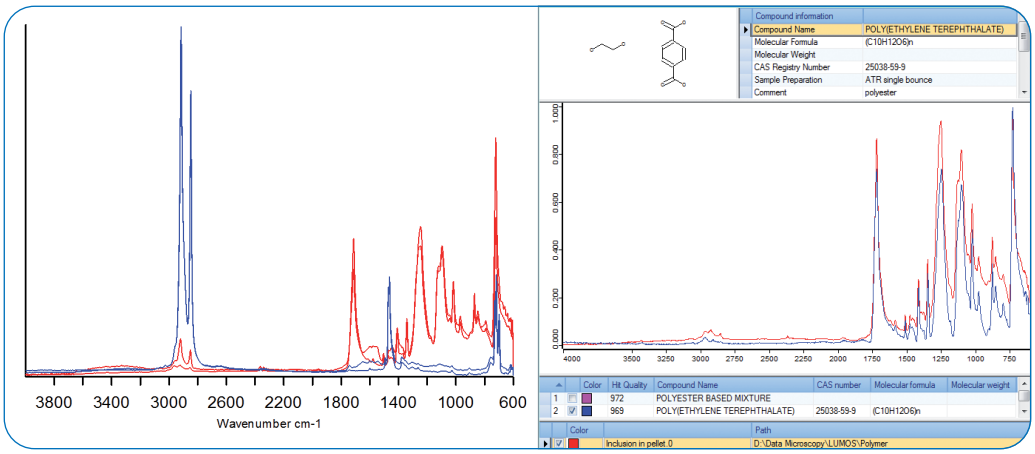


Fig. 6: The IR-spectra show clearly different characteristics on the PE pellet matrix (blue spectra) and on the unknown inclusion (red spectra). Search in a spectral library identifies the inclusion to be a polyester (PET).

Avoiding Product Defects

The failure of polymer and plastic products is most effectively avoided by a good product design, correct material selection and an appropriate production process. During product development many variables influencing plastic properties have to be considered to ensure a high quality of the final product.

FT-IR Microscopy Made Easy

LUMOS II is a compact stand-alone system with full automation of all hardware components. The intuitive software of the LUMOS II guides the operator step by step through the process of data acquisition. At each step the user interface only provides these functions appropriate to proceed. Although the LUMOS II is designed to be operated by non-experts for routine applications, its exceptional sensitivity makes it also very suitable for high demanding applications.

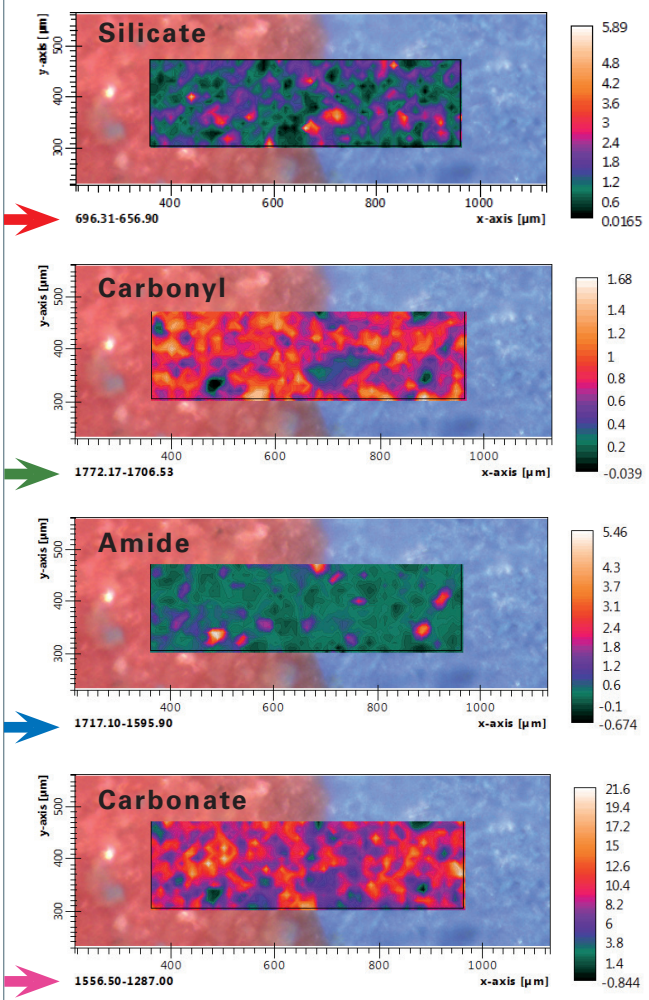
Comfortable Sampling

The LUMOS II provides plenty of space for the sample and a large working distance. In combination with the good accessibility of the sample stage a very convenient sample positioning is achieved.

The large field of view and the high visual quality eases finding the region of interest on the sample.

LUMOS II generates precious information regarding product quality:
Measurements with a local resolution in the micrometer range allow to characterize the composition of plastic materials. Mapping measurements on the sample reveal the distribution of individual components, e.g. the basic polymer, fillers, and plasticizers. The impact of variations in the processing conditions on the material homogeneity can therefore be determined. Events of self-contamination (e.g. due to partly melted granules) are easily detected.

Moreover, LUMOS II is a helpful tool in reverse engineering as it provides information about the composition of products which are already in the market.



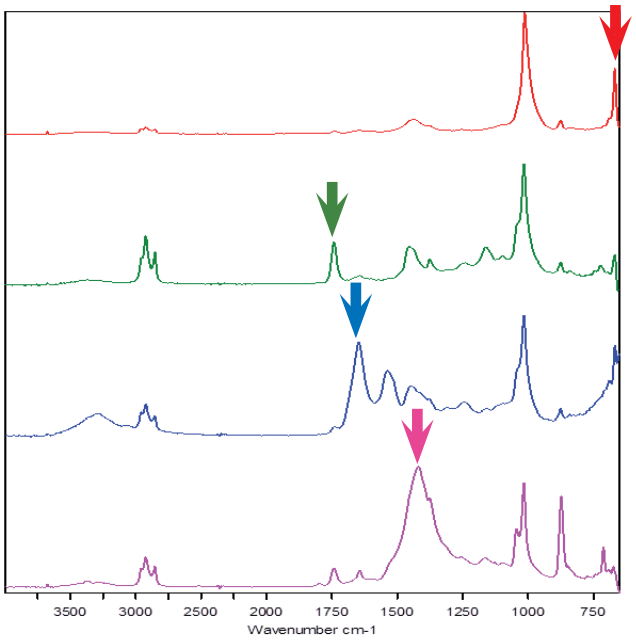


Fig. 7: Microscopic analysis of a rubber eraser. Chemical images on top of the visual sample image show the distribution of individual organic and inorganic components. The concentration is indicated by color coding (white/orange=high; black=low). The chemical images are generated by integration of spectral bands which are specific for a certain component of interest (see indications on selected sample spectra).

Excellence meets Integrity

Optimal measurement equipment for TG-IR analysis originates from our close collaboration between NETZSCH - providing the thermo-balances and Bruker - providing the FT-IR spectrometers. Interface parts like the gas cell, the transfer line or the SW are adopted to each other to guarantee a flawless measurement procedure with highest accuracy and reliability.

Thermal Analysis combines thermal gravimetric methods with IR data of the released gases to provide crucial insights into chemical processes. This way we gain detailed knowledge about thermodynamic transitions, thermal stability, decomposition processes and chemical reactions.

Instrumentation: Full integration with ALPHA II

PERSEUS® series (Fig. 8) offers a very high level of integration such that no transfer line is needed. This allows for a combined system with a small footprint smallest gas cell path length and highest ease of use. The integrated ALPHA II of the PERSEUS® system can be detached and used for stand-alone FT-IR measurements.

Figure 8: PERSEUS® TG 209 ***F1***

Instrumentation: Full flexibility with INVENIO

Various thermobalances with either internal or external gas cells can be coupled to an INVENIO FT-IR spectrometer (Fig. 9) via a transfer line. This combination offers the highest flexibility and performance for TG-IR analysis. Besides doing thermogravimetric measurements the INVENIO spectrometer also allows for independent FT-IR measurements. A multitude of different measurement methods is available by incorporating either various accessories or external modules such as microscopes. Our spectrometers can also be coupled to 3rd party suppliers of thermogravimetric measurement equipment.

Advanced Material Characterization with TG-IR analysis

Operation workflow with Proteus single-point device control SW

Gravimetric and IR measurements are defined and conducted solely via the NETZSCH Proteus® software. The user only needs to input the desired measurement parameters, start the measurement and the devices will be readied with parameter inputs.
Online data collection is simultaneous to guarantee precise time and temperature correlation between all signals from the two coupled instruments for evaluation. The user operates the two software packages from a single computer with access to all SW features from both OPUS and Proteus® SW for data evaluation and results display.

Thermal Analysis

Combined analysis of thermogravimetry (TG) and FT-IR spectroscopy provides all information for chemical identification of polymer decomposition or outgassing over a broad temperature range. TG properties are recorded simultaneously with IR spectra while increasing the temperature of the polymer sample.

Sample Analysis: EVA

Fig. 10 shows the TG data (black curve) together with the IR spectra at various temperatures for an ethylene-vinyl acetate (EVA) sample. As seen from the TG data decomposition takes place in two steps starting at about 300°C. Cutting out spectra at highest mass loss rates and comparing with polymer libraries enables to identify the outgassed substance - in this case we find acetic acid at 350°C and the polymer backbone itself at 470°C.

Figure 9: INVENIO with external gas cell coupled to STA 449 ***F1*** *Jupiter®*

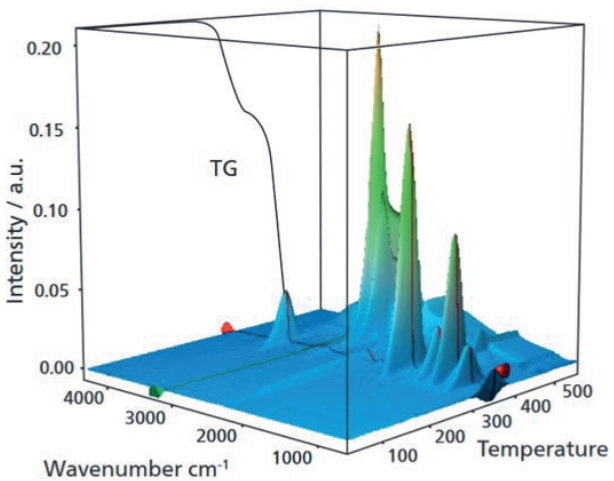


Figure 10: 3D plot of the TG data (black curve) together with IR spectra at respective temperatures taken from a measurement of ethylene-vinyl acetate (EVA).

Dedicated Applications

Differentiation of Polyamides

Polyamides are thermoplastic polymers consisting of monomers joined by an amide bond. The polyamide group includes many different polymers with different chemical and physical properties. Despite a high similarity in their chemical structure IR spectroscopy allows to differentiate between various polyamides like PA6, PA6.6, PA10 and PA12.

Quantification of Fillers, Additives and Blends

Determining the correct composition of plastics is an essential part of quality control. The IR spectrum not only allows the identification but also the quantification of individual components (see Fig. 11). Even the quantification of inorganic fillers is possible with FT-IR spectroscopy when the analysis is expanded to the FIR, e.g. with the optional BRUKER FM package.

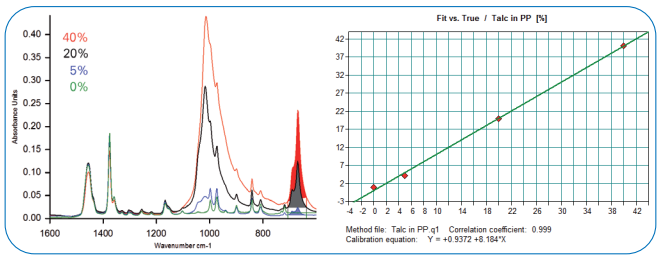


Figure 11: Quantification of talc in polypropylene.

Determination of the Degree of Cure

The curing process has a dcisive impact on the properties of a varnish. FT-IR spectroscopy detects the degree of curing in paints and coatings reliably, quantitatively and within one minute (see Fig. 12).

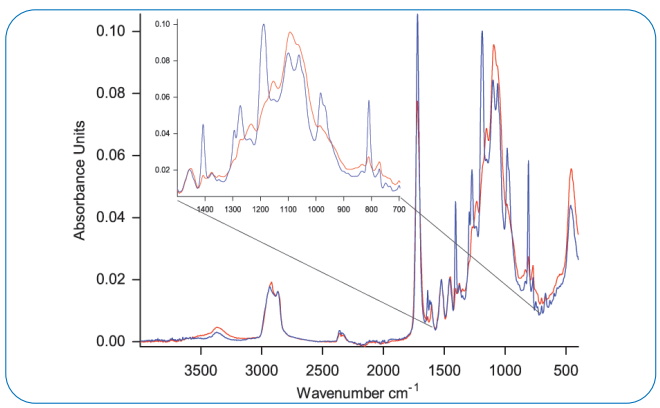


Figure 12: IR spectra of an acrylic varnish before (blue) and after curing (red).

Analyzing Composite Polymer Materials

Creating polymer composites from commercially available raw materials permits a variety of product properties to be defined. Thus, quality control is key with these complex materials, as the bonding of polymers may give rise to product defects, inclusions, contamination and inhomogeneities. IR micro analysis is ideal to identify the constituents (see Fig. 13) and detect material weaknesses.

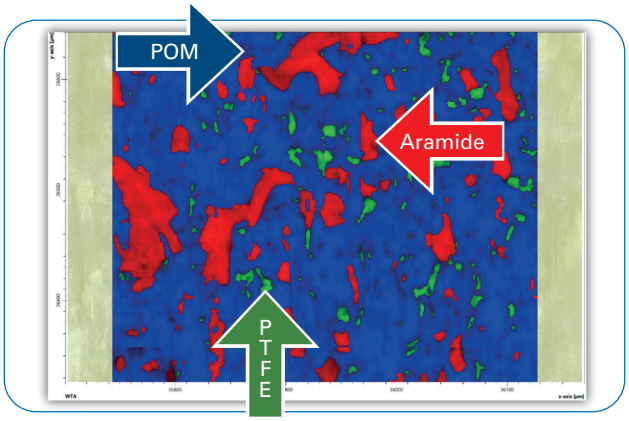


Figure 13: Micro-FT-IR image of a polymer composite material with chemical detection of POM, Aramide, and PTFE.

Analysis of Multi-layered Packaging

Multilayer packaging ensures the integrity of many products, be it food or pharmaceuticals. The development of such high-performance films is a costly process involving many stages. IR microanalysis gives users insight into the structure of such polymer laminates and provides key information on quality, production and efficiency of these products (see Fig. 14).

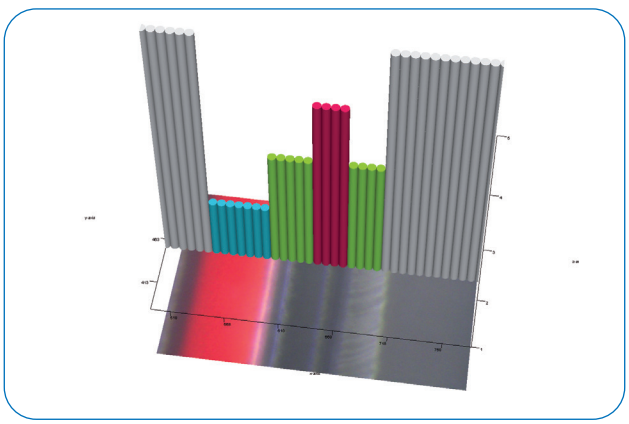


Figure 14: Color coded layer identification and thickness analysis of a cross section of a packaging film.

Outgassing Detection and Control

External environmental conditions such as elevated temperature or the presence of other chemicals can lead to outgassing of polymers.

While regulated outgassing is utilized to obtain final product properties, unintentional outgassing from polymers can deteriorate product properties or even pose health risks.

TGA-FT-IR analysis was applied (see Fig. 15) to learn more about the outgassing behavior.

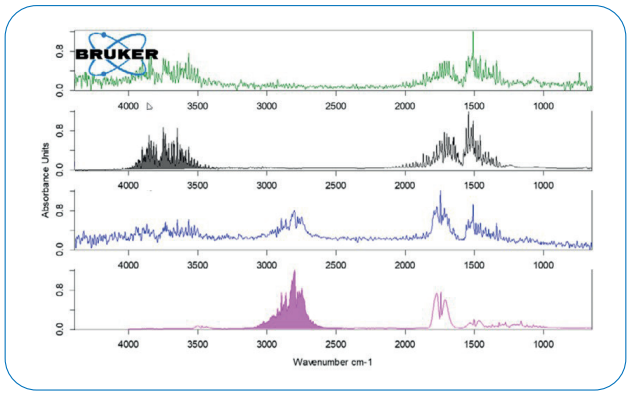


Figure 15: IR spectra taken at 210°C of two kinds of polyoxymethylene during TGA-FT-IR analysis (green and blue curves) together with library spectra from the hazardous substance formaldehyde (pink) and water (black).

Analysis of Polymers and Plastics

with our FT-IR (micro) Spectrometers

ALPHA II

A small, compact benchtop system that can be setup anywhere in your laboratory or even in production. It focuses on ease-of-use, robustness and reliability. Use it for:

- **Quality Control**
- **Analytical Service**
- **Teaching**

LUMOS II

Our fastest FT-IR (imaging) microscope. It provides maximum sensitivity for IR applications and full automation down to the evaluation. Use it for:

- **Quality Control**
- **Root Cause Analysis**
- **Product Development**

INVENIO

Our universal FT-IR platform. With its performance and versatility, it covers research and product development tasks as well as QC/QA applications with high throughput. Use it for:

- **Research and Science**
- **Product Development**
- **Advanced QC/QA**

Experts in Spectroscopy

With decades of experience in spectroscopy of polymers and plastics we support customers in:

- **Quality Control and Quality Assurance**
- **Failure and Root Cause Analysis**
- **Product and Process Development**
- **Applied Sciences and Research**

Watch our video that shows how FT-IR can help in the production of polymer products:

YouTube

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