

Applications of FTIR

Polymer analysis using fourier transform infrared (FTIR) spectroscopy



FTIR Spectroscopy Offers a Wide Range of Analytical Opportunities

Fourier transform infrared (FTIR) spectroscopy is a well-established and powerful analytical technique that allows powerful insights into a wide variety of samples.

Even though FTIR is a mature technology, advances in FTIR sampling interfaces offer great flexibility. Polymer analysis is one application where these sampling interfaces provide opportunities for quick and easy qualitative and quantitative analysis.



Simplify polymer analysis with the Cary 630 FTIR spectrometer

The characterization of polymers by FTIR involves a vast array of analyses, including:

- Studying surface modification and functionalization
- Studying reaction kinetics, and the investigation of thermal effects
- Monitoring additive levels, comonomer content, branching, and tacticity.

The benchtop Agilent Cary 630 FTIR spectrometer is a compact instrument providing rapid quantitative and qualitative information. The Cary 630 has a modular design that allows sampling interfaces to be interchanged within seconds for a wide range of samples and applications. This design makes the Cary 630 ideal for polymer analysis in polymer development, research, and the QA lab.

Applications of FTIR for Polymer Analysis

Additives in polyethylene and polypropylene

Various additives are blended into polymeric materials to modify properties of the polymer. Specific additives and their concentrations in polymer blends are critical when modulating the properties of the polymer. Careful analysis is required to ensure that the additives and levels are appropriate for the intended use.

Irganox 3114, Irganox 1010 and other additives are often used as antioxidants to prevent the degradation of organic polymers (e.g. polypropylene homopolymer formulations) by light, heat, and oxygen.

The Cary 630 FTIR can be used to measure additive content directly in polymer thin films. The sampling capabilities of the DialPath and TumbIIR sampling accessories provide a simple, fast, and reproducible mechanism to mount and measure your sample.

Step-by-step method-driven software, with color-coded results, guides you through your analysis. These design elements ensure that your samples are measured with minimum effort and highest accuracy.

The Agilent DialPath sampling technique simplifies the analysis of polymer coupons and film samples. The DialPath accessory is the silver attachment on top of the instrument. It allows fixed pathlength transmission measurements.

Application notes available for download

Determination of Irganox 3114 in polypropylene by infrared spectroscopy

Determination of Irganox 1010 in polyethylene by infrared spectroscopy

Determination of Irganox 1010 in polypropylene by infrared spectroscopy



The MicroLab software calculates results and presents them in a color-coded format. The screen shows the analysis of 0.16 wt% Irganox 1010 in a polyethylene sample. The green color coding indicates that the sample was within a predefined range.

Copolymer blend determinations: Polyethylene (PE) to polypropylene (PP) ratio

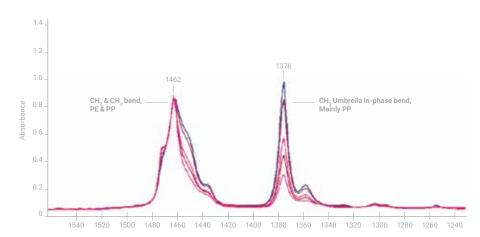
Polyethylene (PE) is the most common group of thermoplastic polymers due to its low cost and versatile physical properties. PE is blended with polypropylene (PP) to improve physical properties, such as low temperature impact performance. The composition of these blends affects performance and the correct mixing of the pure homopolymers can eliminate the need for costly synthesis of new block copolymers. Knowing the composition of these blends is also critical to the recycling and regeneration of polyolefins in waste.

Conventionally, concentrations of 35–85% in PE/PP blends are determined using a cast film technique and a method recommended in ASTM D3900-05a. Following this method, FTIR standard transmission measurements are performed on the polymer films cast onto KBr salt plates. This method requires copolymers to be dissolved and spray coated onto a KBr disk. This procedure is time consuming and requires some skill, creating opportunities for wrong or inconsistent results to be obtained.

Using the Cary 630 FTIR, the PE:PP ratio in blends can be rapidly determined using the DialPath transmission accessory. This method allows the copolymer samples to be measured directly as a film. The polymer can easily be repositioned, allowing measurements in multiple positions on the sample.

This novel method yielded the same excellent calibration and identical correlation coefficient value (R²) as the conventional method using the spray deposition on a KBr cell.

The PE:PP calibration can be implemented into a MicroLab method for routine use with the Cary 630 FTIR. The calibration allows the polymer ratio in unknown samples to be instantly calculated and displayed.



The overlaid aliphatic bend region of the FTIR PE/PP blends calibration spectra. The quantitative method for %PE uses a ratio of the methyl 1376 cm⁻¹ (mainly PP) to the 1462 cm⁻¹ (methyl and methylene bend) band. The peak ratio is used to determine the PE to PP ratio in the copolymer.

Application note available for download

Determination of percent polyethylene in polyethylene/polypropylene blends comparing to cast film FTIR techniques

Copolymer blend determinations

Styrene concentration in Styrene butadiene rubber (SBR) polymer

SBR is the most common synthetic rubber material and its main use is in the manufacture of tires. The properties of SBR rubber can be altered by varying the ratio of styrene to butadiene monomers in the manufacturing process. Higher styrene concentrations make the material harder, but less elastic. Most performance applications, such as racing tires and specialty military applications, require a consistent SBR product. This requirement drives the need for comprehensive quality assurance and control by manufacturers.

A Cary 630 FTIR with a diamond attenuated total reflectance (ATR) sampling module can measure both SBR copolymers. The method has highly linear calibrations with excellent quantitative accuracy and reproducibility.

Application note

Styrene concentration in Styrene butadiene rubber polymer using FTIR (ATR) sampling



The Agilent Cary 630 FTIR spectrometer equipped with single reflection diamond attenuated total reflectance (ATR) sampling module. It can be used for the SBR and polyethylene vinyl acetate (PEVA) copolymer analyses.





The polymer sample is placed directly on the ATR sampling module. Uniform, constant pressure is provided by the sample press, ensuring that high-quality spectra are obtained. Real time analysis software provides an immediate indicator of spectral quality.

Polyethylene to vinyl acetate ratio in polyethylene vinyl acetate (PEVA) polymer

Polyethylene vinyl acetate (PEVA) is common in everyday products used in the home, sports equipment, industrial, and medical applications.

Similar to SBR polymer analysis, PEVA copolymers can be measured using a Cary 630 FTIR with diamond attenuated total reflectance (ATR) sampling module.

Application note

Ratio of polyethylene to vinyl acetate in PEVA using FTIR attenuated total reflectance (ATR) sampling

Copolymer blend determinations

Vinyl content of polyethylene resins

Polyethylene (PE) resins made with chromium catalyst technology, have a vinyl group at the end of each polymer chain. Determining the number of vinyl groups (C=C) in polyethylene resins by infrared spectroscopy can be used to study the effectiveness of the production method. The method is applicable to powder, pellets, or pieces cut from finished parts.

Application note

<u>Determination of the vinyl content of polyethylene resins</u>



The Agilent Cary 630 FTIR can be used measure the component make up of polymer thin films. Step-by-step method-driven software with color-coded, actionable results guides you through your analysis. This approach ensures that your samples are measured with minimum effort and highest accuracy.

Ethylene content in ethylene-propylene statistical copolymers

This method can determine the ethylene content of ethylene-propylene copolymers.

The determination is specific for ethylene and cannot be applied for the quantitation of other comonomers.

The method has been validated over the range of 0.3 to 3.5% statistical content. Samples can be either in powder or pellet form. Both forms are easily handled with the diamond ATR module.

The Agilent Cary 630 FTIR spectrometer equipped with a DialPath or TumblIR sample interface with a 1000 μ m path length was used for the measurement of the polyethylene resins.

Equivalent FTIR spectrometers, such as the mobile or portable Agilent 5500 or the 4500 Series FTIR, shown here, can also be used.

Application note

Determination of percent ethylene in ethylene-propylene statistical copolymers

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