Step-Scan FTIR Spectroscopy solutions for advanced research experiments

Technical Overview

Advantage statement

The Agilent Cary 680 FTIR, Agilent's 'top-of-the-range' FTIR instrument, is in a class of its own. With an air-bearing interferometer, operating in both rapid scan and step-scan modes, the Agilent Cary 680 FTIR is designed for the ultimate in performance and is targeted at high-end research applications such as photoacoustic spectroscopy (PAS), polarization modulated infrared reflection absorption spectroscopy (PM-IRRAS), time-resolved spectroscopy (TRS) and polymer stretching experiments.

The advantage of using an Agilent step-scan system over any other commercially available spectrometer is that it delivers 3 to 4 times more infrared energy to the sample, resulting in higher sampling sensitivity and increasing sample throughput, while providing superior data quality — all of which is required for the challenging step-scan experiments described in this technical overview.

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Key applications

Photoacoustic Spectroscopy (PAS)

PAS is commonly applied to the study of layered or heterogeneous solid samples, as it provides a non-destructive method of obtaining depth-resolved FTIR spectra. Typically, depths of up to a few hundred micrometers can be probed. Common samples studied include:

- Polymers
- Coating layers
- Biological materials
- Intractable materials that cannot be sampled any other way.

The solid sample is held in a small cup within a helium-filled closed cell. Modulated IR from the interferometer is converted into thermal waves by sample absorption. The thermal wave travels to the sample surface where heat is transferred to the buffer gas, resulting in an acoustically-modulated pressure change. That is, sound, which is detected by a sensitive microphone.

The sampling depth is dependent on the modulation frequency. The use of a phase-modulated step-scan interferometer in the PAS experiment offers the significant benefit that, in contrast to rapid-scan experiments, the sampling depth is constant across the IR spectrum. Also, Agilent's square-wave modulation, produced by piezoelectric dithering of the fixed mirror, coupled with our DSP software, enable odd harmonics of the fundamental frequency to be sampled. This capability provides the unique advantage that 5 different depths within the sample can be probed simultaneously, as shown in Figure 1.

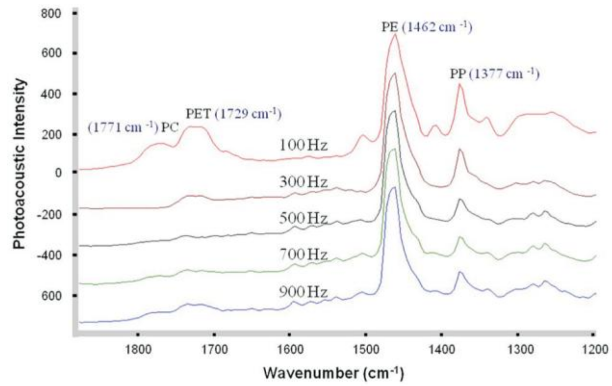

Figure 1. The unique capability to simultaneously collect spectra from five sample depths for a four-layer laminated film, consisting of, from the surface, 10 µm of polyethylene (PE), 10 µm of polypropylene (PP), 6 µm of polyethylene teraphthalate (PET) on a polycarbonate substrate (PC)

Polarization Modulated Infrared Reflection Absorption Spectroscopy (PM-IRRAS)

PM-IRRAS is a highly sensitive method for the characterization of ultra-thin films or monolayers of samples on a metal substrate. This technique enables the identification of adsorbed chemical species, and can also provide information on the orientation of the surface species. A common application is the direct detection of DNA hybridization in self-assembled monolayers of DNA on gold.

The sample is probed at a grazing angle of incidence, taking advantage of the differential absorption of s- and p-polarized IR light. At large angles of incidence, only p-polarized light interacts with the surface adsorbed molecules, allowing s-polarized light to be employed as a reference. This means that a separate background spectrum is no longer required. Furthermore, Agilent's DSP software enables these experiments to be performed without the use of lock-in amplifiers.

A typical PM-IRRAS set up is shown in Figure 2.

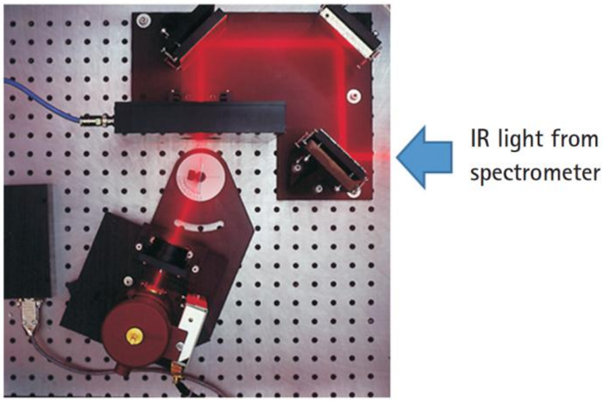

Figure 2. Typical PM-IRRAS set up using the External Experiment Module (EEM). The light enters the accessory from the external port on the instrument

Time-Resolved Spectroscopy (TRS)

Step-scan TRS is an extremely powerful tool for studying spectroscopic changes that occur on the nanosecond or microsecond time scale. Applications include the study of:

- Short-lived transients
- Photochemical reaction mechanisms and kinetics
- Protein folding dynamics.

Step-scan TRS experiments are performed as follows:

1. **Step** the interferometer to a new position.
2. **Trigger** a fast dynamic change using an external device, for example, photolysis by a nanosecond UV laser.
3. **Monitor** detector signal as a function of time using a special mercury cadmium telluride detector designed to give a fast response time.
4. **Step** the interferometer to the next position and repeat.

In this manner, a complete time-resolved interferogram is constructed from which time-resolved spectra can be obtained at any specified time delay following the initiating trigger. All Agilent Cary 600 FTIR Series spectrometers are fitted as standard with a high dynamic range sigma-delta A/D Converter (600 kHz, 24-bit), which affords a time resolution of 1.6 µs in step-scan TRS. An even faster A/D converter (1 GHz, 8-bit) is available as an option, enabling 1 ns time resolution.

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