

Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy exists as one of the most powerful techniques for chemical identification and the most practical for “first step” analysis. Analytical instrumentation such as GC-MS and LC-MS are commonly used for identifying organic compounds. However, these techniques are costly and often require extensive set up time, method development, and sample alteration. Reliance upon FTIR as a robust and versatile tool can be based on its attributes of simplicity, speed, sensitivity, and affordability.

An infrared spectrum represents a “fingerprint” of a sample, with absorption peaks corresponding to the frequencies of bond vibrations. Since different materials have specific arrangements of atoms, the FTIR spectrum of a material is unique. These distinctive traits allows for a diverse range of material identifications for nearly all organic compounds, as well as many inorganics.

FTIR spectroscopy occupies an essential role in material evaluations, cleanliness verifications, and quality control screening. It also is a fundamental step in some failure analysis and allows for the classification of unidentified residues and materials to assist in resolving a root cause. A key advantage of FTIR is its ability to characterize an extensive range of organic chemistries, certain inorganics, polymers, coatings, adhesives, semiconductor materials, and minerals. More significantly, each of these materials can be investigated, in many cases, without destructive analysis. A variety of sampling methodologies exist to allow sample materials to be analyzed “as is,” whether it is a solid, liquid, or gas.

The majority of analyses are accomplished simply by placing the sample directly on the attenuated total reflectance (ATR) crystal (Figure 1).

Fluid samples require a small drop of liquid on the ATR lens while only a single fiber or particle can be adequate for a material identification. Sample spot size allows for a minimum area of analysis in the range from microns to millimeters. Certain samples may require preparation using salt plates, mineral oil (Nujol), potassium bromide (KBr) pellets, films, and gas or liquid cells. In some instances the sample is generated as a result of an extraction procedure. For example, identifying the existence of residues on printed wiring boards can be

accomplished by means of a solvent extraction procedure (as defined in IPC-TM-650, Test Method 2.3.38). In this method, the sample is extracted from the board using acetonitrile and collected on the slide. After solvent evaporation the sample residue is ready for analysis (Figure 2).

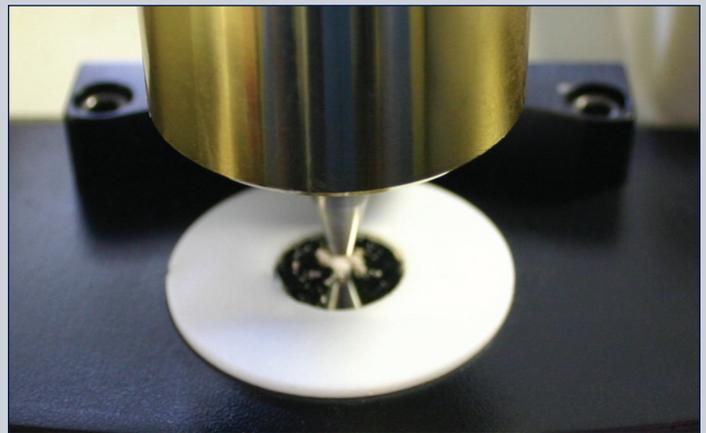


Figure 1: Image of residue on an ATR crystal of a bench top FTIR spectrometer.

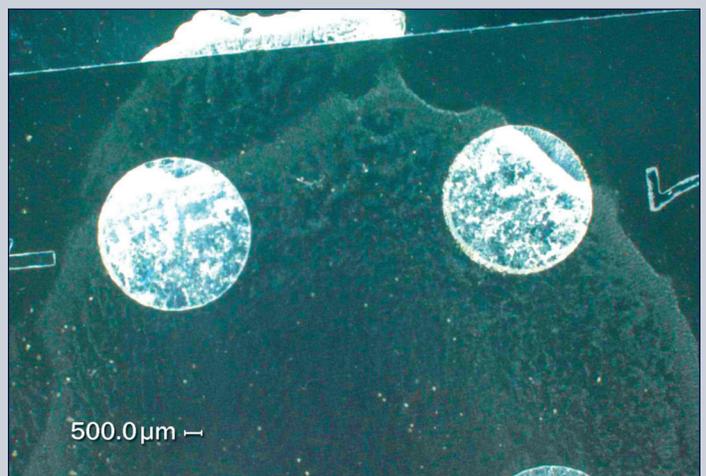


Figure 2: Image of residue on slide obtained after extraction of residue from a bare board.

Minimal sample preparation time allows for “real time” results to be achieved within a matter of minutes. The speed of obtaining FTIR spectra is supplemented by the “ Fellgett or multiplex advantage.” By simultaneously measuring all the frequencies of the sample, the analysis is accomplished in seconds. This also increases sensitivity by decreasing detection limits through signal to noise ratio enhancement.

A search of the sample spectra (Figure 3) against a library of known compounds is essential for spectral interpretation. External commercial spectral databases containing thousands of compounds (Figure 4) are currently available through the internet and the cost associated with utilizing local software spectral libraries can be mitigated with the use of “pay as you go” search methods.

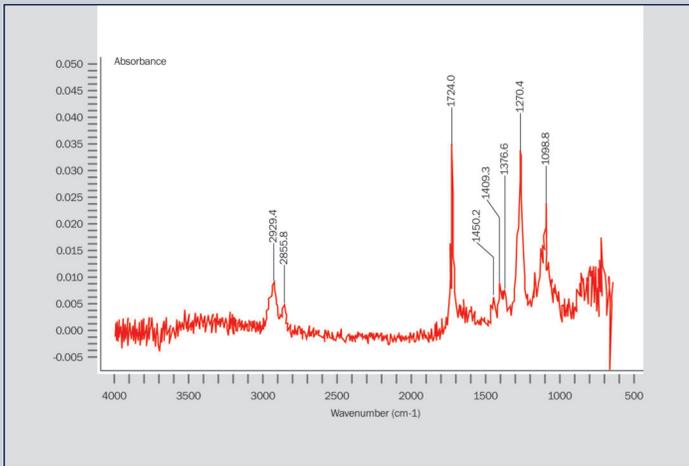


Figure 3: FTIR spectrum of residue from a bare board.

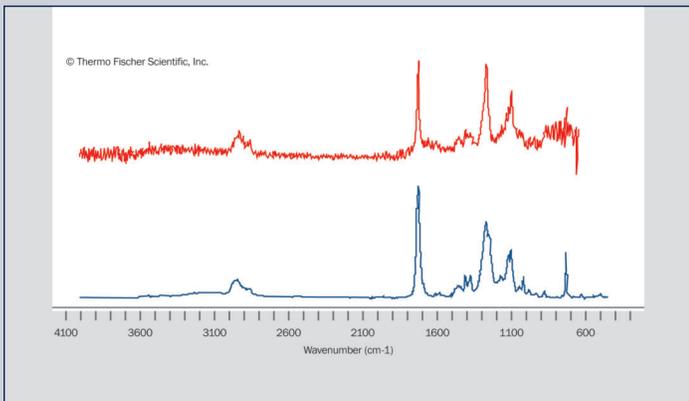


Figure 4: FTIR spectra from residue of a bare board (red) with a known polyester resin (blue) found in the library search.

FTIR spectroscopy remains a cost-effective analytical tool when compared to instruments which yield similar results, such as mass spectroscopy which is an order of magnitude more expensive. Minimal maintenance is required and the likelihood of instrument downtime is reduced due to the mechanical simplicity of the FTIR instrument. Furthermore, FTIR spectrometers are self-calibrated by using a HeNe laser as an internal wavelength standard. Therefore, expenditures after the initial purchase of the instrument can be limited to mostly consumables.

In the realm of failure analysis, FTIR spectroscopy is often used as an accompaniment to Energy Dispersive X-ray Spectroscopy (EDX). EDX analysis is employed primarily for the identification of inorganic atomic species on an elemental level. EDX complements FTIR analysis by confirming organic species, such as carbon, oxygen, silicon and sulfur while also analyzing inorganic species undetected by FTIR.

While FTIR is excellent analyzing molecular species, it is deficient in detecting atomic species. For example, homonuclear diatomic species such as H₂, Cl₂, N₂, and O₂, have no infrared absorption bands and provide no signal. FTIR functions ideally with homogeneous matrices, or those composed of only a small number of constituents, such as pure compounds. Complex mixtures are inclined to confound library search functions due to the intricacy of overlapping spectral fingerprints. However, even when spectral patterns do not yield adequate results, partial information can be gained from the individual peaks.

FTIR spectroscopy persists as the most established instrumental analysis method used in laboratories today. Its versatility finds it well established in all industries including semiconductor, pharmaceuticals, biotechnology, forensics, healthcare, and petroleum industries. Its simplicity, in addition to its characteristics of speed, accuracy, and affordability enable it to be the cornerstone instrument of a laboratory.

ACI Technologies offers a variety of analytical instrumentation and techniques to aid in failure analysis, cleanliness determinations, and material characterization. Optical microscopy, FTIR, and SEM/EDX capabilities are all on hand to assist in resolving root cause. For more information, please contact the Helpline at 610.362.1320.

ACI Technologies



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