Application Note 52416

Thermo Scientific Nicolet iS50 FT-IR Spectrometer: Improving Productivity through Compact Automation

Key Words
Automation, Far-IR, FT-IR, Full-spectral, Infrared, Mid-IR, Multi-range, Multiple Methods, Near-IR, Workflow Optimization

Challenges Facing Industrial Analytical Labs

Many routine QC/QA laboratories can perform material analyses with single range, basic Fourier transform-infrared (FT-IR) instrument configurations. However, modern analytical laboratories face increasing workloads from a broad range of sample types with a simultaneous drive for faster results and more complex sample characterization needs. Flexibility to analyze multiple sample types becomes mandatory when rapidly responding to these different application requests. Such diversity requires laboratory instruments to be reconfigured for specific measurements multiple times per day, taking time away from other critical activities. This also implies that laboratory personnel possess the necessary skills and experience to make decisions on how best to configure the instrument for a given application. In addition, frequent handling of delicate optics components presents a costly risk for instrument failure. As a result, many industrial laboratories choose to outsource complex analyses. These limitations inevitably slow the laboratory's ability to respond to urgent business needs.

The Thermo Scientific™ Nicolet™ iS™50 FT-IR spectrometer alleviates many of these productivity concerns by automating setup of the FT-IR system for multi-spectral range experiments (>20,000 cm^{-1} to 80 cm^{-1}) and for integrating techniques like FT-Raman, near-IR and mid/far-IR attenuated total reflectance (ATR) into a single workflow. Intelligent design behind the Nicolet iS50 spectrometer permits unattended, risk-free operation, increasing lab efficiency, sample throughput, and operational consistency between users. This capability is delivered in an economical, compact system (63 cm of linear bench space) enabling any laboratory to employ multiple techniques for their analysis.

Flexibility and Value-added Activities

Working labs need analytical flexibility to respond to a variety of situations where answers are critical for decision-making. Examples include deformulating mixtures to build a case for patent infringement, identifying counterfeit materials for product safety alerts, analyzing forensic samples for criminal investigations, performing failure analysis to minimize production run delays, assessing process scale-up options for a new product launch, or troubleshooting customer complaints. Such diversity of applications requires the selection and installation of the correct instrument accessory as well as choosing the optimal source, beamsplitter, detector, optical path, and experimental conditions. Manually changing components and sampling parameters requires skill and may risk exposure of expensive optics to the external environment (i.e., dust, fingerprints or water vapor). In addition, changing these parameters can result in extensive wait times to equilibrate the instrument before the next measurement.

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These manual reconfigurations provide little added value to the laboratory workflow. Users must plan and set up batch experiments to minimize the number of steps. This creates bottlenecks, limiting access to the full capability of the instrument. As a result, labs are less able to address "emergency situations" without interrupting the batch run and resetting the instrument parameters. For instance, analysis of a polymer with additives requires mid-IR and far-IR plus Raman spectroscopy. This would entail three beamsplitter changes with associated risks in handling expensive components and instrument recovery times between changes.

The productivity improvements with the Nicolet iS50 FT-IR spectrometer come from two main sources. First, the internally mounted iS50 ABX Automated Beamsplitter Exchanger uses one-button simplicity (described as a Touch Point) to perform instrument setup and operation, providing a "one touch and done" workflow. The removal of manual handling and exposure of the optics to the environment means instant readiness. Second, the user need no longer care about which optics are installed. As seen in Table 1, the potential for error in manual operations is apparent when the array of possible component combinations is considered. With the Nicolet iS50 spectrometer, however, a user simply presses the Touch Point on the instrument to automate the configuration and ready the instrument for the experiment. For example, pressing the Touch Point on the iS50 NIR module automates the setup without requiring any understanding of which optics are used. What matters is performing NIR analysis – not worrying about choosing the right components. The instrument takes care of this step. Integration of the spectrometer with its modules and components allows the user to expand capabilities, increasing productivity with tools such as:

- Up to three detectors (such as near-, mid- and far-IR)
- The iS50 Raman sample compartment module
- The built-in diamond iS50 ATR sampling station
- The iS50 NIR module with integrating sphere or fiber optics
- The iS50 GC-IR module
- A sample compartment thermal gravimetric analysis-IR (TGA-IR Interface)

Figure 1 describes the analytical power the user can achieve with the iS50 spectrometer to obtain answers needed for time-sensitive decisions. With a single user interaction, the instrument can perform multiple measurements and analyses, resulting in a final report, even when unattended. The Thermo Scientific OMNIC™ software provides a user-friendly interface to set up applications quickly and generate spectra for definitive answers. By adding powerful analytical tools like the Thermo Scientific OMNIC Specta™ software with a library of over 30,000 spectra and multi-component searching (or the TQ Analyst™ software for chemometrics), a complete analytical workflow from sampling to results can often be achieved in less than 60 seconds.

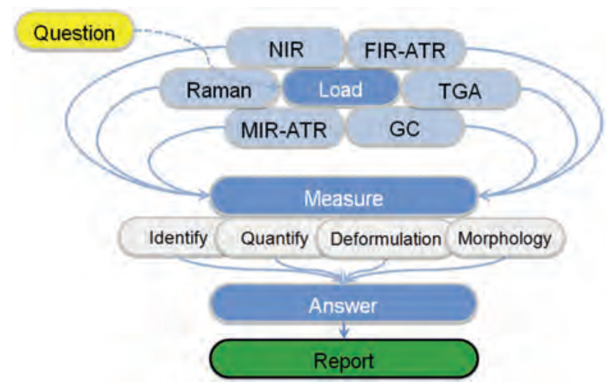

Figure 1: Nicolet iS50 analysis workflow

This paper will demonstrate how the integration and automation of the Nicolet iS50 spectrometer leads to new levels of productivity, while minimizing risk to costly components. Unlike most spectrometers, operating the Nicolet iS50 instrument becomes simpler as modules are added and as more manual steps are removed even when unattended.

Experiment	Source	Beamsplitter	Detector	Accessory
Mid-IR Transmission	Thermo Scientific Polaris™	KBr	KBr-DLaTGS	Standard Cells
Far-IR Transmission	Polaris	Solid Substrate	Polyethylene DLaTGS	Cells w/Far-IR Windows
Near-IR Transmission	White Light	CaF_2	InGaAs	Cuvettes
Mid-IR ATR	Polaris	KBr	Dedicated DLaTGS	iS50 ATR
Far-IR ATR	Polaris	Solid Substrate	Dedicated DLaTGS	iS50 ATR
FT-Raman	Raman Laser	CaF_2	Raman InGaAs	iS50 Raman

Table 1: Experiments made possible with the Nicolet iS50 FT-IR Spectrometer

Automated Multi-spectral Analysis: Mid- and Far-IR ATR plus Near-IR

Most FT-IR users understand the utility of the mid-IR spectral range for qualitative and quantitative analyses. Less well known, the far-IR region can provide new and unique information. Simply put, as the mass of atoms involved in vibrations increases, the wavenumber decreases.[1] Thus, for materials like organometallics or metal oxides, the IR absorption shifts below 400 cm^{-1} and below the range of standard KBr optics. Numerous polymers, sugars, and other large molecules also have far-IR information which may be useful or definitive to the analyst. Traditionally, collecting FT-IR spectra in both the mid-IR and far-IR region entailed significant sample preparation. This included changing hygroscopic optics and multiple detectors, and risking altered system performance from water vapor. The Nicolet iS50 spectrometer enables rapid analysis over the full mid-IR and well into the far-IR region (4,000 cm^{-1} to 80 cm^{-1}) when equipped with the iS50 ABX, iS50 ATR, and the correct beamsplitters.

The typical, multi-range FT-IR application requires opening the spectrometer to swap beamsplitters. This requires care in handling costly components and exposes the internal optics to the environment by disrupting purge or desiccation. This activity adds a recovery period to re-equilibrate the instrument before quality data can be collected. These wait times add no value to operations, wasting the analyst's precious time. Integration and automation on the spectrometer eliminate non-productive wait times, improving efficiency.

As an example, Table 2 compares the steps needed to perform a full spectral analysis from far-IR to near-IR between the manual method (Typical) and the Nicolet iS50 method with built-in iS50 ATR and iS50 NIR module. This represents three spectral ranges in one sampling operation, a unique power of the instrument. Most important the built-in iS50 ATR optics and detector permit spectral data collection in both the mid- and far-IR regions. The analysis time decreases from around 30 minutes to less than seven. With the Nicolet iS50 spectrometer, the user is able to load two sampling locations (the built-in ATR and the Integrating Sphere module), start the macro and walk away, while in the manual operation, continuous attention is needed to swap the beamsplitters at the right moments. This seemingly hidden improvement allows unattended operation, permitting productivity through automation.

Figure 2 shows just the mid- and far-IR spectra collected from acetylferrocene analyzed using an OMNIC macro-controlled workflow. The additional information from the far-IR spectra is clear – the low end triplet verifies that the iron is sandwiched between the cyclopentadiene rings. The NIR data is not shown, but the entire process required seven minutes, including collection of the mid- and far-IR backgrounds. Automation also reduced the total hands-on time of the user (pressing buttons, loading sample) to ≈20 seconds.

Process Step	Typical	Time (minutes)	Nicolet iS50 with Built-in ATR	Time (minutes)
Sample Preparation	Grind, Mix	10	**None**	0
Mid-IR Background	Collect BKG	0.5	Collect BKG (2nd)*	1.
Mid-IR Collect	Load Sample, Collect Spectrum	2	Load Sample, Collect Spectrum	1
Change Optics	Manual Exchange	0.5	Automated	0.5
Recovery Time	Wait for Purge	5–10	**No Recovery Time**	0
Far-IR Background	Collect BKG	0.5	Collect BKG (1st)*	0.5
Far-IR Collect	Load Sample, Collect Spectrum	2	Load Sample, Collect Spectrum	1
Change Optics (NIR)	Manual Exchange	0.5	Automated	0.5
Recovery Time	Wait for Purge	5	**No Recovery Time**	0
Collect Background	Collect BKG	0.5	Collect BKG	0.5
Collect Sample	Load Sample, Collect SAM	1	Collect SAM	0.5
Data Analysis (Search)	Perform Search	2	Automated Search	0.5
Total Time		**29.5–34.5**		**6.5**

Table 2: Far-infrared analysis: Typical versus Nicolet iS50 process

** With the iS50 ATR present, the far-IR background (BKG) is collected, the iS50 ABX swaps beamsplitters, and the mid-IR background is collected in <1.5 minutes. The sample is loaded and the spectra are collected in sequence. All times are approximate.*

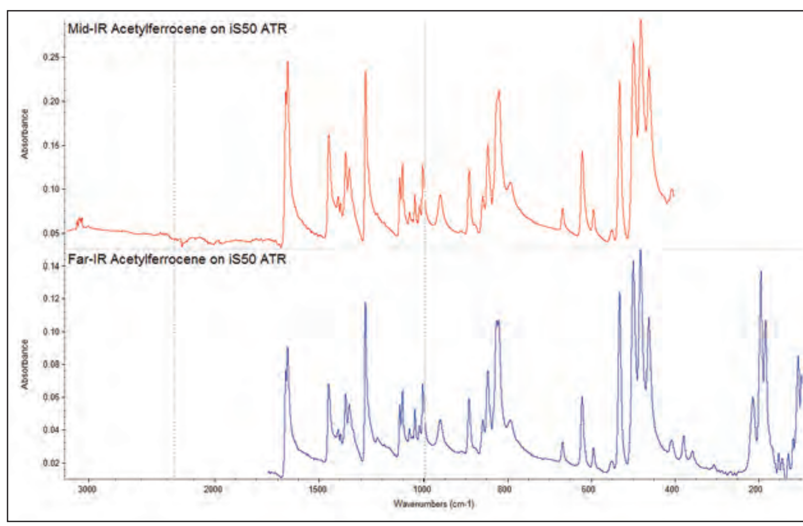

Figure 2: Mid-IR and far-IR spectra of Acetylferrocene. The far-IR optics permit collection to 1700 cm^{-1}, which may be sufficient (fingerprint and far-IR) for many applications.

Figure 3: The Thermo Scientific Nicolet iS50 FT-IR spectrometer configured for FT-Raman, near-IR, and mid/far-IR ATR with the automated beamsplitter exchanger.

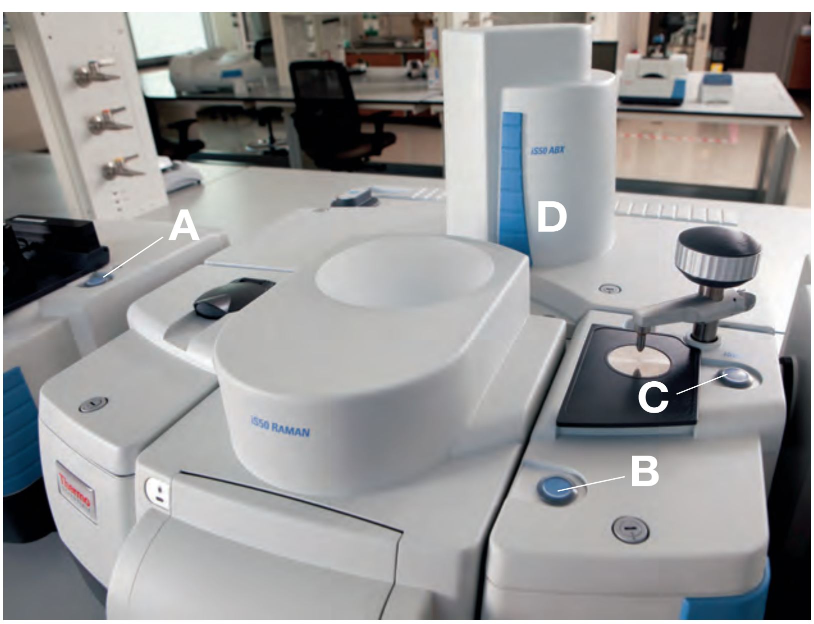

Figure 4: Touch Points on the Nicolet iS50 spectrometer employ one-button switching between modules and the iS50 ABX automates optics set-up

Touch Point A – NIR module
Touch Point B – Raman module
Touch Point C – Built-in diamond ATR
Component D – ABX Automated Beamsplitter Exchanger

Multiple Techniques and Multi-range Analysis: Enhanced Flexibility

The Nicolet iS50 spectrometer can be configured with FT-Raman, NIR, and wide-range diamond ATR. Switching between these experiments raises concerns of instrument recovery time (purge), exposure/handling of optics, and potential confusion or user error. The experiments are often seen as independent activities for these reasons. The spectrometer with iS50 ABX simplifies this apparently complex situation to one step – initiation of a macro. The Nicolet iS50 instrument shown in Figure 3 is configured with the iS50 NIR, iS50 Raman, iS50 ATR and the iS50 ABX modules and shows how easy sample loading and analysis can be done.

For operating one module at a time, the user need only press the associated Touch Point. Seen more closely in Figure 4, Touch Points make one-button operation effortless when switching between modules (sampling stations) and automating optics exchange. Rather than thinking through the components needed (light source, beamsplitter, optical path and detector) to run an experiment, the user simply presses the Touch Point to switch from an ATR to an NIR measurement and waits until the instrument indicates that it is ready to begin. This error-free operation is done in 30 seconds.

The Nicolet iS50 analytical power in Figure 1 becomes clear when the four data collections – mid-IR and far-IR ATR, NIR, and Raman – are performed in one workflow. Collecting spectra from each of these modules using a conventional manual approach required about 50 minutes, including sample loading, optical changes, time for equilibration, and optimization of the Raman signal. The analyst needed to be present throughout the experiment to perform the beamsplitter changes and collect various backgrounds for each sampling station. At the end of the 50 minutes, four spectra and their analyses were available. Actual data collection took 5 minutes and total hands-on time was 45 minutes, representing inefficient use of the analyst's time.

In contrast, the results shown in Figure 5 emerged from a single OMNIC-macro operation. The macro was programmed to begin by collecting backgrounds for the mid- and far-IR ATR, and then switched to the iS50 Raman module. Next the samples were loaded on the ATR, NIR, and Raman sampling stations. After optimizing the signal using the autofocus feature of the Raman module, the macro was initiated, and the analyst walked away. From starting the macro to completion of the final report, the analysis took less than 12 minutes, representing a time savings of over 70%. The actual data collection time was again 5 minutes, however, total hands-on time for the analyst was only ***2 minutes*** – a highly efficient use of the analyst's (and the instrument's) time.

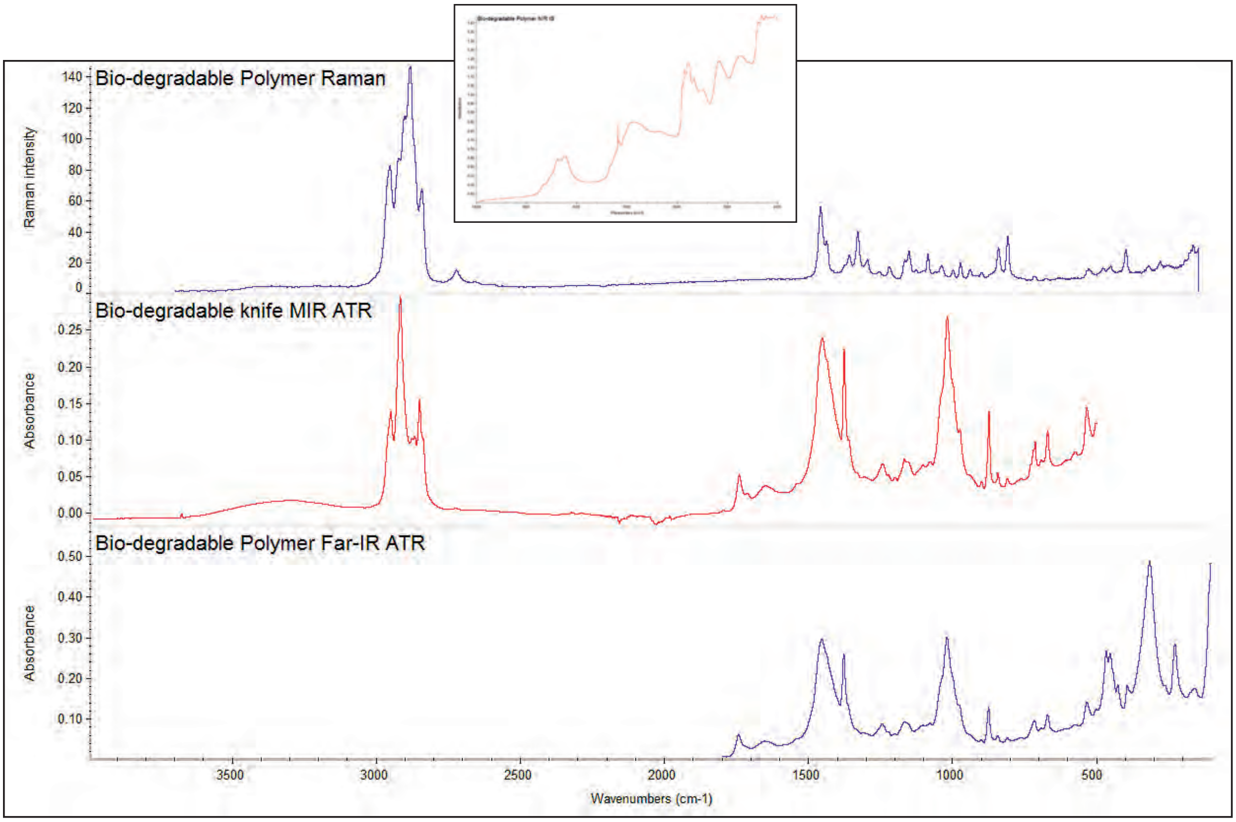

Figure 5: Multi-technique data for a recyclable plastic component using the spectrometer pictured in Figure 3. Inset shows NIR independently for clarity.

Conclusion

Many forces contribute to new pressures on industrial analytical laboratories: increased sample loads, decreased staffing, retirement of experts, and shrinking budgets. The Thermo Scientific Nicolet iS50 FT-IR spectrometer makes a significant contribution to alleviating these challenges through automation in a multi-tasking, single platform instrument. The Nicolet iS50 spectrometer greatly simplifies and streamlines workflows by decreasing the number of steps with one-button ease and macro operations performed by the analyst. In addition, risks inherent in manual operations (e.g., user error, environmental exposure) and long recovery times are eliminated. Analysts of any skill level can successfully obtain meaningful results with minimal hands-on time.

Technology designed to improve workflow can be found in the iS50 ABX and task-specific modules (i.e., Raman, NIR, TGA-IR etc.). The Touch Point operation simplifies access to the full range of capabilities by automatically configuring the optics (near-, mid- and far-IR) and switching between sampling stations (modules) in seconds for enhanced productivity. For the modern industrial lab, the Nicolet iS50 FT-IR spectrometer offers a powerful new tool that goes beyond routine FT-IR to more comprehensive analyses (e.g., FT-Raman and far-IR), adding value to laboratory activities in a compact, easy-to-operate platform.

References

1. Heavy atoms or groups of atoms shift the IR wavenumber value lower, according to the relationship

$$\tilde{\nu} \; \alpha \; \sqrt{1/\mu}$$

where $\tilde{\nu}$ is the IR wavenumber (cm^{-1}) and μ is the reduced mass. As the mass (μ) increases, the IR peak shifts to lower wavenumbers.

Glossary

CaF_2 – calcium fluoride
DLaTGS – deuterated L-alanine doped triglycene sulphate
InGaAs – Indium gallium arsenide
KBr – potassium bromide

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