Technical Note: 51513

Room Temperature Vs. LN_2-Cooled Detectors Infrared Microscopy Cost/Performance Analysis

Federico Izzia, Claude Robotham, Ph.D., Thermo Fisher Scientific, Madison, WI, USA

Key Words

- Cost Per Analysis
- Integrated FT-IR Microscope
- Microscopy Optimized Room Temperature Detector
- Reflection Absorption Spectroscopy (RAS)
- Sensitivity
- Spatial Resolution

Introduction

Infrared microscopy has become a valuable analytical technique in research, analytical services and forensic laboratories. Infrared microscopy measurements can be classified into two main categories; single "point-and-shoot" analysis and multiple-point analysis. The first category applies to homogeneous samples, where a single spectrum is necessary for sample identification, while the latter applies to heterogeneous samples where multiple discrete points, line scans or area maps are required.

In the latter case, the time and spatial resolution (the ability to distinguish small, adjacent sample features) can require several hours to collect for high spatial resolution approaching 10 microns. For this type of application, liquid nitrogen-cooled detectors provide the highest speed, sensitivity and spatial resolution. Liquid nitrogen (LN_2) cooled detectors are used in most infrared microscopes. They are available in both single-element and multi-element configurations. The multi-element detector is popular for chemical imaging applications. However, there are many instances where samples are larger (> 50 microns) and do not require high spatial resolution data collection, such as contaminates or evidence obtained from a crime scene. In these cases, liquid nitrogen can be an unnecessary expense and a bottleneck in the microscopy laboratory. In other circumstances, liquid nitrogen may be difficult to obtain or pose health and safety restrictions, limiting a laboratory's ability to use microspectroscopy.

The Thermo Scientific Nicolet™ iN™ 10 infrared microscope can provide a solution for both scenarios by providing the ability to switch between a routine room temperature detector and a high sensitivity liquid nitrogen cooled MCT detector.

This technical note explains the differences in the two detector modes, helping the user to identify the best solution for their cost, uptime, safety and performance requirements.

Cost and Time Per Analysis Comparison

Infrared room temperature detectors typically do not require any supply of gas or liquid coolant to operate at the proper temperature, resulting in the most convenient, economical way to collect infrared spectra. As long as the spectrometer is at thermal equilibrium with its environment, room temperature detectors are "ready to use."

The only limitation of room temperature detectors is their sensitivity and response speed, which is typically one order of magnitude lower than cooled detectors. However, the high efficiency of the Nicolet iN10 microscope optics, and the proprietary design of the room temperature detector, a microscopy-optimized DTGS (Deuterated TriGlycine Sulfate detector), provide enough sensitivity to measure samples as small as 50 microns in only few minutes.

Table 1: Cost analysis of cooled and room temperature detectors, IR microscopy operation

		Microscope Detectors	
		LN_2-cooled	Room Temperature
Liquid nitrogen volume required to fill the detector	Liters	1	0
MCT detector cooling and stabilization time	Minutes	30	0
Collection time per spectrum	Minutes	0.5	3.0
25 liters liquid nitrogen Dewar refill + delivery cost	**Local Currency**	180.00	0.00
Laboratory cost/hour	**Local Currency**	200.00	200.00
Days per month of microscope usage	**Days**	4	4
Number of samples per day	**Samples**	2	2
Initial cost of 25 liters Dewar, withdrawal and protective tools	**Local Currency**	2,700.00	0.00
Years of capital equipment amortization	**Years**	3	3
Total Savings in 3 Years	Local Currency	**No savings**	**19,640.00**

The average LN_2 hold time of a 25 liters dewar is about one month: depending on usage could be less

Samples of 100 µm of size of larger, require less than a minute collection time with room temperature detector

One year of utilization is calculated as 11 months

Thermo SCIENTIFIC

The cost analysis in Table 1 assumes the following infrared microscopy weekly utilization (which for simplification purposes are express in USD):

- Average liquid nitrogen volume required to fill the detector: 1 liter
- Average liquid nitrogen cost per 25 liters refill: $180.00
- Time required to spill LN_2, fill and thermally equilibrate the detector: 30 minutes
- Average laboratory analysis cost per hour: $200.00
- Days per week of microscope utilization: 1
- Number of samples per day: 2
- 3-years capital equipment amortization period

While the cost of cooled detector operation is mostly driven by the time required for the "first filling of the day," and associated costs of liquid nitrogen, the cost of room temperature detector operation is only due to the longer collection time necessary to achieve the sensitivity level. This "return on investment" evaluation shows thousands of dollars of savings per year assuming that all measurements are conducted on either one or the other detector. A combined utilization of the two would result in an immediate cost benefit. A higher number of "days per week utilization" would further increase the savings per year. However, a much greater number of samples per day could reverse the picture to the point where the cooled detector becomes more economical.

As a final consideration, it should be noted that liquid nitrogen handling and protective tools are not included in the calculation. For best convenience and safety operation in the infrared microscopy laboratory, the typical storage of LN_2 is accomplished by using a 25-liter capacity dewar equipped with liquid withdrawal and protective safety gloves/goggles. This equipment can cost an additional $1500. No additional equipment is necessary for a room temperature detector operation. Every Nicolet iN10 FT-IR microscope is equipped with a microscopy-optimized room temperature detector. An additional liquid nitrogen-cooled MCT detector is also available as an option for those laboratories, providing both the convenience of a room-temperature detector and the speed and sensitivity of an MCT detector.

Performance Analysis Comparison

Infrared microscopy offers three sampling modes; transmission (through the sample) is the most energy efficient mode, reflectance (beam reflected off the sample surface) only utilizes half of the infrared beam, and attenuated total reflectance (infrared is internally reflected via a sample-crystal interface).

While the performance of the microscope decreases respectively from transmission to ATR mode, ease-of-use follows the opposite order, since micro-ATR and reflection require little or no sample preparation. Each sampling mode offers different information (from a sample's bulk or from its surface), which explains why infrared microscope performance is typically evaluated in all sampling modes.

The following tests show the difference in performance obtainable from room temperature and cooled detectors on the Nicolet iN10 FT-IR microscope, at various sample sizes and sampling modes.

Transmission Mode

Some crystals of an active pharmaceutical ingredient (API) were placed on transparent substrate (13 x 2 mm NaCl disc). Room temperature DTGS detector spectra were collected using varying aperture sizes as per Figure 1. The performance of the room temperature detector was compared to a "reference spectrum," collected using the LN_2-cooled detector (MCT) at a smaller aperture size. It is important to note that the unaided human eye can only see objects as small as 150 - 200 microns. Therefore, the following test provides a clear understanding of the performance that can be obtained when measuring trace evidence, contaminants or other small particles found by visual observation.

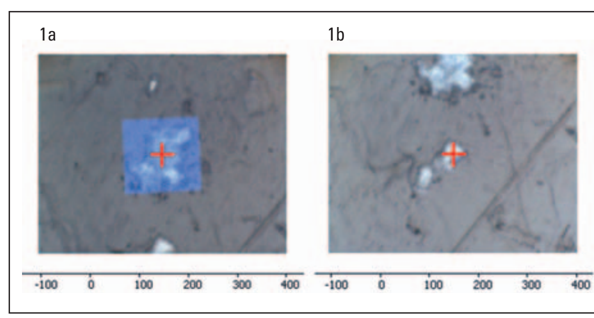


Figure 1: API particle taken from a pharmaceutical tablet and placed on an infrared transparent support (NaCl window) for transmission measurements. Figure 1a shows a 150 x 150 micron aperture (physical size of infrared beam interacting with the sample, illuminated in blue); Figure 1b shows a 50 micron particle of the same material (the aperture – sized at 25 x 25 microns – is hidden by the particle).

The top spectrum of Figure 2 (the reference) was obtained with the LN_2-cooled MCT detector at an aperture of 15 x 15 microns, in only 8 seconds. The middle spectrum was collected from the sample area of Figure 1a in only 2 seconds, with the DTGS room temperature detector. The resulting spectra are perfectly comparable with the top reference spectrum. The room temperature DTGS spectrum, which is comparable to the spectrum obtained by the LN_2-cooled MCT detector, were obtained from the smaller sample in Figure 1b (25 x 25 microns) in 1 minute.

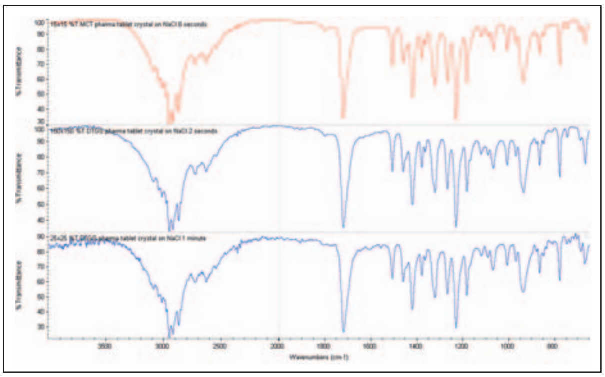


Figure 2: Top spectrum; reference spectrum of the API collected with LN_2-cooled detector in 8 seconds at 15 x 15 microns aperture. Mid spectrum obtained from the crystal shown in Figure 1a, in 2 seconds by room temperature detector. Last spectrum obtained from the crystal shown in Figure 1b, in 1 minute using the room temperature detector.

In transmission mode, the room temperature detector provides excellent signal to noise, requiring only a few seconds of collection time at large apertures or a few minutes at smaller sized apertures. The quality of the spectra obtainable with the room temperature detector in transmission analysis is sufficient to successfully identify materials down to 50 microns in size. Figure 3 demonstrates the excellent library search correlation match of the 25 x 25 micron sample spectrum collected by the DTGS detector.

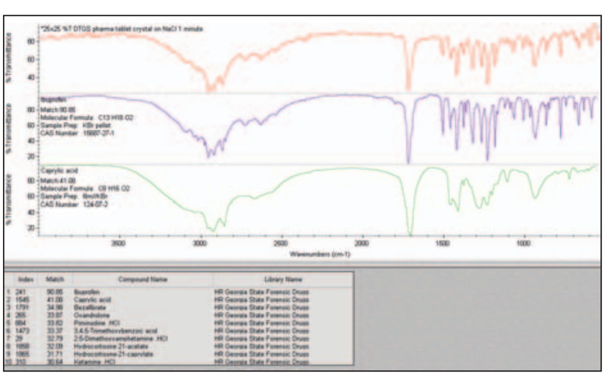


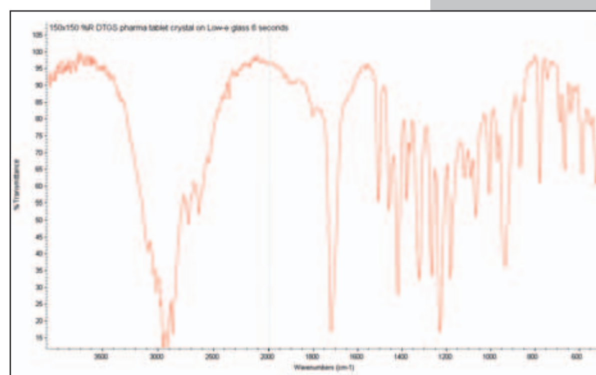
Figure 3: Identification of a 25 x 25 microns particle size spectrum obtained in one minute collection time with room temperature detector (with no use of LN_2). The correlation of 90.85 shows a perfect match with Ibuprofen.

Reflection Mode

Reflection mode infrared microscopy can be used as a substitute for transmission by mounting the sample on a reflective surface. This technique, known as reflection absorption spectroscopy (RAS) or transflectance, facilitates sample preparation and provides transmittance-like spectral quality. In this case, the room temperature detector provides the sensitivity to measure samples quickly as shown in Figure 4.

Reflection mode may also be used to measure diffusely-scattering materials, in which case the sensitivity of LN_2-cooled detector is recommended.

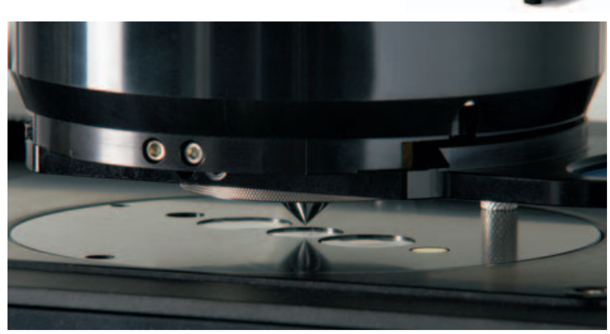
Figure 4: 150 x 150 microns particle size spectrum obtained in 6 seconds collection time with room temperature detector in reflection. Particle crystals were placed on low-e glass slides and flattened with a roller-knife.



ATR Mode

Micro-ATR has emerged as a very popular infrared micro analysis technique due to its ability to obtain high-quality spectra from samples with the minimum amount of sample preparation. Micro-ATR is a non-destructive technique, which expands the analytical power of the infrared microscope where sample evidence must be preserved. Another important feature of micro-ATR is the inherent increase in spatial resolution as a function of the refractive index of the ATR crystal. When using a germanium crystal (approximate refractive index = 4), the size of the aperture that is physically effective in reflection or transmission is zoomed by a factor of 4X. Therefore an aperture size of 100 micron becomes 25 micron at the sample.

All these advantages allow the collection of difficult samples (like fibers, for example) with no preparation.



Germanium Tip-ATR crystal slide of Nicolet iN10 FT-IR microscope; the quick release dovetail mechanism allows for easy crystal cleaning while the rugged crystal design provides excellent sensitivity and spatial resolution.

A set of three polymer fibers (taken from the Nicolet iN10 fiber sampling kit) was used to compare micro-ATR performance between the liquid nitrogen cooled MCT detector and the DTGS room temperature detector. The only collection parameter changed was time; one second for the MCT and one to two minutes for the room temperature. As shown in Figure 5, the quality of spectra, obtained in only a few seconds with the LN_2-cooled MCT detector indicates that a longer collection time would be unnecessary, while spectra of comparable quality can be obtained by the room temperature detector in few minutes (Figure 6).

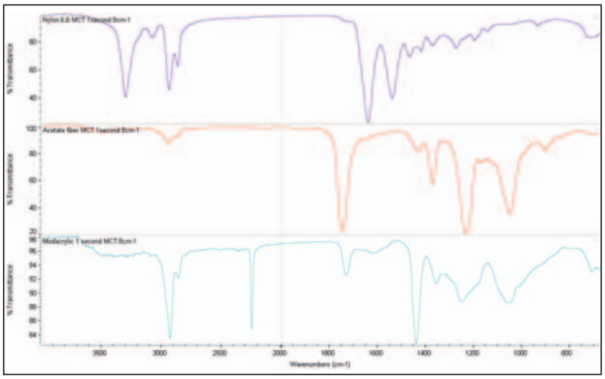


Figure 5: Spectra of polymer single-fibers collected in 1 second with LN_2-cooled MCT detector

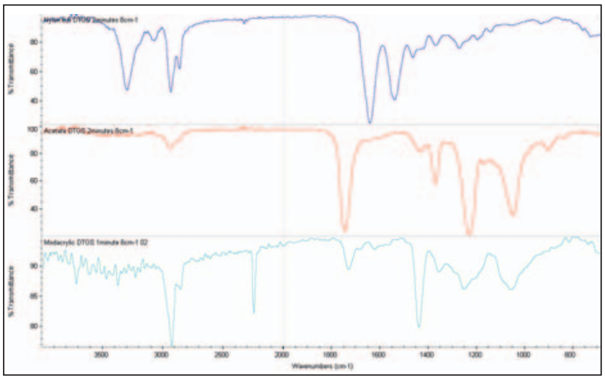


Figure 6: Same set of fibers measured in 2 minutes with room temperature detector

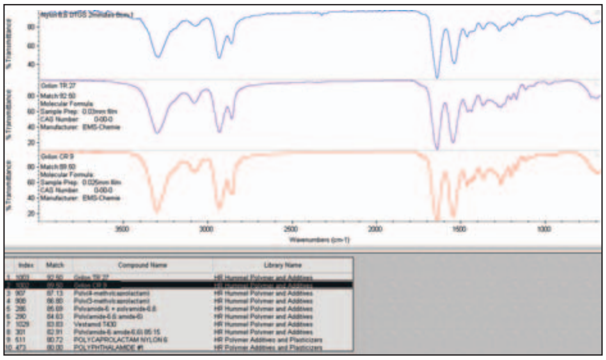


Figure 7: Search identification of a Nylon 6,6 fiber collected in two minutes using a room temperature detector and Tip-ATR

The spectral identification shown in Figure 7 shows that high-quality spectra, which yield high match values against reference spectra, can be obtained simply and easily using a Tip-ATR in combination with a room-temperature detector.

Conclusions

The integrated optical design of the Nicolet iN10 FT-IR microscope provides excellent sensitivity in any sampling mode. In many situations, the use of the microscopy-optimized room temperature detector saves a significant amount of set-up time, effort and laboratory budget.

	Cooled MCT	Microscopy Optimized DTGS
Point and shoot cost per analysis	Good	Best
Limited number of samples cost per analysis	Good	Best
Liquid nitrogen detector hold-time, per filling	16 hours	Not Needed
Point and shoot sample size down to 25 micron	Good	Good
Point and shoot sample size down to 10 micron	Best	N.A.
Small size area map imaging collection	Best	Good
Large size area map imaging collection	Good	N.A.
25 micron spatial resolution imaging collection	Good	N.A.
10 micron spatial resolution imaging collection	Good	N.A.

Table 2: Performance and application characteristics of liquid nitrogen cooled MCT and room temperature DTGS microscopy analysis

When exceptional sensitivity, spatial resolution and speed are necessary, the Nicolet iN10 can be equipped with a cooled detector, which provides a long-lasting liquid nitrogen hold-time of 16 hours or better thanks to a patented dewar design. The Nicolet iN10 FT-IR microscope combines all the values of infrared microscopy the best in economy, speed, spatial resolution and sensitivity.

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

Africa
+43 1 333 5034 127
Australia
+61 2 8844 9500
Austria
+43 1 333 50340
Belgium
+32 2 482 30 30
Canada
+1 800 530 8447
China
+86 10 5850 3588
Denmark
+45 70 23 62 60
Europe-Other
+43 1 333 5034 127
France
+33 1 60 92 48 00
Germany
+49 6103 408 1014
India
+91 22 6742 9434
Italy
+39 02 950 591
Japan
+81 45 453 9100
Latin America
+1 608 276 5659
Middle East
+43 1 333 5034 127
Netherlands
+31 76 579 55 55
South Africa
+27 11 570 1840
Spain
+34 914 845 965
Sweden/Norway/Finland
+46 8 556 468 00
Switzerland
+41 61 48784 00
UK
+44 1442 233555
USA
+1 800 532 4752

www.thermo.com

Thermo Electron Scientific Instruments LLC, Madison, WI USA is ISO Certified.

TN51513_E 02/08M

Part of Thermo Fisher Scientific