Technical note

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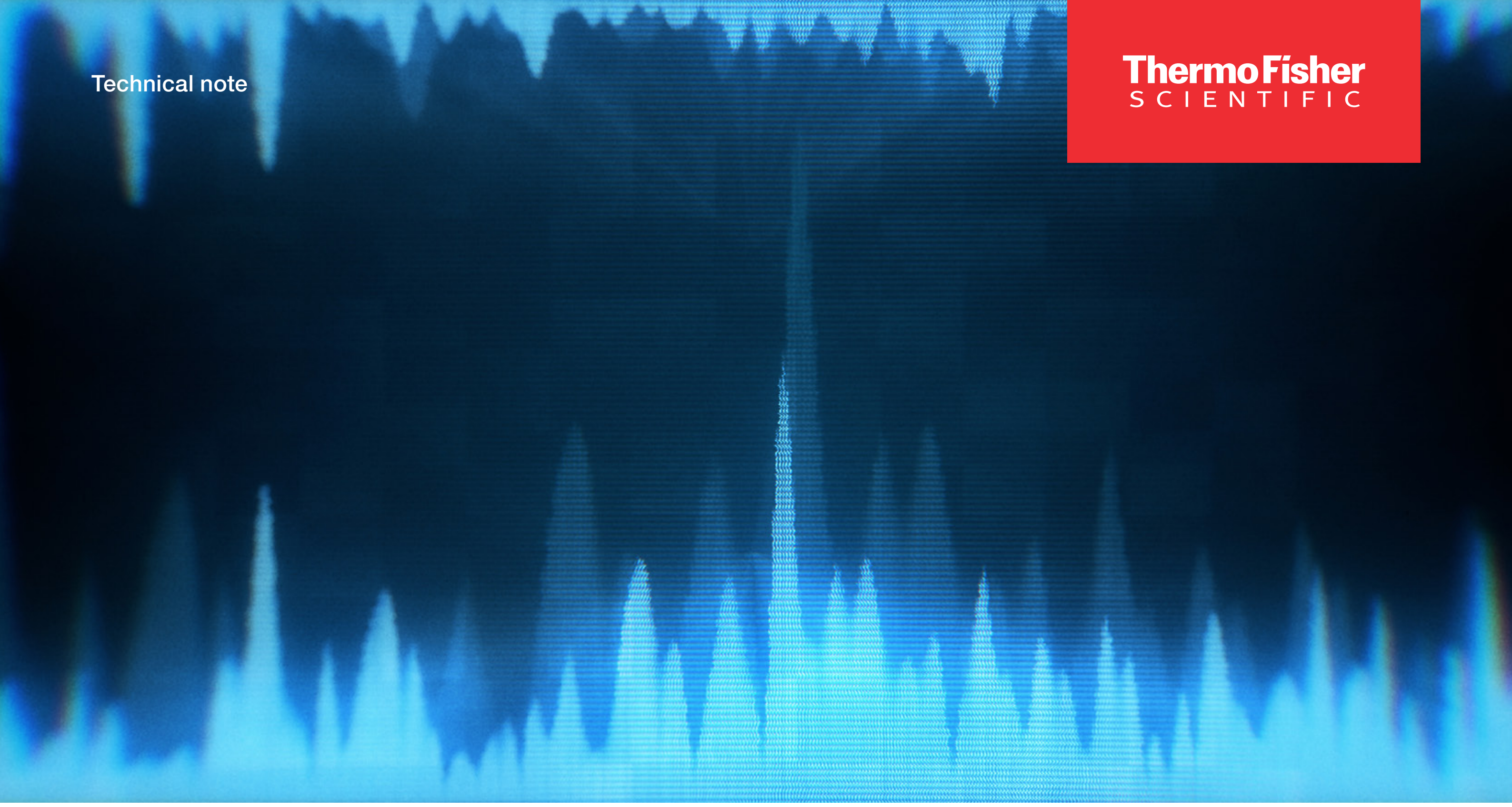

The Thermoelectrically cooled MCT (TEC-MCT) used in an FTIR microscope

Introduction

Fourier transform infrared (FTIR) Microscopy is a workhorse analytical technique in laboratories worldwide. Analysis with FTIR microscopy enables studies of microparticles, multi-layer films, fragments and layers of art or archeologic materials, forensic samples, pharmaceuticals, contaminations, and much more. When using the technique to analyze a sample, certain key steps must be executed: The user must see the sample, target the IR beam accurately upon it, and then collect useful spectra for analysis. This last step requires a high-quality infrared system, from beam source to detector. We focus here on detector options for IR microscopy.

The liquid nitrogen-cooled mercury cadmium telluride (LN2-MCT) detector has been the de facto standard detector in IR microscopes for decades due to its high responsivity and rapid response. The responsivity (D*) is the amount of signal generated per incident photon. A high D* typically yields a good sensitivity and high signal-to-noise ratio (SNR), both essential in IR microscopy where the small aperture limits the photon flux.

The D* for an MCT detector increases as the detector is cooled to ultra-low temperatures (LN2 can achieve -196°C). Critically, the D* generally drops as the spectral range increases. Thus, implementation of any cooling system on an FTIR microscope requires compromises between spectral range and responsivity. As an example of this, the LN2-MCT-A has a D* around 5×10^{10} cm $Hz^{1/2}$ W^{-1} and a spectral range from 11,700 to 600 cm^{-1}, covering most of the mid-infrared region. This is considered the gold standard for microscope detectors: good range and high sensitivity. The alternative LN2-MCT-B has a lower D* around 8×10^{9} cm $Hz^{1/2}$ W^{-1}, but a wider spectral range of 11,700 to 400 cm^{-1}, providing data below 600 cm^{-1} at the cost of lower sensitivity.

While LN2-MCT detectors are well established, some facilities have no access to LN2, while others are striving to reduce or eliminate its use for safety or cost reasons. This makes an alternative to the LN2-MCT system desirable.

A viable alternative involves thermo-electric cooling (TEC) of the MCT detector. Since the D* of MCTs becomes favorable only near -180°C or lower, multi-stage TE coolers have been employed to provide the needed low temperature.

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TEC-MCTs are characterized by different spectral ranges and D* values. The critical factor in choosing a TEC-MCT therefore lies in balancing the same trade-off between spectral range vs D*. While it is possible to obtain TEC-MCTs that “go-low,” the loss in sensitivity becomes steep. However, an effective balance can be found: The Thermo Scientific™ Nicolet™ RaptIR+™ FTIR Microscope uses a TEC-MCT with a cutoff around 900 cm^{-1} but a D* of approximately 3×10^{10} cm $Hz^{1/2}$ W^{-1}, between the liquid nitrogen-cooled MCT-A and the MCT-B.[1]

We describe here the implementation and performance of the TEC-MCT on the Nicolet RaptIR+. Our work in selecting a TEC-MCT was driven by the research emphasis of the Nicolet RaptIR+. High performance was the primary driver. The data shown will provide insight into the impact of this trade-off in real situations.

[1] The values of D* and spectral range provided here for all the detectors are typical, not guaranteed. A wide range of TEC-MCT options were tested during development, and the detector selected was found to be optimal.

Experimental

A Nicolet RaptIR+ coupled with a Thermo Scientific™ Nicolet™ iS50 FTIR Spectrometer and user-swappable LN2-MCT-A and TEC-MCT detectors were used to collect all data. Swapping of detectors can be done without shutting down the instrument, in less than a minute. The LN2-MCT-A can be cooled before insertion (with careful handling) and the TEC-MCT requires about 2 minutes to chill from room temperature to operational temperature. Thermo Scientific™ OMNIC™ Paradigm Software was used to collect and analyze the data. The software automatically recognizes the detector installed and optimizes the collection parameters accordingly.

The first sample was a 50-micron particle of a silane, placed on a KBr window for transmission analysis. A 10×10 micron aperture was used for high spatial resolution, reducing the signal to a very low level. The second sample was a plastic film for transmission analysis. The data was collected using a 20x20 micron aperture. The third sample, used in mapping, was a multilayer film on a gold slide, analyzed via reflection.

The Thermo Scientific Nicolet RaptIR+ FTIR Microscope (left) coupled with the Thermo Scientific Nicolet iS50 FTIR Spectrometer (right).

Results

Figures 1 shows spectra collected from a particle of a silane measured in transmission with a 10×10 micron aperture. The top spectrum is the TEC-MCT result. Despite the limited range and slightly reduced SNR at this small aperture setting, the search result (middle) is still definitive and correct. The bottom spectrum of Figure 1 shows the same particle detected with the LN2-MCT; the wider range and higher SNR are apparent. Of specific interest in comparing these two spectra is the absence, in the TEC-MCT data, of the 800 cm^{-1} peak which is evident in the LN2-MCT-A data.

Figure 2 shows the same comparison with a polymer film. With the slightly larger aperture used, the SNR is very comparable between the top (TEC MCT) and middle (LN2 MCT) spectra. Again, the low wavenumber peaks below 800 cm^{-1} are clearly seen with the LN2 MCT and the search results, but the search against the TEC MCT data yielded an accurate result.

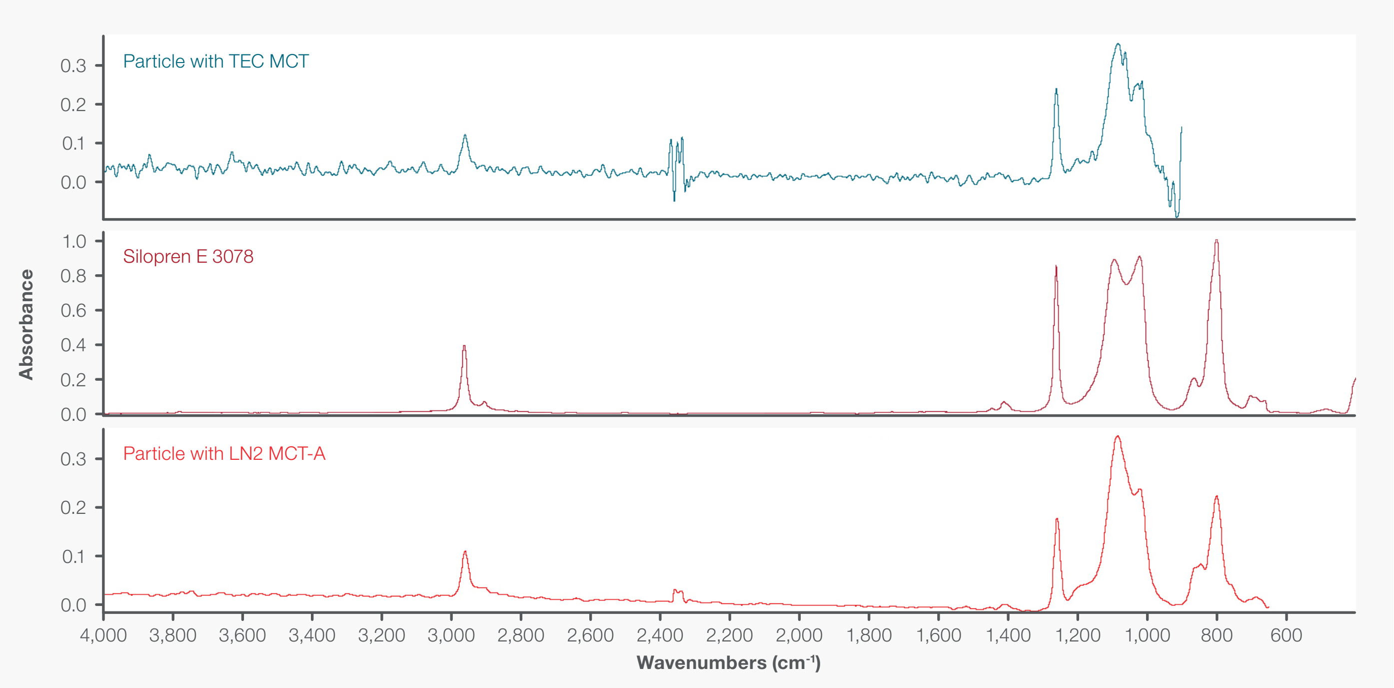

Figure 1. (Top) Siloprene particle collected with a 10×10 micron aperture using the TEC-MCT. (Middle) Library spectrum from the search result against the TEC data. (Bottom) Spectrum using the same particle and aperture but using the MCT-A.

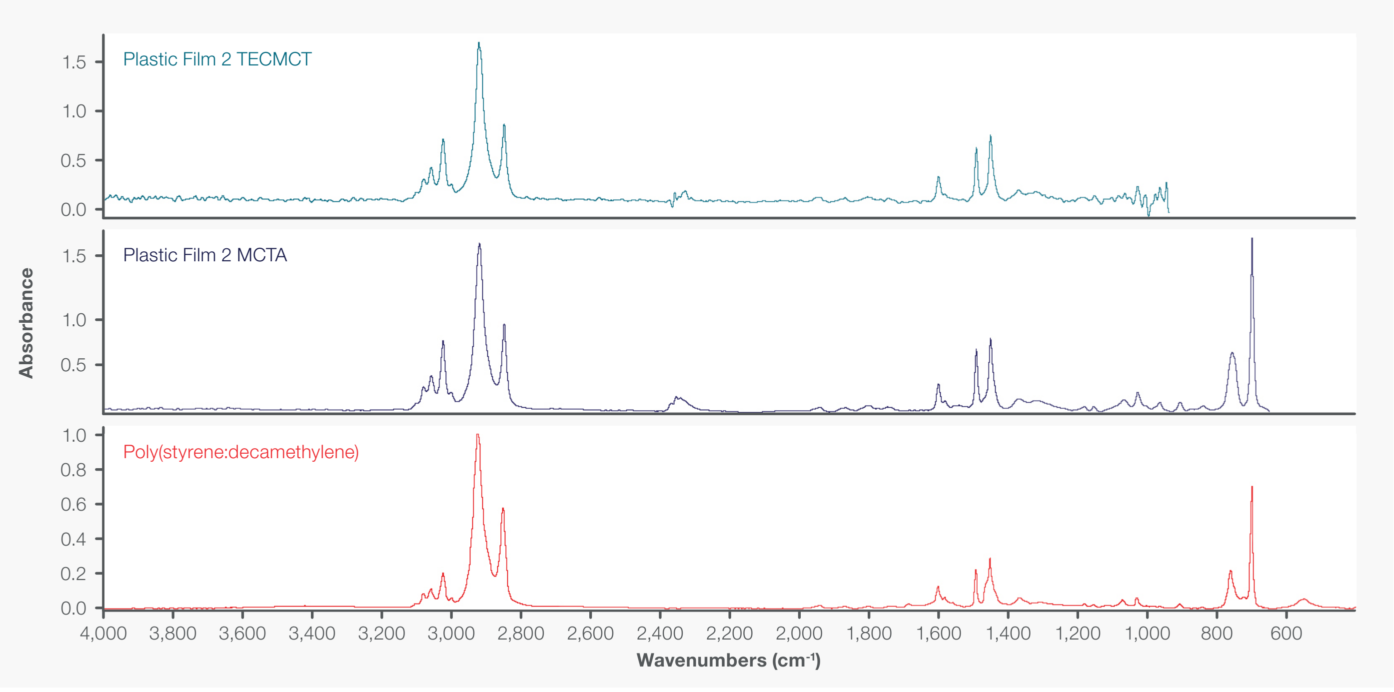

Figure 2. (Top) Plastic film data collected using a 20x20 micron aperture and the TEC-MCT. (Middle) Same sample, 20x20 aperture using the MCT-A. (Bottom) Library spectrum from the search result against the TEC-MCT data.

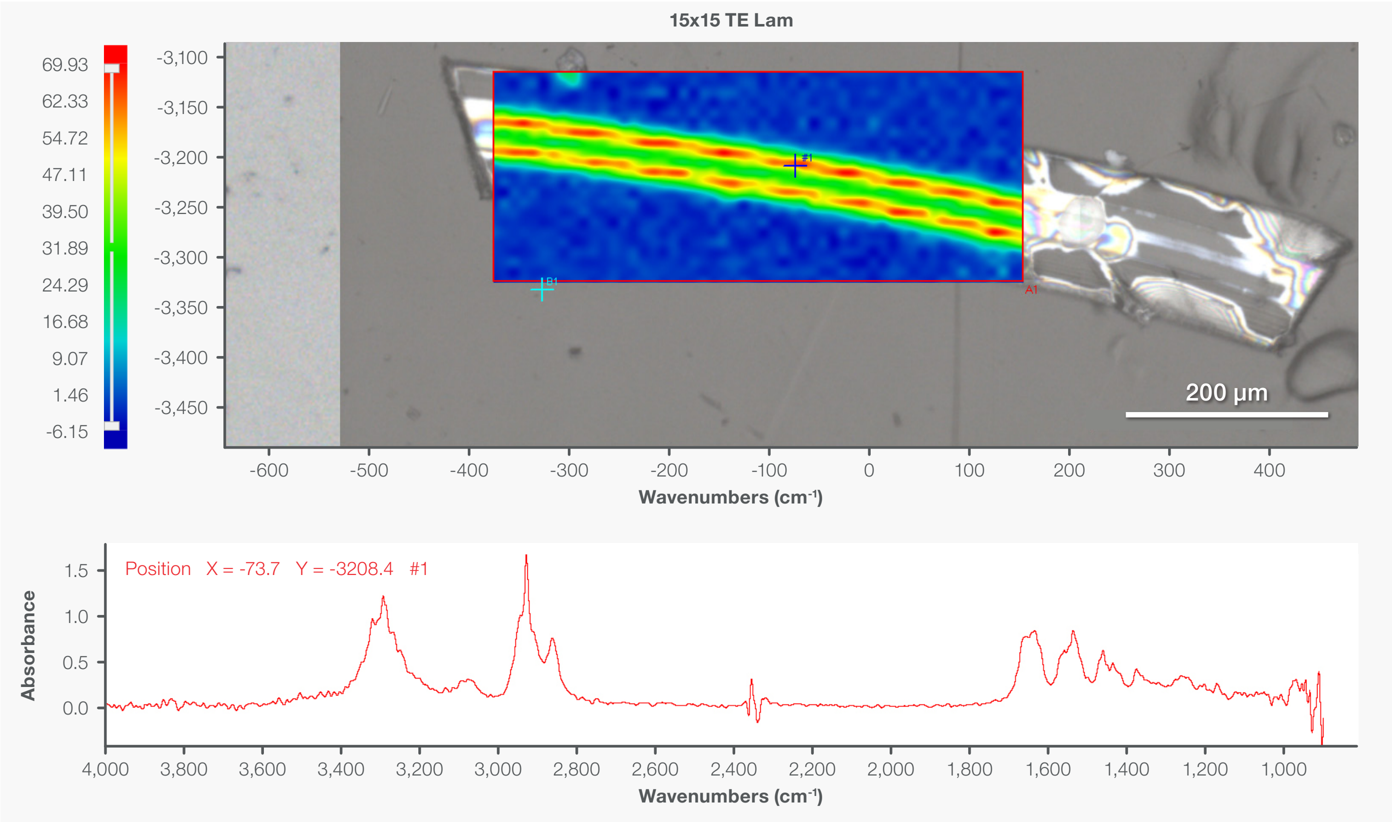

Figure 3. Area map from a laminate, in reflection mode, using the TEC-MCT and a 15x15 micron aperture. B1 was the background point and the blue crosshair indicates where the shown spectrum originates (from a nylon+polypropylene layer). Spectral resolution was set at 8 cm^{-1} and 8 scans were co-added for each spectrum.

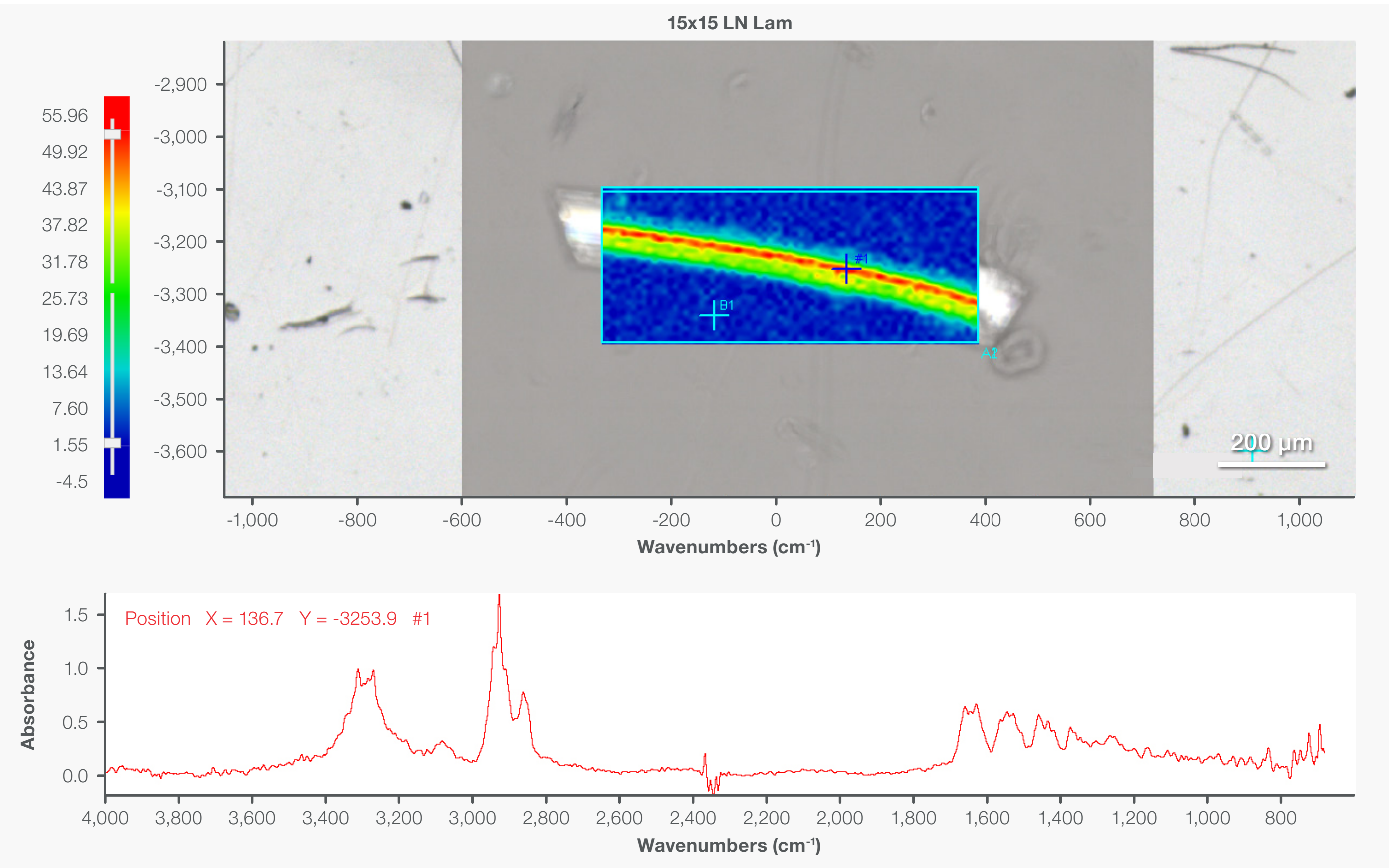

Figure 4. Area map from a laminate, in reflection mode, using the LN2-MCTA and a 15x15 micron aperture. B1 was the background point and the blue crosshair indicates where the spectrum shown originates (from a nylon+polypropylene layer). Each spectrum is a single scan with the spectral resolution set at 8 cm^{-1}. The images are profiles (red high, blue low) of correlation with the nylon spectra.

Figures 3 and 4 compare maps collected in reflection mode using the two different detectors. The lower part of each figure shows a spectrum extracted from the nylon portion of this laminate. Both were collected at 8 cm^{-1} resolution but with 8 scans (TEC) versus 1 scan (LN2) per spectrum. The SNR is excellent in both cases. Further, the maps profiled in color show the same structure of the laminate. All five layers (PP, PP:nylon, PVOH, PE:nylon, and PE) are visible and the overlay with the visual image is excellent. Essentially, there is no difference in either the maps or the underlying data (identification, layer thickness), showing that the TEC-MCT provides an excellent tool for this type of work.

Conclusions

The LN2-MCT-A remains the highest-performance choice for FTIR microscopy, with both a high SNR and wide spectral range. However, the TEC-MCT can provide excellent results for most applications if the limitations are understood and acceptable. Having the thermoelectric cooling option is critical for laboratories lacking access to liquid nitrogen, as other non-LN2 detectors are often significantly inferior in performance. The ability to quickly swap detectors provides Nicolet RaptIR+ users with a wide range of potential solutions with no need to compromise.

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