

# Mid-IR PAT with 10 meter fiber probes

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Fiber optics, coupled to an ATR probe are often used in combination with infrared spectroscopy for industrial reaction monitoring purposes. While fibers in the visible and near-IR spectral regions can have a length of many tens or even hundreds of meters, mid-IR fiber technology is more limited. Even the leaders in mid-IR fibers typically provide only 2-4 m long fiber probes, as the attenuation at longer distances is simply too great for them to be used with commercial FTIR spectrometers.

A solution to this limitation is to increase the power of the spectroscopic light source.

IRsweep's IRis-F1 is based around quantum cascade laser frequency combs. You can [learn](#)

[more about this technology here](#). The most important feature of the IRis-F1, as far as this application note is concerned, is that its lasers can provide a very high power output.

As part of a collaboration with [Pfizer](#) and [art photonics](#), we have investigated the possibility of using very long mid-infrared fibers in a spectroscopic setup. To the best of our knowledge, these are the world's longest mid-infrared fibers ever used for spectroscopy. The purpose of this measurement is to identify the limits of detection for such a measurement in comparison to more traditional techniques.

Please also watch our short video on these measurements, which shows a live measurement through this “record breaking” fiber.

## Experimental setup

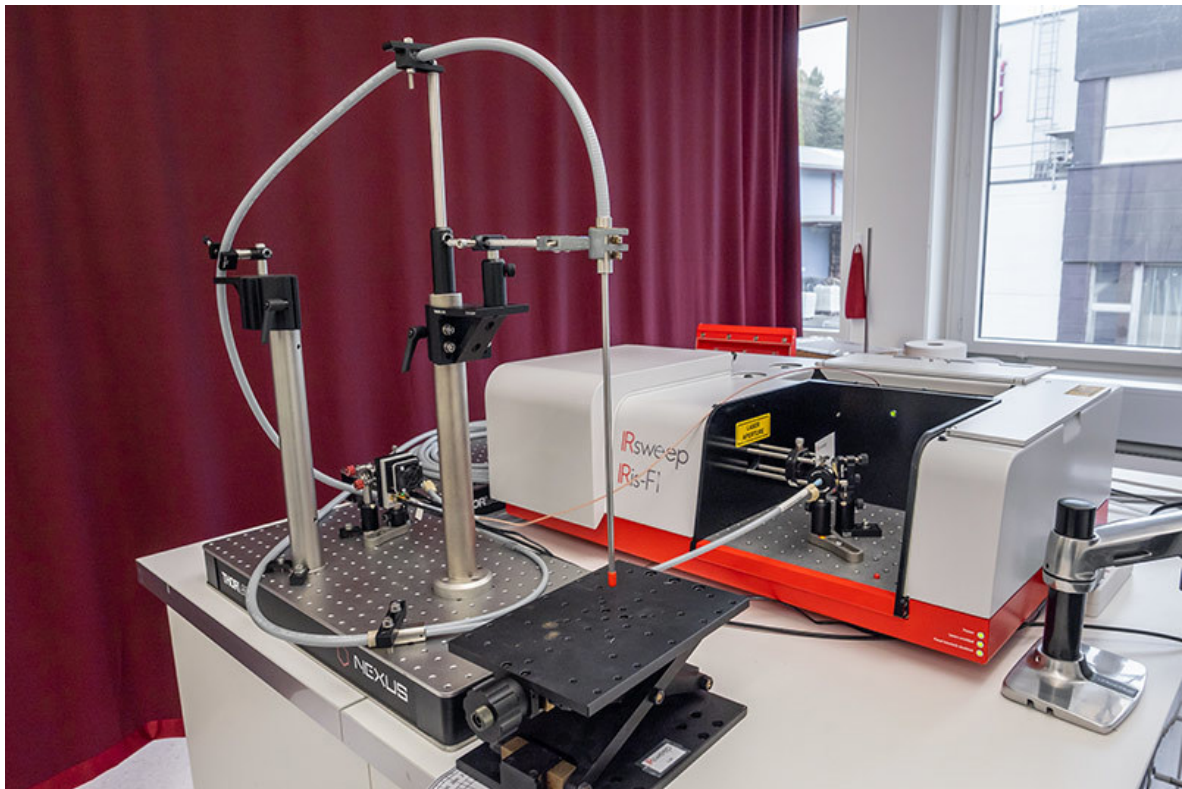


Figure 01: IRis-F1 with coiled up 10 m ATR fiber probe.

The spectrometer used is an IRis-F1 dual comb mid-infrared spectrometer from IRsweep, here with a laser spanning the  $1200 - 1290\text{ cm}^{-1}$  spectral range. The fiber is a custom-made 10m unit from [art photonics](#) (Berlin, Germany) with a two-bounce diamond ATR probe. From input in the spectrometer to output at the detector, the light traveled through 20 m of fiber.

Input coupling was achieved by focusing on the fiber end using a 75 mm ZnSe lens, and output coupling was done with an off-axis parabolic mirror. Here an external detector was used for ease of alignment, but the internal detector of the IRis-F1 would be equally suitable for these measurements.

The fiber was held in the position shown and the samples were exchanged on the lab jack.

Because of space constraints the bulk of the fiber was coiled up for these measurement, but the video also shows a measurement extended on the fully extended fiber.

A test matrix which is representative of reaction monitoring and also relevant to process analytics is mixtures of acetone in toluene. We measured several concentrations to determine the limit of quantification. Each measurement consisted of 15 samples, corresponding to an acquisition time of approximately 250 ms and a processing time of <1 minute.

## Results

The spectra of a range of concentrations of acetone in toluene are shown below. A clear band is seen at  $1215\text{ cm}^{-1}$ , corresponding to the asymmetric C-C stretch of acetone. Gaussian wavenumber averaging has been performed, resulting in a full-width at half maximum spectral resolution of  $3.5\text{ cm}^{-1}$ . A linear baseline correction was done with two points on either side of the analyte peak to isolate the feature of interest.

It is evident that this acetone band is visible until a concentration of approximately 0.1%. This is similar to FTIR measurements, but at a far greater distance. At lower concentrations, we can see baseline fluctuations at similar intensities to the acetone band. We can do a simple linear regression analysis to quantify where the limit of detection lies.

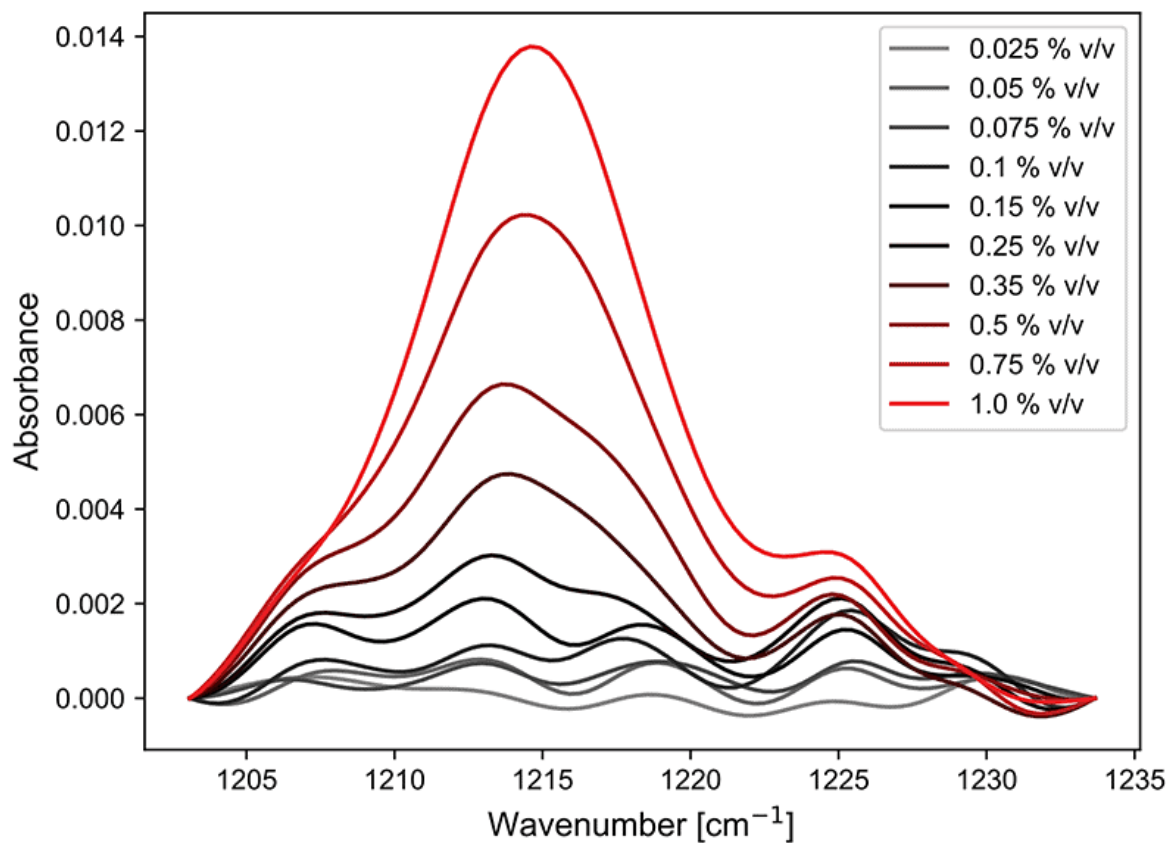


Figure 02: Concentration series of acetone in toluene after linear baseline correction.

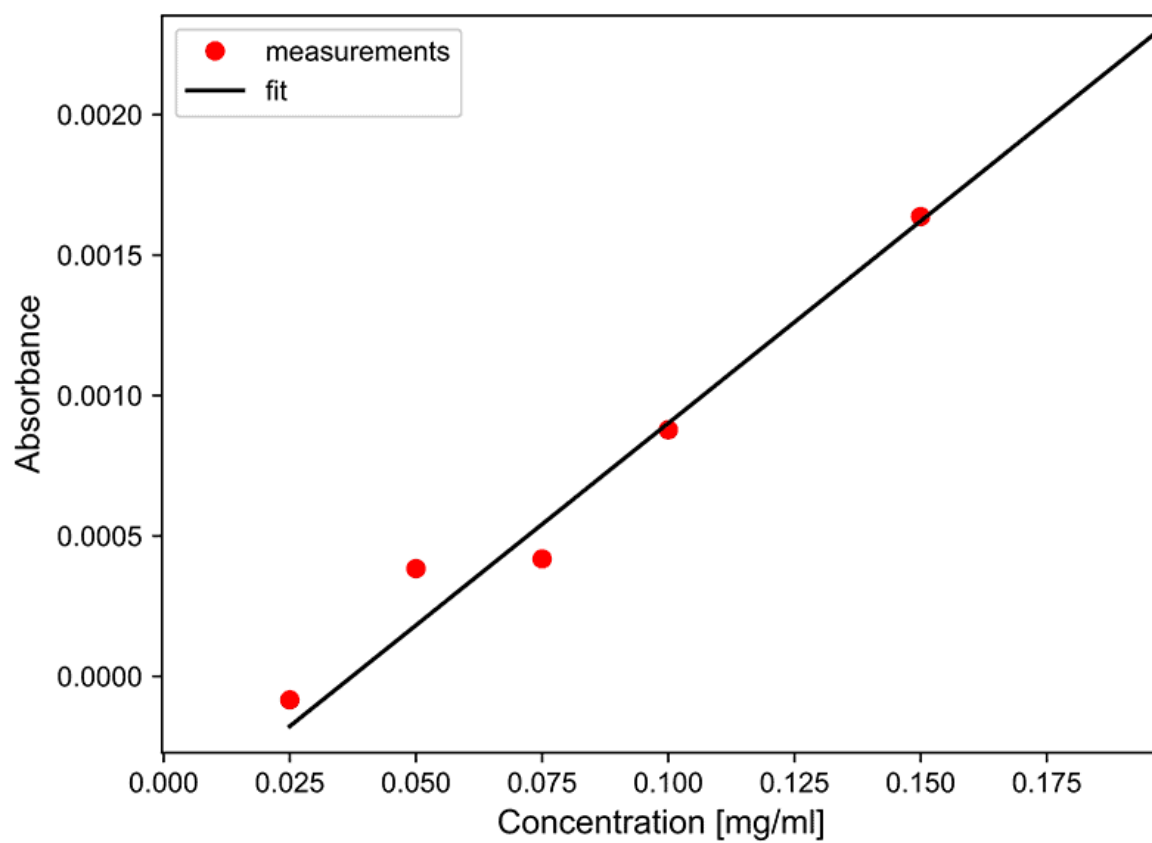
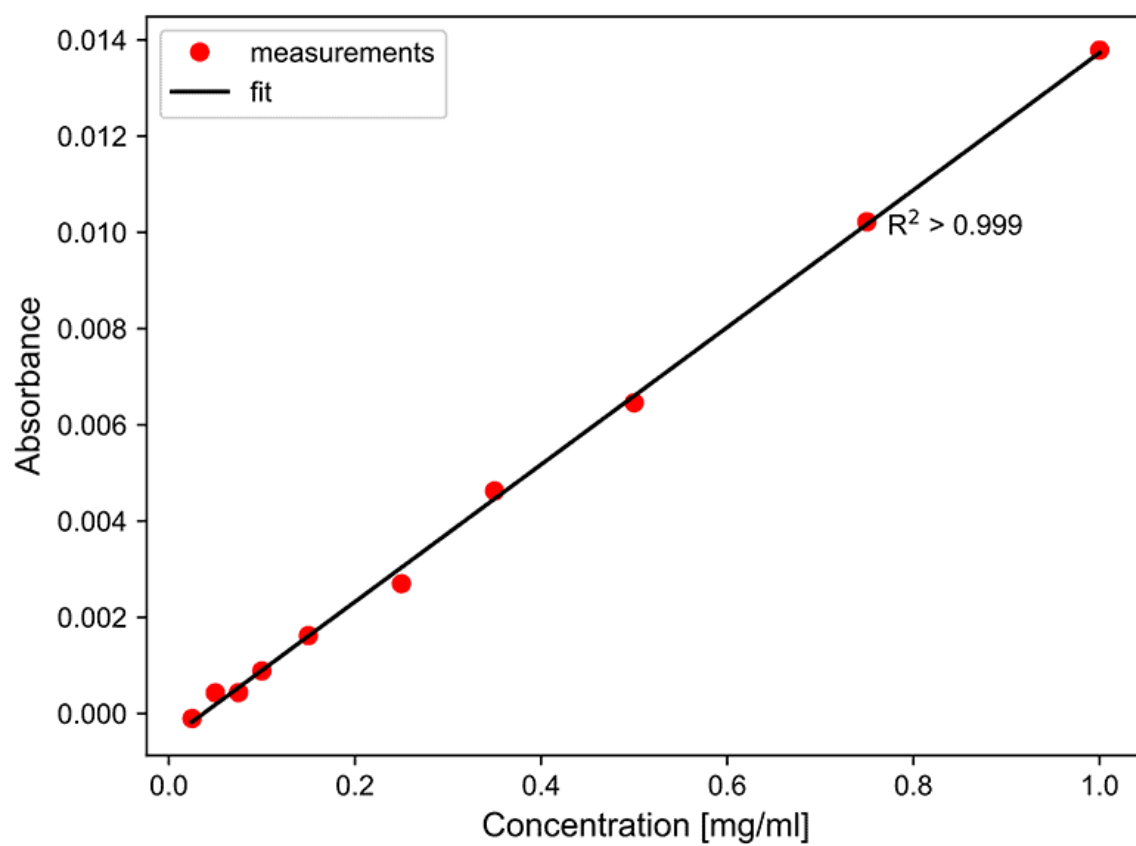


Figure 03 Left panel: Single point linear regression of full concentration range. Right panel: Zoom into low concentrations.

The left panel of figure 03 shows a linear fit of the point at  $1215\text{ cm}^{-1}$  over the entire tested concentration range and the right panel shows a zoom-in of the lower concentration regions. A near-linear relationship is retained even for concentrations below 0.1%. This may be because baseline-fluctuations are wavelength dependent. Although this method can already be used to quantify low concentrations of analyte with low errors, more sophisticated processing that takes into account multiple spectral points is expected to produce even more robust results for future experiments.

## Conclusions and outlook

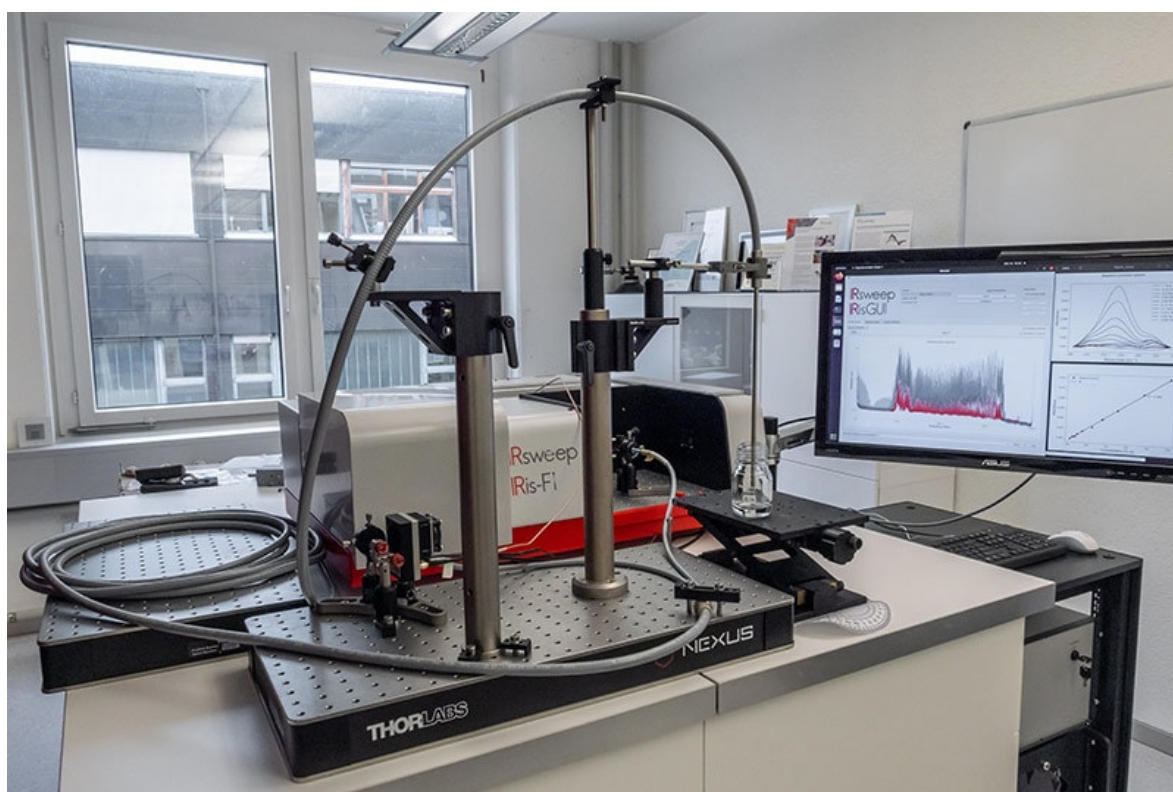


Figure 05: IRsweep IRis-F1 with coupled 10 meter fiber ATR probe.

This application note showcases spectroscopic measurements through a 10 m mid-

infrared fiber designed and built by [art photonics](#). We report good sensitivity of our test system, which is acetone in toluene. The performance is in line with existing FTIR methods but the length of the fiber used is unprecedented.

Although an internal detector could be used, for the measurements shown here we used an external detector for ease of alignment. If an external detector is application-compatible, a future version of the fiber could be used to make the light-return unnecessary by simply moving the detector assembly close to the probe. To operate, it would need a low-voltage DC power supply and a signal cable back to the spectrometer. Using this approach, even longer fibers – up to 20 m – would be feasible without changes to the [IRis-F1](#).

## Additional content:

» Video: [Mid-infrared measurement demo through 10 m long optical fiber](#)

## Related product



### IRis-F1

The IRis-F1 spectrometer offers you an unmatched combination of speed, brightness and multi-color measurements. The quantum cascade laser

frequency comb spectrometer is the first turn-key frequency comb spectroscopy system. As opposed to single-frequency operation, which necessitates tuning over your spectral range of interest, you will achieve a broad spectral coverage in a single-shot measurement.

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