

## APPLICATION NOTE PA301

Measuring single particles or fibers with cantilever-enhanced photoacoustic spectroscopy



### Application

Photoacoustic infrared spectroscopy is a valuable tool for examining bulk of materials and has some advantages against conventional infrared techniques such as no need for sample preparation, contactless measurement and depth profiling capabilities. However, in some cases a bulk of the sample material is not available and single particles have to be measured. If the particles are very small, they are often called microsamples.

Infrared analysis of single particles is an established area in both science and industry, and there are numerous applications, which require the analysis of microsamples. The problem in conventional infrared sampling techniques of microsamples is that the sampling often leads to low reproducibility and signal-to-noise ratios (SNR). Photoacoustic spectroscopy (PAS) of microsamples with normal sampling is not optimal either. The local heating of the sample does not converge into the surrounding air but instead into the sample cup. Therefore, the observed photoacoustic signal is lower than it should be.

A solution to this problem is to elevate the sample a few millimeters above the sample cup surface. This can be done with the Micro Sampling Kit, developed by S.T. Japan for Gasera PA301 photoacoustic detector. This kit consists of two different sampling heads, a tungsten needle for single particles and a special clamp for fiber samples. It also contains fine tweezers, magnifying glass, a magnetic rod for the tungsten needle for picking up the single particles and a light and dark background for the fiber sampling.



Fig 1. Gasera PA301 photoacoustic detector for the analysis of solids, semi-solids and liquids.



Fig 2. S.T. Japan Micro Sampling Kit for Gasera PA301 photoacoustic detector. The kit contains all the necessary parts for sampling and measurement of microsamples.

## Measurement results

The microsampling capabilities of PA301 were demonstrated with hair fibers. Hair is a troublesome sample material for conventional infrared techniques, especially when measuring single fibers. All samples were measured using a Bruker Tensor 37 FTIR spectrometer, PA301 and the Micro Sampling Kit. The photoacoustic cell was purged with helium for enhanced signal. The measurement parameters are presented in Table I.

Table I. Measurement parameters.

Number of scans	100	
Measurement time	4	min
Mirror velocity	2.2	kHz
Resolution	8	cm <sup>-1</sup>
Spectral range	400-4000	cm <sup>-1</sup>

The photoacoustic mid-infrared spectrum of a 5 mm long piece of hair fiber was measured with a regular PA301 sample cup and with the Micro Sampling Kit. The signal increase achieved with the microsampling accessory can be seen in Fig 3, and is quantified in Table II. Table II shows three times higher signal and almost five times higher SNR when using the fiber sampler. Also, the features in the spectrum are much more pronounced with the fiber sampler as seen in Fig 3. The measurements were extremely repeatable with less than 1 % deviation in the highest photoacoustic signal intensity between consecutive measurements.

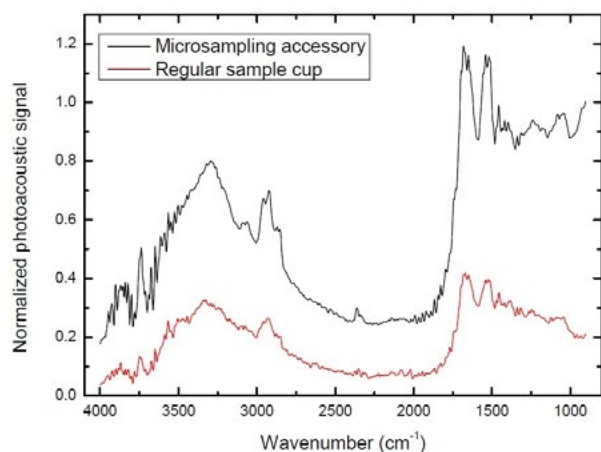


Fig. 3. The signal increase achieved with the Micro Sampling Kit when measuring a 5 mm long hair fiber.

Table II. Photoacoustic signal level and SNR between different sampling methods.

Sample holder type	Highest signal intensity (a. u.)	SNR
Regular sample cup	0.42	23
Micro Sampling Kit fiber holder	1.19	107

In another study, the effect of the sample size to the photoacoustic signal was studied. Five different sample sizes were measured with the same FTIR-PAS setup and the spectra are shown in Fig 4. Fig 4 shows that with only 0.5 mm of sample material, a good quality photoacoustic spectrum that accurately retains the features of the sample can be obtained. Based on the spectra in Fig 4, the sample material could easily be identified from a single piece of fiber. The 0.5 mm hair sample in the fiber holder is photographed in Fig 5.

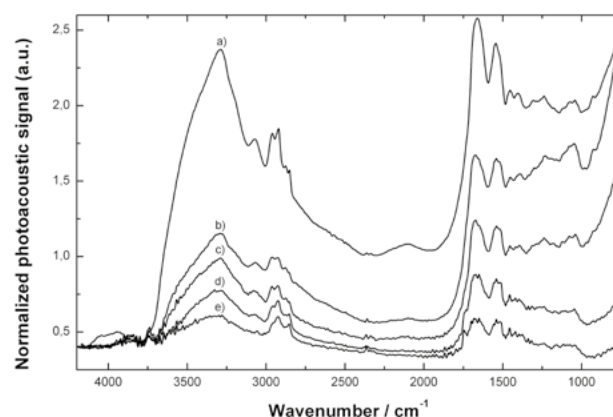


Fig. 4. Photoacoustic spectra of hair samples of different sizes. a) A bulk of hair, b) 8 mm, c) 5 mm, d) 2 mm and e) 0.5 mm piece of hair fiber.

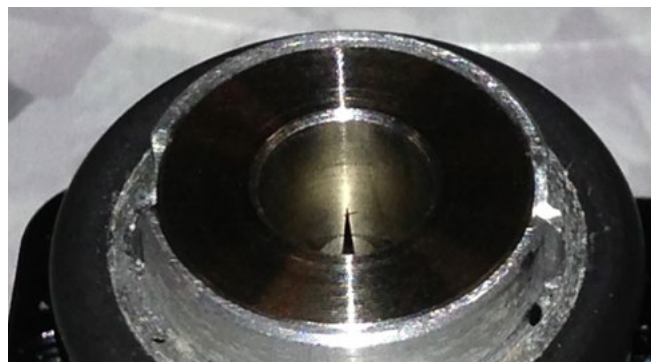


Fig 5. A 0.5 mm piece of hair fiber in the tip of the sharp tungsten needle of the particle holder.

## Conclusion

This application note shows that the microsampling capabilities of PA301 can be further enhanced by using special sampling tools such as tungsten needle. The S.T. Japan Micro Sampling Kit opens up new possibilities for cantilever-enhanced PAS, and PA301 can be used to improve the results in infrared spectroscopy of single particles, for example in unknown material characterization and quality control in industrial processes. Photoacoustic measurement is contactless, which is a key benefit with fragile microsamples. Also, sample material is not consumed during the measurement. Due to its high sensitivity, cantilever-enhanced PAS can work in a small scale formerly reached only by infrared microscopy.