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DISPERSIVE INFRARED PHOTOTHERMAL BEAM DEFLECTION
SPECTROSCOPY OF SOLIDS

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Key Words: Photoacoustic Spectroscopy, Infrared Photothermal Beam
Deflection Spectroscopy, Dispersive Spectroscopy

INTRODUCTION

Since the time the first¹ dispersive broad-band infrared (IR) photoacoustic spectra of solids were reported, practically all IR photoacoustic and photothermal beam deflection work has been done with interferometers. The sample illumination and consequent spectra obtained with interferometers are, of course, much better than those obtained with a dispersion instrument²⁻⁵. However, continuously-scanning interferometers lead to continuously-varying modulation frequencies with attendant difficulties involving penetration depth and signal phase⁶, and spectral profiles can change when different scan speeds are used⁷. With such instruments one has, essentially, little control over some parameters which might be vital in certain circumstances; a step drive would be better.

With these difficulties in mind, and also because of the mundane but practical problem that FT instruments are expensive and beyond the means of many workers, we thought it worthwhile to re-activate our dispersion spectrometer⁸. It was rejuvenated, fitted with a "mirage" detector⁹, and used to explore the feasibility of observing broad-band IR photothermal beam deflection signals. Some initial results are reported.

EXPERIMENTAL

A dispersion photoacoustic spectrometer⁸ based on a Perkin-Elmer Model 421 spectrophotometer was modified. The original source (a Nernst glower) as well as the two source mirrors used to provide the "sample beam" were re-installed so that a light beam passed directly through the entrance slit to the interchange collecting mirror. A chopper operating at 19 Hz was mounted just past the exit slit, and the mirage detector shown in Fig. 1 was installed. The path length from the sample, which was mounted on a micrometer-adjustable platform, was about 70 cm. As the mirage detector is extremely sensitive to vibration, the entire system was mounted on an air table and well shielded.

The probe beam deflection was monitored with a PIN-spot 2D (United Detector Technology) detector; one of two channels of the electronics described elsewhere¹⁰ was used and signals were processed by a lock-in amplifier. The step drive⁸ was retained. The system was managed by an Apple II+ microcomputer plus peripherals.

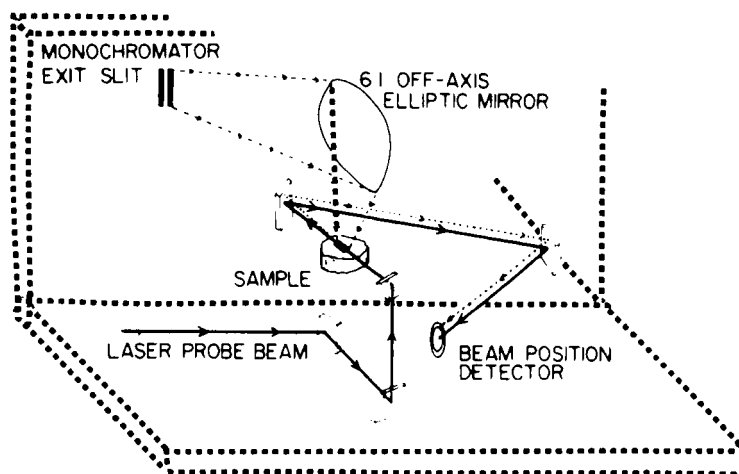


Fig.1 Mirage detection system

RESULTS

Some initial results are shown in Fig. 2. For convenience in handling, the sample powders were lightly pressed to form cylinders (about 3 mm dia. x 3 mm high). The spectra were recorded with the slits fixed and "wide open", i.e., 2 mm, the resulting resolutions of approx. 36, 19, 28 and 7 cm^{-1} at 4000, 3000, 2000 and 1000 cm^{-1} , resp.. The spectra were recorded in two segments; because of the poorer performance after the grating change, different numbers of scans were used. Savitzky-Golay noise reduction was done by a single pass of the data through a 7-point digital filter¹². The measurements employed a time constant of 3 secs., $6\text{ cm}^{-1}/\text{step}$, and a delay of 6 secs. between steps (to allow noise produced by the stepper to die out).

Spectrum A of the reference carbon shows the summation of the effects of the variation of the source with wavenumber and the

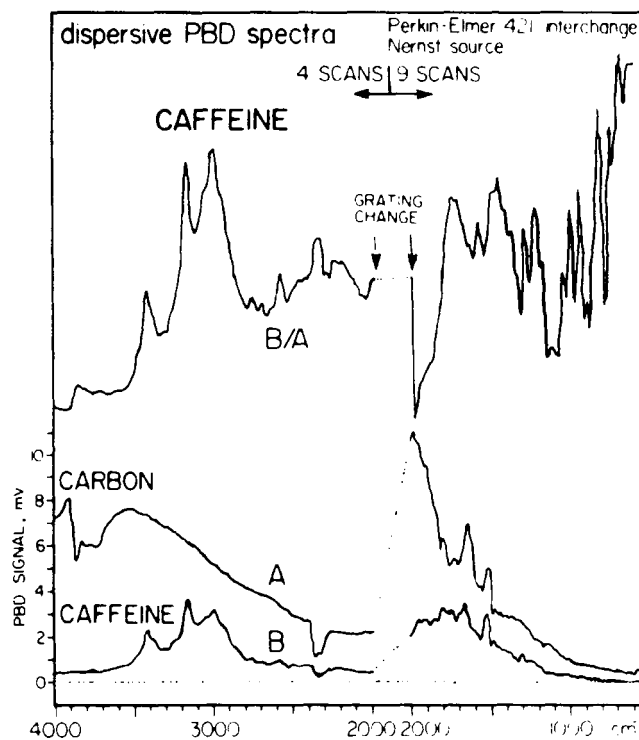


Fig.2 Dispersive IR beam deflection spectra

characteristics of the interchange, and is much like the photo-acoustic spectrum measured with the same instrument prior to modification. The much less intense spectrum B of the caffeine is quite similar to the FT-PBD spectrum¹¹. The compensated spectrum B/A shows the band structure and relative band intensities of the caffeine spectrum quite well, except below about 800 cm^{-1} (the relatively large increase in the apparent signal was probably caused by a drift of the intensity of the source).

CONCLUSIONS

The results show the feasibility of recording usable spectra of particulate solids over the $4000 - 700 \text{ cm}^{-1}$ range, and it seems possible and probable to us, after subduing the instrument's environment, to improve the performance somewhat and also to extend the range to 500 cm^{-1} . Considering that a conventional IR source was used, the results are perhaps astonishingly good.

The rate at which spectra are recorded over an extended range seems painfully slow in comparison to the performance of an FT spectrometer. That, however, is of little importance for studies of signal strength as function of chopping frequency, phase, bed packing, and so on, which can indeed be carried out with the instrument. A bonus is that the instrument's components are relatively cheap.

Dispersive IR-PBDS can be a useful tool.

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