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## **Infrared Photoacoustic Spectra of Solids**

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## INFRARED PHOTOACOUSTIC SPECTRA OF SOLIDS

Key Words: Photoacoustic Spectroscopy. Infrared

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### INTRODUCTION

Although photoacoustic spectroscopy (PAS) has become a valuable analytical and research technique for optical studies of solids,<sup>1,2</sup> almost all work has been confined to the visible range of the spectrum. The absorptions of solids in that range are mainly broad, structureless and non-specific, so that visible-PAS is of limited utility for chemical analysis; the infrared range is required for that.

Several successful attempts have been made to use PAS in the IR range, e.g.,<sup>3-6</sup> (also, several IR-PAS experiments are outlined in the digest of papers of the Topical Meeting on Photoacoustic Spectroscopy<sup>7</sup>). However, these studies employed lasers as excitation sources, e.g., a cw HF laser was used in a study of the OH region of the spectrum of chrysotile asbestos,

the region from  $\sim 3800$  to  $\sim 3400 \text{ cm}^{-1}$  being covered by 12 HF laser lines.<sup>6</sup> Only very small portions of the IR range were examined, so that IR-PAS via presently available tuned lasers remains of limited utility.

Coverage of a broad IR region is needed for chemical studies, and this requirement caused us to examine the feasibility of recording IR-PAS spectra of surface species, employing a broad-band source<sup>8</sup>. We now show that relatively good results can be obtained in the  $4000\text{-}880 \text{ cm}^{-1}$  region.

### Experimental

The PAS system was based on a Perkin-Elmer Model 421 spectrometer. The entire source assembly, recorder, detector and mirrors past the exit slit were removed, and all functions of the instrument except the slit drive were disabled. Radiation from a carbon rod furnace IR source similar to that described by Boyd et.al.<sup>9</sup> was focussed directly out the entrance slit by two mirrors (a hole was cut in the case) so that the mirrors which normally fold the beam were bypassed. The optical path thus contained only 6 reflecting surfaces, the last of which was an off-axis ellipse which gave a 6:1 reduction of the exit slit image at the sample position. The signal from a 1/2" B&K Model 4165 condensor microphone (or from 2 microphones used in a differential mode) operated with a Type 2804 power supply<sup>10</sup> was fed to a Model 116 differential amplifier and Model 124A amplifier locked to a Model 192 variable frequency chopper<sup>11</sup>

operating at 45 Hz, though a 15-bit A/D converter incorporated in a generalized I/O to a Nova 2 computer fitted with peripherals and suitable software. A P&E Model 621 interchange was fitted with end-of-travel switches and a fiduciary marker, and driven by a step drive<sup>12</sup> operable by the computer. The spectra shown were recorded with dual, minimum volume, non-resonant cells used in a differential mode. The slits were constant at 1950 microns, yielding resolution computed at 31, 29, 21, and 6  $\text{cm}^{-1}$  at 3000, 2000, 1600 and 1000  $\text{cm}^{-1}$ , resp. The scan rate was 50  $\text{cm}^{-1}/\text{min}$ , so that the spectra shown required  $\sim 1$  hr. The samples were  $\sim 1$ -3 mg of powder. Operation from 4000 to 700  $\text{cm}^{-1}$  was possible. Merely for convenience, to save time because there were no spectral features worth recording at the higher wave-numbers, scans were started near 3700  $\text{cm}^{-1}$ . The region below  $\sim 880$   $\text{cm}^{-1}$  was too noisy to be useful.

## Results

Trace A of Fig. 1 is the "background," i.e., the PAS signal obtained using a charcoal sample with the 2000  $^{\circ}\text{K}$  source. The abrupt increase in signal at 2000  $\text{cm}^{-1}$  is caused by the automatic change in grating, and many other changes in signal are caused by automatic changes of the order-sorting filters. The spectrum, which is stored in core, disc, and magnetic tape memory, also shows the absorption of atmospheric  $\text{CO}_2$  and water vapor. Trace B of Fig. 1 is a similar single-beam spectrum

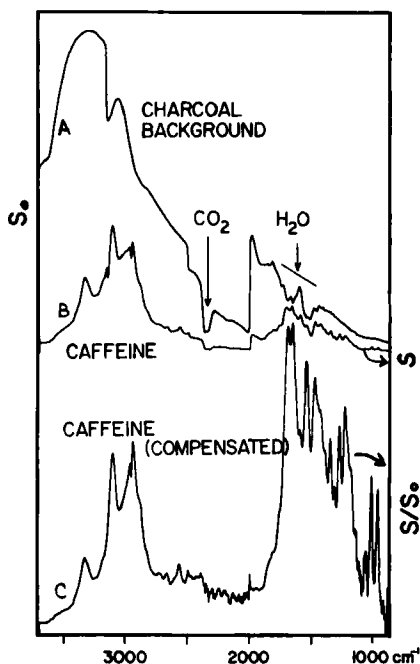


Fig. 1. Single-beam and corrected spectra

A: "background" spectrum of charcoal sample. B: spectrum of caffeine, uncorrected. C: spectrum of caffeine, corrected for background by ratioing against spectrum A, then ordinate scale-expanded.

obtained with caffeine powder. Correction for "background" is made by ratioing the two stored spectra by digital computation. The resulting spectrum C of caffeine, ordinate scale-expanded to arbitrary extent, is shown in Fig. 1.

Some other spectra are shown in Fig. 2. Each spectrum resulted from a single scan (like those of Fig. 1) and is scale-expanded to the same arbitrary extent. The signal-to-noise ratios are reasonably good except in the  $\sim 2400\text{--}2000\text{ cm}^{-1}$  region where

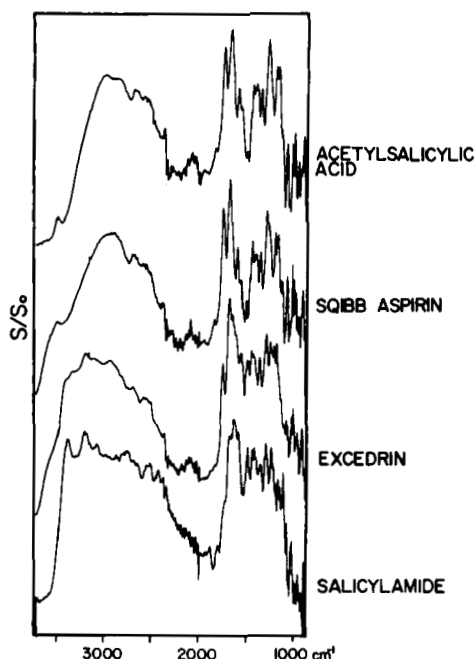


Fig. II. Infrared Photoacoustic Spectra

Each spectrum has been corrected for background and scale-expanded.

the signal strength declines markedly (cf. trace A, Fig. 1) and beyond  $880\text{ cm}^{-1}$  (not shown), so that large scale-expansions are feasible (Fig. 3).

Cursary comparison of the fingerprint spectra of the two commercial mixtures shows that there are substantial differences in composition: comparison of those spectra ( $3700\text{--}3000\text{ cm}^{-1}$ ) with that of acetylsalicylic acid (the main active component of both products) suggests the presence of inorganic fillers; segments of the spectra of aspirin and pure acetylsalicylic acid

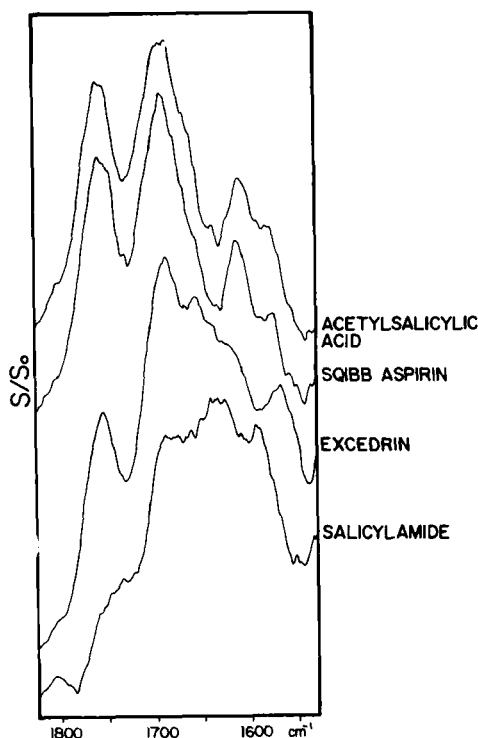


Fig. III. Sections of IR-PAS Spectra

Scale-expanded segments of the spectra of Fig. 2.

show little difference (Fig. 3); that of Excedrin suggests the presence of salicylamide (Fig. 3); and so on.

### Conclusions

The examples shown indicate that it is possible to obtain PAS data of reasonable quality in the IR range and, even with the present limited resolution and noise, spectra which are useful can be recorded. It would obviously be better to be able

to do so with a high power laser source. However, until lasers tunable over a substantial portion of the IR range become practical, it seems worthwhile developing the IR-PAS technique with a broad-band source. With its capability or being able to examine thick, highly scattering samples, IR-PAS can become a valuable ancillary IR technique.

#### ACKNOWLEDGMENT

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