TECHNICAL NOTE

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Use of a Silicon Carbide Sampling Accessory for the Diffuse Reflectance Infrared Fourier Transform Analysis of Samples of Interest to Forensic Science

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ABSTRACT: An infrared spectroscopy method is described which requires little sample preparation and may be used for analysis of a wide range of samples of interest to forensic science. A small quantity of a sample is rubbed onto an abrasive silicon carbide disk, which is then measured by diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS). The technique has been successfully applied to the infrared analysis of paint, synthetic rubber, cosmetics, corrector fluid, and adhesives.

KEYWORDS: criminalistics, spectroscopic analysis, diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS), infrared spectroscopy, Fourier transform infrared (FTIR) spectroscopy, Fourier transform, diffuse reflectance, silicon carbide paper

Since the early development of the technique by Fuller and Griffiths in 1978 [*1*], diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) has found numerous applications in criminalistics. The introduction and acceptance of the technique in forensic science has been largely due to work published by Suzuki and Gresham [*2–4*], by Suzuki alone [*5*], and by Suzuki and Brown [*6*]. Some types of samples, such as tablets, powders, polymeric foams, and metallic paint, can be measured directly by DRIFTS. Other solid samples should be ground and diluted with powdered potassium bromide (KBr) before analysis by infrared (IR) spectroscopy. Liquid and semiliquid samples (such as oils and greases) can also be measured by DRIFTS but must first be diluted with a solvent before being applied to a bed of powdered KBr.

The use of abrasive silicon carbide (SiC) coated disks is an alternate sampling technique for DRIFTS which has not, to our knowledge, been directly applied to the treatment of samples of interest to forensic science. In this technique, the sample is simply rubbed on

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the SiC paper until sufficient material has been deposited. The diffuse reflectance infrared spectrum of the sample is then measured *in situ* on the surface of the paper. Spragg [7] first suggested this method in 1984 as a rapid sample preparation technique, and he demonstrated its usefulness for analysis of polymeric materials. Some industrial applications of the SiC sampling technique have been reported in the literature, such as that reported by Chalmers and MacKenzie [8], in which the cladding on corrugated glass-reinforced plastic sheeting was analyzed. A sampling kit using silicon carbide disks is now commercially available from Spectra-Tech Inc. The method is extremely rapid, and the spectra obtained are generally in good agreement with traditional transmission spectra [7]; however, a relatively large sample surface is normally required.

We have found that smaller SiC disks can be conveniently made and used to perform DRIFTS measurements on relatively small quantities of sample. Preliminary results with adhesives, paint, cosmetics, synthetic rubber, and corrector fluids have indicated that the sampling technique using small SiC disks is a simple and rapid method for obtaining IR spectra of good quality.

Materials and Methods

A silicon carbide sampling kit was obtained from Spectra-Tech Inc. (Article No. 0030-130). Smaller disks were made from the 12-mm SiC disks provided with the kit, using a

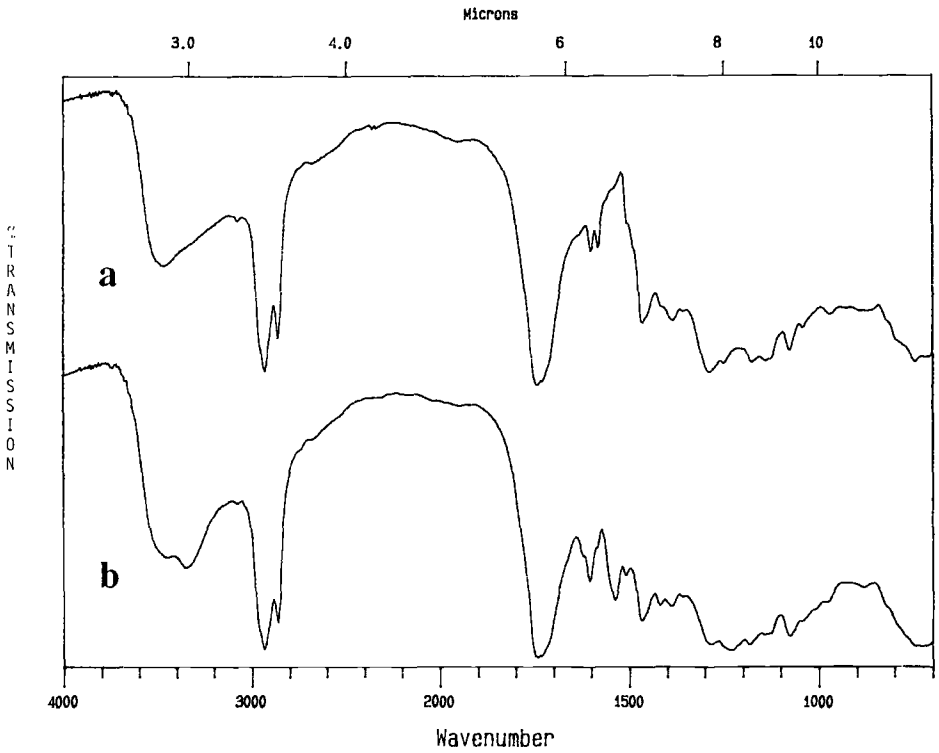

FIG. 1—*Infrared spectra obtained from the SiC/DRIFTS analysis of two samples of white domestic paint:* (a) *alkyd based and* (b) *polyurethane based.*

5-mm-diameter hole punch. Infrared spectra were recorded using a Collector diffuse reflectance accessory (Spectra-Tech Inc.) on a Mattson Cygnus 100 Fourier transform infrared (FTIR) spectrometer fitted with a narrow-range mercury/cadmium telluride (MCT) detector. Measurements were made by fixing the 5-mm SiC disks to the top of the 3-mm sample cup provided with the Collector diffuse reflectance accessory. Spectra were calculated from 200 accumulated scans at a resolution of 8 cm^{-1} and a moving mirror velocity of 2.53 cm/s (34 s of total scan time). A background spectrum was measured on the SiC paper before application of the sample. In cases where specular reflectance bands were visible in the spectrum, powdered potassium bromide (KBr) was applied to the surface of the SiC disk and the infrared measurement was repeated. In most cases, this significantly reduced the amount of specular reflectance while still providing good-quality spectra.

Samples of synthetic rubber (from shoe soles), white domestic paint, red automobile paint, lipstick, glue (the samples had been left to harden on a glass surface), and corrector fluid (applied to a paper surface) were analyzed by DRIFTS after being transferred to a 3-mm SiC disk by rubbing. The resulting IR spectra, although recorded by diffuse reflectance, are displayed in the "% transmission" format (except for Spectrum 5b, where a transmittance to absorbance conversion was used) and are uncorrected. The "% trans-

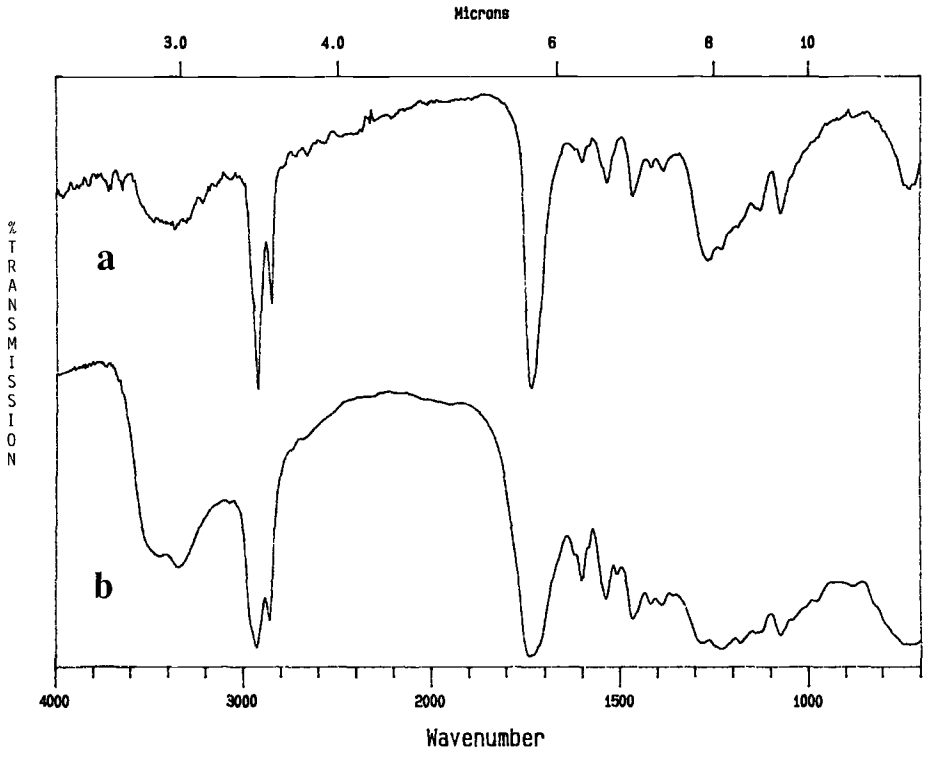

FIG. 2—*IR spectrum of a sample of alkyd-based white paint measured:* (a) *by transmission FTIR microspectrometry and* (b) *by DRIFTS after transfer to a SiC disk.*

mission" ordinate scale was chosen for the practical reasons outlined by Suzuki and Gresham [2].

Results and Discussion

The SiC/DRIFTS analysis of samples of white domestic paint provided good results, as can be seen in Fig. 1—in this example, one spectrum shows characteristics typical of a polyurethane-based paint, while the other shows an alkyd base. The spectra were in agreement with those obtained by transmission FTIR microspectrometry (Fig. 2). Automobile paints were equally well suited to analysis by this method (Fig. 3). The lipsticks tested were not easily differentiated by DRIFTs, but this was probably due to their similar composition. Specular reflection was often a problem with the DRIFTS analysis of lipstick samples—the specular component of the reflection, however, was found to be efficiently reduced by dusting the surface of the sample with powdered KBr (Fig. 4). Figure 5 shows the IR spectrum of a cyanoacrylate-based adhesive sample, measured using the SiC method, compared with the spectrum of a similar adhesive taken from a commercially available database (Adhesive and Sealants Library, Sadtler Research Laboratory, Bio-

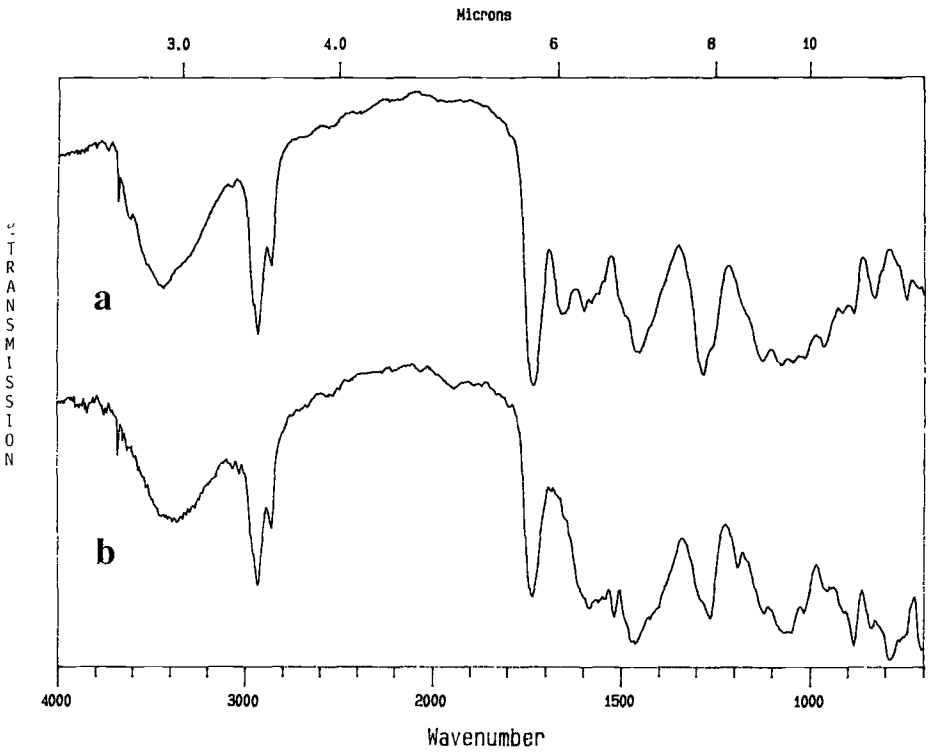

FIG. 3—*Infrared spectra of two samples of red automotive paint measured by DRIFTS after transfer of small quantities to SiC disks.*

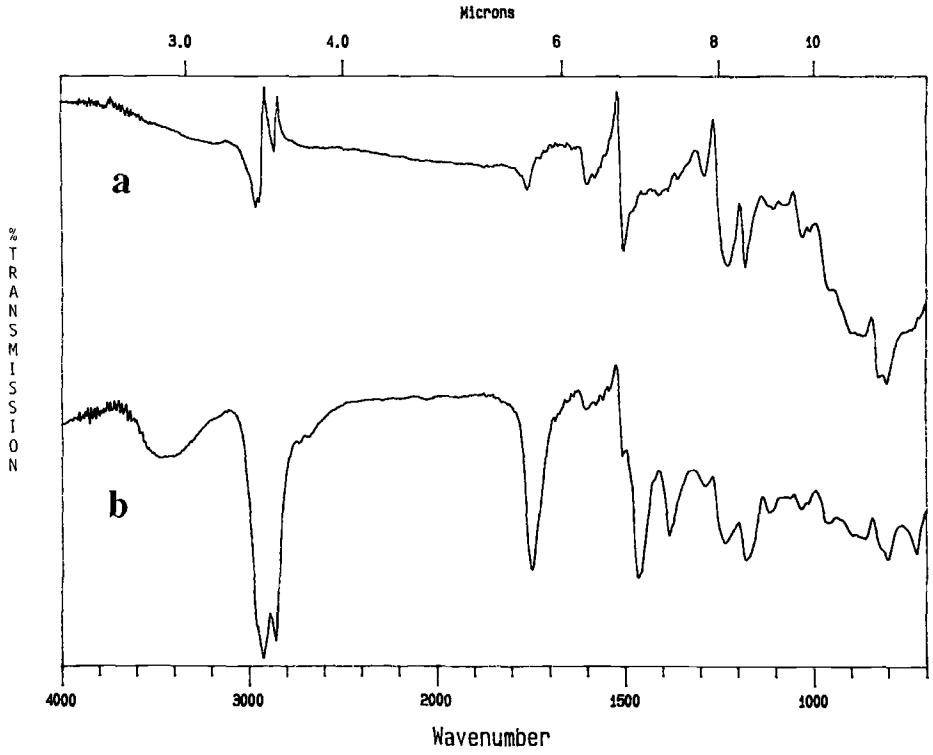

FIG. 4—*DRIFTS spectrum of a lipstick sample on SiC paper:* (a) *showing the effect of specular reflection and* (b) *after dusting with KBr powder.*

Rad Laboratories, Inc., Philadelphia, Pennsylvania). This comparison illustrates the high quality of the IR spectra obtained by the SiC sampling technique. Different brands of corrector fluid could also be differentiated after measurement by DRIFTS (Fig. 6). Figure 7 shows spectra obtained from the IR analysis of synthetic rubber taken directly from the soles of two different pairs of athletic shoes.

Conclusions

The results demonstrate the value of this simple and rapid sampling technique for analysis of a wide range of samples. Although the technique is relatively sensitive (only a small quantity of sample, approximately 5 $\mu g/mm^2$ [7], needs to be transferred to the SiC paper), particularly small samples (less than about 0.5 mm^2) are difficult to apply to the surface of an SiC disk. In such cases, more appropriate sampling techniques, such as using the FTIR microscope, should be employed. If sufficient sample is available, however, DRIFTS analysis using abrasive silicon-carbide-coated disks is a practical alternative.

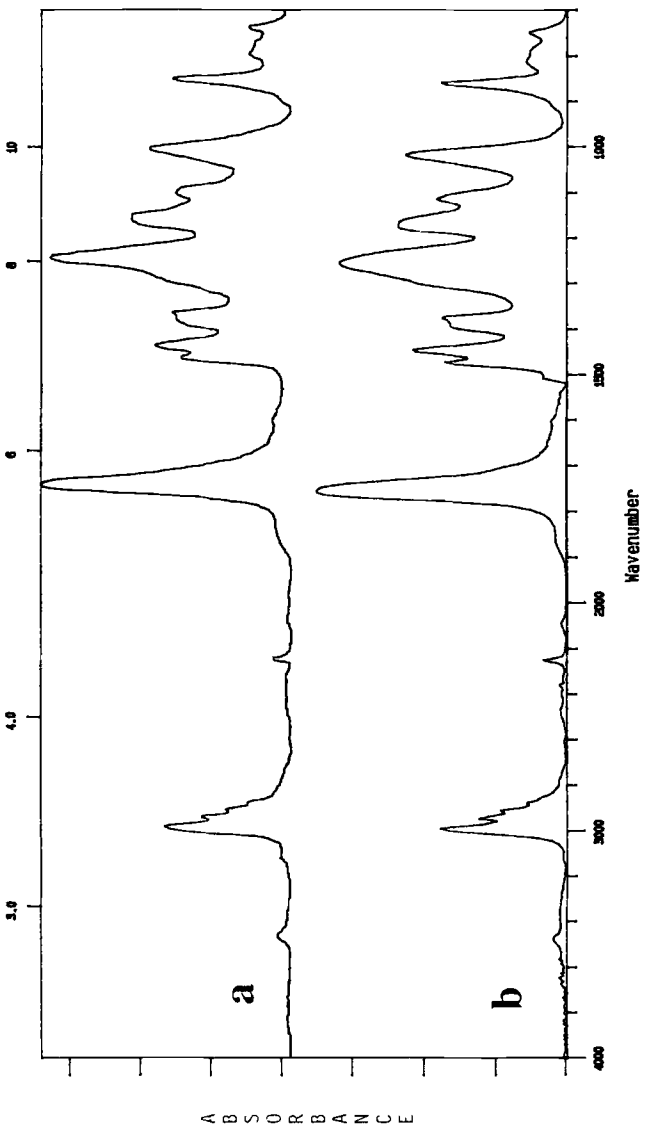

FIG. 5—*Comparison between* (a) *the IR spectrum of a cyanoacrylate-based glue taken from a commercial database (Sadtler Adhesives and Sealants Library) and* (b) *the SiC/DRIFTS spectrum of a similar adhesive (Pattex Super-Gel).*

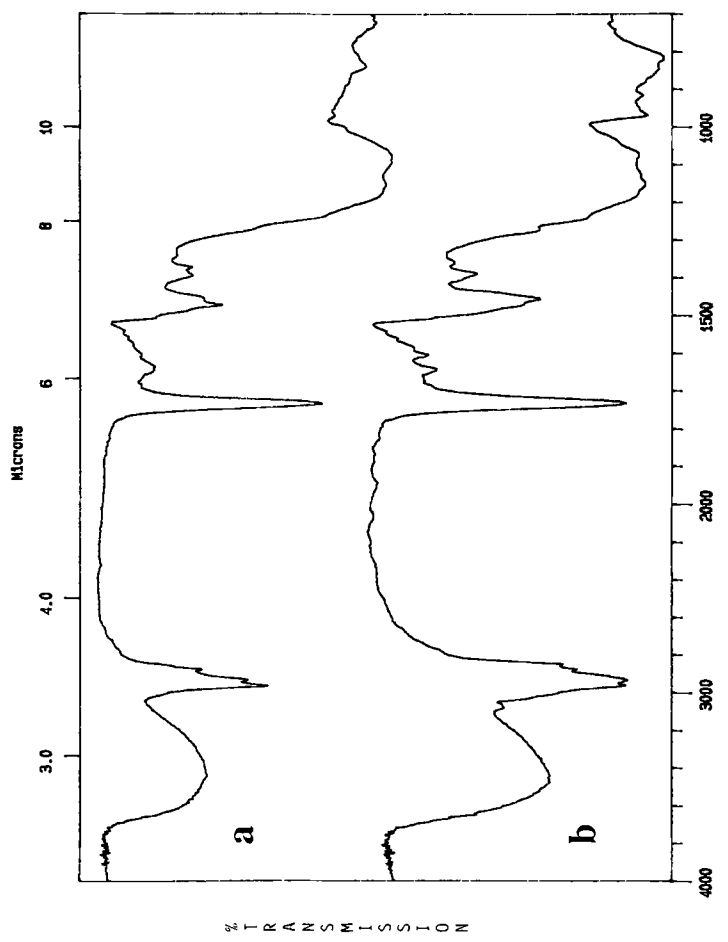

FIG. 6—*Spectra recorded using the SiC/DRIFTS method for two samples of corrector fluid:* (a) *Tipp-Ex Cop-Ex Fluid and* (b) *Pentel Correction Pen.*

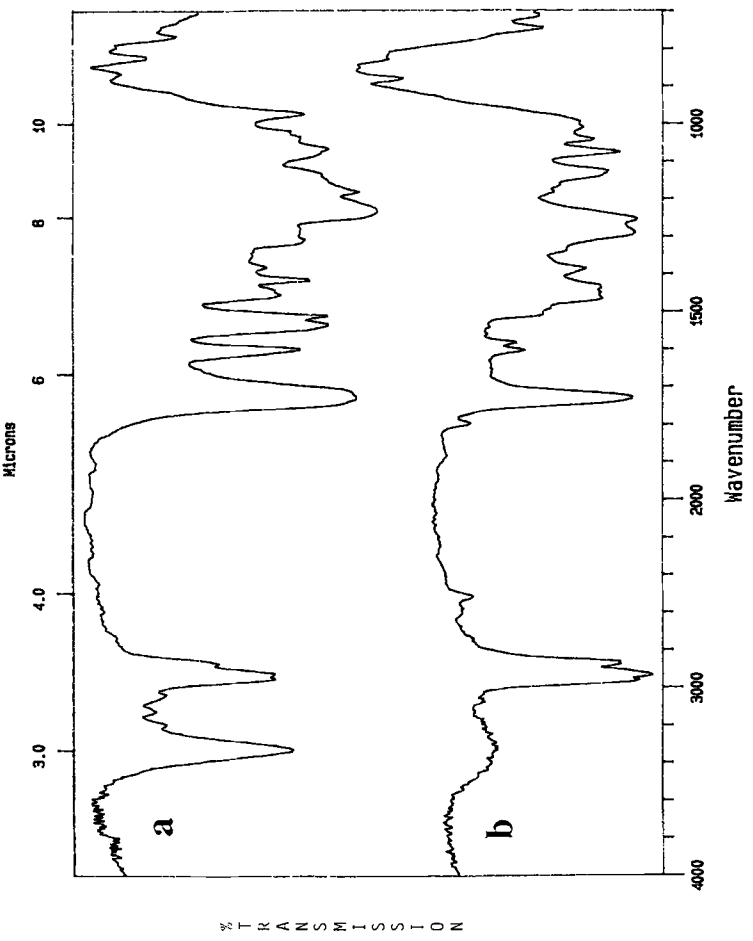

FIG. 7—*IR spectra of two samples of synthetic rubber taken from the soles of two different pairs of athletic shoes using SiC disks.*

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