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# Analysis of Pressure Sensitive Adhesive Tape: I. Evaluation of Infrared ATR Accessory Advances*

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**ABSTRACT:** Attenuated total reflection (ATR), also known as internal reflection spectroscopy (IRS), is a forensically accepted method for infrared (IR) analysis of pressure sensitive adhesive tapes. Advancements of ATR accessories in the last decade have provided the forensic examiner several ATR methods to choose from. These accessories offer a variety of ATR crystal choices with a variety of prices and capabilities. Four different types of pressure sensitive adhesive tapes including duct tape, electrical tape, packaging tape and office tape have been used to compare six different ATR methods. Each of the methods tested offers both benefits and limitations which must be considered for the type of sample to be analyzed. The intent of this paper is to assist the reader in better understanding ATR techniques, the many differences among currently available ATR accessories and how the method is applied to the analysis of pressure sensitive adhesive tape.

**KEYWORDS:** forensic science, tape, adhesive, polymer backing, infrared spectroscopy, FT-IR, attenuated total reflectance, internal reflection spectroscopy, criminalistics

The characterization of pressure sensitive adhesive tapes for forensic purposes has been explored using both physical and analytical techniques (1–8). General information on tape adhesives has also been published (9,10). This paper is the first in a series intended to present a detailed study on the forensic analysis of several types of pressure sensitive adhesive tapes. Infrared (IR) analysis of pressure sensitive adhesive tapes is an established procedure within the FBI Laboratory and attenuated total reflection (ATR), otherwise known as internal reflection spectroscopy (IRS), is an often chosen technique to obtain the IR spectrum of both the adhesive and backing sides of a tape sample. ATR is a surface analysis technique that requires little sample preparation. The authors developed a method during the late 1980s which involved the use of a standard size, multireflection ATR accessory for analysis of electrical tapes (11). Over the past decade, ATR techniques have improved dramatically with the development of numerous, commercially available, micro ATR accessories. Bartick et al. described microscopical ATR and its application to the analysis of several types of forensic evidence including electrical tape (12). Such equipment is a great asset to forensic laboratories expected to provide detailed analyses of small fragments of polymeric evidence. The availability of so many ATR accessories, each with their own set of benefits and limitations, has created some confusion among forensic examiners as to which accessory is best suited to their needs. Overall, ATR has proved useful for the analysis of both the adhesive and backing sides of several pressure sensitive tape types including polyethylene coated cloth tape commonly referred to as duct tape, polyvinyl chloride backed electrical tape, polypropylene backed packaging tape and clear cellulose acetate backed office tape. A study was undertaken to determine which type of ATR device is best suited for forensic tape analyses.

*Equipment and Samples*

All studies were conducted at 4 $cm^{-1}$ resolution using a Nicolet (Madison, WI) Magna 760 IR spectrometer equipped with a deuterated triglycine sulfate (DTGS) detector, cesium iodide (CsI) beamsplitter and a Nic Plan microscope with a mercury-cadmium-telluride-A (MCT-A) detector. ATR methods tested included two accessories for the main bench and two accessories for the IR microscope. Two different internal reflection elements (IREs) or ATR crystals were tested for each of the microscope techniques. Thus, a total of six different methods were studied. Spectra-Tech Inc.'s (Shelton, CT) variable angle ATR with a macro KRS-5 IRE and the DuraSamplIR™ by SensIR Technologies, LLC (Danbury, CT) with a diamond/KRS-5 IRE were tested within the main bench compartment. The IR microscope ATR techniques included Spectra-Tech's ATR slide-on with germanium (Ge) and silicon (Si) IREs and Spectra-Tech's ATR objective with zinc selenide (ZnSe) and diamond IREs. All of the techniques offer single reflection, microanalysis capabilities except the variable angle ATR which is a 17 reflection, standard accessory. These six ATR methods were compared by analyzing both the adhesive and backing sides of six electrical tapes, four duct tapes, one packaging tape and two office tapes as listed in Table 1.

## Results and Discussion

Each of the six ATR methods tested offer both benefits and limitations but all produced usable spectral information. Figure 1 shows baseline adjusted adhesive spectra from a Shuford Mills duct tape, Shurtape PC 620, obtained by the six ATR methods studied. The most effective technique for a particular tape examination will depend on the type of tape being analyzed, the size of the available sample and financial considerations. Table 2 summarizes the properties of the ATR techniques studied. Selection of the ATR crystal should be based on the refractive index of the sample being analyzed. The refractive index of the ATR crystal, $\eta_1$, should be

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significantly higher than the refractive index of the material being tested, $\eta_2$, in order to obtain an undistorted spectrum (13). Typically, polymeric materials used in the manufacture of tapes, have a refractive index near 1.5. All the IRE materials considered in this study have significantly higher refractive indices as shown in Table 2. If $\eta_1$ and $\eta_2$ are close, evidence of dispersion may appear in the resulting spectra. Dispersion effects appear as severely sloping baselines and downward peaks prior to strong absorption bands. The presence of carbon black in a tape adhesive increases $\eta$ of the adhesive and can cause such effects. Use of a Ge IRE ($\eta = 4.0$) for such samples will reduce the effects of dispersion. With other IREs, the presence of dispersion bands offers information as to the adhesive composition, but some spectral details may be suppressed.

TABLE 1—*Tapes used for initial studies to determine the best technique for analysis.*

| Tape Type | Manufacturer or Distributor | Model |
|---|---|---|
| Duct Tape | 3M | Scotch Cat. 131 |
| Duct Tape | Manco | B-200 |
| Duct Tape | Shuford Mills | Shurtape PC 620 |
| Duct Tape | Tuck Tape | 9260 |
| Electrical Tape | 3M | Scotch Super 33+ |
| Electrical Tape | 3M | Scotch Super 88 |
| Electrical Tape | 3M | Temflex 1700 |
| Electrical Tape | Industron | 440 Series |
| Electrical Tape | Manco | 667 |
| Electrical Tape | Plymouth | Premium 85 CW 4240 |
| Packaging Tape | 3M | 3750 |
| Office Tape | 3M | Scotch Magic Tape |
| Office Tape | 3M | Scotch Double Stick Tape |

The variable angle ATR, with a multireflection KRS-5 crystal, was considered the technique of choice in the late 1980s for IR analysis of electrical tape within the FBI Laboratory (11). It provides a method of rapid, nondestructive analysis with high quality spectral results. However, this technique requires a clean piece of tape at least 5 to 10 mm wide. Laboratory submissions are often received as wadded masses of tape that, with some effort, can be separated to locate a clean piece within the mass. Under such circumstances, the single reflection ATR accessories may be applied to case specimens with greater ease than the standard, multireflection accessories. A significantly smaller clean area of the tape is required with the small contact areas of the single reflection accessories.

Spectra-Tech's ATR slide-on and ATR objective both provide single reflection techniques through the IR microscope. The actual circular spot size for these accessories is dependent on the size of the aperture used, the objective magnification and the refractive index of the ATR crystal ($\eta_1$). The small spot size offered by these accessories can be beneficial, but the need to perform the analysis through the IR microscope sets the lower analysis limit to 650 $cm^{-1}$ due to the cutoff point of the MCT-A detector. This eliminates spectral information below 650 $cm^{-1}$ that may be useful for indicating the presence of some inorganic materials used in tape adhesives.

Use of a single reflection ATR accessory for within-compartment operation combines the benefits of the microscope accessories with the benefits of the multireflection bench accessories. Other brands of single reflection ATR accessories that operate within the sample compartment are commercially available but only the SensIR accessory was tested as part of this study. For this accessory, the effective contact area is a round spot, 0.75 mm in diameter. The durability of the diamond IRE allows the user to apply pressure for suitable sample contact and cleanup can be done with

TABLE 2—*ATR techniques compared for analysis of pressure sensitive adhesive tapes.*

| Accessory and Crystal | Used with | Range Limit* | $\eta$ | $d_p$ @ 1000 $cm^{-1}$† | Contact Dimensions | Cost§ | Remarks |
|---|---|---|---|---|---|---|---|
| Variable angle ATR with KRS-5 (multi-reflection) | Bench DTGS | 260 $cm^{-1}$ | 2.37 | 2.1 µm | 17 reflections across 4.5 cm length | * | KRS-5 deforms under pressure. |
| Single reflection ATR with Diamond/KRS-5 | Bench DTGS | 260 $cm^{-1}$ | 2.4 | 2.0 µm | 0.75 mm spot size | ** | KRS-5 strengthened by diamond presence. |
| Slide-on with Ge | Microscope MCT-A 15X obj. | 650 $cm^{-1}$ | 4.0 | 0.69 µm | 0.053 mm spot size‡ | ** | Ge is hard and brittle. |
| Slide-on with Si | Microscope MCT-A 15X obj. | 650 $cm^{-1}$ | 3.4 | 1.1 µm | 0.063 mm spot size‡ | ** | Si is hard and brittle. |
| ATR Objective with ZnSe | Microscope MCT-A 15X obj. | 650 $cm^{-1}$ | 2.4 | 2.0 µm | 0.089 mm spot size‡ | *** | ZnSe is hard and brittle. |
| ATR Objective with diamond | Microscope MCT-A 15X obj. | 650 $cm^{-1}$ | 2.4 | 2.0 µm | 0.089 mm spot size‡ | **** | Diamond is very hard and withstands high pressure. |

* The lower detection limit for instrument conditions used in this study.

† The depth of penetration (dp) was calculated at 1000 $cm^{-1}$ ($\lambda$) with a 45° incident angle ($\Theta$) for a sample material whose refractive index ($\eta_2$) is 1.5.

‡ Contact dimensions are based on the use of the microscopical ATR accessory with a 3.2 mm circular aperture and an objective magnification of 15 times.

§ Cost comparison is based on the assumption that an IR microscope is currently available in the laboratory.

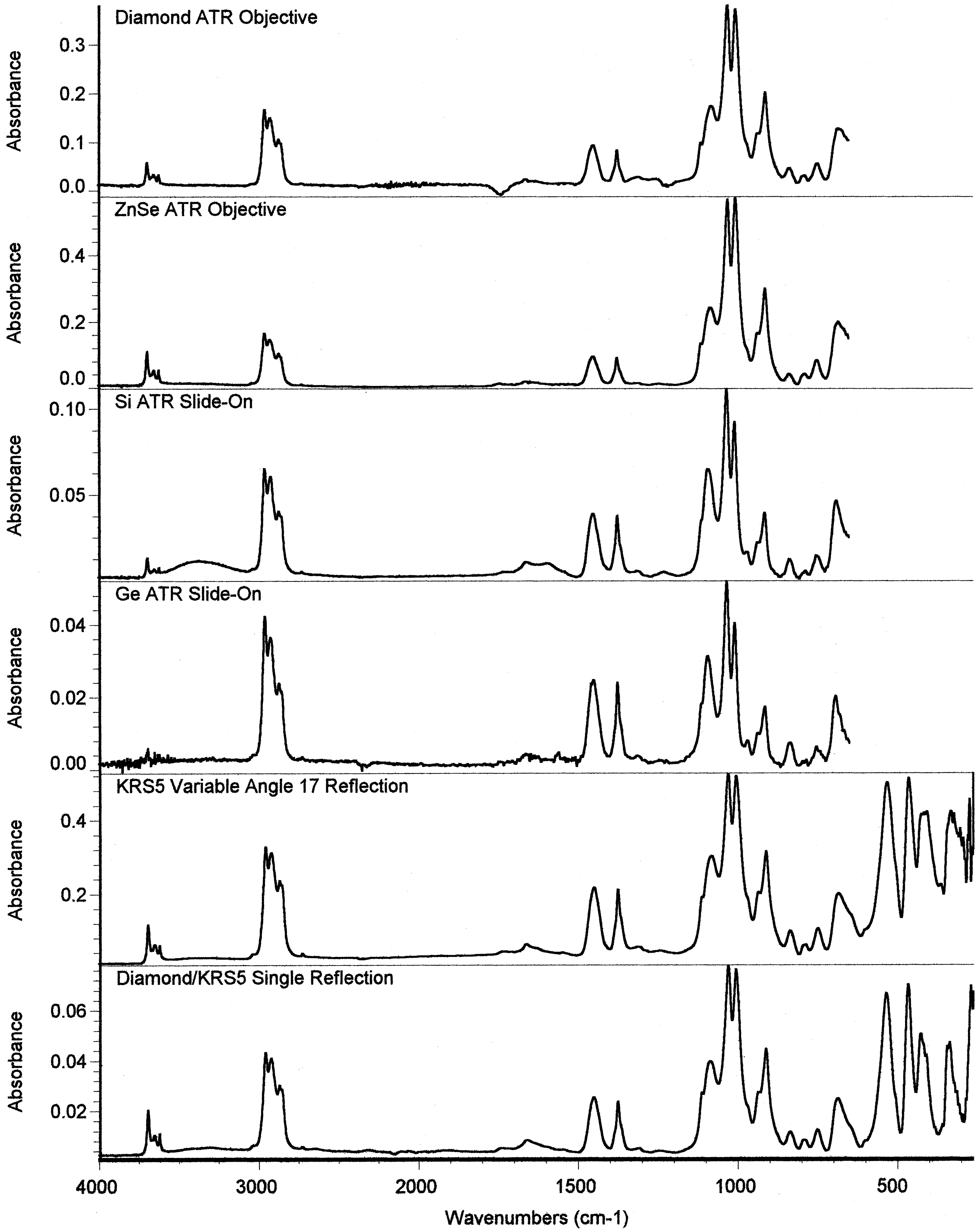


FIG. 1—*Adhesive spectra from a Shuford Mills duct tape, Shurtape PC 620, obtained by six different ATR techniques.*

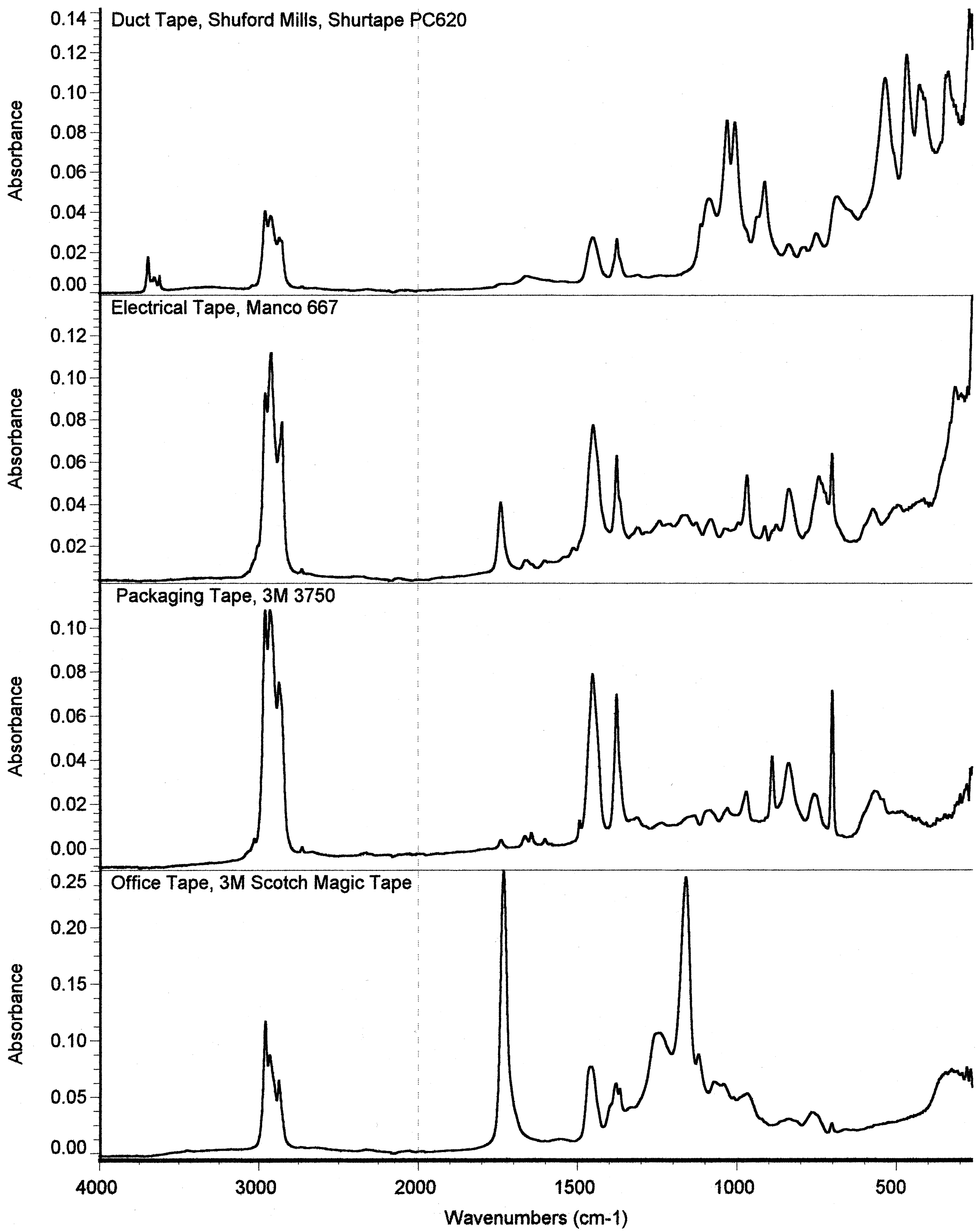


FIG. 2—*Adhesive spectra from four different types of pressure sensitive adhesive tapes obtained with a single reflection, within-compartment ATR accessory equipped with a diamond/KRS-5 IRE.*

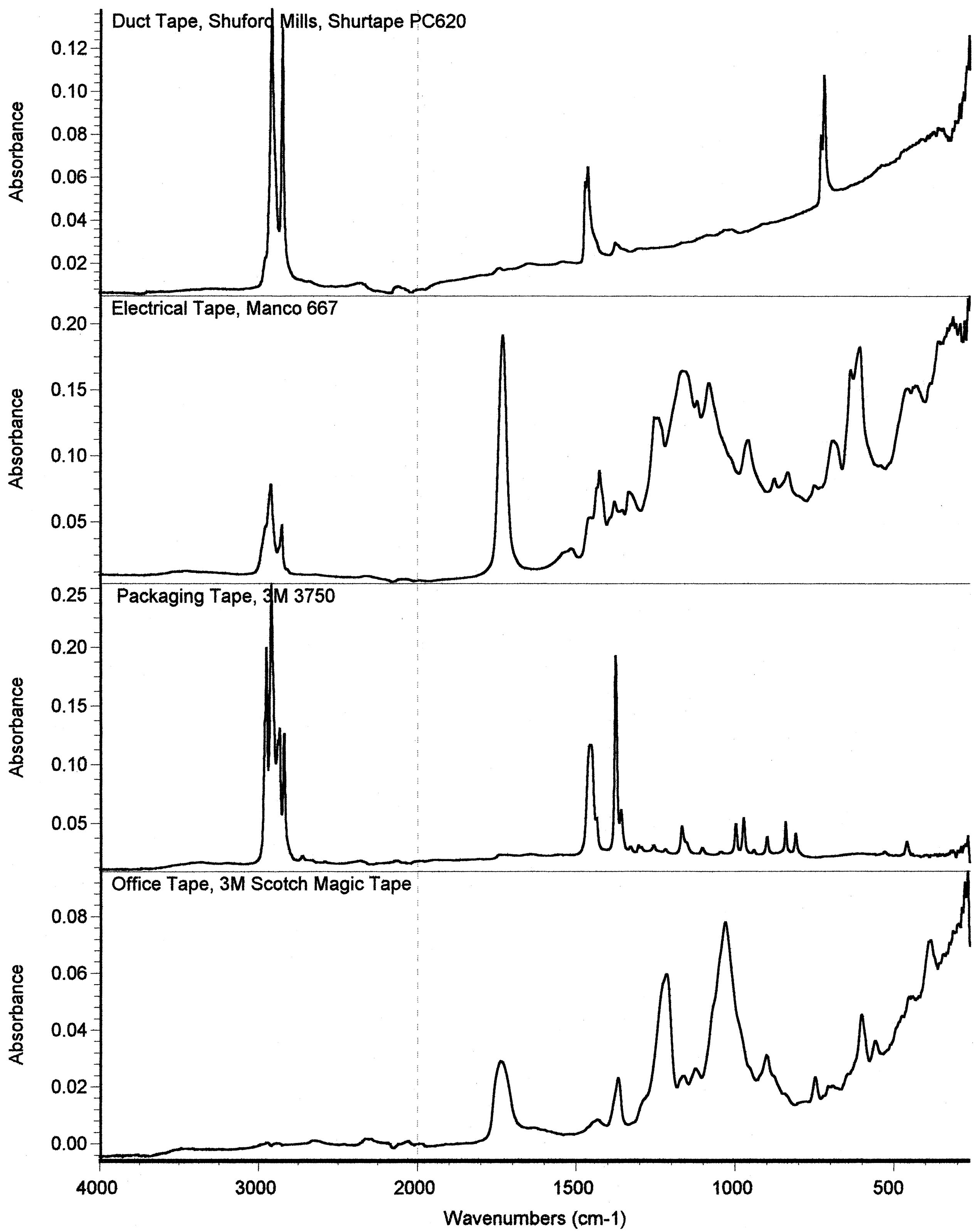


FIG. 3—*Backing spectra from four different types of pressure sensitive adhesive tapes obtained with a single reflection, within-compartment ATR accessory equipped with a diamond/KRS-5 IRE.*

numerous solvents without concern for crystal damage (14,15). Use of such an ATR accessory, with a diamond/KRS-5 element in an IR bench configured with a DTGS detector and CsI beamsplitter, offers extended range capability to approximately 260 $cm^{-1}$ (16). This extended range may be especially useful for analysis of duct tape adhesives which contain inorganic materials as fillers, cross-linkers and color enhancers. Extended range information is also available when the variable angle ATR is used with the proper instrumental conditions. The noise below 400 $cm^{-1}$, seen in the spectrum from Fig. 1 obtained with the variable angle ATR, is probably due to a decrease in energy at the longer wavelengths as a result of the increased number of reflections across the KRS-5 crystal. Noise was not seen in this region when the single reflection accessory was used.

The purge option, with the single reflection, within-compartment accessory, reduces interference from water vapor and carbon dioxide, producing high quality spectra without need for subtraction techniques. Figures 2 and 3 show adhesive spectra and backing spectra respectively, prior to baseline adjustment, from four different tape types obtained using the single reflection, within-compartment ATR accessory with a diamond/KRS-5 IRE. All of these spectra show good signal-to-noise ratios that are free of interference from water and carbon dioxide. The technique provides a fast, nondestructive surface analysis and generates high quality spectra.

## Summary

Overall, within-compartment ATR, with a single reflection diamond/KRS-5 IRE, is the technique of choice for forensic tape analyses where limited sample size may be a concern. The single reflection technique allows the user to perform rapid, nondestructive surface analyses of small areas on either the adhesive or backing side of any type of pressure sensitive adhesive tape examined in this paper. The area to be analyzed should be free of dirt and debris in order to obtain the best results. Tape evidence, submitted to a laboratory for examination, may be crumpled and soiled, but it is usually possible to find a suitable location for analysis since the spot size for the accessory is only 0.75 mm in diameter. Further studies have been conducted using this technique for detailed analysis of duct tape and spectra of possible adhesive components have been collected for the planned second paper in the series on analysis of pressure sensitive adhesive tape. Similar studies are planned for both electrical tapes and packaging tapes.

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