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Studies of Techniques for Analysis of Photocopy Toners by IR

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ABSTRACT: A study was undertaken to examine three measurement techniques for the analysis of photocopy toners by microscopical infrared spectrometry (IR) and to compare these techniques to studies previously done by Diffuse Reflectance (DR). Based on our preliminary studies, analysis of photocopy toners may be successfully performed by DR, microscopical Attenuated Total Reflectance (ATR) with a germanium Internal Reflection Element (IRE) or by microscopical Reflection-Absorption (R-A) techniques with a variety of reflecting media including mirrored slides, low emissivity glass, and aluminum foil. Selection of the analysis technique can be made based on equipment availability, budgetary constraints, ease of sample preparation, speed of analysis, and importance of the original photocopied document to determine if a mildly destructive technique can be used.

KEYWORDS: forensic science, questioned documents, photocopy toner, infrared spectroscopy, FTIR, infrared microspectroscopy, FTIR microscopy, infrared microscopical spectrometry, diffuse reflectance, attenuated total reflectance, reflection-absorption

Interest in the forensic analysis of photocopy toners has increased over recent years with the proliferation and improvement of photocopy machines and other office equipment utilizing the photocopy process. The increasing popularity and quality of both standard and color copiers and laser printers clearly demonstrates the need for forensic scientists to stay abreast of the changing technology in order to keep up with criminal activities involving photocopies. A basic knowledge of printing processes and mechanical designs of specific photocopy machines is invaluable to the examiner of photocopied documents. Since 1980, a great deal of work has been published involving identification and classification of photocopies based on physical characteristics (1–8). For general information, Totty has written excellent reviews on the forensic analysis of photocopies (9,10). The desire to use nondestructive techniques for the analysis of forensic evidence has encouraged the use of optical techniques such as infrared luminescence, infrared reflectance, and laser luminescence for the examination of color photocopies (11,12). Scanning electron microscopy (SEM) with energy dispersive X-ray (EDX) has been used to study the surface morphology and elemental composition of photocopy toner on documents (13,14). A brief study using Differential Scanning Calorimetry (DSC) showed potential usefulness for thermal analysis but was not pursued due to the time-consuming nature of the technique (14). Recently, attention has turned to the molecular analysis of the organic binders to assist with the forensic examination of photocopies. The scientific literature includes several papers on the analysis of photocopy toners by gas chromatography/mass spectrometry (GC/MS) (15–20) as well as multiple papers involving analysis by infrared spectrometry (IR) including standard transmission with potassium bromide (KBr) disks (20–22), and diffuse reflectance (DR) (11,23–25). Some work in the area of infrared spectral interpretation has been discussed by Williams (26) based on IR studies using KBr disks. These references clearly illustrate differences among photocopy toners produced by different manufacturers.

IR has developed into a useful technique for analyzing the chemical composition of photocopy toners. Unfortunately, the sampling techniques previously cited involve tedious sample preparations that are destructive, though minimal damage is done to the document. The continued advancement in IR sampling accessories has now yielded microscopical Attenuated Total Reflectance (ATR) which permits a nondestructive surface analysis of a microscopic area (27,28). Andrasko reported a method of microscopical reflection-absorption (R-A) for the rapid analysis of photocopy toner (29). Application of R-A offers a variety of sampling techniques which look promising for the analysis of photocopy toners. These techniques involve lifting the copy toner from a questioned document onto a reflecting medium for analysis. The lifting process is quick and easy and the document sustains minor damage which is only visible microscopically. A study was undertaken to examine several measurement techniques for the analysis of photocopy toners by microscopical IR and to compare these techniques to studies previously done by DR.

Materials and Methods

The samples used in the study included three toners received from Europe; IBM 3-20, Konica UBIX 160 and Minolta EP 550Z, and five toners from the FBI Laboratory collection; Cannon LBP-4, IBM Laser Printer 4019, Konica LP-3015, Konica LP-3110 and Xerox 4197. Each of the photocopy samples was analyzed with a Nicolet 20SX Fourier transform IR (Madison, Wisconsin) using

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DR, microscopical ATR and microscopical R-A. All resulting spectra were baseline adjusted as needed using the Nicolet software.

Diffuse Reflectance

The technique for analysis of toners by DR has been described in several papers (23–25). To prepare the samples for analysis by DR it was necessary to remove a portion of the copy toner by slicing off the top layer of fibers from a small area of the document to be analyzed. Approximately half of a single letter was required for sufficient sampling. This sample was then placed in a limited volume insert in a sample vial and was extracted with 10 μL of acetone for 10 minutes. Three μL of the extract was pipetted onto a KBr-filled microsample cup 3 mm in diameter and 2 mm deep. The acetone was allowed to air evaporate for 15 minutes and the microsample cup was then placed in an oven at 100°C for 15 min to ensure complete removal of the solvent. The sample was analyzed with a Spectra-Tech Collector™ (Shelton, Connecticut) using a wide-band mercury cadmium telluride (MCT) detector operating at 4 cm^{-1} resolution with a full aperture. Each sample was scanned 256 times from 4000 to 450 cm^{-1}.

Microscopical ATR

A Spectra-Tech IR-Plan® microscope with a medium-band MCT detector was used for all the microscopical IR studies including both ATR and R-A. No sample preparation was necessary prior to analysis by the ATR technique. At the time of the study, two different microscopical ATR accessories were commercially available, the Spectra-Tech ATR Objective (Patent 5093580) and the Spectra-Tech ATR Slide-On which fits over the 15X Reflachromat™ objective. A selection of Internal Reflection Elements (IRE) was available from Spectra-Tech for each of these two ATR accessories. Our tests were limited to use of the diamond IRE with the ATR Objective and the germanium IRE with the ATR Slide-On. The penetration depths for these accessories at 1000 cm^{-1} were 2 μm for the diamond ATR Objective and 0.69 μm for the Germanium ATR Slide-On (30). Spectra-Tech's Contact Alert System, which provides both an audible and visual indication that contact has been made with the sample, was used with both of the ATR accessories. The alert system makes it possible to obtain reproducible contact force and to prevent crystal damage by accidentally applying too much pressure. All samples were analyzed with a pressure indication of eight showing on the Contact Alert System.

The photocopies tested contained both text and graphics so it was possible to position each of the documents such that a large area of toner was under the IRE without use of the visual capabilities of the microscope. This was necessary since germanium is not transparent in the visible range and the diamond offers poor visual clarity. When necessary, a smaller area of toner may be precisely positioned under the IRE by locating the area with either the visible objective or the standard casegrain objective prior to moving the ATR into position. Spectra were acquired at 8 cm^{-1} resolution using each of the two ATR techniques. The samples were scanned from 4000 to 650 cm^{-1} and 512 scans were co-added for each spectrum.

Microscopical Reflection-Absorption

For the R-A studies, the photocopy toner was lifted off the sample document onto a reflecting medium using a heat transfer process. Several reflecting media were examined including heavy duty aluminum foil affixed to a standard microscope slide with double-sided tape, aluminum-coated mirrored slides (Spectra Tech), and low emissivity (low E) glass. The photocopy toner was transferred to these reflecting media using a temperature regulated soldering iron equipped with a screwdriver tip that had been ground off leaving a flattened round head with a 4.8 mm diameter. Initial transfers were done with the soldering iron set at 371°C but this temperature was found to easily scorch the paper document. Better results were obtained using the soldering iron set at 260°C. This temperature worked well for transferring toner to mirrored slides or to Low E glass, but 288°C was necessary to consistently transfer toner to aluminum foil. This was probably due to the rapid heat loss by the thin aluminum foil. To transfer the toner to either the aluminum foil or to the mirrored slides the heat may be applied through the reflecting medium to the document underneath to melt the toner and lift it off the document. This technique does not permit the analyst to visualize the exact area from which the toner is being lifted. Use of the low E glass as a lifting medium with this technique permits visualization of the document through the glass, but the material is only available in 3 mm thicknesses which is too thick to adequately transfer the heat to the document to lift the toner without breaking the glass. To transfer the toner to low E glass it was necessary to place the document, with the printed side down, on top of the glass and heat the back of the document with the soldering iron set at 260°C. By working over a light box, it was still possible to see through the document and visualize the sampling area. This technique of applying heat to the document itself also works well with both the aluminum foil and the mirrored slides and by working over a light box, enough light bends around the reflective medium for the analyst to visualize the sampling area. Care must be taken to lift the toner onto the reflective side of the medium being used. Successful transfers have also been made to the reflective surface of disposable scalpel blades by heating the back of the blade with a soldering iron at 300°C for 20 seconds.[3] Once the toner was lifted onto the reflective medium, the sample was analyzed by R-A at 4 cm^{-1} resolution from 4000 to 650 cm^{-1}. Each spectrum was scanned 256 times.

Results and Discussion

Spectra resulting from each of the analysis techniques required varying amounts of baseline adjustment. Necessary adjustments were minimal for those spectra acquired by DR. However, analysis by both microscopical ATR and microscopical R-A resulted in spectra with baselines requiring considerable adjustment to flatten them as is illustrated in Fig. 1 showing a R-A spectrum of Xerox 4197 both before and after baseline adjustment.

Analysis of photocopy toners by DR offers high quality reproducible spectra which are suitable for spectral libraries. Some skill is required to remove a portion of the toner for extraction and the simple act of sampling the toner causes some destruction to the document. The sample preparation required for this technique is substantial and requires hood space for solvent extraction and an oven to ensure complete evaporation of the solvent. Once the sample is prepared, the analysis is fast and is run with the DR accessory in the main bench without the need of an IR microscope.

Microscopical ATR offers an alternate technique for the analysis of photocopy toners which is totally nondestructive and has no requirement for sample preparation. The area of toner to be sampled is simply positioned under the IRE and the stage is moved upward until physical contact is made between the sample and the IRE

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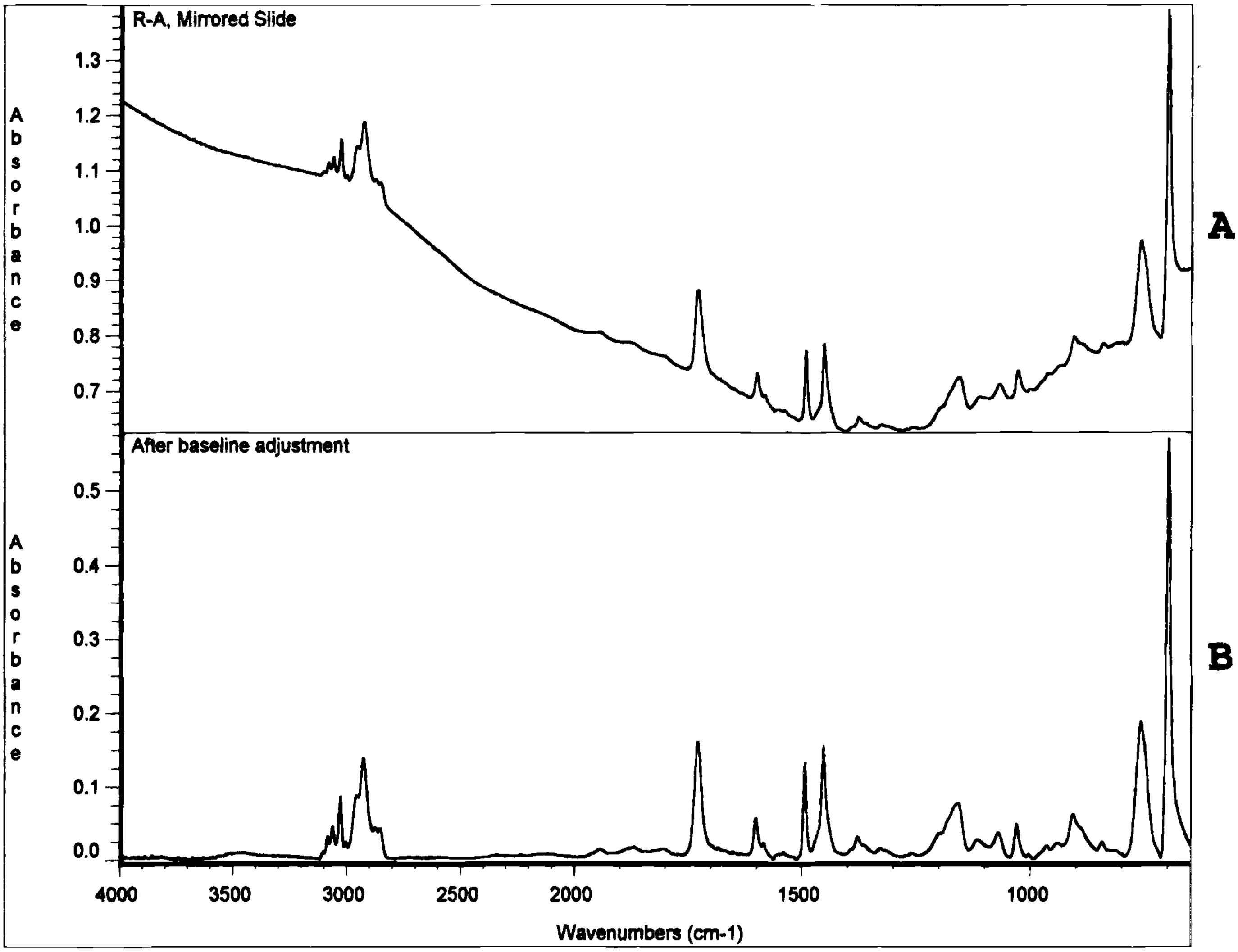

FIG. 1—*Spectra of Xerox 4197 toner obtained by reflection-absorption with an aluminum coated mirrored slide before* (A) *and after baseline adjustment* (B).

and a reading of eight is obtained on the pressure meter of the Contact Alert System. This technique utilizes a single reflection ATR accessory to analyze microscopic size samples, thus less reflective energy is available to the detector and the resulting spectra show lower signal-to-noise ratios (S/N) than the DR and R-A spectra of the same samples. ATR spectra characteristically show stronger bands at longer wavelengths due to increased penetration depth of the evanescent wave with increasing wavelength (31,32). As a result, spectra obtained by this technique were ATR corrected using the Nicolet conversion software in order to make the resulting spectra more like transmission spectra for comparison purposes.

Each of the eight samples were analyzed using the ATR Objective with a diamond IRE. The resulting spectra were somewhat noisy due to the low absorptions. It was necessary to blank out the region between 2400–1800 cm^{-1} in all of these spectra where a noncompensated diamond absorption appears. Figure 2 shows a single beam spectrum of the diamond ATR along with the noncompensated diamond absorption present in a ratioed spectrum of Konica LP 3110 toner and the final spectrum obtained by blanking the region between 2400–1800 cm^{-1}. This noncompensated diamond absorption seems to be characteristic for copy toners but its cause is uncertain. Possibly, a minor shift in the IRE position on contact with the sample causes a change in incident angle with a subsequent change in penetration depth and since the background spectrum was obtained with no pressure on the diamond IRE, incomplete compensation occurs.

Cellulose absorptions from the paper beneath the toner layer appear in several of the spectra obtained with the ATR objective due to the penetration depth of the evanescent wave with the diamond IRE. Figure 3 shows a spectrum of paper obtained with the diamond ATR objective and a spectrum of the IBM 320 toner analyzed on the paper by the same technique. The cellulose absorptions in the sample spectrum indicate penetration of the evanescent wave through the toner layer to the paper beneath.

The high concentration of carbon black in the toners caused a great deal of dispersion in the ATR spectra displayed as sharp, downward inflections just prior to large absorption bands. This is particularly obvious in Fig. 2C. In order to obtain an internal reflection spectrum, the IRE crystal must have a significantly greater refractive index (RI) than the absorbing sample. The angle of incidence must also exceed the critical angle in which total reflection occurs. As the angle of incidence approaches the critical angle for the IRE, dispersion in reflectivity occurs resulting in

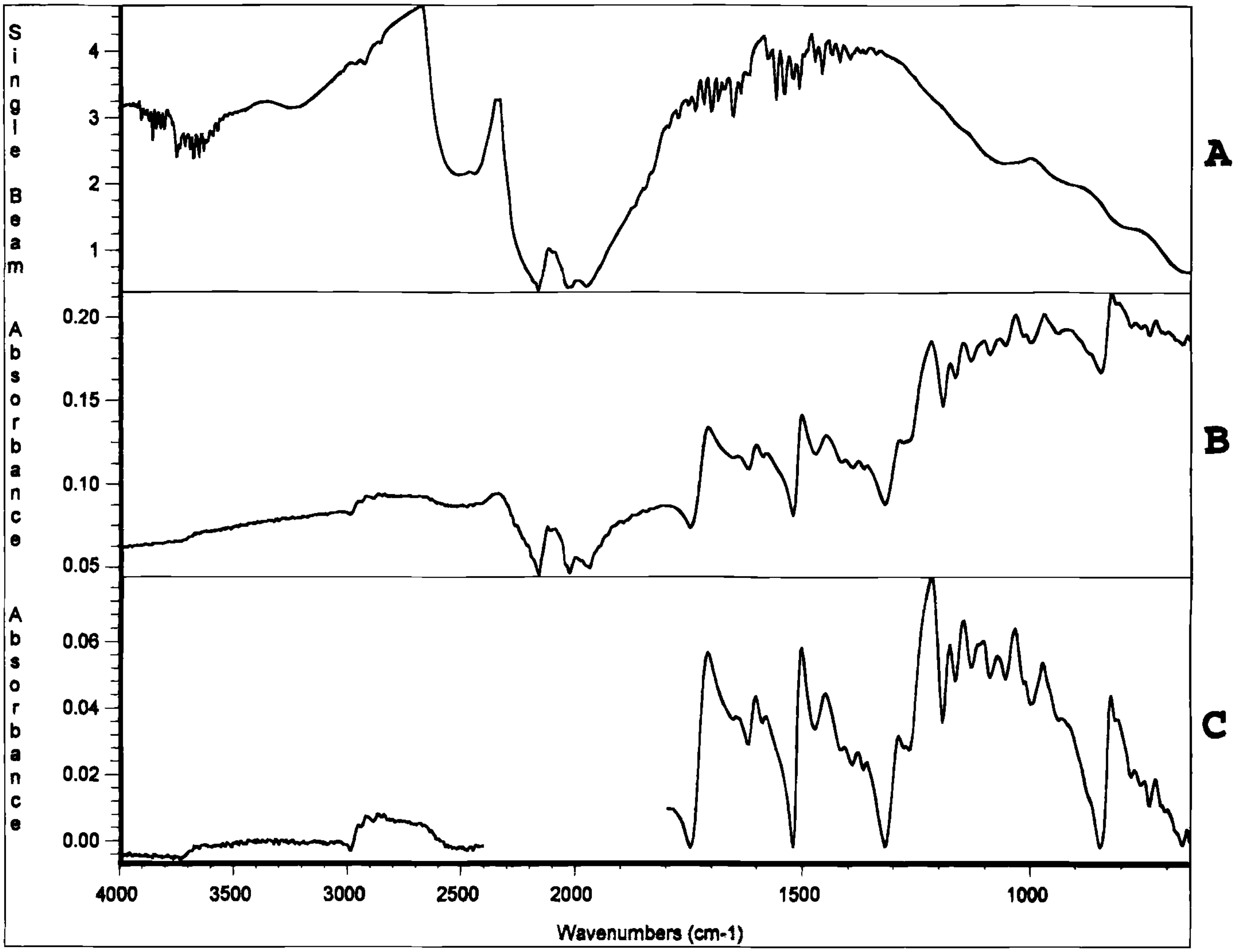

FIG. 2—(A) *Single beam spectrum of the diamond ATR crystal showing diamond absorptions between 2400–1800 cm*$^{-1}$. (B) *Ratioed spectrum of Konica LP-3110 toner obtained by ATR with a diamond IRE showing noncompensated diamond absorption.* (C) *Resulting spectrum of Konica LP-3110 toner after baseline adjustment and blanking the region between 2400–1800 cm*$^{-1}$ *to remove the noncompensated diamond absorption.*

distorted spectra (31,32). To reduce the problem of dispersion in reflectivity, an alternate IRE was selected.

Use of germanium as the IRE permits analysis of samples with high refractive indices making it an ideal crystal for analysis of photocopy toners. Because there is less penetration of the evanescent wave, theoretically there should be less cellulose interference from the paper beneath the toner layer. The germanium IRE is available in two different accessories, the ATR Objective and the Slide-On ATR which was used for our studies.

Analysis of the same eight photocopy toners using the Slide-On ATR with the germanium IRE yielded much better spectra than the spectra obtained with the diamond IRE. The improved results, with reduced dispersion and less interference from cellulose absorptions, are indicative of the benefit of the germanium IRE. Though still a little noisy, these spectra are of a quality approaching those obtained by diffuse reflectance, but without the sample preparation. The Slide-On ATR is easy to use since it mounts onto the standard 15X Reflachromat™ objective and can be moved in or out of position based on the needs of the analysis. With the germanium IRE mounted on the ATR Slide-On, the diamond IRE can remain in the ATR objective offering the availability of two separate IREs without the need to remount and adjust a different crystal. Replacing the crystal in the ATR objective can be a tedious and time-consuming project.

Analysis of photocopy toner by microscopical R-A requires use of an IR microscope but involves no additional expensive accessories as with the ATR analysis. The technique requires considerably less sample preparation than DR and yields equivalent quality spectra. Lifting the toner onto a reflective medium causes some minor damage to the document, but generally this damage can only be seen microscopically.

This technique offers flexibility in selection of reflective media since analytically indistinguishable results were obtained whether the sample was analyzed on aluminum foil, low E glass, or a mirrored slide. Because of its pliable nature, aluminum foil is easier to work with if it is affixed to a standard glass slide with double-sided tape. This makes the lifting process easier as well as providing a base for use with the microscope.

Occasionally, the lifting technique pulls paper fibers from the document. Care must be taken during the analysis not to focus on an area of the sample that includes these fibers. They are generally very noticeable under the microscope but in the event they go unrecognized visually, cellulose fibers have a very distinct, easily identified spectrum, shown previously in Fig. 3.

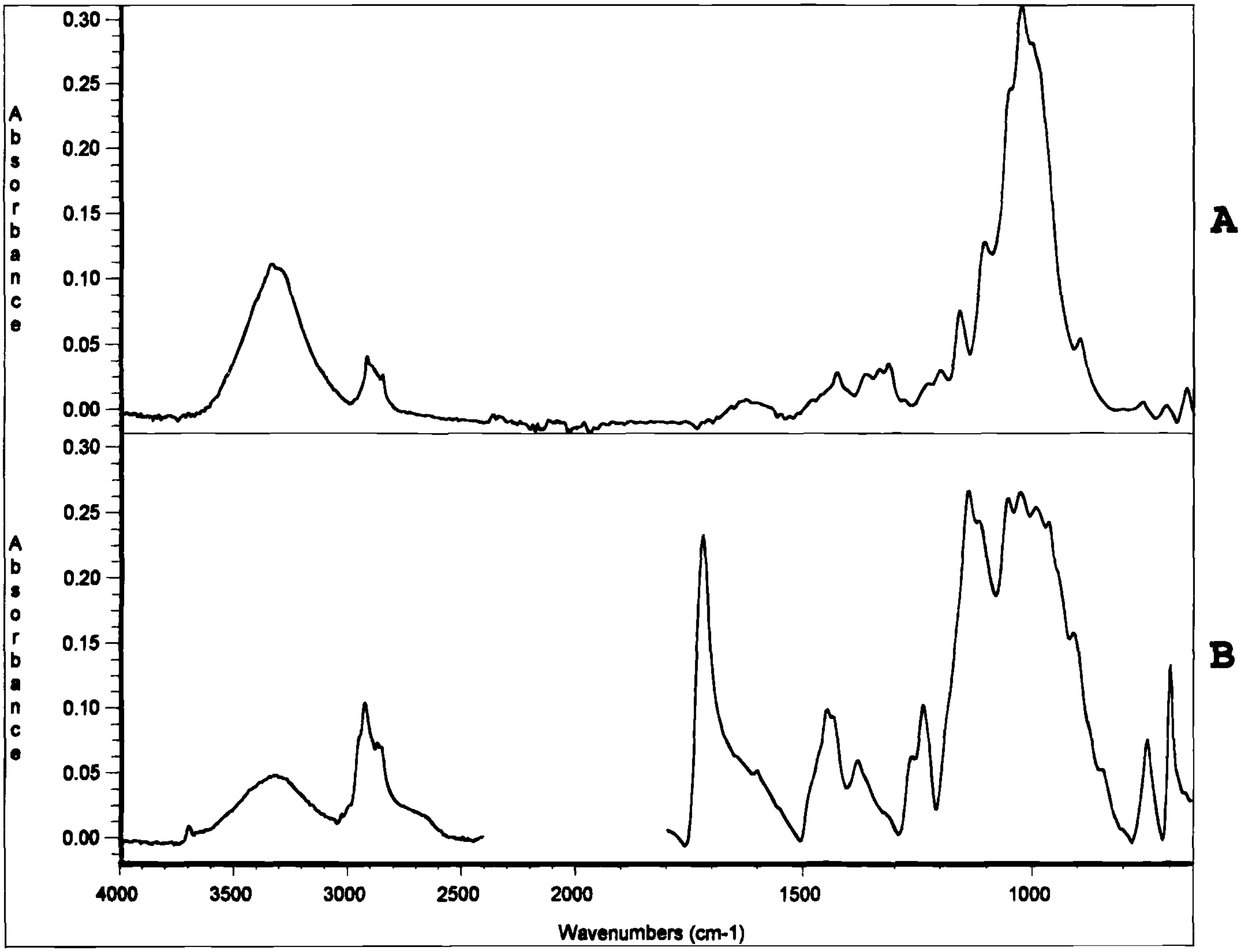

FIG. 3—(A) *Spectrum of cellulose obtained by ATR with a diamond IRE.* (B) *Spectrum of IBM 3-20 toner obtained by ATR with a diamond IRE showing both dispersion and cellulose interference from the paper beneath the toner.*

Difficulties could arise using the lifting technique where heat is applied through the document. If the document is a double-sided copy, the soldering iron could come into contact with toner on the back of the document. In these situations it is necessary to apply the heat through the reflective medium to lift the toner, thus limiting the lifting medium to either aluminum foil or a mirrored slide and reducing visualization of the sample site. For double-sided copies, if specificity in sample site is imperative to the document examination, heat may be applied through a small piece of aluminum foil, cut to the appropriate sample size, to lift the toner, allowing the analyst better visualization of the sample site. After lifting the toner, the aluminum foil must be carefully pressed flat and adhered to a glass slide for analysis.

Figures 4 and 5 show comparison spectra obtained by DR, ATR objective with a diamond IRE, ATR Slide-On with a germanium IRE, and R-A using a mirrored slide for two of the eight samples analyzed. Based on our preliminary studies, analysis of photocopy toners may be successfully performed by DR, microscopical ATR using the Slide-On ATR with a germanium IRE or by microscopical R-A with a variety of reflecting media. Figure 4, showing analyses of IBM Laser Printer 4019 toner, illustrates that comparable spectra can be obtained by each of these methods. The same spectral peaks are present in spectra obtained by each technique. Differences lie in baseline, resolution and S/N. Selection of the analysis technique can be made based on equipment availability, budgetary constraints, ease of sample preparation, speed of analysis, and importance of the original document to determine if a mildly destructive technique can be used.

Analysis by DR yields high quality spectra without the need of an IR microscope. The resulting spectra are free of interference from cellulose absorptions. Unfortunately, the required sample preparation is tedious and the extraction process is mildly destructive to the document. Spectral distortions can also occur as shown in the DR spectrum in Fig. 5. These distortions, visible as dips below the normal spectral baseline near 3400 cm^{-1} and 1000 cm^{-1}, are specular reflections (reststrahlen bands) caused by front surface reflections, possibly due to silica content in the toner.

Analysis by microscopical ATR requires use of an IR microscope as well as the ATR accessory. Both the ATR objective with the diamond IRE and the slide-on ATR with the germanium IRE offer fast, easy and totally nondestructive methods for screening photocopy toners, though the resulting spectra have lower S/N than the other methods. Use of the diamond ATR objective results in loss of information between 2400–1800 cm^{-1} due to an uncompensated diamond absorption. It also has the disadvantage of producing spectra with dispersions and cellulose interference making

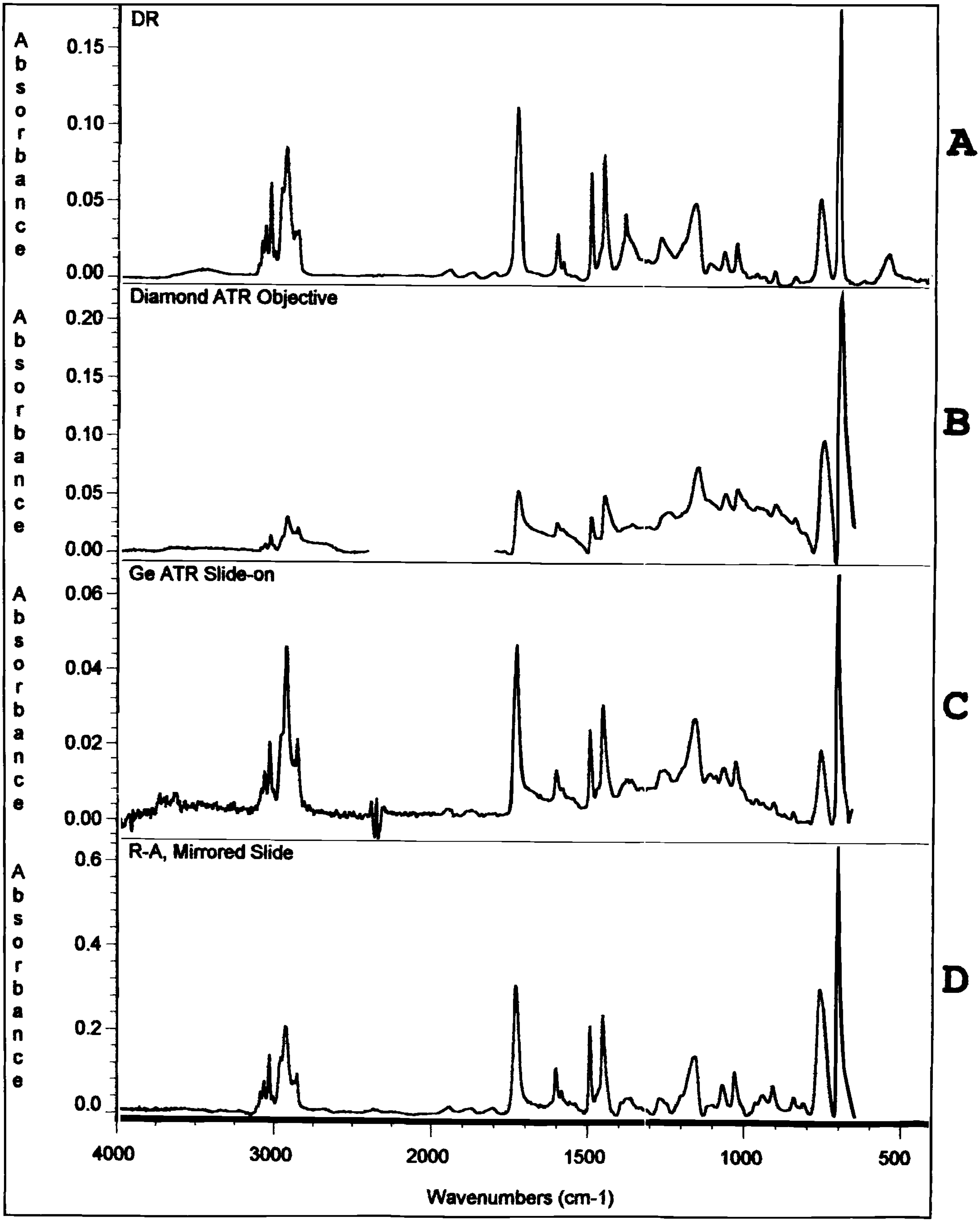

FIG. 4—*Spectra of IBM Laser Printer 4019 toner obtained by four different techniques.* (A) *Diffuse reflectance.* (B) *ATR objective with a diamond IRE.* (C) *ATR Slide-On with a germanium IRE, ATR corrected.* (D) *Reflection-absorption with an aluminum coated mirrored slide.*

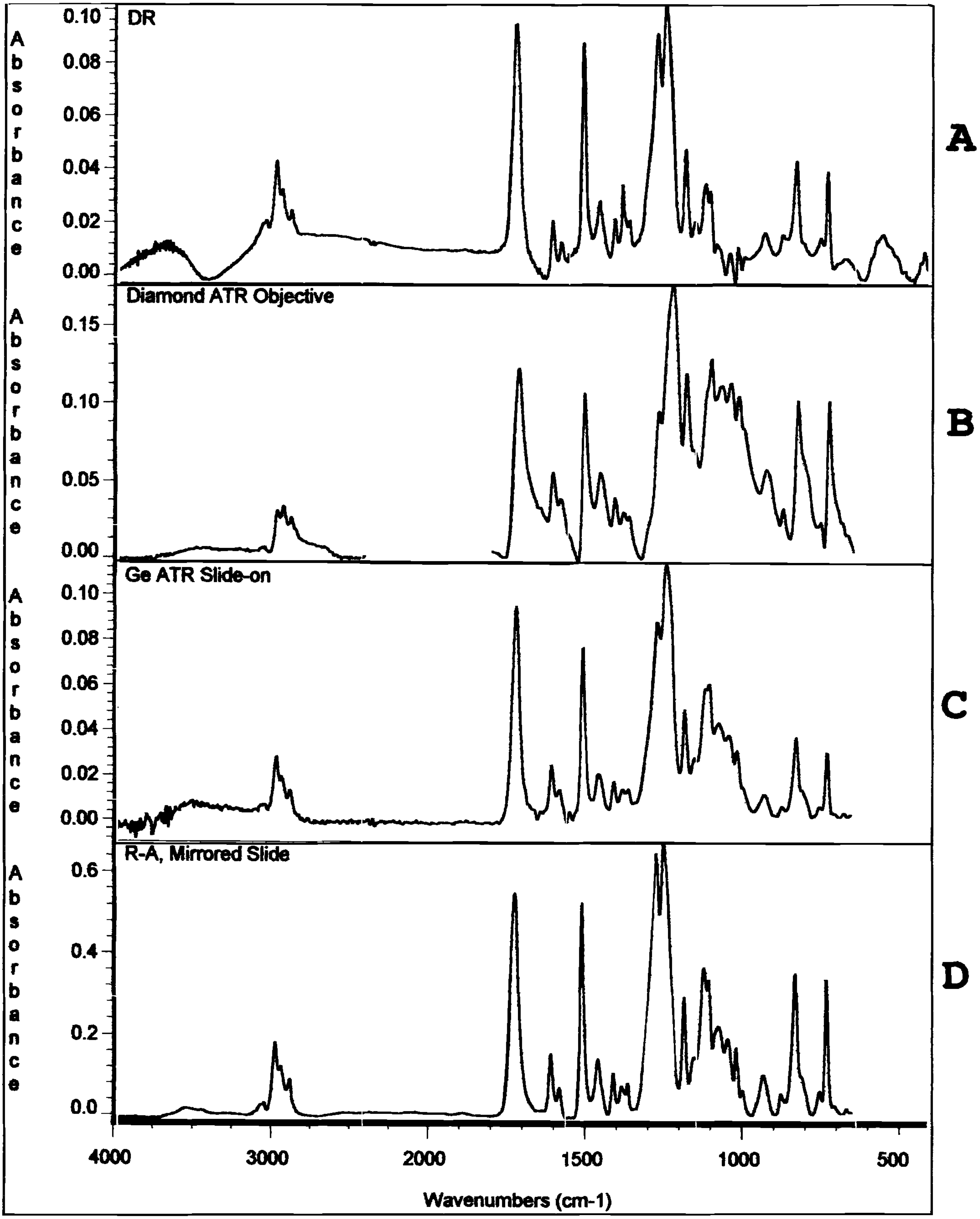

FIG. 5—*Spectra of Minolta EP 550 Z toner obtained by four different techniques.* (A) *Diffuse reflectance.* (B) *ATR objective with a diamond IRE.* (C) *ATR Slide-On with a germanium IRE, ATR corrected.* (D) *Reflection-Absorption with an aluminum coated mirrored slide.*

it undesirable. The spectrum shown in Fig. 5*B* clearly illustrates these problems.

Use of the germanium Slide-On ATR provides good quality spectra with only minor cellulose interferences. After initial nondestructive screening using either of the microscopical ATR techniques, specific toner samples can be extracted and analyzed by DR or lifted and analyzed by R-A if more precise information is needed.

Conclusion

Analysis by R-A appears to be the overall optimum method for analyzing photocopy toners when considering quality of spectra, speed, cost of accessories and destructiveness. DR generally provides the highest quality spectra with the least cost in measurement accessories; however, the technique is time-consuming due to the elaborate sample preparation required prior to the analysis. Though the accessories are expensive, microscopical ATR with a germanium IRE was the fastest and least destructive technique examined and it resulted in good quality spectra. R-A is a compromise of these two methods producing good quality spectra with little sample preparation and no additional cost when an IR microscope is available, as it is in most forensic laboratories today. Following analysis of a photocopy toner by R-A on aluminum foil, the same sample may be analyzed by pyrolysis GC or Pyrolysis GC/MS without additional sampling as indicated by Chang et al. (19). Thus, IR spectroscopy offers viable sample measurement alternatives to obtain the molecular composition of the organic binders in photocopy toners.

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