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Classification and Identification of Photocopying Toners by Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS): I. Preliminary Results

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ABSTRACT: A diffuse reflectance infrared Fourier transform technique is described for the analysis of raw photocopying toners and small samples of toner removed from the surface of a photocopied document. The technique gives reproducible results and makes possible differentiation of toner samples obtained from different photocopying machines. In addition, toners may be classified into a number of groups, based on the presence or absence of certain absorption bands in their diffuse reflectance spectra. Preliminary results indicate the feasibility of a database of infrared spectra for the photocopying toners available on the market. Such a database would be a valuable aid to the document examiner in the increasing number of cases involving photocopies.

KEYWORDS: questioned documents, diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS), toners, photocopiers, photocopies, diffuse reflectance, infrared spectroscopy, Fourier transform infrared (FTIR) spectroscopy, criminalistics

With the massive increase in the availability of photocopying machines over the last several years, the document examiner is more than ever faced with the necessity of examining photocopied documents. A wide range of brands and models of photocopiers is now available from numerous distributors. In addition, banks, post offices, schools, shopping centers, and department stores now make available photocopying machines for self-service use by clients. As a result of this increased ease with which documents can be copied, there has been a shift in the way falsified documents and anonymous letters are being constructed. The typewriter and the collage of letters cut from magazines have largely given way to the use of photocopies, which offer certain advantages to the criminal. The ease with which original documents can be modified or falsified has been aided by the fact that modern plain paper photocopying machines allow copies to be made on a wide range of paper and plastic supports. The danger is especially evident with color photocopiers, which make possible the fabrication of high-quality forgeries of documents such as bank notes and checks [1].

The increase in the number of crimes linked to the use of photocopies has stimulated a search for efficient analytical techniques for classification and identification of photo-

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copying machines. The development of such techniques will only be possible after detailed study of the electrostatic transfer technique employed by most modern copiers.

The physical characteristics of photocopies (the paper type, toner type, toner application, fusion method, magnetic properties, and other characteristics) have been proposed by a number of authors for use in classification of photocopy machines [2-7]. Chemical analysis of the toner appears to be a useful and logical addition to classification by physical characteristics. The toners employed in modern photocopiers are composed of organic resins, such as synthetic polymers and copolymers, mixed with carbon black (or colored pigments in the case of toners for color copiers). As a result, the chemical analysis of toners can be conveniently performed using established analytical techniques such as pyrolysis gas chromatography and infrared spectroscopy.

In 1983, Kemp and Totty [8] showed that toners used in plain paper photocopying machines could be distinguished from each other by infrared spectroscopy, using small samples of solid toner removed from the surfaces of photocopied documents. The sample of toner was ground with potassium bromide (KBr) and then pressed into a pellet. The infrared spectrum of the pellet was then measured in the transmission mode on a dispersive infrared (IR) spectrometer. Kemp and Totty found that a given toner sample could be placed in one of several groups, based on the recorded IR spectrum of the toner. Some differences were observed between the spectra of bulk toner and spectra of toner removed from paper. These differences were attributed to the fusion process employed by the photocopier (which may use heat, pressure, or both). Williams [9] took the results obtained by Kemp and Totty and found that it is possible to identify and characterize the chief chemical constituents of a photocopying toner by using infrared group frequency analysis. In 1985, Horacek and Müller [10] employed a Fourier transform infrared (FTIR) spectrometer for the analysis of toners. Samples of toner were obtained by grinding KBr directly on the document. The KBr was then pressed into a pellet and measured by transmission IR spectrometry. The spectrum of the paper support (without toner) was obtained in a similar fashion and subtracted from the toner spectrum. In this manner, any influence from the paper could be overcome. The technique proposed by Horacek and Müller, however, lacks sensitivity.

In 1986, Levy and Wampler [11] proposed the use of pyrolysis gas chromatography/mass spectrometry (GC/MS) for the analysis of toner materials from photocopies. Their technique required pyrolysis of the photocopied paper with and without toner. A number of pyrolysis products were identified by GC/MS and associated with various toner systems. Zimmerman et al. [12] analyzed a number of raw toner powders and toners extracted from photocopied documents by IR spectrometry. (The toner powder or toner extract was mixed with powdered KBr and pressed into a pellet, which was then analyzed by transmission IR spectrometry.) Of 35 toners analyzed, 18 different categories could be established. Zimmerman et al. found that the toners in some of these categories could be further subdivided by pyrolysis gas chromatography (GC). Interestingly, good agreement was observed between the results obtained from raw toner powders and those obtained from toners extracted from photocopied documents. Zimmerman et al. suggested that a library of toner spectra could be constructed from the analysis of raw toners collected from various companies. More recently, Munson [13] made a comparison of photocopies from 62 different photocopy machines by pyrolysis GC/MS. A sample of toner was removed from each photocopy by heat transfer onto a section of a glass microscope slide. In this manner, the toner could be separated from the paper before analysis by pyrolysis GC/MS. Based on the presence or absence of peaks in the pyrograms, the toners could be separated into 18 classes.

Since the early development of the technique by Fuller and Griffiths in 1978 [14], diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) has found numerous applications in criminalistics. The introduction and acceptance of the technique

in the forensic sciences has been largely due to work published by Suzuki and Gresham [15–17], by Suzuki alone [18], and by Suzuki and Brown [19]. DRIFTS has already proven to be a practical and sensitive method for infrared analysis of a wide range of samples of interest to forensic science. However, apart from an unpublished report [20], the analysis of photocopying toners by DRIFTS has not, to our knowledge, been investigated.

The purposes of this project were the following:

(a) to determine if DRIFTS can be applied to the analysis of raw toner powders and toners extracted from photocopied documents and if the IR spectra obtained can be used to differentiate samples from different machines;

(b) to determine if the spectrum of a toner extracted from a photocopy and measured by DRIFTS is consistent with the spectrum of the raw toner powder from the same photocopying machine;

(c) to devise a classification scheme for photocopying toners based on their diffuse reflectance infrared spectra; and

(d) to investigate the feasibility of constructing a database of IR spectra based on the photocopying toners currently available on the Swiss market.

Only plain paper copiers, using the electrostatic transfer technique, have been considered in this study.

Experimental Procedure

Apparatus

Diffuse reflectance infrared spectra were recorded using a Spectra-Tech Inc. diffuse reflectance accessory (the Collector) fitted to a Mattson Cygnus 100 FTIR spectrometer. The spectra were calculated from 400 accumulated scans at a resolution of 8 cm^{-1} and a moving mirror velocity of 2.53 cm/s . A narrow-range mercury/cadmium telluride (MCT) detector was employed and spectra were collected between 4000 and 700 cm^{-1} .

Toner Samples

Photocopies produced on 130 different photocopying machines and 119 different samples of bulk toner powder were obtained from company representatives in Switzerland. Information on the compatibility of each toner was also obtained, where possible. (For example, the toner Agfa CB-749 is compatible with the Agfa photocopiers X-29, X-40, and X-41.) In addition to toners produced for specific photocopying machines, it was also possible to obtain 50 different samples of toners produced in Switzerland which have been adapted for use in a variety of photocopiers (that is, in different brands and models).

Of the samples at our disposal, a small subset was arbitrarily chosen for evaluation of the DRIFTS analysis technique. This report is based on the results obtained from the analysis of photocopies from 33 different machines (Table 1) and of 25 different raw toner powders (Table 2). This subset represents 26 photocopy/raw toner pairs and 32 different toners, according to information received from the manufacturers.

Infrared Analysis of Toner on Photocopied Documents

The toner on photocopied documents was analyzed by DRIFTS after removal of a small amount of toner (equivalent to a line of about 1 cm length) from the surface of the copy by scraping it with a scalpel. This sample (toner plus paper fibers) was then extracted with $8\text{ }\mu\text{L}$ of acetone (spectroscopic grade). The extraction was performed in a capillary tube (1 mm in diameter) to limit evaporation of the solvent. After a few

TABLE 1—*List of the machines used to produce the 33 different photocopies from which toner samples were extracted and analyzed in this preliminary study.*

Brand of Copier	Models ^a
Agfa	X-29, X-35, X-41, X-45, and X-88
Canon	NP-210, NP-300, NP-1215, NP-3225, NP-4035, NP-4540, NP-7050, NP-7550, and NP-9030
Gestetner	ZDF-2355
Kodak	90-E
Konika	100
Minolta	EP-30, EP-270, EP-350Z, EP-410Z, and EP-450Z
Nashua	4550Z
Oce	1850
Panasonic	FP-2520
Ricoh	FT-7770
Sharp	SF-8200
Toshiba	5110
Utax	C-110, C-220, and C-230
Xerox	5052 and 9200

^aThe models in bold type indicate the 26 cases in which both the toner on the photocopy and the raw toner powder were analyzed.

TABLE 2—*List of the 25 different raw toner powders analyzed, together with the list of photocopying machines using these toners.*

Bulk Toner	Compatibility
Agfa CB-749	Agfa X-29, X-40, and X-41
Agfa CB-752	Agfa X-55 and X-88
Agfa CB-755	Agfa X-35 and X-45
Canon NP-210	Canon NP-210, NP-270, NP-271, NP-300, NP-305, NP-400, and NP-500
Canon NP-1015	Canon NP-1015 and NP-1215
Canon NP-3025	Canon NP-3025, NP-3525, NP-3225, and NP-3725
Canon NP-3325	Canon NP-3325 and NP-3825
Canon NP-4035	Canon NP-4035, NP-4040, and NP-4540
Canon NP-4335	Canon NP-4335 and NP-4835
Canon NP-6000	Canon series NP-6000 to 8000
Canon NP-9030	Canon NP-9030
Konika 100	Konika 100, 150, and 250Z
Minolta EP-30	Minolta EP-30
Minolta EP-270	Minolta EP-270, EP-370, and EP-370Z
Minolta EP-350Z	Minolta EP-350Z
Minolta EP-410Z	Minolta EP-410Z, EP-415Z, and EP-425Z
Minolta EP-450Z	Minolta EP-450Z
Oce 1900	Oce series 1700 to 1900
Pelikan ^a Canon 270	Canon NP-210, NP-270, NP-271, NP-300, NP-305, NP-400, and NP-500
Pelikan ^a Konika 100	Konika 100, 150, and 250Z
Pelikan ^a Minolta EP-450Z	Minolta EP-450Z
Ricoh 4000	Ricoh FT-7770
Utax C-110	Utax C-110
Utax C-220	Utax C-220, C-221, C-222, C-225, C-230, and C-233
Xerox R	Xerox 1040, 1050, and 5052

^aThe Pelikan brand toners are made in Switzerland and are not the original brands of toner suggested for use with the corresponding photocopying machines.

minutes, the extract was deposited on the surface of a bed of powdered potassium bromide (15 mg), previously prepared in a 3-mm-diameter DRIFTS sample holder (Fig. 1). The sample holder, containing the KBr and the sample extract, was then heated at 100°C for 15 min to evaporate the solvent and dry the KBr. A second sample holder, containing only KBr, was similarly prepared to be used as a reference for the DRIFTS measurements. The sample of toner and the KBr reference were then measured in the DRIFTS accessory using the conditions listed above.

Infrared Analysis of Raw Toner Powder

A few milligrams of raw toner powder were dissolved in acetone. Approximately 8 μL of this solution was then deposited on a bed of KBr prepared, as before, in a 3-mm diffuse reflectance sample holder. The sample was dried and measured by DRIFTS (using a KBr reference) as described for the analysis of toners on photocopied documents.

General Procedure

The analysis of each toner was repeated a number of times (taking samples from different parts of the photocopied document and varying the quantity of the sample) in order to assess the reproducibility of the method. In addition, the spectra obtained for toners extracted from photocopied documents were compared with those measured for equivalent raw toner powders. Acetone extracts of the paper supports (without toner) were also analyzed to evaluate any influence from this source.

The infrared spectra, although recorded by diffuse reflectance, have been displayed in the transmission format (%T) for the practical reasons outlined by Suzuki and Gresham [15]. Although all spectra were measured between 4000 and 700 cm^{-1} , the majority of spectra in this report are displayed in the range of 2100 to 700 cm^{-1} , since most of the absorption bands of interest are found in this region.

Results

The infrared spectra of the toner samples, obtained by the described diffuse reflectance technique, were of high quality and showed good reproducibility. Sampling of different parts of the same photocopy, of different photocopies produced on the same machine, or of photocopies from different machines of the same type (brand and model) had little

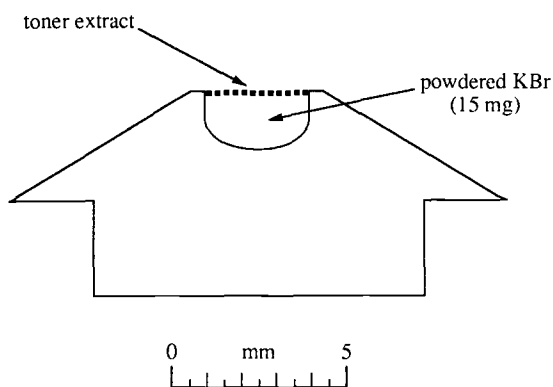


FIG. 1—Cross section of the 3-mm diffuse reflectance sample cup used for DRIFTS measurements of toner extracts.

effect on the spectra. The analysis of toners from photocopies produced on different models of machine that use the same toner also gave identical results (Fig. 2). In addition, no differences were observed between the spectra of bulk toner and those of toner extracted from the photocopied documents (Figs. 3 and 4). (This is contrary to the results reported by Kemp and Totty [8] but in agreement with those reported by Zimmerman et al. [12].) The results indicate that neither the paper nor the photocopying process (that is, the heat or pressure, or both, applied to the toner) has an effect on the infrared spectrum of the toner. (A number of tests using different brands of white and colored—blue, green, and yellow—photocopy paper confirmed that the support does not influence the spectra as measured by the DRIFTS technique.)

Variations in the quantity of toner analyzed did not affect the spectral reproducibility (Fig. 5). It was found that the removal of approximately 1 mm of a photocopied line from a document was sufficient for extraction and analysis by DRIFTS. The reproducibility also appears to be unaffected by the age of the photocopy, as toners on documents over 12 months old have been analyzed without producing any significant differences in the infrared spectra recorded.

The toner samples taken from the photocopied documents and the raw toner powders could be classified into 16 distinct groups according to their infrared spectra. The classification scheme is based on the presence or absence of certain infrared absorption bands (Table 3). Figures 6 through 13 show typical spectra for each of the 16 groups, with the important absorption bands indicated. It is evident that the differences between certain groups are sometimes minimal but nevertheless reproducible. These slight differences are probably due to the presence of minor additives mixed with an otherwise common resin base.

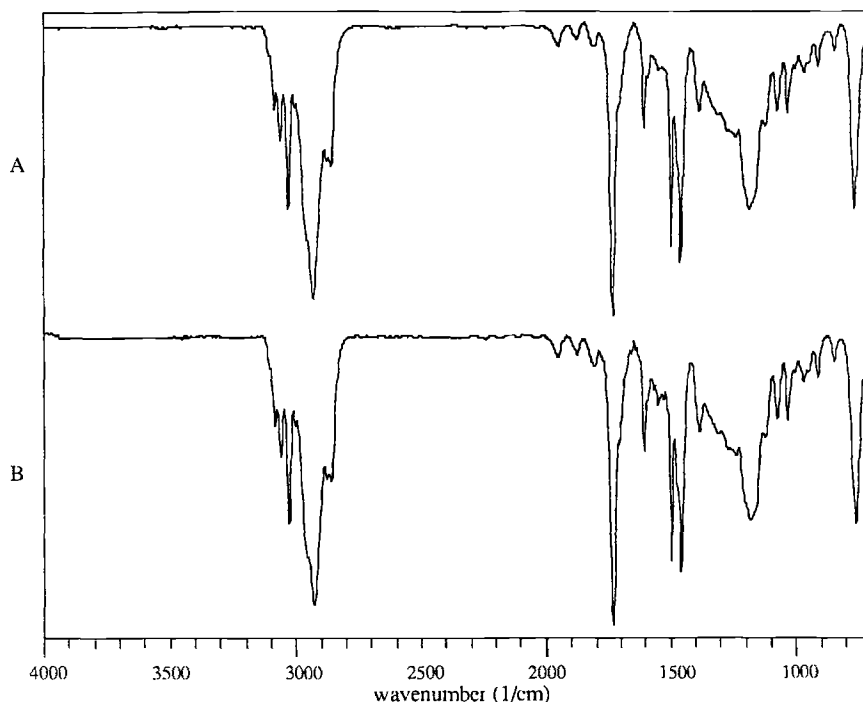


FIG. 2—DRIFTS spectra of toners extracted from photocopies produced on two different models: (a) Agfa X-35 and (b) Agfa X-45. (These two machines use the same Agfa CB-755 toner powder.)

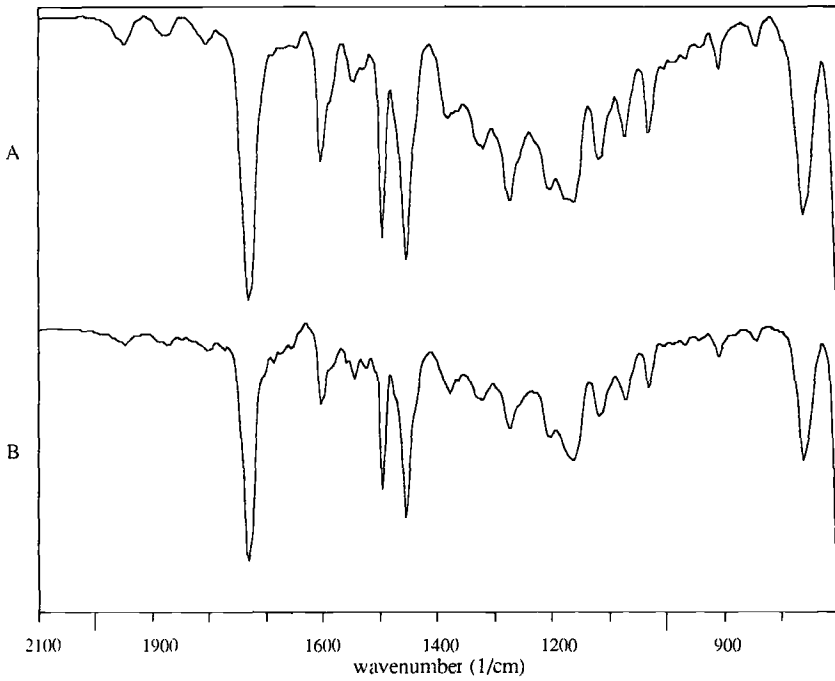


FIG. 3—Comparison between the DRIFTS spectra of (a) a raw toner powder (Minolta EP-350Z) and (b) the corresponding toner extracted from a photocopy (Minolta EP-350Z).

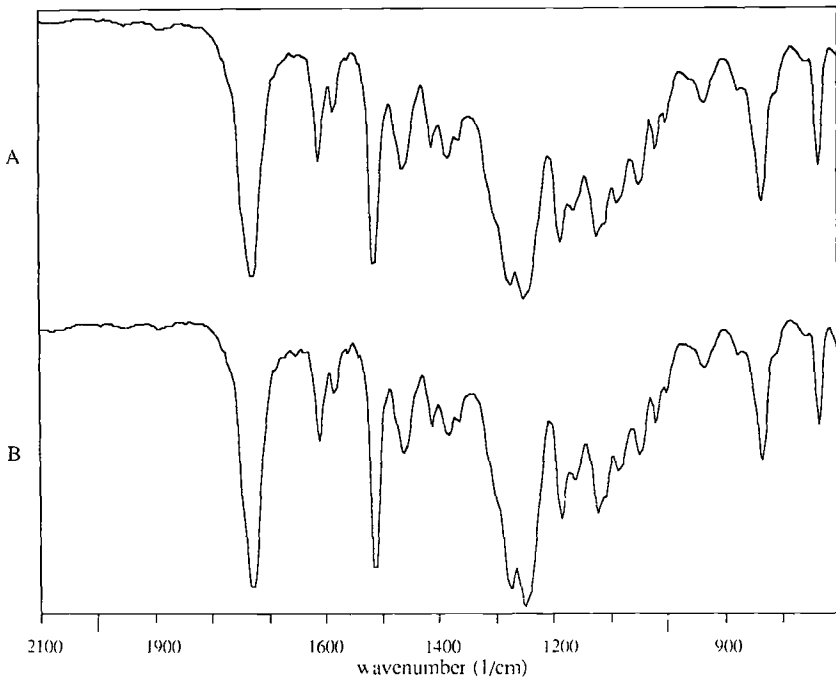


FIG. 4—Comparison between the DRIFTS spectra of (a) a raw toner powder (Agfa CB-752) and (b) the corresponding toner extracted from a photocopy (Agfa X-88).

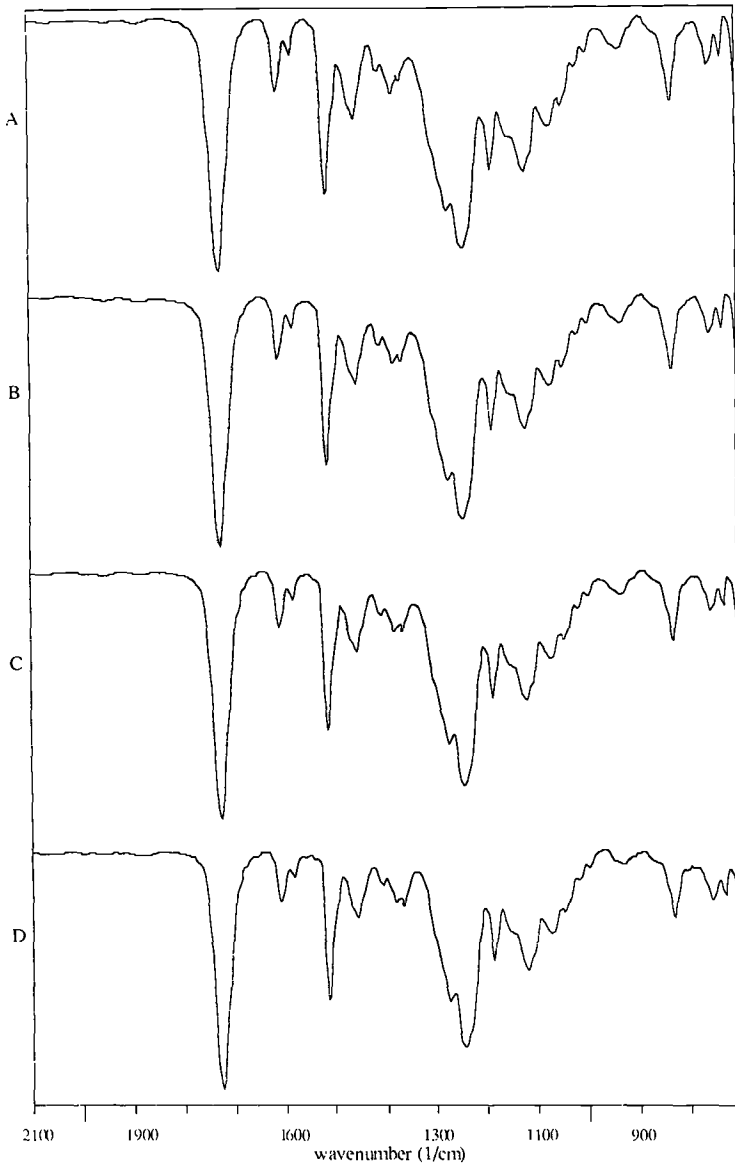


FIG. 5—Example of the reproducibility of the DRIFTS technique for toner analysis. Spectra were recorded for toner samples extracted from four different regions of the same photocopy (Konika 100): (a) from 10 mm of a photocopied line, (b) from 5 mm of a photocopied line, (c) from 3 mm of a photocopied line, and (d) from 1 mm of a photocopied line.

The company Pelikan produces toners in Switzerland for certain brands of copiers to be used in place of the original brand of toner suggested by the manufacturer. Some of these toners are chemically similar to the original brand used and are, therefore, difficult to differentiate by IR spectroscopy (Figs. 14 and 15). Other Pelikan toners are easily distinguished from the original brand of toner (Fig. 16). It is also interesting to note that although most photocopier manufacturers also make their own toners, some toners sold under different brand names are actually produced by the same company; for example,

TABLE 3—*The classification scheme, according to characteristic absorption bands in the diffuse reflectance infrared spectra, for the 32 different toners analyzed.*

Group No.	Characteristic IR Absorption Bands, cm^{-1}	Toner
1	1783, 1246, 843	Canon NP-210
2	1495, 1000, 970, 910	Pelikan Konika 100
3	1410, 1273, 1110, 1085, 1019, 934, 875, 734	Agfa CB-752 Canon NP-6000 Kodak 90-E
4	2973, 1649, 1297, 1159, 985, 775	Oce 1900
5	3030, 1456, 1274, 1074, 1019, 999, 760, 734	Konika 100
6	2928, 1161	Canon NP-1015 Canon NP-3025 Canon NP-3325 Canon NP-4035 Canon NP-4335 Canon NP-9030
7	1704, 1180	Utax C-110 Agfa CB-755 Minolta EP-30 Minolta EP-270 Minolta EP-410Z Minolta EP-450Z Minolta EP-350Z
8	1318, 1272, 1202	Nashua 4550Z
9	1327, 1267, 1179, 799	Ricoh 4000
10	1272, 1202, 1180	Panasonic FP-2520
11	1271, 1162, 1070	Pelikan Canon 270 Sharp SF-8200
12	2094, 1183	Toshiba 5110
13	1185, 1158, 1116	Pelikan Minolta EP-450Z Utax C-220
14	2935, 1272, 1184	Xerox R Xerox 9200
15	1327, 1258, 1200, 1164	Gestetner ZDF-2355
16	1328, 1294, 1161	Agfa CB-749

some Kodak toners are made by Canon and some Agfa toners are made by Minolta [27]. This can sometimes explain why different brands of toner can give identical infrared spectra (for example, Fig. 17) and are therefore classified in the same group.

Discussion and Conclusions

The DRIFTS technique has been shown to be a powerful technique for the analysis of raw toner powders and toners extracted from photocopied documents. The technique is sensitive (between 50 and 100 times more sensitive than transmission methods using KBr pellets) and rapid, and the toner does not need to be physically separated from the paper, as it does in other reported techniques. (Kemp and Totty [8] found that a broad band at 1020 cm^{-1} resulted in infrared spectra of toners contaminated with paper.) The preliminary results have allowed the classification of 32 different toners (raw toner powders and toners extracted from photocopies) into 16 distinct groups, which indicates a high capability of discrimination. It has also been possible to show that no significant differences exist between the spectra of toners extracted from photocopied documents and the spectra of equivalent raw toner powders. This finding indicates that a database

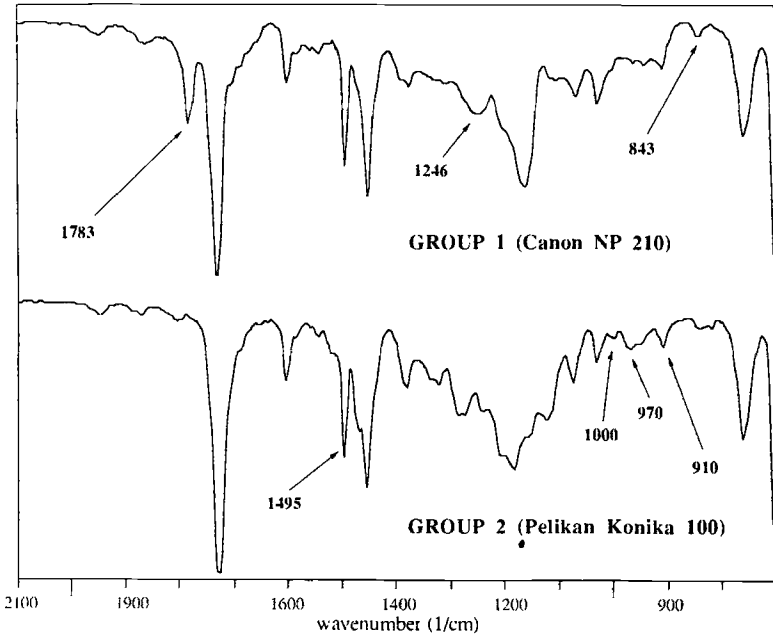


FIG. 6—Examples of spectra from Group 1 (Canon NP-210) and Group 2 (Pelikan Konika 100), showing characteristic absorption bands.

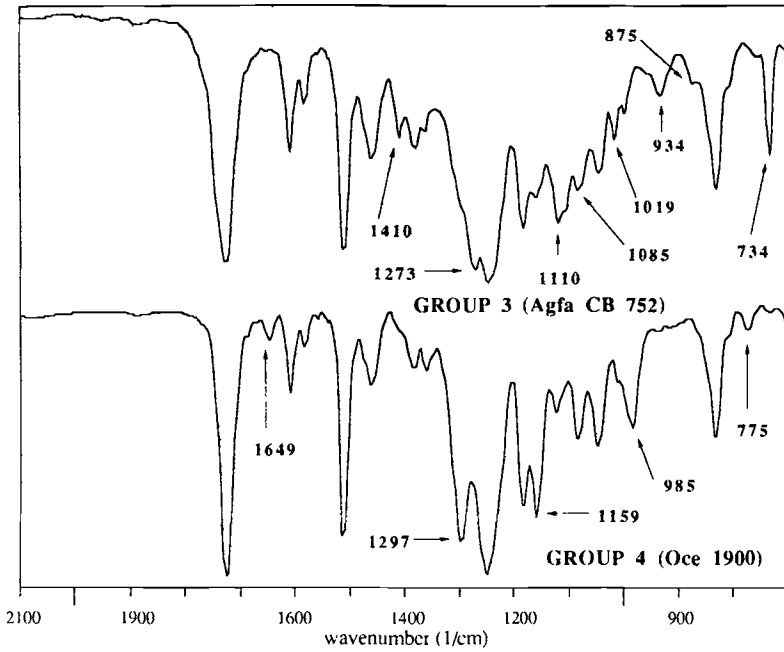


FIG. 7—Examples of spectra from Group 3 (Agfa CB-752) and Group 4 (Oce 1900), showing characteristic absorption bands.

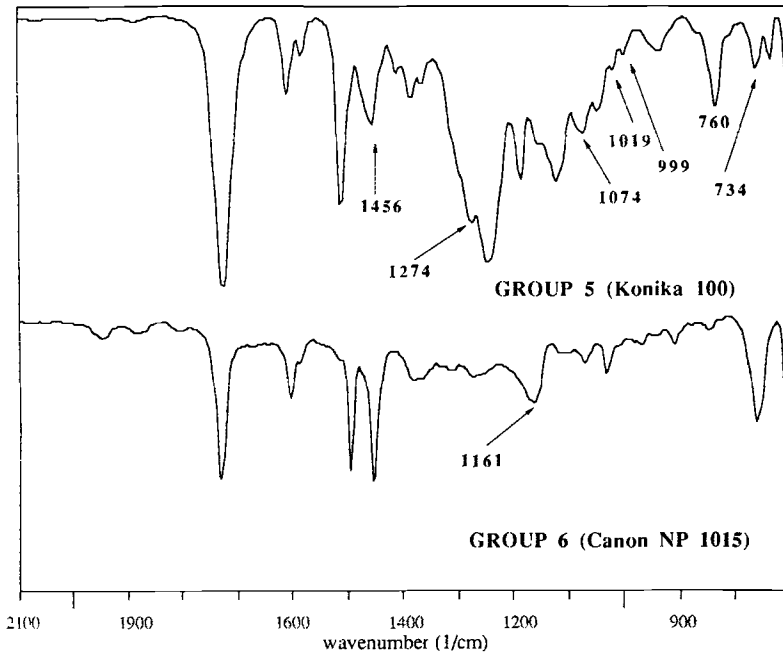


FIG. 8—Examples of spectra from Group 5 (Konika 100) and Group 6 (Canon NP-1015), showing characteristic absorption bands.

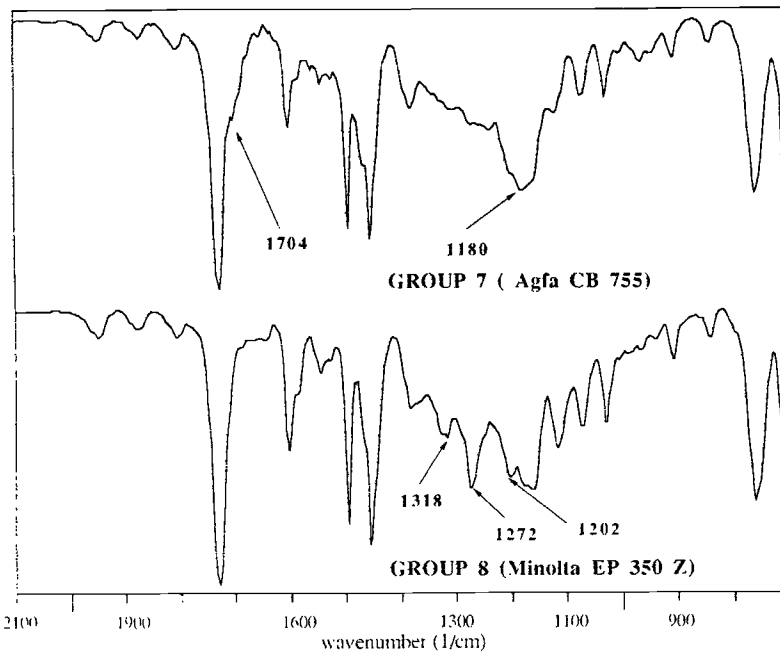


FIG. 9—Examples of spectra from Group 7 (Agfa CB-755) and Group 8 (Minolta EP-350Z), showing characteristic absorption bands.

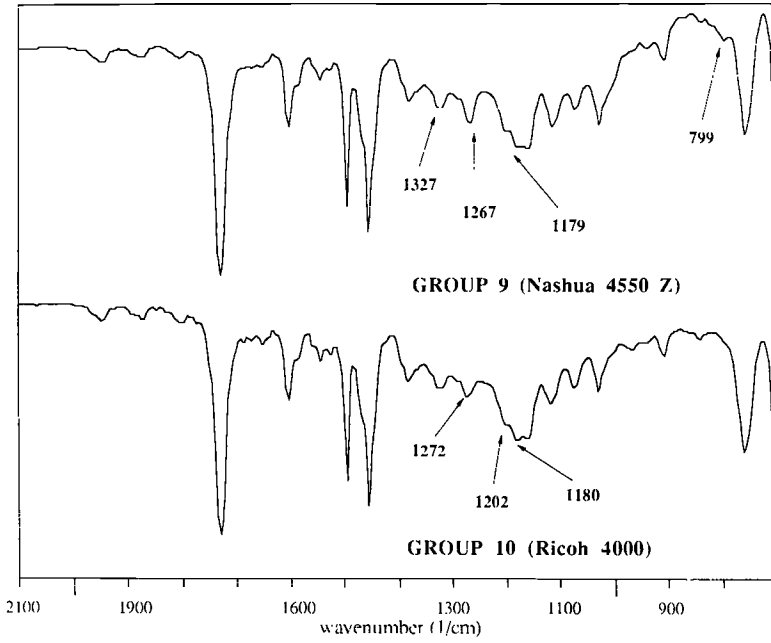


FIG. 10—Examples of spectra from Group 9 (Nashua 4550Z) and Group 10 (Ricoh 4000), showing characteristic absorption bands.

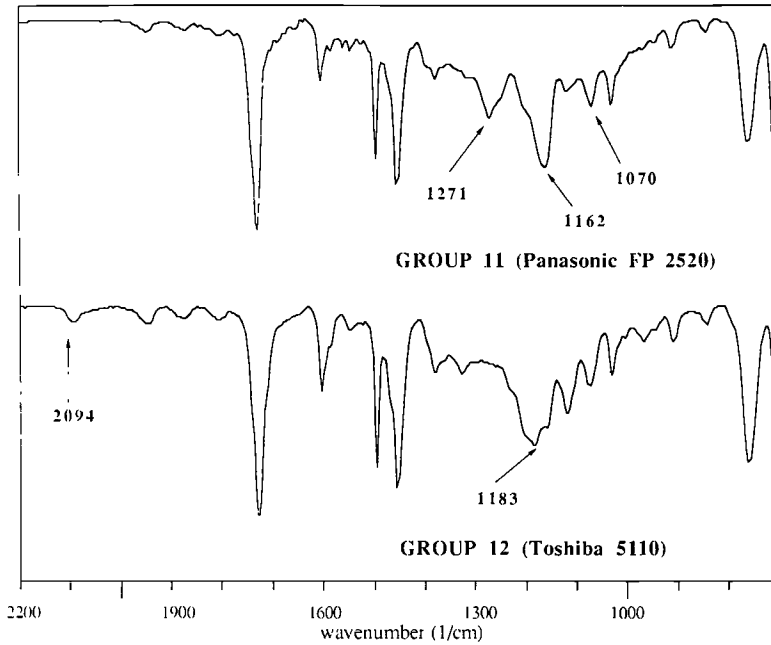


FIG. 11—Examples of spectra from Group 11 (Panasonic FP-2520) and Group 12 (Toshiba 5110), showing characteristic absorption bands.

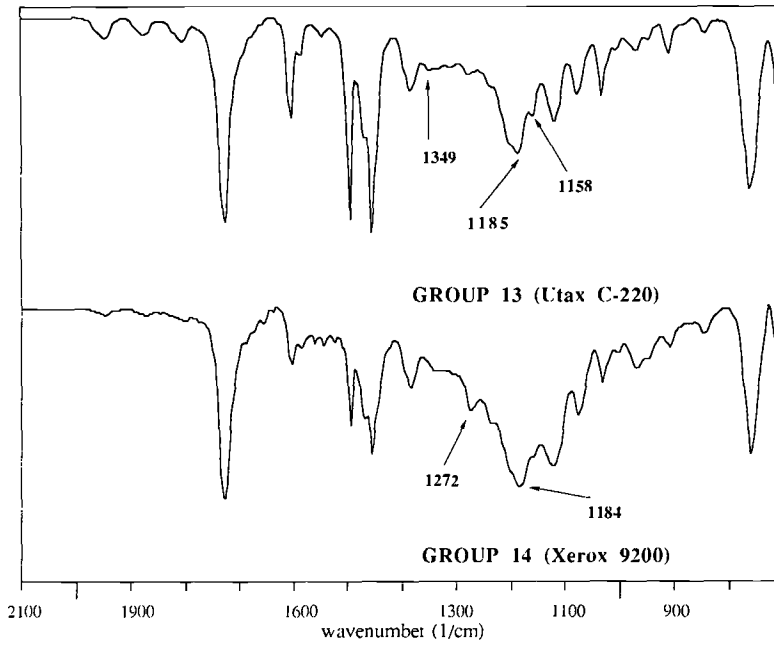


FIG. 12—Examples of spectra from Group 13 (Utax C-220) and Group 14 (Xerox 9200), showing characteristic absorption bands.

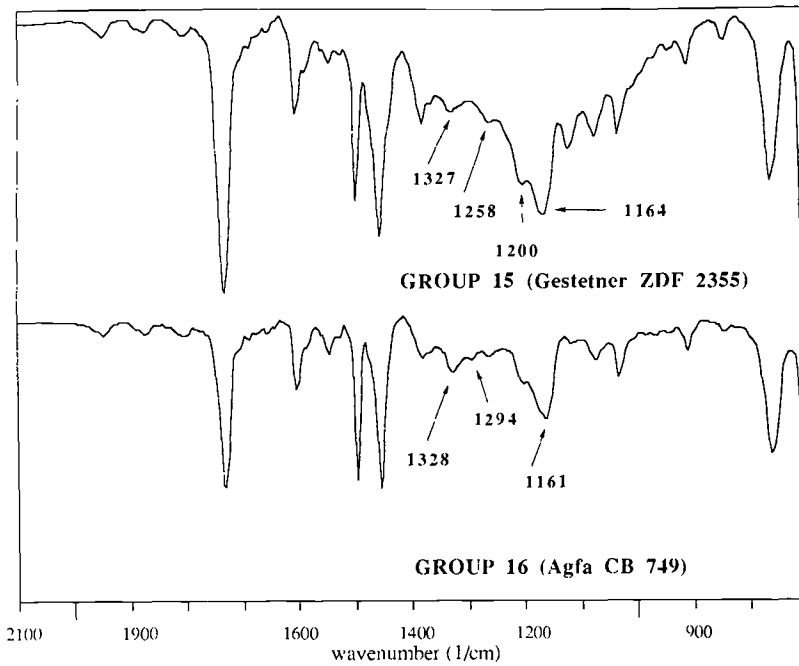


FIG. 13—Examples of spectra from Group 15 (Gestetner ZDF-2355) and Group 16 (Agfa CB-749), showing characteristic absorption bands.

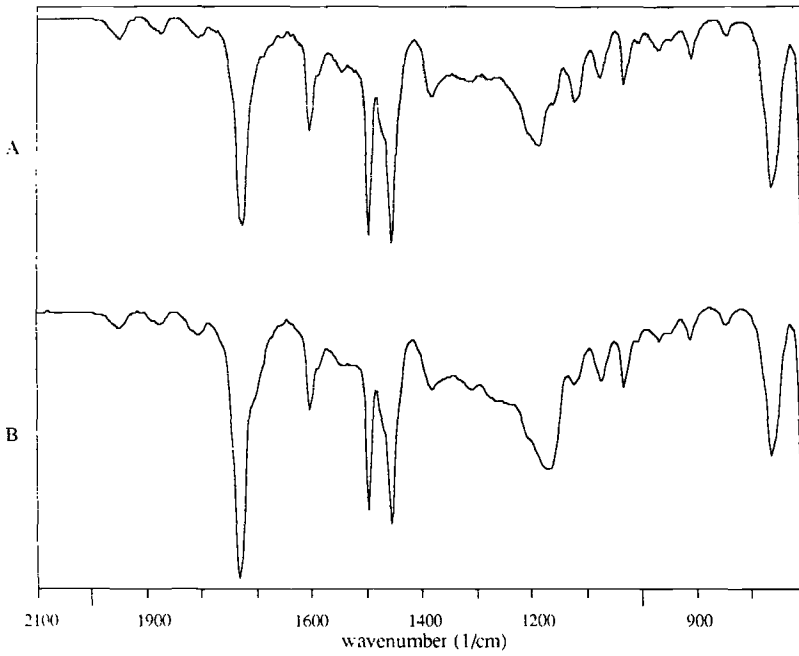


FIG. 14—Comparison between the diffuse reflectance spectra of (a) the toner Pelikan Minolta EP-450Z and (b) the toner Minolta EP-450Z.

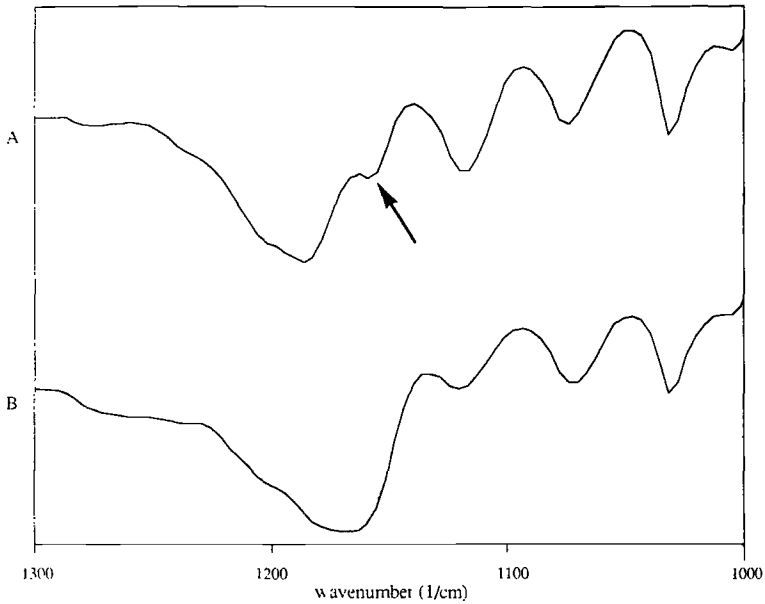


FIG. 15—Comparison between the diffuse reflectance spectra of (a) the toner Pelikan Minolta EP-450Z and (b) the toner Minolta EP-450Z. The spectra have been expanded to highlight the slight, but reproducible, difference between the toners.

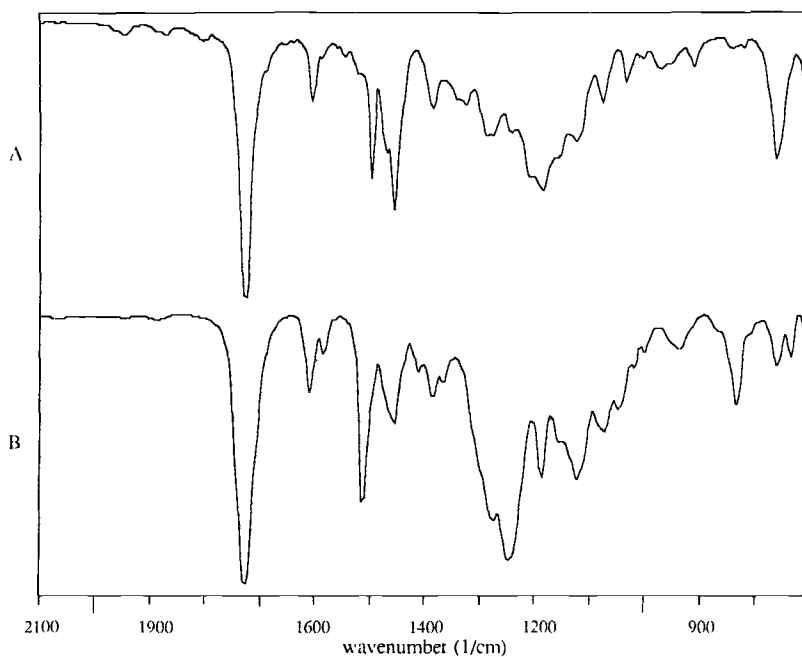


FIG. 16—Comparison between the diffuse reflectance spectra of (a) the toner Pelikan Konika 100 and (b) the toner Konika 100.

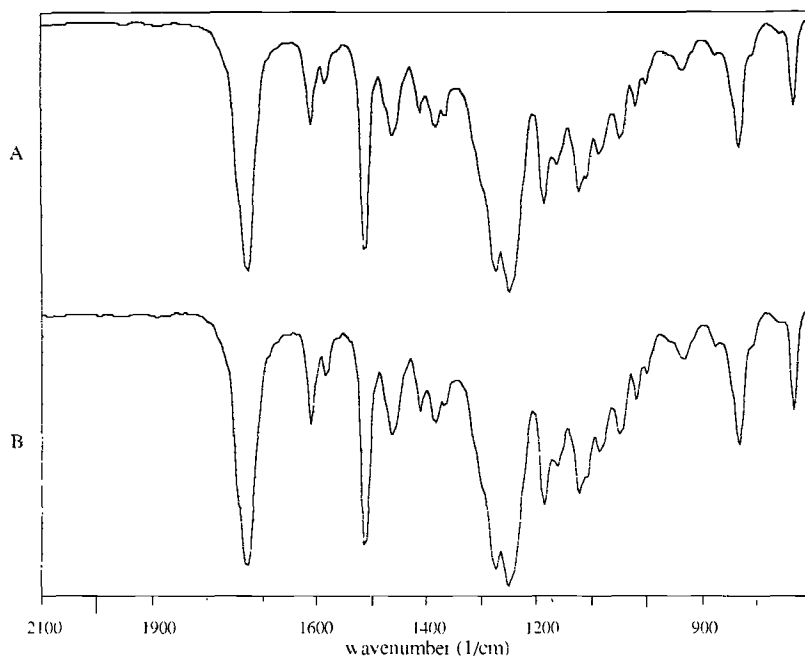


FIG. 17—Comparison between the diffuse reflectance spectra of (a) the toner Canon NP-6000 and (b) the toner Kodak 90-E. Both of these toners are produced by Canon.

constructed from the spectra of raw photocopying toners could, in theory, be used for analysis of toners extracted from photocopied documents.

Because of the encouraging results presented in this preliminary report, a database of infrared spectra for the different bulk photocopying toners available on the Swiss market is in the process of being constructed. A photocopied document can be analyzed by extraction and infrared (DRIFTS) measurement of the toner, and the resulting spectrum can be compared with the spectra in the database. In this manner, a list of likely brands of photocopying toners can be obtained. A knowledge of the compatibility of each individual toner can then be applied to construct a list of photocopying machines (brands and models) that may have been used to produce the questioned document. This information is of direct interest in the investigation of various crimes (anonymous and threatening letters, blackmail, ransom demands, and others). This approach should give satisfactory results in the majority of cases. (Indeed, a number of blind trials in the authors' laboratory have indicated a 100% success rate under ideal conditions.) Problems may arise, however, if toners not normally recommended for use in a particular photocopier are employed. This will always remain an uncontrollable variable in this type of analysis.

The results have allowed us to arrive at the following conclusions:

1. The DRIFTS technique can be successfully, simply, and rapidly applied to the analysis of both raw toner powders and toners extracted from photocopied documents—the IR spectra obtained can, in some cases, differentiate photocopies produced on different machines (photocopies produced on different machines that use the same toner cannot be differentiated by this method).

2. The spectrum of a toner extracted from a photocopy and measured by DRIFTS is identical to the spectrum of the raw toner powder.

3. Toners may be classified into a number of groups according to characteristic absorption bands in their diffuse reflectance infrared spectra.

4. A database of infrared spectra of toners available on the market could be used to obtain a list of likely machines used to produce a questioned photocopy. If a particular case involves copies produced on a number of different machines, the list of suspects can be restricted to those having access to such a combination of photocopiers (in a certain office or department, for example).

A number of questions, however, remain to be answered. Are there significant variations between batches of the same brand of photocopying toner that can be detected by the DRIFTS technique? Do toner samples from photocopies produced over an extended period of time on the same machine give identical infrared spectra? Is the toner analysis affected by prior treatment of the document with reagents used for chemical development of latent fingerprints (ninhydrin, for example)? These questions are currently under investigation, and the results, together with the results from DRIFTS analysis of a wider range of toners, will be reported.

Acknowledgments

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