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An Improvement on Pyrolysis Gas Chromatography for the Differentiation of Photocopy Toners

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ABSTRACT: During the past two decades, the great increase of the use of photocopying machines has given the forensic scientists many practical difficulties in identifying the origins of copied documents. Analysis of chemical properties of toners can be helpful for photocopier discrimination. Several techniques including infrared spectroscopy (IR) pyrolysis gas chromatography/mass spectrometry (Py-GC-MS) and scanning electron microscope-energy dispersive X-ray analysis (SEM-EDX) have been extensively used.

In this study, a Curie-point pyrolyser, a dual wide-bore capillary column GC equipped with dual flame ionization detectors (FIDs) and a personal computer were connected on-line. The differentiation of photocopier toners, however, was based both on the comparison of retention indices and on conventional pattern recognition. The proposed method including the library search is quite simple, reliable and low-cost.

KEYWORDS: questioned documents, Curie-point pyrolyser, retention index, dual wide-bore capillary column GC, photocopier, toner analysis

Over the past two decades there has been a tremendous increase in the use of office and personal photocopying machines [*1,2*]. The speed, simplicity, accessibility and low-cost have made photocopying very convenient not only for legitimate use but also for illegal activities.

Of the various crimes associated with photocopied documents, the most important are the ones concerning terrorism, fraud, counterfeit currency, false insurance certificates, anonymous letters, secret drawings, and other confidential materials. Being aware of the possibility of their being identified through the examinations of handwriting, typewriting, ink or paper, the perpetrators have often resorted to the use of photocopies to obscure the identifiable features of the original documents. Consequently, forensic examiners of questioned documents, in an effort to determine the source of the photocopied products, are encountering, with increasing frequency, photocopies rather than original documents.

There are eleven class characteristics which the FBI laboratory uses to classify photocopies: paper type, toner type, toner application, reduction, enlargement, magnetic properties, paper supply, marks, fusion method, color and laser [*3*]. Holland reviewed the photocopy processes and machines available in Australia [*4*]. The author described

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how the seven features of photocopiers—electrostatic process, paper type, paper feed mechanism, toner type, drum, fixing system, and maximum copy size—can be used to characterize and classify photocopies. Most of these characteristics or features are fairly obvious and can be easily discerned in somewhat nondestructive fashions. The fusion (fixing) method is considered relatively difficult to determine. Crown has given the details of the techniques for differentiating various types of photocopy machines with an emphasis on fusing methods [5]. Very often, after the classification procedure based on the above characteristics, the number of possible source machines can still be large. Therefore, any additional examination of both the toners and photocopy papers would be useful [6–9].

While the comparison of defect marks serves to establish the link between a questioned document and a photocopier, it is often necessary to eliminate a large number of possible source machines before commencing this detailed examination. Usually, differentiation of toners offers a more effective means of achieving this elimination and is carried out prior to the examination of defect marks [10,11].

Totty listed in a relatively recent review [12] ten major methods that had been applied to the problem of toner differentiation: visual examination, optical microscopy (low and high power), scanning electron microscopy, energy dispersive spectrometry (X-ray microprobe analysis), detection of magnetic material, infrared spectroscopy, pyrolysis gas chromatography, pyrolysis gas chromatography/mass spectroscopy, differential scanning calorimetry, and maximum grey scale density determination. After a thorough and objective evaluation, the author proposed a general and sequential approach to toner analysis

1. Determination if photocopy is on plain or coated paper by visual examination.
2. Determination if toner is dry or liquid by low power microscopy.
3. Determination if toner is magnetic or nonmagnetic by a magnetic viewer.
4. Determination of toner fusion method/examination of surface morphology of toner by medium power microscopy and/or scanning electron microscope.
5. Determination of maximum optical density of toner by a microdensitometer.
6. Analysis of inorganic content of toner by energy dispersive microscopy.
7. Analysis of organic content of toner by infrared spectroscopy or pyrolysis gas chromatography.

These statements along with several other studies [12] indicated that the classification of photocopies by chemical analysis of the toners might be a useful addition to the classification by physical characteristics.

Three instrumental techniques, infrared spectroscopy (IR), pyrolysis-gas chromatography (Py-GC) and pyrolysis gas chromatography-mass spectrometry (Py-GC-MS), have been reported for the analysis of the complex organic mixtures, mainly polymeric resins, of toner [13–18]. Py-GC-MS is becoming more common and easier to use in the forensic science area. While it will provide detailed information on the organic components of the toner, the level of detail is, in practice, more than usually required. As to the relative merits of Py-GC and IR, Zimmerman et al. implied that greater discrimination of toners is achieved by the former. It was for this reason that we chose Py-GC as the method for the current study.

Dual wide-bore capillary column GC equipped with dual FIDs has been used in our previous study of arson analysis, the match of chromatograms being based on both retention indices comparison and pattern recognition [19]. The success of that study prompted us to connect a Curie-point pyrolyzer with the established GC system to see if the modified Py-GC can serve as a better method for the differentiation of photocopy toners. During the course of this study, we found a report in the literature which described an automatic sampler for Curie-point Py-GC and measured the retention indices of the pyrolysates of such polymers as poly(methyl methacrylate), poly(ethylene) and poly(propylene) [20]. However, some critical points which we are concerned about with regard to the measurement of retention index were not given.

Presumably because most of the conventional pyrogram comparisons were based on the patterns and retention times, the lack of responsibility has often been considered as one of the drawbacks of Py-GC. Moreover, visual comparisons of a vast amount of Py-GC pyrograms are time-consuming and unreliable. By using relatively reproducible retention indices comparison, library search with the aid of a computer becomes more feasible.

Experimental Procedure

As dry toners are more common than liquid toners in Taiwan, this study dealt mainly with the dry. Should the liquid come to be the analyte in any real casework, the analytical procedure would remain the same except a more destructive removal of the fused toner (for example, by scraping it with a scalpel) from the copy and an extra pyrolysis for the paper fibers blank are required. Solvent extraction method usually does not perform well for the pretreatmental purposes. Photocopies produced on different photocopying machines and different samples of bulk toner powder were obtained from company representatives in Taiwan. Information on the compatibility of each toner was also obtained, where possible.

For the photocopy samples, the fused toner specimens to be analyzed were removed from the photocopies using a technique developed by Munson [18] but with a little modification. A piece of foil (23 by 9 mm) instead of a glass microslide was used directly as the sample lifter. An ordinary iron rather than a soldering iron was used to melt the toner. However, two glass slides were put on top of the foil and at the bottom of the copy, respectively, to assure the compactness between the foil and the copy. A substantial portion of the toner released from the paper and adhered to the foil. The foil with the attached toner was then slightly wound and inserted into the quartz sample tube for the pyrolysis. It is advantageous that the remaining characters on the photocopy are still legible. Figure 1 shows two typical examples of this technique.

FIG. 1—*Portions of the toner on the photocopies,* (a) *have been removed with the foil,* (b).

TABLE 1—*Experimental conditions.*

Pyrolysis	
Foil temperature	590°C
Time for pyrolysis	3 s
Transfer line temperature	200°C
Carrier gas (N_2) flow rate	~ 12 mL/min
Gas chromatography	
Injector temperature	290°C
Detector temperature	280°C
Oven temperature program	40°C initial 2°C/min to 90°C 12°C/min to 150°C 4°C/min to 250°C hold 250°C for 5 min
Carrier gas (N_2) in each column	~ 12 mL/min
Splitless	

For the bulk toner powder samples, approximately 0.5 mg of each sample was tightly sealed with a piece of foil in a manner described in the operator's manual. The foil with its filling was then inserted into the quartz sample tube for the pyrolysis.

Paraffins n-C_6 through n-C_{20} were used as standards for retention indices calculation and were obtained from Merk Co. in Taiwan. A 0.3 to 0.5 μl aliquot of CS_2 solution (approximately 0.001 v/v with respect to each alkane component) of the paraffins was routinely injected into the pyrolyser via the injection port immediately after finishing the pyrolysis of each toner specimen.

Pyrolysis was performed on a Model JHP-2 Curie-point pyrolyzer (Japan Analytical Industry Co.) according to the procedure given in the operator's manual. The pyrolysates along with the paraffins standard were swept by the carrier gas via a transfer line into the injection port of an HNU Model 421 GC, which is equipped with dual wide-bore capillary columns (SPB-1 and SPB-20, 30 m by 0.53 mm ID, 1.5 μm film) and dual FIDs. The pyrograms from the two columns were displayed simultaneously on a personal computer terminal. The software used for the direction of the analysis and the calculation of the retention indices was obtained from Shiunn Hua Co. Ltd. The experimental conditions used are presented in Table 1.

A dBase III Plus program was used to compile a retention indices data bank for all toner samples. This program should also be able to undertake searching for any matching source photocopier.

Results and Discussion

Several modifications were made on the previously reported Py-GC methods for toner analysis and differentiation. First of all, retention indices of the Py-GC peaks were measured as an additional parameter for the comparisons of pyrograms. While conventional pattern recognition and retention time comparison have proved useful to discriminate different toners, these visual methods are usually tedious and lengthy. The reproducibility of the retention indices of very complex GC peaks have been previously verified with various arson accelerants [*19,21*]. In this study, it was further demonstrated that the coupling of a pyrolyzer to a conventional GC would not reduce the reproducibility of the retention indices based on the examination of several toner samples. Table 2 shows the very small variations of fourteen arbitrary Py-GC retention indices from toner Sharp SF7750 in five discrete runs under the same conditions. The reproducibility of R.I. makes a computer-assisted library search more feasible for toner identification. Based on the theory, and our previous experiences, we set ± 2 as the window for a typical match of

TABLE 2—*The reproducibility (precision) of the retention indices of 14 selected Py-GC peaks from toner Sharp SF-7750 (raw powder) in 5 discrete runs on column SPB-1 under the condition described in Table 1.*

No. of Peaks	Max. of R.I.	Min. of R.I.	Mean of 5 tests	Standard deviation	Relative S.D. (%)
1	648.6	647.0	647.6	.626	.09
2	755.8	754.9	755.4	.327	.04
3	849.5	848.9	849.2	.224	.02
4	974.5	974.0	974.1	.260	.02
5	1049.3	1048.9	1049.1	.164	.01
6	1127.9	1127.5	1127.7	.148	.01
7	1215.2	1214.7	1214.9	.192	.01
8	1329.1	1328.4	1328.8	.250	.01
9	1455.3	1454.8	1455.1	.207	.01
10	1537.3	1536.9	1537.0	.167	.01
11	1631.4	1630.8	1631.1	.239	.01
12	1763.9	1763.2	1763.6	.292	.01
13	1873.9	1873.1	1873.5	.328	.02
14	1961.1	1960.4	1960.7	.370	.01

two peaks under comparison. To compile an R.I. data library, as many toner samples as possible should be collected and as many as possible peaks' R.I. values should be included. When doing search, one first picks up manually most of the distinct peaks' R.I. from the pyrogram from the suspect sample, then inputs the R.I. values to the searching program and finally lets the computer search through the R.I. library. The program will do the search in a peakwise fashion, but the R.I. values to be searched will be input to the program all at once. The most critical thing is that all the peaks on the pyrogram of a suspect sample should find their equivalents in one or more R.I. value sets in the library. For an R.I. library to be efficient, the number of peaks taken from a standard sample's pyrogram should always be larger than that taken from a corresponding suspect sample's pyrogram. In fact, conditions associated with the toner analysis are different from those of the arson analysis because a positive answer must exist under any circumstances with regard to whether some kind of toner has been used although the arson analysis may sometimes not involve any arson accelerant at all. Ideally, a certain search made for a certain suspect sample should always lead to one and only one matching toner—that, however, may be used for more than one machine model. The dBase III Plus program used for the searching purpose is listed in Table 3. On the other hand, pyrogram pattern recognition can still be referred to whenever needed.

Dual wide-bore capillary columns and dual FIDs were used throughout this study. The two different pyrograms obtained along with two sets of R.I. data double the discriminating power of each run. The 30 m by 0.53 mm ID wide-bore capillary columns offer a larger sample capacity than narrow-bore capillary columns while maintaining some degree of resolution of complex toner pyrolysates.

Curie-point pyrolysers have previously been used in arson analysis [*22*]. It was, however, in this study that this type of pyrolyser was for the first time used to replace other types of pyrolysers for toner analysis. The pyrolysis temperature can be reached in as short a time as 0.2 s and then maintained constant. The sample is completely pyrolyzed within a few seconds and the pyrolyzates are swept instantly away from the foil so that secondary reactions are minimized. As a result, much better accuracy and reproducibility can be achieved. The modified sample-lifter method further facilitated the analysis process with a Curie-point pyrolyser. Moreover, the foil is reusable.

Thus far, although only 14 bulk toner powder samples as well as some of the corresponding photocopy samples have been analysed under the conditions described in Table

TABLE 3—*The dBase III Plus program used for the R.I. library search.*

```
set talk off
set echo off
set stat off
set device to print
restore from pei20 additive
use ci20
i = 1
do while i < 101
key1 = 1trim(str(i,3))
mspb_1_&key1 = SPB_1_&key1
i = i + 1
enddo
use chow20
replace all qualify with .f.
1
do while .not. eof()
a = 1
i = 1
do while i < 101
key1 = ltrim(str(i,3))
key2 = ltrim(str(a,3))
if mspb_1_&key2 <= SPB_1_&key1 + 2
if mspb_1_&key2 >= SPB_1_&key1 - 2
a = a + 1
key2 = ltrim(str(a,3))
if a = 101
replace qualify with .t.
exit
else
if mspb_1_&key2 = 0
replace qualify with .t.
exit
endif
endif
else
exit
endif
endif
i = i + 1
enddo
skip
enddo
list model for qualify
set device to screen
wait
use
set stat on
set talk on
set echo on
```

1, many preliminary tests under other conditions had been carried out on these two forms of samples. No significant differences in either pyrogram patterns or R.I. values were observed between the two forms of samples. Figure 2 shows an example of this type of comparisons. Also, for fused toner, sampling from different parts of the same photocopy, sampling from different photocopies from the same machine and sampling from different photocopies from different machines using the same toner had little effect on the reproducibility of the pyrogram patterns and the R.I. values if the slight variations of some

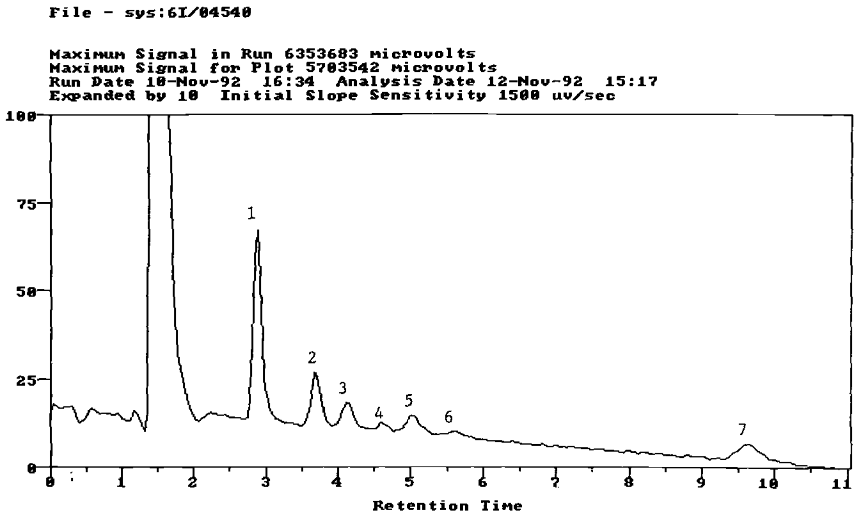

FIG. 2—*The pyrogram pattern (shown for SPB-20 column only) from a Xerox 3990 photocopier raw toner powder sample,* (a) *is nearly the same as that from a corresponding fused toner sample,* (b).

very weak peaks were ignored. These were in agreement with those previously reported [*18*]. Accordingly, we simply used the bulk toner powder samples' data to compile the library. Table 4 displays the R.I. values obtained from the Py-GC analyses of the 14 toners. Based on the above stated method, all of the 14 toner samples could be unambiguously differentiated from one another. Also, ten simulated blind tests based on the established R.I. data and the dBase III Plus searching program have been successfully conducted. In fact, for each pyrogram only 10 R.I. value were arbitrarily picked up to input to the searching program, and for each sample either pyrogram out of the two would be sufficient to definitely achieve the searching purpose.

In order to get the best results, some preliminary tests on such experimental conditions as pyrolysis temperature, pyrolysis time and GC conditions were also carried out. Figure 3 shows the five pyrograms obtained from toner Mita 5585 at five different pyrolysis temperatures, with other conditions being the same. The 358 and 445°C pyrograms contain too small a number of peaks to be efficient for toner differentiation. The 590 670 and 764°C pyrograms are all well suited for toner differentiation. However, the 590°C foil is cheaper and more durable than the latter two. Moreover, higher temperatures like 670 or 764°C may yield more secondary products and cause the analyses to be less reproducible. Therefore, the 590°C foil is recommended for use.

It was reported that the homogeneity of the sample is very important for the pyrolysis to be complete, which in turn is critical for a good reproducibility to be achieved [*18*]. However, this is not really the case for a Curie-point pyrolysis because of the different properties previously mentioned in this paper. In fact, as short as 3 s is usually enough for a 590°C pyrolysis of a toner sample to be comparatively complete, and no significant interference due to time lag for pyrolysis process will occur within such a short period of time. Figure 4 shows three pyrograms obtained from 1, 3 and 5 seconds, respectively, of pyrolysis of toner Xerox 3990, with other conditions being the same. We chose 3 seconds for this study.

Many efforts were made to choose an appropriate GC condition that can be applied to all of the toner samples. That is, the resolution of the GC peaks should always be

TABLE 4a—*The R.I. values recorded from the Py-GC analyses of 14 toners on SPB-1 column.*

Photocopier models	Retention indices[a] (SPB-1 column)									
Cannon np5540	615.4	645.7	656.4	671.6	685.8	716.1	755.5	787.6	826.6	840.9
	848.9	888.9	910.7	933.5	944.7	951.2	972.7	978.5	981.1	983.5
	992.2	1010.8	1021.8	1029.5	1033.6	1040.8	1049.3	1058.7	1067.8	1074.4
	1081.3	1085.0	1090.4	1095.8	1107.2	1116.5	1128.4	1133.9	1142.6	1146.7
	1156.4	1165.3	1182.7	1188.8	1213.7	1225.7	1237.3	1249.9	1258.1	1262.5
	1271.2	1282.4	1308.5	1315.6	1320.8	1335.7	1343.3	1350.9	1361.1	1372.1
	1390.0	1410.6	1426.1	1431.0	1444.4	1455.6	1465.7	1475.7	1482.1	1517.4
	1535.1	1565.0	1579.4	1587.7	1614.9	1631.7	1646.3	1657.1	1662.6	1670.6
	1683.7	1724.8	1732.1	1740.4	1750.9	1763.0	1774.6	1830.8	1837.4	1851.6
	1872.6	1909.5	1917.8	1927.7	1946.4	1960.0	1971.1	1985.0		
Canon np4835	633.3	647.1	755.3	827.1	841.5	849.6	858.1	888.4	896.3	914.1
	924.4	939.1	951.8	958.7	976.6	980.8	985.9	988.0	1010.5	1031.0
	1038.2	1046.1	1071.6	1081.0	1084.4	1109.3	1129.9	1135.2	1149.9	1168.8
	1177.5	1184.6	1215.2	1225.5	1238.7	1250.7	1257.1	1263.2	1272.9	1284.0
	1309.0	1316.5	1324.8	1336.7	1351.8	1363.4	1375.3	1392.8	1413.2	1469.3
	1505.6	1539.8	1582.3	1590.5	1610.7	1618.4	1635.3	1648.2	1671.7	1687.1
	1711.1	1726.9	1735.0	1765.8	1778.1	1789.6	1875.4	1912.5	1961.5	
Konica 1012	628.2	646.8	671.1	686.3	725.3	754.1	827.3	842.2	849.4	874.9
	931.6	971.3	980.6	1009.9	1041.5	1062.1	1086.5	1090.5	1112.9	1129.9
	1147.0	1163.3	1168.9	1192.6	1229.3	1236.2	1248.6	1264.2	1276.3	1287.8
	1309.1	1348.7	1361.4	1382.2	1389.6	1412.5	1418.6	1473.7	1490.9	1563.1
	1627.7	1791.8	1830.5	1876.5						
Konica 3042 3142 4000	626.9	646.9	655.9	686.1	725.1	754.2	787.8	849.5	874.8	888.9
	920.8	935.0	974.0	983.2	992.8	1010.2	1039.3	1044.5	1060.7	1090.2
	1113.9	1129.0	1153.6	1162.1	1169.4	1177.6	1192.1	1212.9	1230.6	1236.6
	1249.6	1266.5	1276.1	1288.6	1361.7	1382.8	1388.8	1419.8	1474.6	1490.0
	1569.1	1589.1								

Konica	626.5	645.9	654.9	670.8	685.5	710.3	724.6	753.6	787.7	841.8
1600	849.2	874.5	889.0	918.4	930.9	969.7	978.9	991.6	1040.8	1058.6
	1062.8	1086.3	1090.2	1111.6	1129.1	1145.4	1160.3	1167.8	1191.6	1227.7
	1234.9	1247.2	1262.6	1275.7	1287.4	1347.3	1360.0	1381.3	1388.6	1412.6
	1417.6	1427.2	1472.8	1489.6	1546.6	1561.4	1589.2	1791.9	1828.9	1875.1
	1919.0	1927.6								
Minolta	647.4	671.1	685.9	710.0	754.5	761.5	769.0	812.6	815.0	817.2
ep270	834.5	841.6	849.7	857.0	887.7	892.9	906.1	911.7	936.0	947.8
ep370	957.1	978.0	984.0	985.9	992.8	1010.7	1032.7	1039.3	1048.1	1058.0
ep370e	1066.7	1082.6	1085.5	1090.4	1093.0	1109.5	1127.1	1134.8	1148.3	1156.5
	1167.6	1182.5	1192.2	1215.3	1224.4	1233.3	1238.1	1249.5	1255.6	1264.9
	1272.3	1282.1	1288.4	1308.5	1327.0	1363.1	1380.1	1388.9	1412.6	1436.9
	1519.8	1527.1	1538.2	1566.9	1580.5	1589.7	1610.5	1632.5	1647.9	1654.3
	1672.7	1687.2	1711.4	1730.9	1739.1	1745.9	1760.4	1771.8	1784.1	1808.3
	1823.3	1869.3	1876.2	1891.0	1926.8					
Minolta	649.1	757.0	788.1	811.5	816.2	836.7	850.9	888.9	893.9	908.3
ep300	912.3	917.2	939.7	948.9	953.6	963.8	981.8	995.3	1011.2	1031.9
ep300re	1046.6	1084.5	1109.9	1126.9	1134.5	1148.9	1155.8	1167.5	1179.2	1191.9
ep310	1210.8	1238.1	1249.1	1258.4	1265.6	1272.4	1282.5	1288.2	1308.2	1325.2
ep320	1362.2	1378.8	1383.4	1411.7	1468.7	1492.2	1519.3	1537.0	1565.8	1582.1
ep530	1590.6	1612.6	1632.2	1649.4	1657.1	1672.5	1687.6	1710.0	1728.8	1736.4
ep530r	1744.3	1764.8	1777.9	1812.5	1829.9	1874.8				
Mita[b]	626.9	646.3	656.7	667.8	709.8	755.4	786.1	811.0	820.3	841.8
5585	849.6	889.0	909.9	913.2	918.3	921.7	926.8	929.3	936.4	943.2
	948.3	955.0	966.7	975.6	983.2	992.8	1011.3	1029.5	1033.0	1037.4
	1048.6	1056.4	1066.3	1071.1	1077.6	1083.2	1085.4	1109.8	1130.1	1134.9
	1143.2	1148.7	1158.3	1167.6	1177.4	1184.4	1215.3	1227.6	1239.4	1249.8
	1257.9	1264.3	1273.2	1278.7	1281.9	1309.5	1317.9	1326.4	1337.6	1357.1
	1364.2	1381.4	1410.0	1426.5	1434.2	1453.8	1463.8	1474.4	1481.1	1512.0
	1535.1	1553.4	1565.4	1570.0	1572.6	1580.9	1589.7	1616.1	1630.6	1646.2
	1652.0	1669.7	1684.2	1704.5	1723.9	1736.8	1751.7	1763.9	1776.6	1808.8
	1819.5	1833.7	1853.9	1874.2	1911.1	1928.9	1947.3	1959.7	1969.5	1986.5

TABLE 4a—*Continued.*

Photocopier models	Retention indices[a] (SPB-1 column)									
Ricoh	646.3	695.0	754.4	786.5	806.9	849.6	882.8	912.2	934.3	941.1
nc100	945.9	964.2	974.2	981.8	1010.7	1029.4	1033.3	1040.2	1049.1	1059.1
	1074.4	1085.4	1108.5	1112.7	1127.8	1133.9	1147.4	1164.0	1186.5	1214.8
	1230.3	1236.3	1241.8	1248.1	1278.1	1308.9	1357.6	1372.4	1382.0	1410.1
	1426.7	1435.9	1455.0	1469.4	1486.8	1514.8	1522.6	1527.7	1555.3	1570.8
	1581.0	1590.5	1611.5	1623.4	1633.7	1648.1	1659.1	1666.3	1688.1	1709.1
	1764.6	1776.6	1817.3	1833.0	1871.6	1883.4	1888.8	1947.6	1959.3	1984.7
Sharp	610.3	627.4	647.9	717.3	755.4	841.7	849.4	887.9	987.0	912.5
sf7750	934.8	941.3	946.3	950.8	974.0	982.7	1011.0	1029.6	1033.5	1040.4
	1049.1	1058.8	1067.1	1072.2	1083.1	1084.0	1109.2	1127.6	1134.3	1143.2
	1148.1	1166.8	1177.0	1183.8	1191.9	1214.9	1224.2	1238.8	1249.6	1257.8
	1263.7	1272.4	1278.8	1281.4	1309.2	1316.4	1328.4	1336.2	1356.1	1362.9
	1411.5	1434.7	1455.0	1467.2	1474.3	1483.4	1503.1	1519.6	1537.0	1554.4
	1566.6	1580.7	1588.8	1610.1	1631.0	1646.8	1652.2	1657.2	1671.0	1684.7
	1709.2	1725.9	1740.6	1763.6	1776.4	1822.2	1832.4	1839.1	1852.1	1873.5
	1910.7	1938.8	1946.6	4960.7	1976.2	1986.5				
Sharp	647.1	721.2	754.9	786.2	841.1	849.4	889.6	906.3	912.2	918.1
sf770	935.7	947.8	975.4	983.0	986.6	1010.9	1032.7	1039.8	1048.0	1055.2
sf771	1065.6	1072.6	1080.4	1085.2	1108.7	1128.4	1134.9	1148.8	1160.9	1167.4
sf781	1177.0	1184.0	1215.1	1223.6	1238.5	1272.1	1282.0	1308.7	1316.4	1325.9
sf7400	1336.2	1363.4	1412.8	1469.2	1519.9	1538.2	1581.5	1589.3	1611.7	1632.2
	1647.8	1658.7	1672.0	1686.2	1727.3	1739.8	1764.7	1776.6	1815.9	1838.9
	1873.4	1911.8	1946.6	1959.2	1986.0					

Toshiba	611.5	627.9	647.6	755.5	786.7	841.7	849.7	887.0	907.2	913.0
bd9100	917.0	919.9	935.6	947.8	955.5	974.9	982.6	1010.7	1029.3	1032.4
	1037.4	1048.3	1056.3	1082.8	1109.5	1129.6	1134.7	1148.2	1167.5	1210.8
	1215.1	1227.2	1238.9	1249.6	1281.5	1287.2	1308.6	1325.4	1363.6	1384.2
	1409.7	1435.7	1455.7	1466.3	1476.5	1485.5	1517.9	1536.7	1555.6	1571.9
	1581.9	1616.8	1631.8	1648.1	1654.6	1659.7	1672.7	1687.3	1709.0	1728.1
	1739.2	1753.9	1764.5	1777.5	1833.2	1837.0	1874.0	1911.3	1947.7	1960.9
	1987.9									
Xerox	629.2	646.9	755.0	811.2	828.8	841.9	850.0	888.0	892.9	907.0
3990	913.5	919.2	936.9	949.0	967.0	980.3	985.0	1011.2	1032.6	1039.2
5026	1048.2	1058.0	1065.9	1072.7	1082.7	1086.0	1093.7	1110.1	1127.9	1135.6
5030	1149.0	1157.0	1168.4	1183.8	1188.9	1193.4	1211.7	1215.9	1224.6	1239.1
	1250.3	1256.1	1264.5	1273.2	1282.9	1293.0	1308.9	1317.7	1327.3	1337.3
	1345.4	1364.1	1377.8	1384.7	1389.6	1413.5	1438.3	1469.4	1477.7	1487.1
	1527.1	1538.7	1567.0	1635.6	1649.0	1659.6	1672.4	1687.7	1710.2	1735.2
	1746.2	1753.0	1762.7	1777.9	1811.9	1849.9	1875.2	1912.6	1961.7	
Xerox	625.0	645.8	694.2	754.3	837.6	849.6	886.0	892.3	912.8	918.7
5043	921.8	936.2	948.2	980.0	984.5	1010.4	1029.6	1031.8	1039.0	1047.6
5990	1070.7	1084.8	1109.7	1126.5	1134.7	1148.6	1156.1	1167.2	1181.1	1192.2
	1215.2	1227.2	1238.7	1249.5	1264.4	1272.1	1282.0	1308.1	1316.1	1325.7
	1411.8	1537.9	1566.2	1581.4	1589.4	1611.4	1635.2	1648.2	1657.7	1672.5
	1676.8	1687.4	1710.0	1728.9	1735.7	1752.7	1765.1	1777.3	1810.9	1818.2
	1835.3	1872.9	1911.8	1947.9	1960.4	1987.3				

[a]Most of the R.I. values listed in this table are the means of three independently obtained values. For some weak Py-GC peaks, the R.I. values may be the means of two independently obtained values or just a single-run value.

[b]A few weak peaks' R.I. values are not included in this block as we believed the 100 vacancies provided in the dBase III Plus program would be definitely enough for the searching purpose.

TABLE 4b—*The R.I. values recorded from the Py-GC analyses of 14 toners on SPB-20 column.*

Photocopier models	Retention indices[a] (SPB-20 column)									
Canon	643.4	706.6	738.3	813.2	846.7	860.0	864.8	874.5	908.1	941.8
np5540	964.7	979.5	984.6	1015.7	1033.6	1047.7	1060.1	1064.0	1074.1	1090.5
	1117.8	1121.2	1131.6	1136.5	1206.5	1211.4	1215.6	1225.9	1243.2	1252.5
	1322.2	1334.3	1340.4	1358.2	1381.4	1391.3	1411.3	1426.3	1464.0	1471.0
	1488.3	1514.7	1520.6	1533.9	1539.8	1554.8	1613.5	1621.6	1626.9	1644.2
	1654.6	1671.0	1679.4	1722.5	1730.0	1735.7	1744.7	1762.7	1773.5	1814.6
	1855.0	1876.2	1885.7	1914.4	1925.1	1934.7	1943.1	1967.1	1983.3	
Canon	635.7	707.6	812.9	847.7	860.0	865.7	908.2	913.7	964.4	983.6
np4835	1037.2	1053.4	1064.3	1067.1	1075.7	1080.6	1088.4	1091.2	1116.9	1121.7
	1129.7	1148.2	1154.9	1164.8	1184.2	1186.2	1212.6	1214.4	1227.9	1245.4
	1256.0	1264.8	1268.7	1286.6	1315.7	1323.2	1341.0	1358.9	1382.5	1411.7
	1428.3	1434.9	1461.6	1467.7	1522.3	1558.4	1647.3	1658.0	1674.5	1682.5
	1709.1	1734.1	1740.1	1749.3	1766.5	1779.7	1806.6	1821.5	1849.4	1859.7
	1879.6	1891.6	1909.6	1917.0	1926.9	1937.8	1947.2	1971.5	1989.6	
Konica	661.7	708.1	761.9	812.3	836.2	848.0	908.7	949.1	972.1	1046.1
1012	1055.1	1067.3	1077.6	1087.7	1090.1	1123.4	1140.1	1161.9	1167.4	1177.1
	1187.9	1214.5	1222.1	1247.2	1255.0	1258.5	1284.3	1318.2	1331.0	1352.8
	1368.0	1382.5	1409.3	1418.6	1460.2	1479.4	1484.5	1522.4	1537.9	1616.6
	1661.5	1669.7	1718.9	1852.8	1952.3					
Konica	615.8	637.7	707.7	740.3	812.4	835.6	908.5	948.9	971.1	1049.1
3042	1061.0	1070.3	1079.9	1122.4	1135.4	1147.6	1159.4	1165.0	1175.5	1188.3
3142	1215.6	1221.8	1255.5	1258.7	1275.8	1284.6	1318.4	1331.3	1340.3	1353.1
4000	1360.4	1368.3	1382.0	1409.4	1419.6	1460.2	1481.1	1538.6	1618.8	1729.3
	1855.9									

Konica	635.5	657.3	663.8	706.5	740.7	759.5	811.5	835.2	847.0	907.8
1600	913.5	948.1	960.8	971.7	994.2	1044.3	1053.1	1065.5	1075.8	1086.5
	1122.8	1140.2	1148.9	1161.8	1167.1	1176.4	1182.1	1187.1	1213.1	1220.4
	1231.6	1237.6	1245.8	1253.2	1282.8	1316.6	1329.7	1351.2	1361.8	1381.1
	1407.6	1417.0	1435.0	1444.6	1450.5	1458.4	1476.8	1482.4	1519.9	1536.0
	1547.4	1615.4	1639.8	1660.2	1668.4	1715.3	1730.8	1769.6	1851.0	1871.7
	1919.2	1946.1	1989.7							
Minolta	636.3	658.7	708.3	768.3	812.5	847.1	860.4	871.1	879.4	888.5
ep270	908.9	963.6	985.7	1041.6	1052.5	1063.0	1066.4	1080.4	1088.4	1091.1
ep370	1117.5	1121.4	1131.0	1151.1	1155.4	1166.1	1212.2	1226.8	1234.3	1243.4
ep370e	1254.7	1260.7	1266.3	1286.4	1314.2	1322.8	1342.1	1372.3	1392.8	1411.2
	1424.7	1454.4	1479.0	1520.8	1530.3	1545.5	1556.3	1644.8	1656.1	1672.6
	1680.7	1732.7	1740.8	1747.1	1763.9	1780.3	1816.3	1845.5	1855.3	1877.5
	1889.3	1915.5	1936.0	1946.4	1955.9	1969.6	1986.7			
Minolta	635.9	652.2	709.4	757.3	814.4	862.4	872.0	892.4	909.1	914.2
ep300	918.4	964.8	978.5	992.5	1049.3	1050.0	1057.1	1065.4	1070.4	1075.1
ep300re	1081.7	1083.2	1088.4	1092.0	1117.8	1123.0	1129.3	1148.8	1154.0	1164.4
ep310	1188.2	1213.4	1224.4	1228.3	1235.3	1242.4	1257.7	1262.3	1276.3	1287.1
ep320	1316.2	1323.1	1342.2	1352.7	1358.7	1372.3	1380.9	1414.9	1426.2	1453.1
ep530	1536.3	1557.0	1616.1	1644.8	1656.2	1673.4	1680.9	1733.0	1742.8	1747.2
ep530r	1766.3	1783.2	1807.4	1821.2	1845.3	1857.8	1879.0	1890.5	1916.6	1937.0
	1947.6	1957.4	1970.8	1988.3						
Mita[2]	634.9	656.2	669.1	707.0	722.0	739.6	760.0	788.8	813.7	847.2
5585	860.5	908.1	964.0	967.4	982.1	987.5	992.8	994.9	1035.9	1051.7
	1067.1	1076.2	1075.9	1080.8	1088.9	1091.6	1118.4	1122.5	1131.9	1135.3
	1142.2	1152.1	1175.7	1181.9	1188.9	1212.5	1216.1	1227.5	1235.9	1245.6
	1254.8	1264.0	1268.9	1285.9	1290.2	1315.8	1324.0	1335.3	1342.7	1348.0
	1353.1	1360.1	1365.9	1375.6	1384.6	1415.4	1427.0	1436.0	1443.5	1456.2
	1470.9	1509.9	1520.5	1531.6	1538.1	1543.7	1550.4	1559.7	1570.9	1578.1
	1587.9	1609.6	1628.6	1646.2	1657.8	1664.8	1673.0	1681.9	1691.5	1710.3
	1715.4	1725.4	1733.1	1738.8	1747.8	1754.6	1764.8	1777.2	1805.2	1817.4
	1858.5	1875.9	1886.5	1893.9	1907.3	1923.4	1933.1	1944.4	1969.5	1986.3

TABLE 4b—*Continued.*

Photocopier models	Retention indices[a] (SPB-20 column)									
Ricoh	659.9	707.1	756.8	765.1	813.2	819.9	851.6	861.0	870.2	909.0
nc100	958.1	972.5	1035.6	1049.2	1059.5	1067.8	1090.0	1118.3	1121.7	1131.3
	1141.8	1151.1	1157.9	1183.2	1188.4	1215.1	1226.9	1253.7	1259.0	1277.0
	1288.8	1314.9	1330.3	1347.7	1378.5	1410.8	1420.2	1425.3	1442.8	1459.4
	1490.5	1515.2	1525.9	1531.0	1543.5	1553.7	1561.0	1577.1	1589.3	1617.2
	1636.5	1645.0	1653.8	1662.7	1671.9	1677.2	1684.2	1731.4	1739.4	1768.7
	1775.6	1818.4	1854.3	1876.8	1888.9	1915.3	1934.7	1944.9	1968.4	1984.8
Sharp	633.7	654.5	667.4	706.0	756.8	811.8	846.8	859.9	908.0	963.9
sf7750	972.7	981.1	983.2	987.1	1017.5	1023.8	1034.3	1049.3	1060.5	1067.4
	1075.6	1091.2	1118.5	1122.3	1131.8	1136.3	1151.2	1156.8	1182.4	1189.0
	1212.0	1216.2	1227.3	1234.6	1244.7	1254.1	1261.9	1268.8	1285.3	1315.3
	1323.8	1335.3	1342.0	1353.2	1353.9	1365.1	1374.1	1381.7	1415.9	1426.0
	1435.1	1443.5	1448.8	1519.1	1537.1	1542.4	1549.2	1556.0	1559.1	1587.0
	1614.8	1645.6	1656.0	1672.3	1681.2	1713.7	1730.5	1737.4	1744.5	1762.3
	1773.2	1813.0	1855.8	1875.5	1889.0	1914.7	1935.5	1946.1	1969.5	1986.6
Sharp	633.8	656.0	760.8	761.0	783.7	812.1	846.6	859.7	907.8	916.6
sf770	928.9	1033.0	1051.2	1062.0	1066.8	1090.4	1117.2	1121.5	1130.9	1133.9
sf771	1149.6	1156.8	1172.0	1183.9	1187.8	1211.9	1225.9	1245.1	1252.8	1264.3
sf781	1270.2	1283.8	1314.4	1322.9	1414.8	1424.9	1555.3	1645.0	1655.6	1671.5
sf7400	1680.3	1732.6	1741.1	1748.1	1765.2	1776.7	1804.6	1818.7	1848.6	1856.6
	1877.4	1889.4	1907.6	1914.8	1935.3	1945.6	1968.3	1985.6		

Toshiba	636.6	656.9	670.2	708.1	722.5	757.9	813.1	847.6	860.3	908.8
bd9100	963.0	971.8	1034.1	1050.2	1054.9	1061.4	1088.2	1090.9	1117.1	1121.5
	1131.4	1133.3	1151.5	1188.6	1211.9	1226.6	1234.9	1244.7	1254.0	1268.0
	1285.0	1289.3	1314.9	1323.0	1413.9	1425.7	1434.8	1442.9	1510.5	1518.3
	1549.5	1551.1	1569.8	1587.0	1609.7	1645.0	1657.2	1672.5	1681.1	1733.5
	1739.9	1747.5	1765.1	1777.6	1806.1	1818.9	1848.1	1857.4	1877.9	1889.7
	1906.7	1915.1	1936.1	1946.4	1970.0	1987.2				
Xerox	636.9	657.9	708.3	761.7	813.1	847.8	860.9	909.1	964.2	971.9
3990	984.5	993.8	1017.1	1045.4	1056.1	1064.4	1089.5	1091.6	1118.3	1122.7
5026	1131.4	1151.9	1157.4	1166.3	1189.8	1213.8	1228.1	1235.0	1243.1	1250.9
5030	1256.1	1267.2	1287.0	1323.8	1342.9	1352.8	1359.4	1367.6	1373.2	1381.6
	1410.7	1413.2	1425.7	1428.2	1463.6	1466.1	1479.3	1520.8	1531.0	1537.2
	1547.6	1558.0	1570.8	1646.8	1657.0	1673.5	1733.5	1739.6	1742.2	1749.1
	1766.0	1779.1	1805.6	1821.0	1843.9	1857.4	1878.9	1880.1	1890.7	1919.6
	1937.8	1947.1	1957.3	1970.4	1988.0					
Xerox	634.8	707.2	755.9	812.2	885.0	892.8	908.6	961.4	972.5	993.0
5043	1044.6	1054.3	1061.4	1068.3	1090.9	1116.6	1121.1	1129.8	1145.6	1149.6
5990	1154.7	1165.4	1188.2	1215.0	1222.4	1227.0	1233.4	1241.6	1254.3	1260.4
	1287.4	1315.5	1342.1	1353.0	1358.9	1366.1	1372.4	1415.1	1426.1	1536.0
	1557.1	1645.4	1656.0	1673.1	1732.4	1738.1	1743.0	1746.9	1749.1	1766.2
	1778.3	1805.7	1820.9	1843.6	1856.8	1878.5	1889.9	1915.9	1936.3	1946.0
	1957.2	1969.0	1986.4							

[a]Most of the R.I. values listed in this table are the means of three independently obtained values. For some weak Py-GC peaks, the R.I. values may be the means of two independently obtained values or just a single-run value.

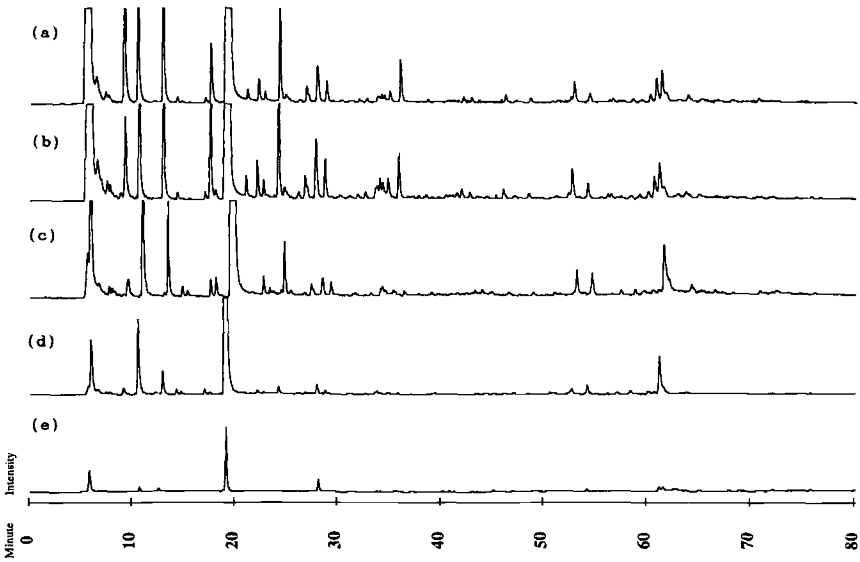

FIG. 3—*Five pyrograms obtained from the pyrolyses of toner Mita 5585 (raw powder) at:* (a) *764,* (b) *670,* (c) *590,* (d) *445 and* (e) *358°C, respectively, with other conditions being the same.*

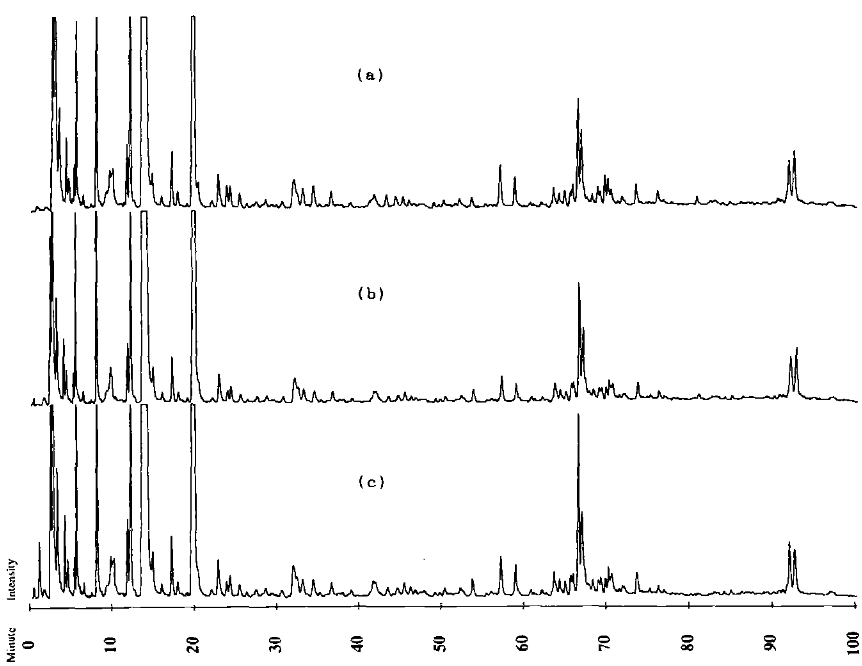

FIG. 4—*Three pyrograms obtained from:* (a) *1,* (b) *3 and* (c) *5 sec, respectively, of pyrolysis of toner Xerox 3990 (raw powder), with other conditions being the same.*

acceptable while the chromatography not too time-consuming. It was the condition described in Table 1 that we finally chose for this study. Displayed in Figure 5 is a typical pyrogram obtained from the Py-GC analysis of toner Toshiba BD-9100. The paraffins reference peaks and the R.I. values of most of the pyrolysate peaks are also shown. It is noteworthy that the real resolution of the chromatographic peaks and the real reproducibility of the R.I. values are both far better than what can be shown in the rough-looking pyrogram. The time (60 min) taken for each run seems too long. However, this is normal for a 30 m by 0.53 mm ID capillary column and a complex sample like photocopier toner.

We have recently purchased a new Model JHP-3S Curie-point pyrolyser, which is in most aspects more sophisticated and more precise than the old Model JHP-2 Curie-point pyrolyser. The major difference between the two pyrolysers is that in the Model JHP-3S design the pyrolysis chamber is located right on top of the GC inlet and does not use a transfer line. This once again considerably reduces the probability of secondary reactions or condensation of the pyrolysates. Therefore, the new Model JHP-3S system was expected to achieve better reproducibility and need smaller sample size than the old JHP-2 system. As far as the sample size is concerned, it turns out that the new Py-GC system usually took only approximately 50 μg of bulk toner powder sample (that is, no more than 5 letters for fused toner, and the foil sample lifter can be cut into even small size.), which is about one tenth that for the old system. Of course, the paraffins injection amount should be reduced proportionally. As for the reproducibility, although the intrasystem reproducibility was improved with the new JHP-3S pyrolyser, the R.I. data were, unfortunately, nontransferable between the two Py-GC systems probably due to the different designs with respect to the transfer line. Figure 6 shows the appreciable variations of the Py-GC data when raw toner powder Canon NP-5540 was pyrolyzed on the two different pyrolyzers but with the same Py-GC condition programming.

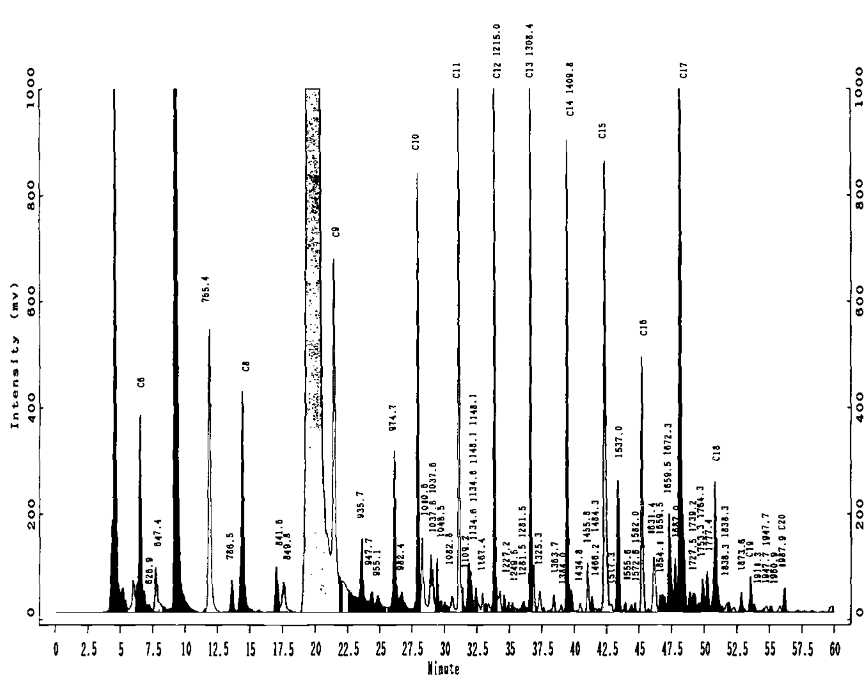

FIG. 5—*A typical pyrogram (shown for SPB-1 column only) obtained from the Py-GC analysis of toner Toshiba BD-9100 (raw powder).*

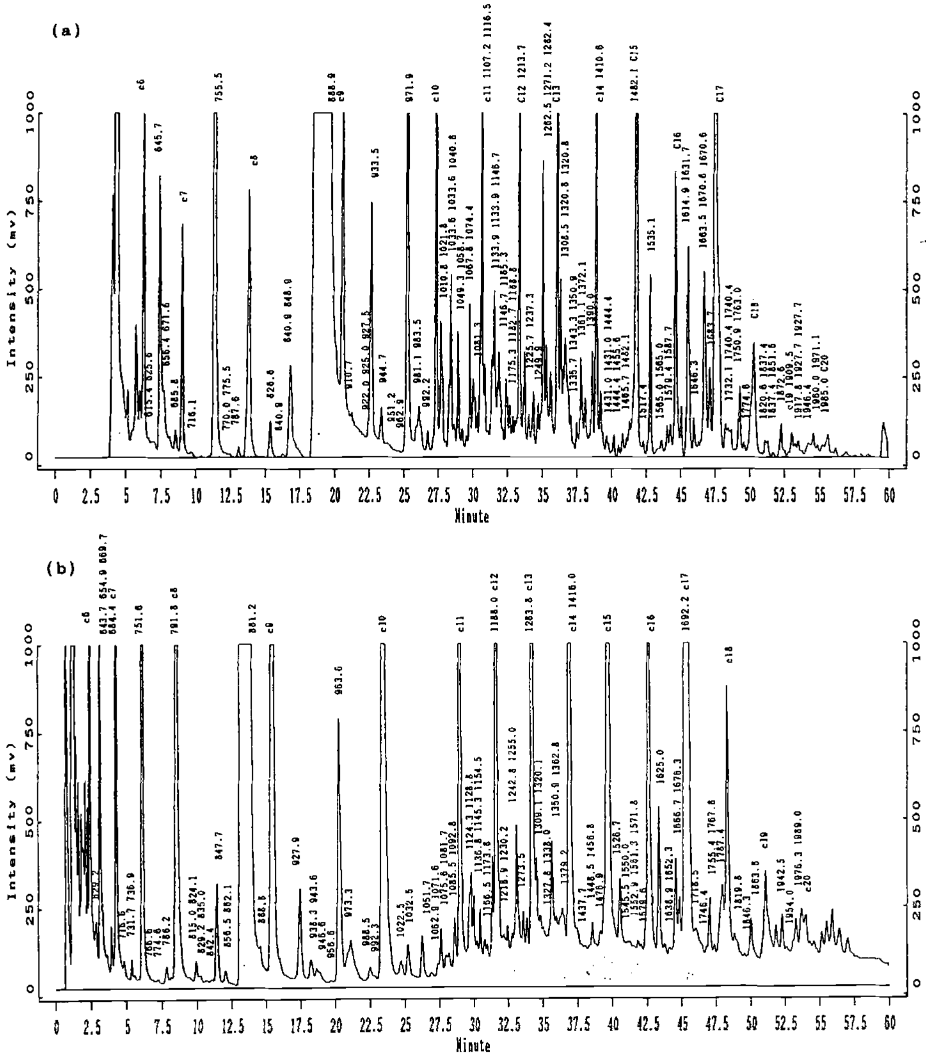

FIG. 6—*The two pyrograms (shown for SPB-1 column only) obtained from the Py-GC analyses of toner Canon NP-5540 (raw powder) on:* (a) *JHP-2 pyrolyser/HNU 421 GC and* (b) *JHP-3S pyrolyser/HNU 421, GC, respectively. The Py-GC conditions used were both the same as those listed in Table 1 except in JHP-3S system there is no transfer line. Sample size:* (a) *0.5 mg,* (b) *50 μg.*

Conclusions

The results presented in this report demonstrated that the modified Py-GC is a useful technique for the differentiation of photocopying toners and that the computer-assisted library search based on retention indices comparison is a powerful approach to the locating of suspect photocopying machines. For a real questioned photocopy casework sample, the proposed Py-GC analytical procedure for the fused toner is summarized in Fig. 7. While it can still be referred to whenever needed, the conventional pyrogram pattern recognition has not been actually used yet so far and probably will hardly find its necessity for most of the samples.

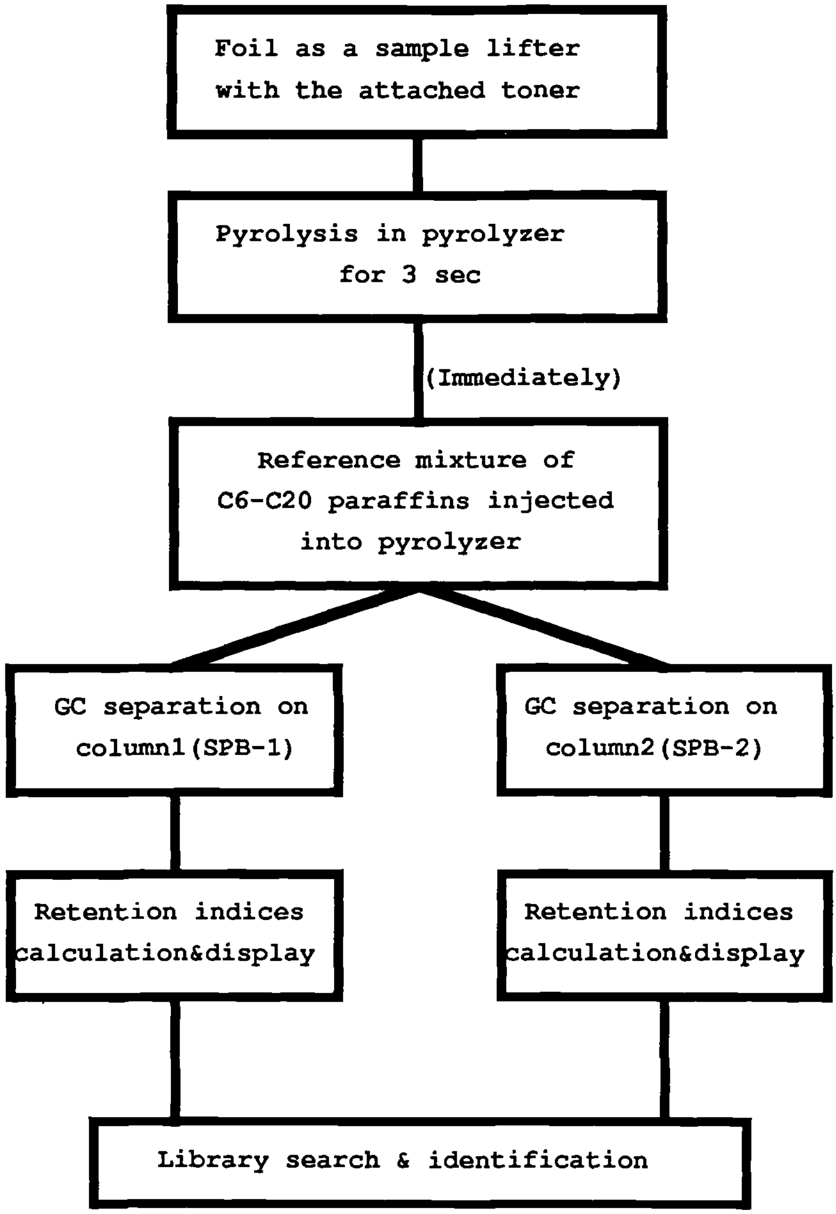

FIG. 7—*The flow chart of the Py-GC analysis.*

For the library to be efficient for searching for possible sources of a questioned photocopy, many more standard toners including those used for laser printers would need to be analysed. In addition, the data would be more realistic if they could be obtained with the above described newer Py-GC device. As academic researchers, however, we are not inclined to start over again or proceed further for the time being. The primary objective of this paper is simply to introduce the novel methodology itself. Those real casework examiners who are interested in this paper may wish to compile their own data library with their own equipments according to the above described procedure. Also, the manner in which the paraffins standard is introduced to the pyrolyser may need to be modified if an automatic sampler is used.

Acknowledgment

The authors wish to thank Mr. Hong-Chen Chou for his assistance at writing the dBase III Plus program, which made the successful library search possible. A financial support under the grant No. NSC-81-0208-M-015-01 from the National Science Council of the Republic of China is also gratefully acknowledged.

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zinc are found together in semen from a number of mammals. The zinc test strips do have the advantage of being cheaper and easier to use than the immunoassays for p30.

Although for several centuries universities have made a fetish of the learned thesis as the *ne plus ultra* of scholarship, it has been clear for some generations that these in themselves make little contribution to fields of knowledge. Scholarly discourse now commonly employs the agency of the journal article. Only rarely is a thesis of such earthshaking import as to be worthy of publication and widespread dissemination. The only example of such a graduate thesis that comes readily to mind is that of Louis de Broglie which laid out the foundations of wave mechanics. Regrettably, the work under review is not of this caliber. Although it was presented as a thesis for a doctoral degree, this is a rather short work (only 133 pages with 203 references) written on the level of a masters dissertation. The quantity and quality of research it represents would also be more appropriate for a masters degree, rather than a doctorate. The author's research would have been better presented as an article in a scientific journal such as the *Journal of Forensic Sciences.*

Although in English, this work was clearly not written by a fluent speaker. It abounds in ungrammatical constructions, incorrect word usages and annoying misspellings. It should have been submitted to the attentions of a competent editor before it was published. Taken as a whole, I can only recommend this work to the zinc-in-seminal-plasma aficionados who simply *must* have every work on this subject. To the average forensic laboratory worker for whom the zinc spot test is merely one in a battery of such tests, it does not offer enough to justify its price.

Erratum

In the July 1993 issue, a misprint occurred. Figure 2 of the paper by Wei-Tun Chang, Chin-Wang Huang, and Yun-Seng Giang should appear as follows.

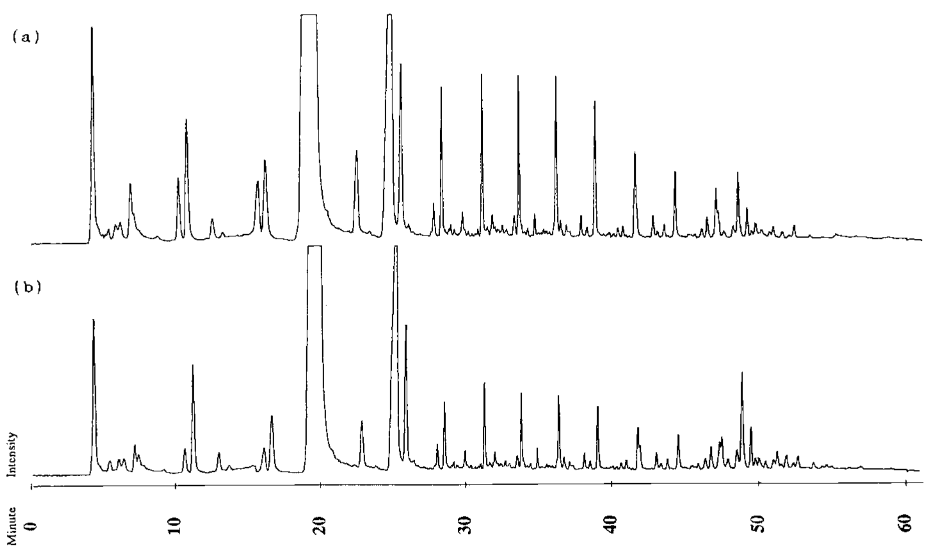

FIG. 2—*The pyrogram pattern (shown for SPB-20 column only) from a Xerox 3990 photocopier raw toner powder sample,* (a), *is nearly the same as that from a corresponding fused toner sample,* (b).