

## TECHNICAL NOTE

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# Classification of Black Powder Toners on the Basis of Integrated Analytical Information Provided by Fourier Transform Infrared Spectrometry and X-Ray Fluorescence Spectrometry

**ABSTRACT:** Differentiating between black powder toners used in laser printers and copiers can be challenging for forensic examiners. One hundred and sixty-two samples from 82 different types of cartridges produced by 21 manufacturers that are currently available on the Polish market were studied using Fourier transform infrared spectrometry (FT-IR) and energy-dispersive X-ray fluorescence spectrometry (XRF). The spectra obtained by these two methods were classified into 14 groups and 28 groups using FT-IR and XRF, respectively. These classifications served as a basis for the development of an analytical scheme for differentiating black powder toners. This scheme can be used with any two methods that supply different information about an examined sample. The research will show that if two samples are similar in polymer composition (e.g., FT-IR spectra), additional quantitative elemental composition from XRF analyses may provide more discrimination. It was possible to differentiate 82.5% pairs of examined samples using only FT-IR, 90.8% pairs of examined samples using only XRF, and 95.8% pairs of examined samples using these two methods. Obtained spectra and all the available information could be used to create/build a database. The results obtained confirm the multiplicity and compatibility of toners. Additionally, it was stated that not all the samples were original (OEM).

**KEYWORDS:** forensic sciences, questioned documents, toner, electrophotography, differentiation, FT-IR, XRF

Determination of authenticity concerning many types of documents can give rise to many different problems. One of them is characterizing some of the materials used to produce the document such as the ink and/or toner.

In the three decades, a significant increase in the number of documents prepared with the use of printers, copiers, and multi-function systems has been noted (1). Widespread access and ease of use of these systems make it simple to create fraudulent documents using a toner (e.g., dry or liquid) and/or ink. As with most materials associated with criminal cases, it may be necessary to collect various analytical and product data, which may help the investigative process.

Verification of authenticity may involve establishing how a questioned document was produced and/or determining the brand of printer or copier. Establishing how a document was produced can be mainly done microscopically (2). Determination of the brand of printer or copier may involve a chemical analysis of the toner present on the document (3).

The current study focused on raw powder toners. Powder toners are mixtures of several types of chemical compounds such as a colorant(s) (e.g., a dye or a pigment), a binding agent(s), and additives. The binding agent allows for permanent contact with the

paper. The additives ensure/improve other physical and chemical properties.

A typical toner should have the following properties: a suitable mass, an electric charge, and magnetic quality. Black toners contain, mostly, soot as the pigment. Also, small quantities of blue-violet organic dyes, e.g. compounds containing cyan ( $\text{CN}^-$ ) groups (see Fig. 1), may be present. The binding material is mainly synthetic acryl, polyester, polyvinyl, and epoxy resin co-polymers. The most frequently applied compounds with magnetic properties are magnetite ( $\text{Fe}_3\text{O}_4$ ) and ferrites ( $\text{MeFeO}_4$ ,  $\text{Me} = \text{Fe}, \text{Co}, \text{Mn}, \text{Ni}, \text{Cu}, \text{Mg}$ ). Fused silica is often used as an additive. All information about chemical composition of toner is protected by patent law.

Various analytical methods can be used for the analysis of toners, such as: thin-layer chromatography (TLC) (4,5), infrared spectrometry (IR)—mainly Fourier transform infrared spectrometry (FT-IR) (6–9), calorimetry, and combined methods, e.g., pyrolysis gas chromatography/mass spectrometry (Py-GC/MS) (10–14), scanning electron microscopy coupled with energy-dispersive X-ray spectrometry (SEM-EDX) (15,16).

Qualitative analysis of IR spectra of toners has been presented by Brandi et al. (17), Mazzela and colleagues (18,19), and Tandon et al. (20). Results concerning examination of toners by several methods such as DRIFTS, SEM-EDX, and Py-MS (16) are described and analyzed separately. However, the authors did not address further discriminatory techniques. Utilizing two mutually exclusive techniques that provide different information about an

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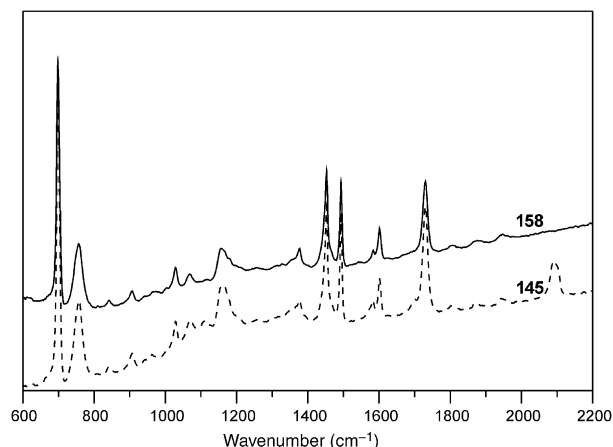


FIG. 1—Infrared spectra of samples 145 and 158.

examined sample may be warranted to help eliminate the possibility of determining whether two samples are the same when they are, in fact, chemically different.

This paper proposes a method of combining two separate analytical methods. This approach is illustrated using information about the polymer composition from FT-IR and the elemental composition of toner from X-ray fluorescence spectrometry (XRF). It was established that the application of both methods enables one to distinguish a greater number of pairs of samples as opposed to using each method separately.

### Material and Methods

A total of 162 samples of black toners originating directly from 82 different types of cartridges were removed from printers and copiers produced by 21 firms. The toners that were analyzed are commonly sold on the Polish market. Data concerning the make and model of the machines, or the type of toner and the corresponding number of samples are listed in Table 1. Catalogues providing data about the mutual compatibility of machines and types of toner in use were also used for compiling information (22,23).

The toners were examined with the use of IR microspectrometry (MK-FT-IR). Measurements were performed in the transmission mode using an FTS 40A spectrometer coupled with a UMA 500, Bio-Rad/Digilab microscope. Spectra were obtained at  $4\text{ cm}^{-1}$  resolution with 128 scans in the range of  $600\text{--}4000\text{ cm}^{-1}$ . Toner particles were removed from cartridges or scratched from paper with a scalpel under an optical microscope to prevent contamination. They were placed onto a KBr window and pressed in order to obtain a suitable thickness. Each spectrum was measured three times and normalized. Two samples were determined to be different if the presence (or absence) of an additional band in the spectrum was detected, or when the difference in relative intensities of spectra was considerable (different quantities of components). It is shown in Fig. 2.

Elemental composition was determined using energy-dispersive XRF. Measurements were performed with an ED 2000 spectrometer by Oxford Instruments Ltd. equipped with a silver cathode. Helium was used as an inert gas. Samples of toner powders taken from cartridges were placed in suitable cuvettes. A quantitative analysis was performed using XpertEase™ software in general conditions (high voltage 25 kV) mode. Each series of measurements were followed by a calibration using a multielemental

TABLE 1—List of model makes and the number of examined samples.

No.	Make	Model or Type of Toner and Sample Number (in Parentheses)	Total Number of Samples
1	Agfa	× 35 (1)	1
2	Brother	TN200 (2–4)	3
3	Canon	A30(5), E30(6–9), NPG 1 (10–20), NPG10 (21), NPG11 (22)	18
4	Develop	1212 (23)	1
5	Epson	EPL 5500(24), 5700(25)	2
6	Hewlett Packard	92274A(26, 27), 92275A(28), 92295A(29), 92298A(30), C3903A(31–36), C3906A(37–40), C4092A(41–46), C4096A(47, 48), C4127X(49, 50), C4129X(51, 52)	27
7	Infotec	887523 (53)	1
8	Konica	947136(54), 947376(55)	2
9	Lanier	1170114(56, 57)	2
10	Lexmark	13T0101(58, 59), 69G8256(60, 61)	4
11	Minolta	Di30 (62), 101B(82, 83), 102B(84), 103B(85), 302B(64), 401B(86–88), 601B(89–91), 4152-303(65–73, 92), 8916102(74), 8931102(75), 8932202(76–78), 8931810(79, 80), 8932004(63)	31
12	Mita	3704601(93), 3702800(94, 95), 37090008(96)	4
13	Nashuatec	3135(1), CT105(98), CT110(99)	3
14	Oce	337050 (100)	1
15	Okidata	09002390(101, 102)	2
16	Panasonic	FQTS15(103, 104), KXP458(105)	3
17	Ricoh	885190(106), 887132(107–109), 887523(110), 887573(111), 887622(112), 887642(113–118), 887659(119), 889490(120), 889857(121)	16
18	Sharp	ZT20TD1(122), SF770AT1(123), SF230ST1(124), SF216LT1(125), SF780CT1(126), SF222LT1(127), SF235LT1(128), AL100TD(129), AR150TD(130)	9
19	Tally	44898(131, 132)	2
20	Toshiba	T1350E(133–138), T1550(139), T2060(140–143), T4550(144, 145), T6550(146)	14
21	Xerox	1R00081(147), 6R90099(148–153), 6R09070(154), 6R90168(155, 156), 6R90170(157), 6R90203(158), 6R90269(159), 6R90270(160, 161), 6R916(162)	16

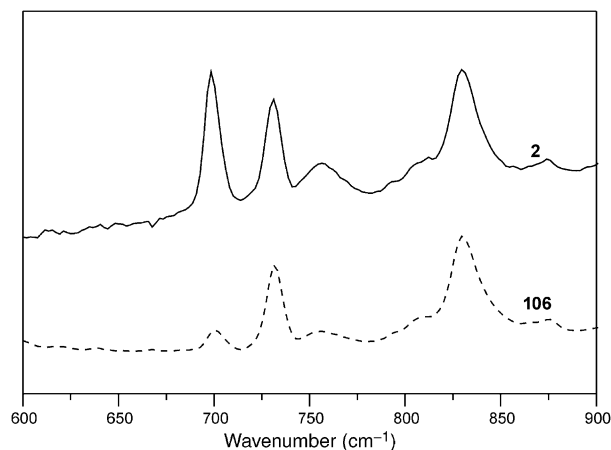


FIG. 2—Differences in component quantity—infrared spectra of samples 2 and 106.

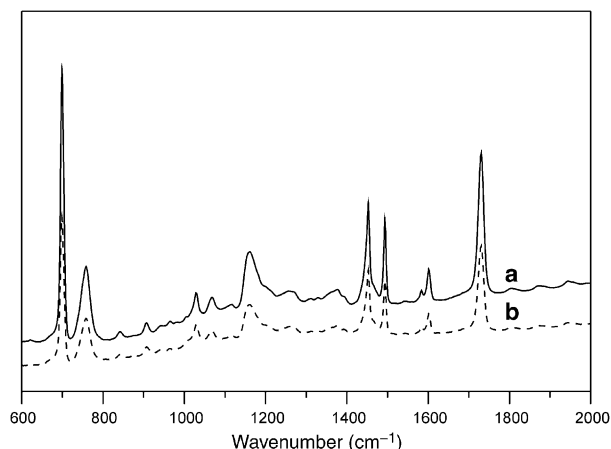


FIG. 3—Infrared spectra of sample 126 from cartridge (a) and paper (b).

standard provided by the manufacturer of the XRF spectrometer; however, no calibration curve was necessary. Measurements for each of the examined toners were performed three times, and standard deviations ( $\sigma$ ) were calculated. Two samples were found to be different, when the ranges of the relative content for at least one element were  $\bar{x} - 3\sigma$ ;  $\bar{x} + 3\sigma$ .

If possible, a printed document was obtained and toner particles from the paper were also examined using FT-IR and by SEM-EDX. Toner particles do not penetrate into the structure of paper, and can be easily separated from its surface without any damage (10,13). It was observed that paper components did not influence the IR spectra (see Fig. 3). Also, no differences were detected in the elemental composition of toner samples from paper and from cartridge.

## Results and Discussion

When analyzing IR spectra of toners, the criterion of division was the presence of characteristic bands. Too little information was available to interpret origin of all the bands. Sometimes, the characteristic bands originated from polymer. These bands are as follows: bands originating from styrene are labeled with the symbol S, while esters of the acrylate or methacrylate type are marked as A and M, respectively. However, in some cases the origin of characteristic bands was not successfully determined. These bands are not labeled. Based on these criteria, the IR spectra were divided into 14 groups, which are presented in Table 2. For four of them (III, IV, VII, VIII), the discriminating feature within the group is the inclination of the base line in the range of 600–800  $\text{cm}^{-1}$ , which can be attributed, to the presence of  $\text{TiO}_2$  (see Fig. 4). It could not be excluded that the bands (730 and 1233  $\text{cm}^{-1}$ ) present in spectra belonging to group I originate from derivatives of isophthalic acid. Their respective positions are the same as characteristic bands for alkyd resins of the isophthalate type. However, unequivocal determination of the kinds of aromatic compounds responsible for the presence these bands was not achieved. The bands at 830 and 1510  $\text{cm}^{-1}$  present in toners spectra of groups I, II, and XII–XIV are due to the presence of Bisphenol A. The same bands can be observed in the spectrum of epoxy resins produced on the bases of this compound. Bands at 730 and 1271  $\text{cm}^{-1}$  present in the spectra of samples belonging to groups II, XII–XIV are characteristic of paint resins such as terphthalate. Bands at 700 and 760  $\text{cm}^{-1}$  present in the spectra of samples belonging to group XIV reveal the same position as bands

TABLE 2—Classification scheme for IR spectra according to characteristic peak positions and the inclination of the baseline.

Group Number	Number of Samples	Samples	Position of Characteristic Peaks ( $\text{cm}^{-1}$ )	Inclination of the Baseline (600–800 $\text{cm}^{-1}$ )
I	10	65–73, 92	730, 1233 830, 1510	Horizontal
II	3	24, 54, 105	730, 1271 830, 1510	Horizontal
III	59	1, 8, 9, 11–14–20, 23, 26, 27, 28, 30, 34, 35, 37–39, 41, 42, 43–49, 51, 52, 55, 58, 59, 78, 80, 81, 82, 84–86, 96, 101–104, 115, 117, 119, 124, 126, 128, 131, 155, 156, 158	S (700, 760), A (1156–1164)	Horizontal or declining
IV	22	6, 60, 83, 63, 74, 79, 89, 91, 112, 125, 130, 133–135, 136, 139, 140–144, 162	S (700, 760), A (1156, 1180, 1200)	Horizontal or declining
V	5	75–77, 94, 95	S (700, 760), A (1156–1164, 1200)	Horizontal
VI	4	7, 10, 21, 22	S (700, 760), A (1156–1164, 1260)	Declining
VII	18	5, 25, 40, 50, 61, 87, 88, 90, 97, 108, 118, 122, 123, 127, 129, 132, 148, 157	S (700, 760), A (1156–1164, 1270)	Horizontal or declining
VIII	6	53, 64, 107, 109, 111, 151	S (700, 760), A (1156, 1180, 1200, 1270)	Horizontal or declining
IX	10	93, 147, 149, 150, 152, 153, 154, 159–161	S (700, 760), M (1156, 1179)	Declining
X	5	29, 31–33, 36	S (700, 760), A (1156–1164); 1780	Declining
XI	6	56, 57, 137, 138, 145, 146	S (700, 760), A (1156–1164, 1260); CN <sup>−</sup> (2092)	Horizontal
XII	9	2–4, 62, 106, 113, 114, 116, 120	S (700, 760), 730, 1271 830, 1510; 1157	Horizontal
XIII	4	98, 99, 110, 121	S (700, 760) 730, 1271 830, 1510; 1162	Horizontal
XIV	1	100	1 (700, 760), 2 (730, 1271 830, 1510;1488)	Horizontal

IR, Infrared; CN<sup>−</sup>, cyan group.

originating from styrene (S); however, in these spectra, the intensity of the band 700  $\text{cm}^{-1}$  is weaker than the intensity of the band 760  $\text{cm}^{-1}$ , unlike in the styrene spectrum (see Fig. 5a and b). The presence of bands 1488 and 1780  $\text{cm}^{-1}$  (groups XIV and X) could not be unequivocally assigned to specific compounds or defined types of bonds.

The classifications performed can serve to discriminate between samples and to classify the type of machine equipped with a given type of toner. As a rule, samples of toners originating from different individual machines of the same model can be different. If as a result of the performed research, samples are ascertained to be

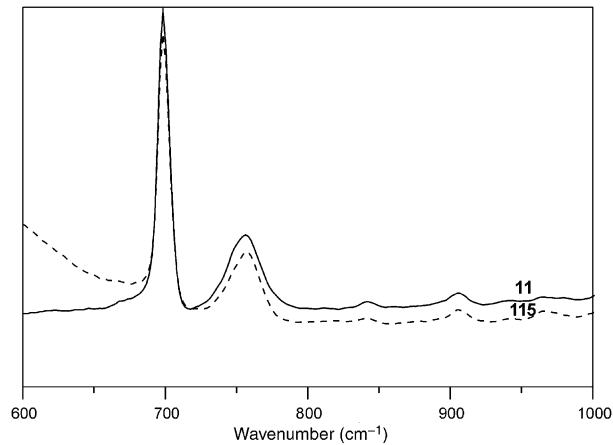


FIG. 4—Infrared spectra of samples 11 and 115 from group III.

indistinguishable (knowing that they originate from different machines), this could result from the compatibility of entire devices or cartridges. Such a possibility should be borne in mind, when, on the basis of analysis of chemical composition, one intends to define the model of a machine working with a given type of toner. The opposite situation is also possible: samples of toners originating from the same machine, but succeeding cartridges, differ from each other. This may result from the fact that some models

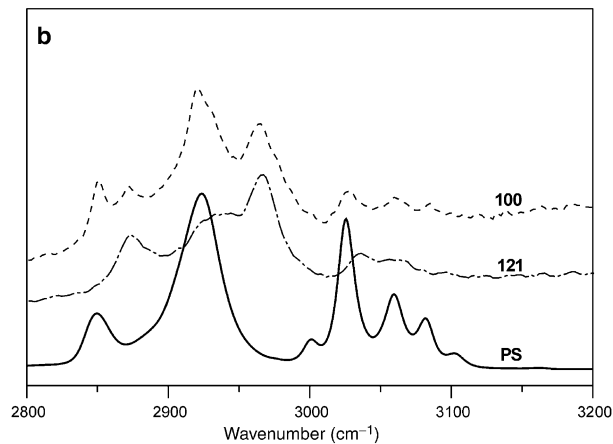
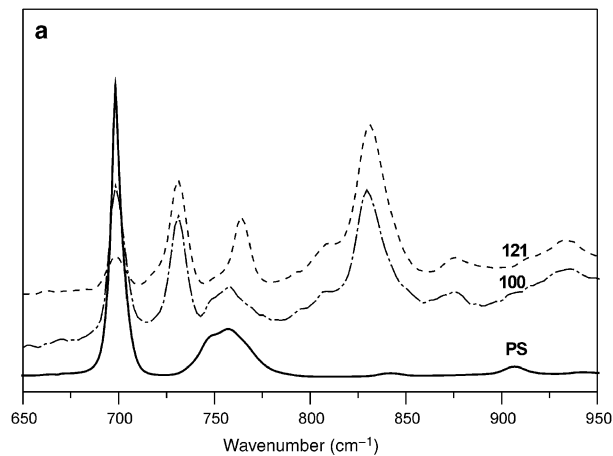


FIG. 5—(a) Infrared (IR) spectra of polystyrene (PS), sample 100 (XIV group), and sample 121 (XII group) in the range 650–950 cm<sup>-1</sup>. (b) IR spectra of PS, sample 100 (XIV group), and sample 121 (XII group) in the range 2800–3200 cm<sup>-1</sup>.

TABLE 3—Classification scheme for XRF spectra according to elemental content.

Group Number	Number of Samples	Sample Number	Dominant Element and its Relative Content	Differentiating Element(s) and its Relative Content
I	5	135, 138, 144–146	Fe > 50%	Cu, Zn 5–50%
II	4	74, 80, 86, 124		Zn
III	6	25, 56, 57, 61, 97, 112		Cr
IV	16	53, 63, 77, 78, 84, 85, 94, 101, 102, 127, 136, 139, 147, 150, 153, 154		S
V	1	159		Ca
VI	1	96		Ti
VII	1	125		Mn
VIII	3	22, 48, 62		Sr
IX	1	137		Mo
X	6	7, 21, 31–33, 41		Sr, Ti < 5%
XI	1	95		Br, Ti
XII	6	10–12, 14–16		Mn
XIII	5	29, 128, 130, 162, 148		Cr
XIV	9	88, 89, 91, 103, 104, 123, 126, 151, 157		S
XV	2	129, 132		Ca
XVI	39	5, 6, 8, 9, 13, 17–20, 26–28, 30, 34, 35, 36–40, 42, 43–47, 49, 50–52, 59, 64, 87, 90, 119, 122, 131, 155, 156,	Fe > 90%	Many others < 1%
XVII	1	60	Zn > 50%	Many others < 50%
XVIII	3	54, 55, 158	Ti	
XIX	2	114, 120	Ca	S > 20%
XX	5	23, 83, 133, 134, 142	S	Fe > 15%
XXI	12	67, 68, 70–73, 75, 92, 106, 107, 109, 111	Cr	Many others < 50%
XXII	1	81	Cu	
XXIII	14	2–4, 98, 99, 117, 118, 121, 140, 141, 143, 149, 160, 161	S 25–50%	Many others < 50%
XXIV	5	24, 66, 69, 93, 105	Cr	
XXV	2	58, 65	Cl	
XXVI	7	1, 76, 79, 82, 110, 116, 152	Fe	
XXVII	1	113	Ca	
XXVIII	3	100, 108, 115	Zn	

XRF, energy-dispersive X-ray fluorescence.

may work using the toner recommended by the producer (OEM), as well as an alternative/universal toner (Table 3).

The analytical procedure of discriminating between two samples of toner is presented in Fig. 6. It is recommended that the comparison should begin with examination by means of IR spectrometry because it is an effective and efficient method to begin classifying samples based on polymer composition. If two samples belong to different groups, there is no need to carry out further examinations. If the two samples are indistinguishable, they can be examined using an alternate method such as XRF, ICP-MS, and SEM-EDX. The author chose to utilize XRF to further discriminate toners. If toners cannot be discerned only by visual

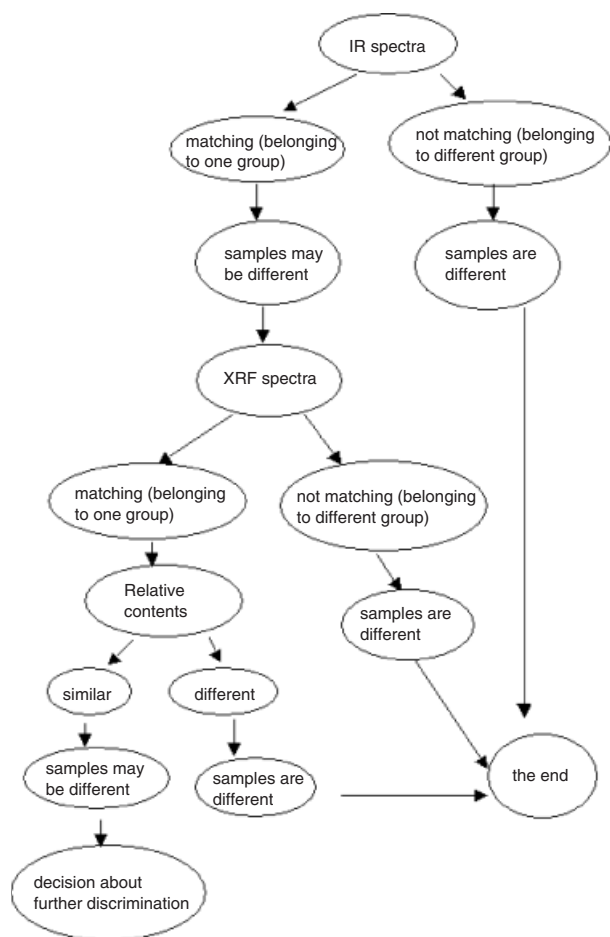


FIG. 6—A flow chart for differentiation of two toner samples.

comparison of XRF spectra, quantitative data concerning elemental composition should be taken into account.

The next stage may require for another analytical method. After completing the procedure, the examiner will have to determine whether the samples are distinguishable.

The usefulness of the proposed scheme for the discrimination of the examined samples is illustrated using the following examples. If two samples could not be differentiated on the basis of IR spectra, data from XRF measurement allow, in many cases, to individualize them. For example, pairs created by samples of the following toners: Brother HL 760 (two samples), HPLJ C3903A (three samples), Minolta 4152-303 (two samples) and 601B (two samples), Oki 52106201 (two samples), Ricoh 887132 (two samples), Xerox 6R90270 (two samples), and 6R90099 (two samples), could not be differentiated on the basis of group membership. Appropriate data are presented in Table 4.

By analyzing numerical data contained in this table, one can ascertain that the qualitative and quantitative composition of toner samples found in the same type of cartridge (e.g. Brother TN200; Ricoh 887622 and Minolta 4152-303) differs considerably. It means that one cartridge was an original (OEM) and the second was refilled. Differences in element contents between two toners arising from the same type of cartridges could also point to batch-to-batch differences.

The 162 samples were grouped into 13,041 pairs. Using FT-IR, 10,755 pairs (82.5%) were differentiated. Among undifferentiated 2286 pairs (17.5%), 1735 (13.3%) were subsequently differentiated by XRF. So, applying the proposed scheme (e.g., the combination of FT-IR and XRF), 12,490 pairs (95.8%) were differentiated. Utilizing only XRF, 11,863 pairs (90.8%) were differentiated. As an example, undifferentiated samples No: 24 and 54 belonging to II IR group (toners used in Epson, Konica models, respectively) are differentiated by the XRF method. These samples belong to various XRF groups (XVIII and XXIV, respectively). In another case, the IR method turned out to be insufficient to differentiate between toner samples 10, 21, and 22 used in various Canon models. Data relating to elemental content allow to differentiate these three samples.

## Conclusions

The following is a summary of the results:

1. There exist differences, both in the polymer composition and in the elemental composition. One hundred and sixty-two sam-

TABLE 4—Elemental composition of some toner samples.

Toner	Sample	S	Fe	Ca	Si	Cu	Zn	Cl	Cr	Group Number	
										XRF	IR
Brother TN-200	2	33.3 ± 1.1	17.9 ± 2	11 ± 0.7	27.5 ± 1.2	4.7 ± 1.4	—	—	—	XXIII	XII
	3	25.1 ± 1.1	17.6 ± 0.7	20.4 ± 1.3	23.3 ± 0.3	4.2 ± 1.1	4.1 ± 2.6	—	—		
Minolta 4152-303	71	7.5 ± 0.3	1.6 ± 0.5	5.0 ± 0.3	4.9 ± 0.2	—	—	27.9 ± 0.5	52.0 ± 0.5	XXI	I
	92	4.9 ± 0.3	5.2 ± 1.1	14.5 ± 0.6	3.4 ± 0.1	—	—	19.8 ± 0.3	51.7 ± 0.7		
Minolta 601B	89	2.1 ± 0.1	94.6 ± 1.0	—	0.9 ± 0.1	1.0 ± 0.2	0.8 ± 0.6	—	—	XIV	IV
	91	2.1 ± 0.1	94.9 ± 0.3	—	0.8 ± 0.1	—	1.3 ± 0.4	—	—		
Oki 52106201	101	13.5 ± 0.2	52.5 ± 0.2	10.7 ± 0.5	4.0 ± 0	—	6.1 ± 0.2	1.4 ± 0.2	11.0 ± 0.2	IV	III
	2	13.3 ± 0.6	55.1 ± 0.3	8.8 ± 0.6	3.7 ± 0.2	—	4.7 ± 0.8	1.8 ± 0.5	11.8 ± 0.2		
Ricoh 887132	107	12.2 ± 0.2	17.1 ± 1.0	5.8 ± 0.2	1.4 ± 0.1	1.8 ± 0.6	3.2 ± 0.8	—	53.6 ± 0.3	XXI	VIII
	109	14.5 ± 1.0	5.3 ± 0.5	1.2 ± 0.5	1.6 ± 0.1	2.4 ± 1.2	—	—	75.0 ± 1.6		
Xerox 6R90099	150	11.9 ± 0.6	73.1 ± 1.5	4.3 ± 0.2	5.7 ± 0.3	2.3 ± 0.8	0.6 ± 0.6	2.1 ± 0.3	—	IV	IX
	153	8.0 ± 0.3	76.8 ± 0.8	3.6 ± 0.4	4.2 ± 0.2	2.0 ± 0.5	0.7 ± 0.6	1.1 ± 0.2	—		
Xerox 6R90270	160	31.3 ± 1.3	18.9 ± 0.3	20.1 ± 0.3	14.9 ± 0.4	4.3 ± 2.7	1.6 ± 1.2	4.5 ± 0.7	—	XXIII	IX
	161	45 ± 1.6	8.2 ± 0.9	17.0 ± 1.1	19.6 ± 1.2	2.2 ± 1.6	1.1 ± 1.1	7 ± 0.8	—		
HPLJ C3903A		Fe	Sr	Mn	Ca	Cu	Zn	Ti			
	31	90.6 ± 0.1	3.1 ± 0.1	2.0 ± 0.1	0.2 ± 0.1	0.3 ± 0.1	0.4 ± 0.1	2.9 ± 0.6	—	X	X
	32	93.9 ± 0.1	1.3 ± 0.1	2.2 ± 0.1	0.1 ± 0.1	0.3 ± 0.1	0.4 ± 0.1	1.3 ± 0.1	—		
	33	92.1 ± 0.1	2.0 ± 0.1	2.2 ± 0.1	0.5 ± 0.1	0.3 ± 0.1	0.4 ± 0.1	2.0 ± 0.1	—		

XRF, energy-dispersive X-ray fluorescence; IR, infrared.

- ples of toners were divided into 14 and 28 groups based on their FT-IR and XRF spectra, respectively.
2. Iron is the dominant element for 106 (65%) samples (groups I–XVI). As many as 39 samples (group XVI) have an iron content higher than 90%. Sulfur is also present in all samples, but is dominant or differentiating for 35 (22%) samples. For 21 (14%) samples (groups III, XXI, and XXIV), chrome is the dominant or differentiating element.
  3. The most frequent binding agents in toners are copolymers produced from styrene and acrylic ester(s). As much as 125 (77%) samples contain an acrylic-styrene copolymer (groups III, IV, and VII) and 10 (16.2%) samples contain methacrylate-styrene esters (group IX). Thirteen samples (8%) contain alkyd and epoxy resin (groups I and II).
  4. Toners for different devices may differ not only in binder and elemental composition, but sometimes toners for different devices can be of the same type (compatible).
  5. Sometimes, it is possible to use a refilled cartridge instead of OEM. In this situation, the created document could have some flaws.
  6. The analytical scheme presented for raw toner from cartridges can be applied for toner removed from questioned documents. In the present work, no significant differences in chemical composition were found in toners removed from paper and toners taken from a cartridge. However, the possibility that toner particles could be contaminated by paper components cannot be excluded. Therefore, it is recommended that examiners conduct examinations on a paper standard.
  7. The use of two methods (FT-IR and XRF) allowed for further differentiation of toners.
  8. Determining the chemical composition of a toner may help in identifying the make or model of a machine; however, one must consider the possibility of using refill toners and the cross-compatibility of various devices and toners.
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