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The Comparison of Black Polyvinylchloride (PVC) Tapes by Pyrolysis Gas Chromatography

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ABSTRACT: Pyrolysis capillary gas chromatography (Py-GC) was used to compare thirty black polyvinylchloride (PVC) tapes. A 0.5-mm square of tape (about 25 μg) was found to be an adequate sample size. Tests of within-roll homogeneity showed sufficiently consistent pyrogram patterns throughout to allow correct matches to be made. Twenty-six of the tapes gave pyrograms which were easily distinguishable from all the others. Fragments of tape recovered after the detonation of four improvised explosive devises were matched to the appropriate tape standards by Py-GC even when the fragments were partially burned.

KEYWORDS: criminalistics, adhesive tapes, polyvinylchloride, chromatographic analysis, pyrolysis gas chromatography

Adhesive tapes are frequently submitted as evidence to the FBI Laboratory. Black polyvinylchloride (PVC) tape is seen most frequently with duct tape and surgical tape second and third, respectively. The black PVC tape is frequently associated with improvised explosive devices (IEDs). The IEDs are often electronically initiated, and the PVC tape is used for insulating, sealing, or securing something within the devices. When a piece of tape is submitted as evidence, the forensic science examiner is often asked to determine whether the tape has originated from a roll of tape found in the possession of a suspect. On occasion, the cut ends will match, providing a positive conclusion that the tape sample came from that roll of tape. If this match cannot be made (possibly as a result of a length of missing tape), sometimes tool marks on the cut ends of the tape (both sample and roll) with match, once again strongly associating the tape sample with the roll of tape and the suspect.

If these types of positive matches do not exist, however, one must relate the tapes to each other using class characteristics of the samples. A very useful method is to make optical comparisons of physical properties such as width, thickness, degree of glossiness, tape edge, and surface characteristics. The surface finish of black PVC tape can vary widely from tape to tape. In addition, the rollers used in the manufacturing of the backing can leave characteristic marks. Many of these physical characteristics of the tape can become sufficiently distorted before the examination that a meaningful comparison is not possible.

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At a recent symposium, a report was presented [*1*] which contained an excellent discussion of various aspects of the manufacture of PVC adhesive tape and methods and measurements which may characterize individual tapes. The use of X-ray fluorescence spectrometry (XRF) and infrared spectroscopy (IR) as instrumental means for tape comparisons was discussed. At this same symposium a second report [*2*] was presented which concluded that black PVC tapes could be distinguished by using stereoscopic examination of surface characteristics in combination with XRF and IR.

The characterization of adhesives by pyrolysis gas chromatography is a well-established technique [*3,4*]. This report presents the results of a study to estimate the degree to which black PVC tapes can be individualized by pyrolysis gas chromatography. The work presented here was performed as a summer intern project at the FBI Forensic Science Research and Training Center in 1984.

Materials and Methods

The tape samples were compared by capillary pyrolysis gas chromatography using a CDS 120 Pyroprobe unit (Chemical Data Systems, Oxford, PA) attached to a Perkin Elmer Sigma 2B capillary gas chromatograph (Perkin Elmer, Norwalk, CT) as described elsewhere [*5*]. The pyrolyzate was separated into components using a 15-m by 0.25-mm inside diameter (ID) SPB-7 bonded phase fused silica capillary column (Supelco, Bellefonte, PA). A flame ionization detector set at 300°C was used as the detector. Data were collected and stored for later recall on a Perkin Elmer 3600 data station equipped with two 64K interface buffers (Perkin Elmer, Norwalk, CT). Instrumental parameters were as shown in Table 1.

Thirty black PVC tapes were used for this study—twenty-seven from a collection in the FBI Materials Analysis Unit, and three from various local sources. Before analysis, short strips of the tape samples were stuck onto 7.5 by 2.5-cm glass microscope slides. For analysis, a 0.5-mm square was cut in the tape on the slide with a scalpel. The square was then removed with pointed forceps and placed in the quartz sample tube of the Pyroprobe for analysis. This sample size corresponded to about 25 μg. Samples of a black PVC tape were pyrolyzed at 550, 650, 750, and 850°C for 5 s, and 850°C was chosen as the most suitable for this analysis because it introduced the largest amount of sample without noticeably altering the pattern of peaks in the pyrolyzate.

Finally, four IEDs were prepared and detonated using several black PVC tapes in their construction. Two of the devices were prepared using high explosives and two with low explo-

TABLE 1—*Experimental conditions.*

Pyrolysis	
Ramp	off
Temperature	850°C
Time	5 s
Interface temperature	200°C
He flow rate	23 mL/min
Gas Chromatography	
Injector temperature	200°C
Detector temperature	300°C
He linear velocity	40 cm/s
Split ratio	20:1
Temperature profile	25°C for 0.5 min to 150°C at 9°C/min to 250°C at 25°C/min 250°C for 10 min

sives, and in all cases the size of the charge was kept fairly small to assure that fragments of the tapes would remain for examination. Each IED was covered with sandbags and detonated in an underground bunker. From the high explosive IEDs, only a few fragments of tape were recovered. To prepare a sample for analysis, the tape fragments were cleaned with a cotton tipped applicator soaked with tap water to remove dirt and explosive residues.

Results and Discussion

Of the 30 different tapes analyzed, 26 of the pyrograms had unique sets of peaks. These pyrograms were relatively easily differentiated by looking for the presence or absence of certain peaks. Figure 1 shows pyrograms for 4 of these tapes. One of the tape pairs which gave indistinguishable pyrograms was, in fact, 2 tapes from the same manufacturer which a factory representative said differed only in the thickness of the backing material and the amount of adhesive applied to the backing. One might expect these 2 tapes to be indistinguishable by Py-GC. The other indistinguishable pair consisted of tapes with different brand names.

As a test of homogeneity within a roll of tape, four samples from within a 1-cm square and five samples at 1-m intervals down the length of the roll (5.0-m roll) were analyzed. The results for all of the samples appeared to be the same—the profiles were nearly identical with a few small differences. Figure 2 shows a typical pyrogram for this tape. The eight labeled peaks were chosen randomly and were used to calculate the deviations of the samples along the length of the roll. Table 2 shows that the percent standard deviations of the relative areas for each of the eight peaks in the five samples ranged from 3.4 to 7.4%.

Two rolls of the same tape used for the tests described above were purchased at the same time from the same hardware store. The pyrogram from the second roll was very similar to those obtained from the first roll. The relative areas of Peaks 1 to 5 and 8 used in the reproducibility study shown in Table 2 fell within the one standard deviation range shown. Peaks 6 and 7 fell within three standard deviations. While one might expect that multiple rolls of the same tape would give very similar pyrograms, particularly, if they were produced within a narrow time frame, this point has yet to be studied in detail. It seems likely that the major differences observed in the pyrograms from the different tapes stem from differences in the adhesives rather than the PVC backing, but tests have not yet been performed to substantiate this claim.

The tape used for these homogeneity tests was produced by a major manufacturer of tapes and might be expected to be of a more uniform quality than other tapes on the market. In fact, during this study, more apparent variations within rolls were observed with some of the other tapes. These differences involved changes in the ratios of some of the components and occasionally the appearance or disappearance of components or both. The inhomogeneities were not a problem in characterizing the tapes in this sample set, but caution must be exercised since inhomogeneities do exist. Figure 3 shows four pyrograms from a tape which exhibited such inhomogeneities. Samples A and B were taken immediately adjacent to each other, and C and D about 0.5 and 5 cm down the roll, respectively. Although the four samples do not exactly match each other, they are easily more similar to one another than to any of the others in the sample set.

The tape fragments recovered from the exploded IEDs were stiffer than the original tapes and had sand and dirt adhering to the remnants of the adhesive. Nonetheless, pyrograms of fragments from each of the four IEDs matched the appropriate tape with only slight differences. Figure 4 shows a recovered tape fragment which was sampled for Py-GC analysis. Figure 5 shows the comparison of the pyrogram from one of the recovered tape fragments to the matching tape. Samples purposefully taken from partially burned portions of the recovered tape fragments also matched the appropriate tapes. While the results with this small sample set look promising, common sense would suggest that when working with recovered

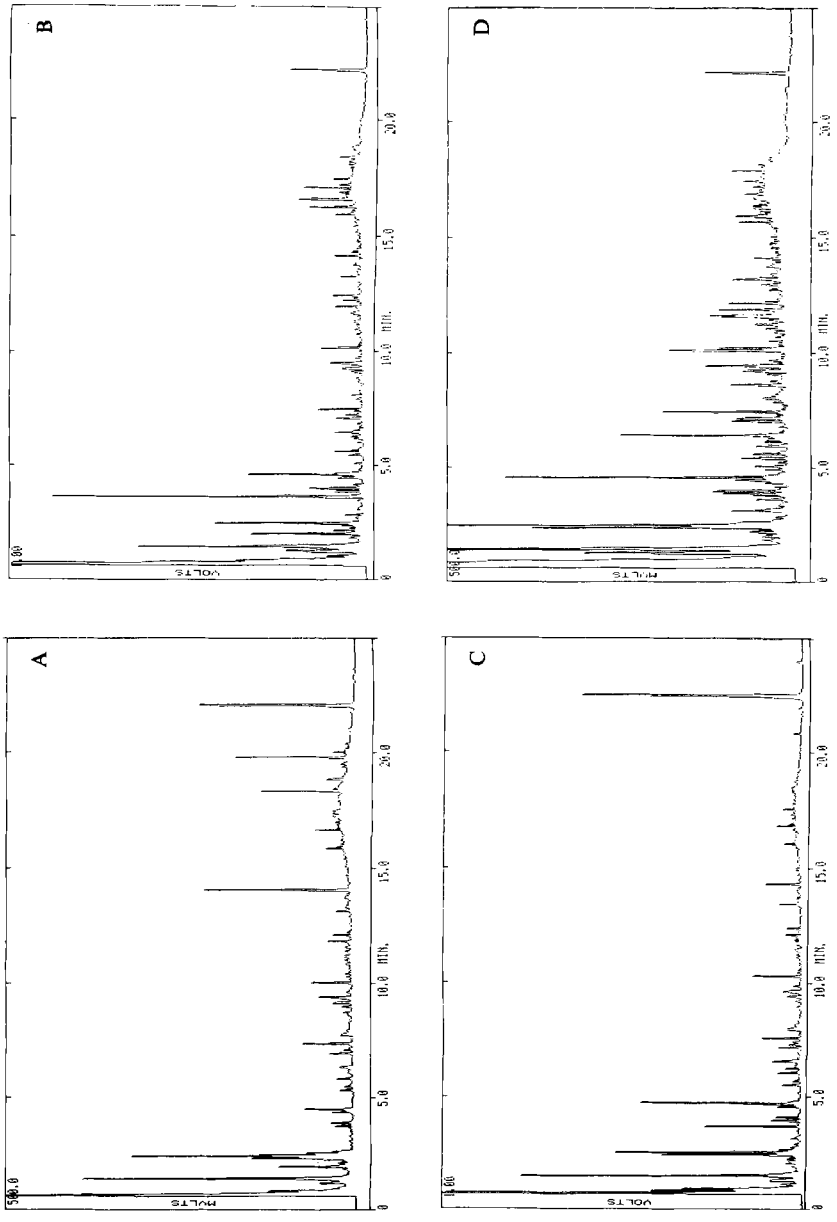

FIG. 1—*Pyrograms of four different tapes:* A, B, C, *and* D.

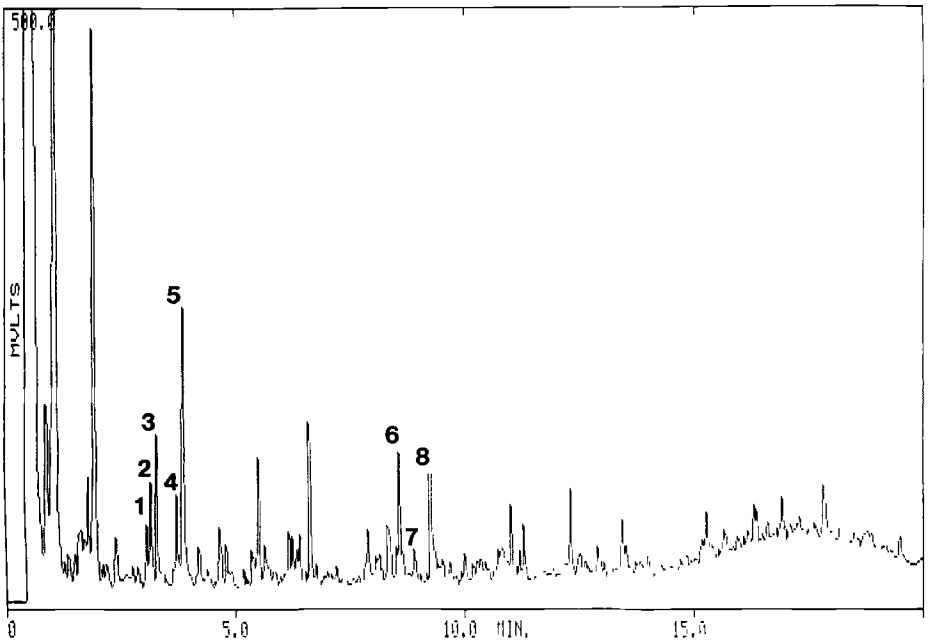

FIG. 2—*Typical pyrogram of the tape used for the homogeneity study. Areas for the numbered peaks appear in Table 2.*

tape fragments such as these, one should compare the least damaged and cleanest tape sample and interpret the results with caution.

The most conclusive results from a comparison of black PVC tapes by Py-GC would come from a comparison where the pyrograms are quite different; in this case, the tapes would almost certainly be of different origin. In the case where the pyrograms are very similar, that is, a "match," the results are said to be consistent with the tapes having a common origin. Unfortunately, this statement may encompass an entire range of occurrences. If the evidential tape matches a fairly rare type of tape (a specialty tape or one of very limited circulation)

TABLE 2—*Deviation of peaks in the pyrolyzate.*

Peak Number[a]	Mean Relative Area, %[b]	Percent Standard Deviation[c]
1	5.50	6.1
2	10.4	3.4
3	15.2	7.2
4	9.3	5.8
5	28.9	5.0
6	12.4	7.1
7	4.26	7.4
8	13.4	4.8

[a]These numbers refer to the numbered peaks in Fig. 2.

[b]The mean of the five relative area values obtained by dividing the area of that peak by the sum of the areas of all eight peaks in the pyrogram and multiplying by 100.

[c]The standard deviation of the mean divided by the mean and multiplied by 100.

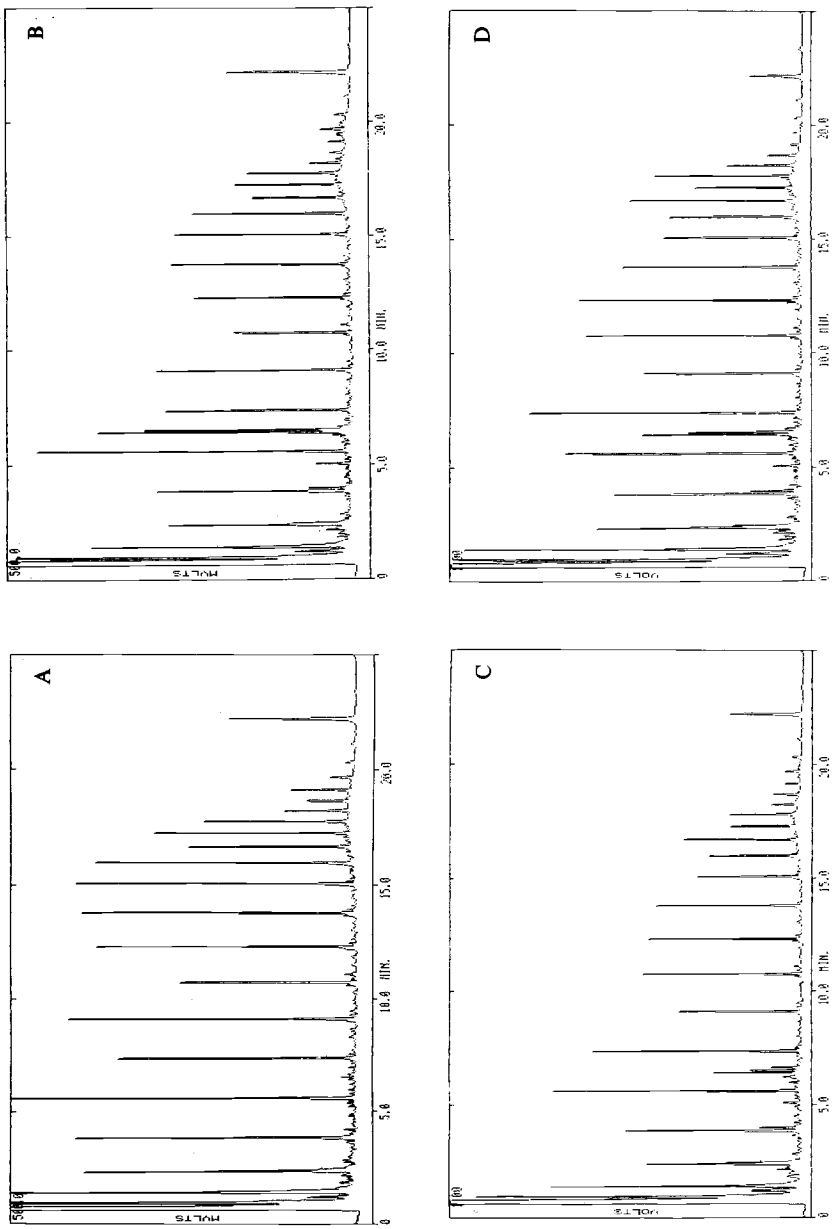

FIG. 3—*Replicate pyrograms of a tape showing some inhomogeneity.*

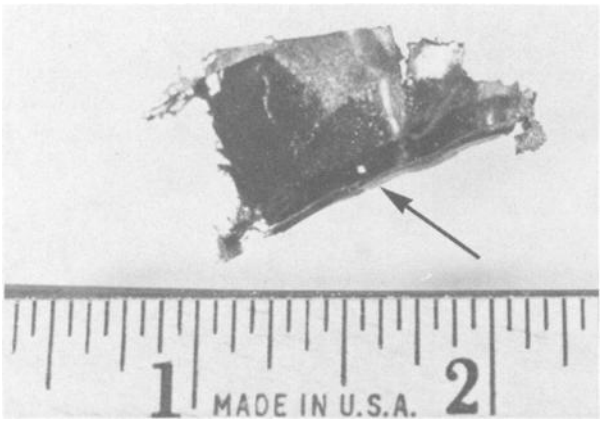

FIG. 4—*Tape fragment recovered from exploded improvised explosive device. The arrow indicates where a sample was removed for analysis.*

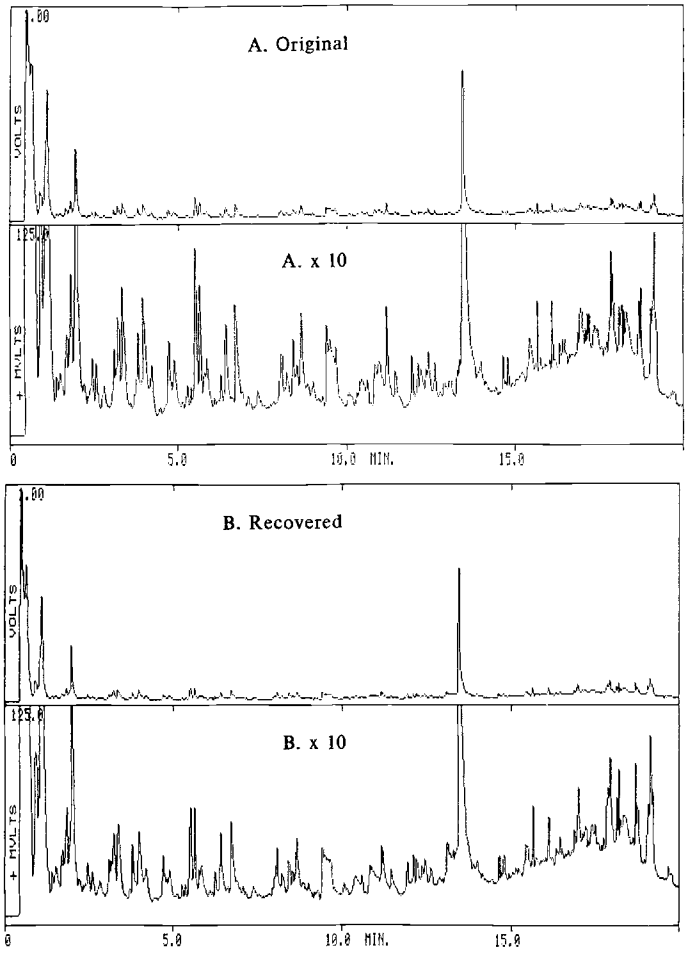

FIG. 5—*A comparison of pyrograms of a tape fragment recovered from a detonated improvised explosive device and the corresponding tape standard (the lower portion of each plot shows the vertical scale magnified tenfold to reveal the smaller peaks).*

in the possession of a suspect, that person is "linked" to the crime with a fairly high probability. On the other hand, if the match is with a brand of black PVC tape which is readily available in most communities in the United States, in the absence of other connecting evidence, a match is not very useful. Many of these rolls of tape would have given an equally good match. If there is other evidence linking the suspect to the crime, however, such a match showing that the suspect had access to the type of tape associated with the crime adds to the weight of the evidence.

Conclusions

The results of this study indicate that Py-GC may offer a high degree of discrimination between different types of black PVC tapes—26 of the 30 tapes examined gave unique pyrograms. Tape samples which have very dissimilar pyrograms almost certainly do not have common origins. Additional studies comparing multiple rolls of the same tape would be useful for estimating the evidential value of a "match."

Useful comparisons seem possible even with highly damaged fragments of tape recovered from exploded IEDs. Note that this portion of the study was performed on a limited data set and interpretation of the data should be approached with some caution.

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