FORENSIC CHARACTERIZATION OF BLACK POLYVINYL CHLORIDE ELECTRICAL TAPE

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Black polyvinyl chloride (PVC) electrical tape is commonly employed in the fabrication of improvised explosive devices as a convenient means of securing components (batteries, timer, circuitry and explosive) into one package. Usually pieces of the tape survive the explosion and can be recovered for comparison with known tape found in the possession of a suspect. In this study, black PVC tapes from six manufacturers were characterized by non-destructive techniques only. These tapes represent types of PVC tapes commercially available. Two rolls from each manufacturer were studied in an effort to determine intra-roll as well as inter-roll variability.

EXPERIMENTAL PROCEDURE

Six brands of black PVC electrical tape (3/4 inch wide by .007 inch thick) were chosen for this work: LePages, Tuck, 3M (Scotch "33"), Nashua, Vanguard and Manco. Three rolls of each brand were purchased simultaneously to ensure a high probability that at least two had come from the same batch of tape. The tapes were initially examined under a low to moderate power stereomicroscope to reveal surface features of the PVC backing (non-adhesive side). Next, non-destructive infrared spectroscopic analyses were performed on the pressure sensitive adhesive of each tape. Approximately 35-40 mm of tape was applied to one side of a 20 x 52.5 x 2 mm 45° KRS-5 internal reflectance crystal (Wilks #005-6001 or equivalent). And, finally, the elemental profiles of the tape backings were obtained with a Philips Model 9500 energy X-ray dispersive fluorescence spectrometer (Philips Electronic Instruments, Inc., Mahwah, NJ). The first revolution of tape was removed from each roll prior to analysis to eliminate any surface contamination. Ten consecutive 10-15 cm strips were cut and analyzed. The instrumental parameters are listed in Table 1. The X-ray energies used for the elemental analyses are listed in Table 2.

Table 1. CONDITIONS FOR X-RAY FLUORESCENCE ELEMENTAL ANALYSIS

Instrument:

Philips Model 9500

Tube:

Rhodium

Voltage:

25 kV

Current:

Sufficient to give ap-

proximately

counts per second

 $(50-100 \mu A)$

Filter:

None

Vacuum:

0.5 torr or better

150 seconds

Table 2. LINES AND ENERGIES FOR 10 ELEMENTS USING X-RAY FLUORESCENCE

Element	Line	KeV
Al	$K_{\alpha,\beta}$	1.49
Si	$K_{\alpha,\beta}$	1.74
S	$K_{\alpha,\beta}$	2.31
Cl	K_{α}	2.62
	K_{β}	2.82
Sb	L_{α}	3.60
	$L_{\beta 1}$	3.84
	$L_{\beta 2}$	4.10
Ca	K_{α}	3.69
	K_{β}	4.01
Ti	K_{α}	4.50
	K_{β}	4.93
Fe	K_{α}	6.40
	K_{β}	7.06
Zn	\mathbf{K}_{α}	8.63
	K_{β}	9.57
Pb	L_{α}	10.55
_ •	$L_{oldsymbol{eta}}$	12.62
	L_{γ}	14.76

RESULTS

Microscopic Examination

Each of the brands appeared to have distinct surface textures sufficiently unique to allow discrimination between manufacturers when examined with a stereomicroscope at an overall magnification of 25x to 50x. The Vanguard, Nashua, LePages and Tuck tapes (Figures 1 through 4) showed different forms of longitudinal stippling or striation that may

or may not be visible on the photographs as printed. The origin of these marks has not been established; however, it is possible that they are associated with the equipment with which the tapes were manufactured. The 3M and Manco tapes (Figures 5 and 6) did not exhibit striations of recognizable form, but did show different forms of irregular cratering.

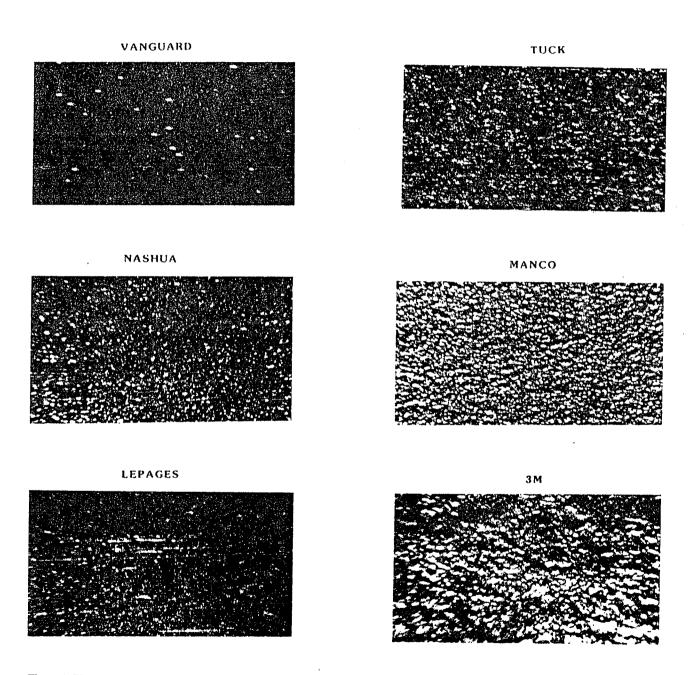


Figure 1. Photomicrographs of black polyvinyl chloride electrical tapes arranged from the smoothest surface (Vanguard) to the roughest (3M), all at the same magnification (40X).

Infrared Spectroscopy

The spectrophotometric curves obtained (Figure 2) show that each of the adhesives could be distinguished from the others. All of the spectra contain the aliphatic C-H stretching absorption band (just below 3000 cm⁻¹) and the CH₂ and CH₃ bending absorptions (1460 cm⁻¹ and 1375 cm⁻¹), indicative of long chain aliphatic hydrocarbons. All spectra also contain carbonyl absorption band (1720 cm⁻¹ to 1740 cm⁻¹) typical of aliphatic or aromatic estertype plasticizers. These two observations are consistent with ingredients known to be used in the manufacture of pressure sensitive adhesives (Satas 1982) (Table 3). No interference from PVC was observed. In the "fingerprint" regions (1700 cm⁻¹ to 500 cm⁻¹), absorption due to various adhesive substrates can be seen. A reliable identification of these substrates is difficult because of interferences from plasticizers and other compounds present. The only unencumbered bands for natural rubber are at 835 and 570 cm⁻¹; those for stryene/butadiene copolymer rubbers (SBR) are at 760 and

700 cm⁻¹; and polybutadiene rubber (PBR) bands are at 965, 910 and 775 cm⁻¹ (Haslam and Willis 1965). Based on these assignments, it may be inferred that the Vanguard and Manco adhesives contain SBR, while Nashua, LePages and Tuck adhesives contain both natural rubber and PBR. Phthalate plasticizers are indicated in the spectra of the Vanguard, Manco and Tuck tapes by the carbonyl band and the ester bands at 1280, 1125 and 1075 cm⁻¹ (Chicago Society for Paint Technology 1969). These adhesives can be distinguished from one another by the ratio of carbonyl to CH_{2,3} (1730 cm⁻¹/1460 cm⁻¹). Differences can also be observed between the Vanguard and Manco adhesives in the minor peaks at 630, 710 and 885 cm⁻¹. The Nashua, LePages and 3M spectra all contain the ester carbonyl band, but do not contain the "fingerprint" bands for phthalate plasticizers. The Nashua and LePages spectra are easily distinguishable by differences in the unassigned bands between 700 and 760 cm⁻¹. The fingerprint region of the 3M spectrum is unlike any of the other five spectra and has not been further elucidated.

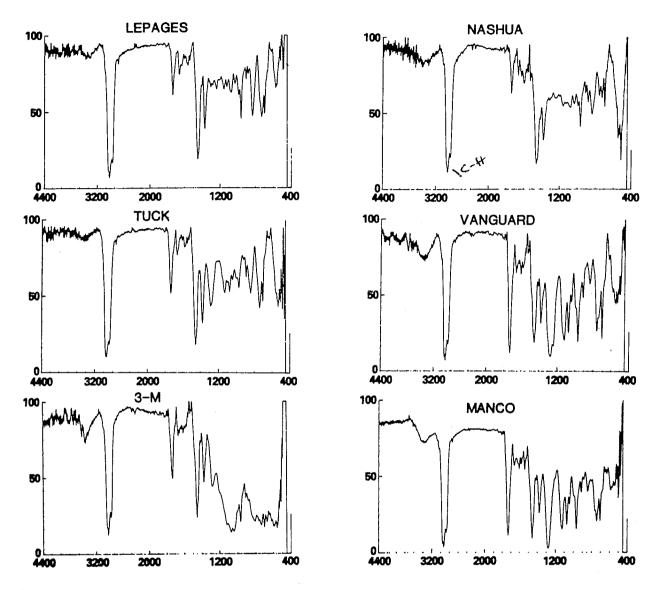


Figure 2. Internal reflectance Fourier transform infrared spectra of tape adhesives.

Table 3. THE COMPOSITION OF PRESSURE-SENSITIVE **ADHESIVES**

- A. Polymer: natural rubber, butyl rubber, polyisobutylene, block copolymers (polystyrene/ polybutadiene/polystyrene; polystryene/ polyisoprene/polystyrene), acrylics, vinyl ethers, silicones
- B. Rubber Tackifiers: wood rosin and rosin esters, terpene resins, terpene phenolics, phenol formaldehyde, petroleum based resins
- C. Plasticizers: mineral oil, lanolin, lecithin, phthalate esters, polybutene, petrolatum
- D. Pigments/fillers: TiO2, silica, CaCO3, kaolin, mica, BaSO₄, ZnO, talc, carbon blacks
- E. Antioxidants: amines, phenolics, dithiocarbamates

Elemental Analysis

The ten elements analyzed by X-ray fluorescence spectrometry were aluminum (Al), silicon (Si), sulfur (S), chlorine (Cl), antimony (Sb), calcium (Ca), titanium (Ti), iron (Fe), zinc (Zn) and lead (Pb). The count rate data for ten elements were accumulated for two rolls of each brand and normalized by dividing the count rate for each element by the total for all ten elements and multiplying by 100. The average normalized count data and standard deviations are shown in Table 4. The count rate data do not necessarily reflect the relative abundances of the elements present. This is especially true for S, Sb and Ca. The X-ray fluorescence peak for S $(K_{\alpha,\beta})$ contains a large contribution from the M line from Pb which cannot be resolved. Likewise, a substantial overlap exists between the Sb L lines used in the analyses and the Ca K_{α} and K_{β} peaks. In this situation, however, a partial resolution is achieved and the computer in the spectrometer decides how much of the area to allot to Sb on the basis of the number of counts in the channel corresponding to the strongest Sb line (L_{α}) . The remainder of the area is assigned to Ca. While these manupulations may not give a true representation of the elements present, they do not affect the comparison of elemental profiles from one sample to another. The elements found in the tapes are generally similar to those present in PVC fillers, stabilizers and lubricants (Satas 1982) (Table 5). Within each manufacturer, the two rolls are basically congruent, considering the standard deviations. Between manufacturers, sufficient variations exist among the ten elements to unequivocally demonstrate differences in elemental profiles. The elemental profiles for each manufacturer are shown graphically in Figure 3. The magnitude of the differences seen obviates any further tests for population overlap.

Table 4. SEMIQUANTITATIVE DETERMINATION OF	F TEN ELEMENTS IN ELECTRICAL TAPES
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Manufac- turer	Roll	Al	Si	S	Cl	Sb	Ca	Ti	Fe	Zn	Рь
Le Pages	A	1.27a(.03)b	3.40(.04)	2.29(.02)	89.61(.14)	.70(.02)	-0-	.27(.02)	.27(.02)	-0-	2.19(.07)
	В	1.33(.04)	3.40(.06)	2.03(.04)	89.60(.22)	.74(.04)	-0-	.30(.02)	.27(.02)	-0-	2.33(.08)
Tuck	Α	.08(.01)	1.12(.03)	2.40(.04)	94.06(.09)	.15(.01)	-0-	-0-	-0-	-0-	2.19(.06)
	В	.09(.02)	1.12(.03)	2.44(.02)	94.04(.10)	.15(.01)	-0-	-0-	-0-	-0-	2.15(.08)
3M	Α	.11(.01)	.26(.01)	3.83(.10)	91.49(.07)	.46(.01)	.13(.01)	-0-	-0-	.43(.01)	3.28(.07)
	В	.10(.01)	.26(.02)	3.75(.03)	91.48(.10)	.45(.02)	.15(.01)	-0-	-0-	.44(.01)	3.36(.10)
Nashua	A	-0-	.81(.02)	2.62(.04)	83.88(.14)	1.05(.04)	1.07(.02)	-0-	-0-	7.35(.13)	3.23(.09)
	В	-0-	.81(.02)	2.62(.03)	83.96(.18)	1.08(.04)	1.04(.02)	-0-	-0-	7.14(.10)	3.35(.11)
Van-	A	-0-	.18(.01)	2.59(.03)	94.45(.09)	.66(.01)	-0-	-0-	-0-	.16(.01)	1.96(.07)
guard	В	-0-	.19(.01)	2.61(.03)	94.44(.09)	.66(.03)	-0-	-0-	-0-	.16(.01)	1.95(.07)
Мапсо	A	.86(.03)	2.18(.04)	1.99(.02)	90.89(.24)	1.53(.06)	1.14(.05)	.14(.01)	.19(.01)	-0-	1.08(.06)
IVIAIICO	В	.87(.02)	2.20(.03)	1.99(.04)	90.91(.08)	1.49(.02)	1.17(.02)	.13(.01)	.18(.02)	-0-	1.06(.04)

^aMean, n = 10

bStandard deviation

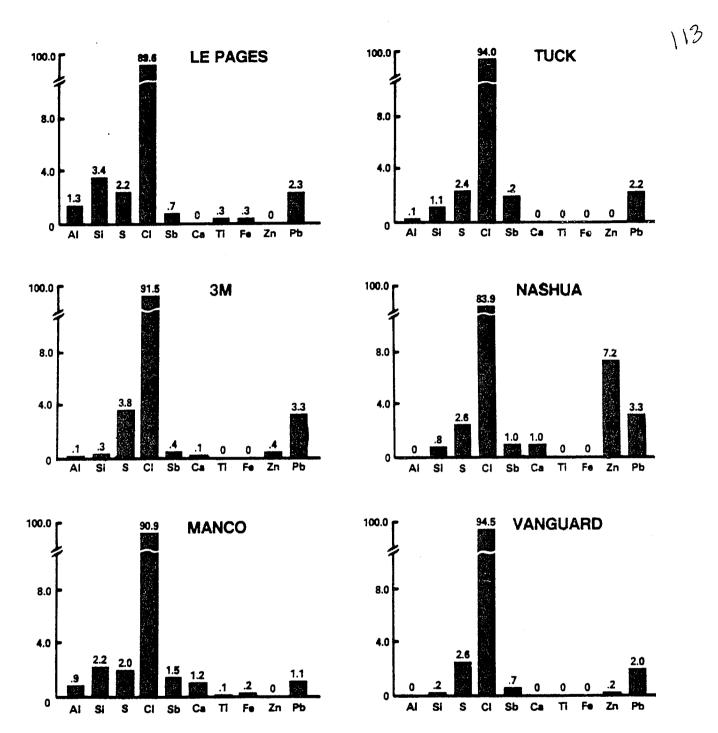


Figure 3. Relative X-ray abundances of ten elements in tape backings.

114

Table 5. THE COMPOSITION OF ELECTRICAL TAPES

- A. Backing: polyvinyl chloride
- B. Plasticizers: phthalate esters, aliphatic fatty acid esters, alkyl/aryl phosphates, dialkyl tin, castor oil
- C. Fillers: TiO2, CaCO3, BaSO4, kaolin, talc
- D. Lubricants: calcium stearate, waxes
- E. Stabilizers: basic lead carbonate, tribasic lead sulfate, Ca, Pb, Cd, Ba Stearate, dibutyl tin dilaurate, diphenyl urea

CONCLUSIONS

All six brands of black PVC electrical tape examined possess unique characteristics which can be determined by non-destructive techniques and used to distinguish one tape from another. These characteristics consist of microscopic surface features, elemental profiles and adhesive composition. Stereomicroscopic examinations showed that all six tape backings have different and distinguishable surface textures. X-ray fluorescence showed that all six tapes have clearly different elemental profiles,

while two rolls from the same manufacturer are indistinguishable. And infrared spectroscopic analyses showed that each manufacturer's adhesive is different and distinguishable from the others. Because each brand of tape can be distinguished from the other five on the basis of any one of the three techniques studied, a combination of all three techniques can lend a very high discriminating power to the examination of evidentiary black PVC tape samples.

REFERENCES

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