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THE DEVELOPMENT OF A COMPREHENSIVE SCHEME FOR THE ANALYSIS OF ELECTRICAL TAPE USING INSTRUMENTAL AND CHEMOMETRIC METHODS

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AMANDA BETH STURDEVANT

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Jay A. Siegel
Major Professor's Signature

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THE DEVELOPMENT OF A COMPREHENSIVE SCHEME FOR THE ANALYSIS OF ELECTRICAL TAPE USING INSTRUMENTAL AND CHEMOMETRIC METHODS

By

Amanda Beth Sturdevant

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ABSTRACT

THE DEVELOPMENT OF A COMPREHENSIVE SCHEME FOR THE ANALYSIS OF ELECTRICAL TAPE USING INSTRUMENTAL AND CHEMOMETRIC METHODS

By

Amanda Beth Sturdevant

Polyvinyl chloride electrical tape is often encountered during explosive investigations. It may be used in the construction of improvised explosive devices as wire insulation, for sealing openings, and for various other purposes. If tape is recovered and submitted as evidence, an analyst may be asked to compare the questioned tape with a known source. When a physical match of tape ends is not possible, the analyst turns to comparisons of class characteristics for the purposes of association or elimination. Most often these class characteristics include physical dimensions, surface texture, and chemical composition.

This study addresses the need for a comprehensive scheme for electrical tape analysis and discusses a preliminary evaluation of the methods currently in use. A physical examination followed by instrumental analysis (ATR-IR, SEM-EDS, HT-GC-MS, and Py-GC-MS) of 38 rolls of black electrical tape separated the sample set into 18 distinguishable classes. Differences in adhesive formulation, elemental composition, and plasticizer type were the major characteristics used for differentiation. Py-GC-MS was found to be the least useful method in electrical tape analysis. Furthermore, the use of statistical methods such as factor analysis allowed for additional discrimination of tape samples (21 classes) based on quantitative differences in their chemical composition.

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1. INTRODUCTION

The philosophy behind any forensic comparison is to utilize methods that differentiate samples from a given population to the limit of the sample population's heterogeneity. In other words, the set of techniques chosen for a comparison should be able to distinguish each member of the population from all others that are, in fact, different. In this way, when two samples are found to have very similar characteristics, the probability of the two samples sharing a common source is increased relative to the probability that the two samples are only coincidentally alike. In the case of polyvinyl chloride (PVC) electrical tape, a successful analytical scheme should discern differences between samples at a relatively small scale, whether that is a tape manufacturer, brand, batch, or individual roll. Ironically, an analytical scheme that could discern differences between samples from the same roll of tape would not be desirable; such a scheme would make associations between a questioned and known length of tape difficult or even impossible.

Regardless, it is not possible to truly evaluate the success or failure of an analytical scheme at meeting this goal without first addressing some problems that are common to many forensic population studies. These include such issues as poor understanding of the inherent heterogeneity of the sample population, not ensuring representative sampling of that population, introducing micro-heterogeneity effects through insufficient sample sizes or excessively precise methods, using only a limited number of analytical techniques, a lack of quantitative comparison of results, and failure to monitor changes in the population.

This study addresses the above issues and includes a preliminary evaluation of some of the techniques used for electrical tape analysis in forensic science laboratories. This is accomplished by gathering as much information as possible about the manufacturing process and distribution of electrical tape, obtaining as large a sample set as possible, avoiding sub-sampling, applying multiple analytical methods to the entire sample set, quantitatively comparing the distinguishing capabilities of these methods, and then monitoring changes in the population.

1.1 Electrical Tape: Importance and Background

In a forensic setting, there are several different instances when electrical tape may be encountered as evidence. The tape can be used to bind victims, to package drugs, or in the construction of improvised explosive devices (IEDs). In the latter scenario the tape can be used to secure separate components of the device together, seal openings, insulate wires, attach the device to the target object, or form a crude container for the explosives themselves. In any of these cases, questioned fragments of electrical tape recovered from a scene can be compared to known samples using individual or class characteristics. Electrical tape is composed of two main layers: backing and adhesive. The backing is composed of approximately 60% PVC resin and 40% liquid and powder additives that are included to adjust the physical and/or chemical properties of the PVC. The most abundant of these additives is liquid plasticizer, generally aromatic or aliphatic in nature, which creates space between the PVC polymer chains and lowers the inter- and intra-chain attractive forces, giving the PVC the flexibility and workability that is common to electrical tape[1]. Examples of some of the more common plasticizers are listed in Table 1. The powder additives, such as stabilizers to prevent oxidation and/or degradation, fillers to reduce the overall cost of the tape, colorants, and flame retardants are used in smaller amounts. Examples of each of these additives are also listed in Table 1.

The two main components of the adhesive layer are an elastomer and tackifier resins. An elastomer is a natural and/or synthetic rubber polymer, which at room temperature is capable of recovering its size and shape after removal of a deforming force[1]. Tackifier resins improve the stickiness, or tack, of the polymer by lowering its viscosity and allowing it to form an immediate bond with a surface under low pressure[1]. Common examples of polymers and tackifiers used are listed in Table 1. As in the backing, there are various other additives that lend certain chemical and physical characteristics to the adhesive layer. These are also listed in Table 1.

There are two other layers incorporated into electrical tape that are used in such small amounts that analyzing them is not practical; these are the primer layer and the release coat. The primer layer is intended to increase the bonding of the adhesive layer onto the backing and is coated onto the backing prior to adhesive application. The release coat is sometimes applied to the top side of the backing to aid in the unwinding of the tape. Examples of these materials are listed in Table 1.

Table 1: Examples of electrical tape components[1]

Electrical Tape Components	**Examples**
Plasticizer Aromatic	phthalate esters
Aliphatic	aliphatic fatty acid esters, alkyl/aryl phosphates, adipates/sebacates, dialky tin, castor oil
Additives	
Stabilizers	$PbCO_3$, $PbSO_4$, stearates, dibutyl tin, diphenyl urea
Fillers	carbon, TiO_2, $CaCO_3$, $BaSO_4$, kaolin ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$), talc ($Mg_3Si_4O_{10}(OH)_2$), dolomite ($CaMg(CO_3)_2$)
Colorants	carbon black, aluminum powder, iron complexes
Flame retardants	Sb_2O_3, chlorinated H-C
Polymer	
Natural rubber	polyisoprene
Synthetic rubber	polybutadiene
Polyacrylates	polybutylacrylate
Copolymers	styrene/isoprene, styrene/butadiene
Tackifier resins	wood rosin, rosin esters, terpene resins/phenolics, petroleum based resins
Primer	rubber, acrylics, starch, polyamides
Release coat	silicones, waxes, acrylics, polyesters, surfactants, polyamide

The first process used in the manufacturing of electrical tape is the calendering of plasticized PVC into a thin film. Calendering is defined as the pressing (of a cloth, rubber, or paper) between rollers or plates in order to smooth and glaze or to thin into sheets[2]. The PVC resin is thoroughly mixed with the additives and then passed through a series of nip rollers that press the resin into progressively thinner sheets that are then cooled and wound onto rolls for future use. At this point, some manufacturers (e.g. 3M) apply an electrical discharge, a "corona," to the PVC sheets in order to increase the surface tension and render the surface more receptive to subsequent coatings. This process is called a Corona Treatment.

The remainder of the manufacturing process involves the roll coating of various layers (primer, release, and adhesive) onto the PVC film. This is accomplished by passing the film between a transfer roller and a pressure roller. The transfer roller is in constant contact with a pick-up roller that is partially immersed in a solution of the desired component. The pressure roller is adjusted to control the thickness of the layer. The PVC film is then passed through an oven to evaporate the solvent. These large sheets are then sectioned into smaller commercial rolls[3]. It is important to note that throughout the entire manufacturing process every piece of machinery that comes into contact with the electrical tape has potential to leave physical and possibly visible markings on the final product. For example, marks such as striations, "divots," "craters," and bubbles can be from rollers, cutters, the coating application, and even the winding of the tape. These markings can sometimes be useful in forensic comparisons (i.e., fast eliminations based on microscopic backing texture). Figure 1 depicts the manufacturing process of electrical tape.

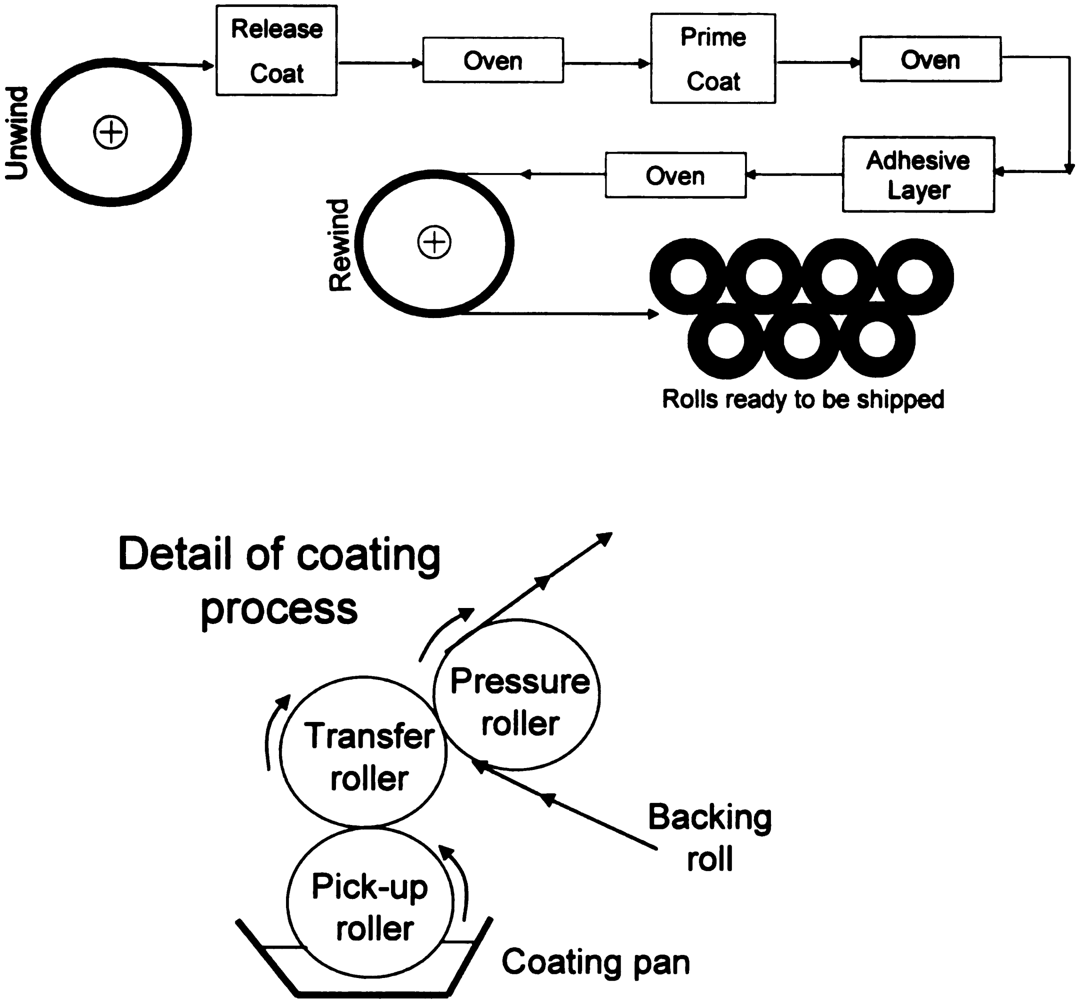

Figure 1: Schematic of the entire coating process of electrical tape and detail of roll-coating process[1,3]

Electrical tape is commercially available in three different grades, depending on the quality and/or quantity of raw materials used in the manufacturing process. These grades can be designated as general, mid-range, and premium. Each grade of tape meets a certain set of standards that affects its suitability for use under various conditions. Examples include temporary versus permanent application, indoor versus outdoor use, voltage levels, application temperature ranges, flexibility, tensile strength, and tack. Table 2 shows the specifications and working conditions of three grades of 3M vinyl electrical tape[4].

Table 2: Specifications of three brands of 3M electrical tape[4]

General Use (Temflex 1700)
Application 0°C-80°C
Temporary applications
Relatively low flexibility
Good initial tack
Agency certified

Mid-range (Highland 700)
Application -10°C-90°C
Permanent Application
Good flexibility
High initial tack
Agency certified

Premium (Super 33+, Super 88)
Application -18°C-105°C
All-weather application
Permanent application
Superior flex/conformability
Excellent initial tack
Multiple-agency certified

1.2 Previous Studies

There have been several articles published regarding the forensic analysis of electrical tape. In 1984 Kee published his work regarding over 100 samples of PVC adhesive tape that had been received into evidence at the Northern Ireland Forensic Science Laboratory between 1980 and 1981. He examined many physical properties of the tapes including their dimensions, surface textures, and edge markings. He used X-Ray Fluorescence (XRF) and Multiple Internal Reflectance-Infrared Spectroscopy (MIR-IR), respectively, to study the inorganic and organic components of the backing side of the tapes. He concluded that by using XRF data to determine the presence and/or absence of calcium (a common filler component) and lead (a common stabilizer component) in the tapes, he could separate the samples into four broad classes. He then further divided these groups based on the presence and/or relative amounts of other elements, specifically silicon, antimony, and phosphorous. Using this method, Kee was able to separate 131 tapes into fifteen different groups based solely on elemental composition. Using MIR-IR and physical characteristics, two of these groups could be divided into two smaller groups, resulting in a total of seventeen distinguishable classes[3].

Also in 1984, Keto purchased eighteen rolls of black PVC electrical tape, three rolls from each of six manufacturers; LePage, Tuck, Manco, Nashua, Vanguard, and 3M. Based on microscopic examination of the backing, Attenuated Total Reflectance-Fourier Transform IR (ATR-FTIR) analysis of the adhesive side, and XRF elemental analysis he concluded that any one of these three methods was sufficient to distinguish each of the six brands from the other five. He noted that the intra-roll variations were much less than the inter-roll variations within a single manufacturer and that the 3M tapes produced a completely unique fingerprint region of the IR spectrum that could not be related to any of the other brands. This aspect was not further examined[5].

In 1988, Williams and Munson analyzed thirty black PVC tapes with pyrolysis-gas chromatography-mass spectrometry (Py-GC-MS) to evaluate the discriminatory power of the method. They were able to distinguish twenty-six of the tapes based on pyrogram pattern analysis, leaving two pairs indistinguishable. One indistinguishable pair was found to be from the same manufacturer with the only differences being in the amount of adhesive applied and the thickness of the backing material used. The tapes in the other indistinguishable pair had different brand names. Although slight differences did exist within a single roll, the samples taken from the same roll were still more similar to each other than to any others in the sample set. All samples recovered and tested post-blast from an IED could be linked back to the appropriate tape, with only small differences noted. Finally, Williams and Munson suggest that experiments comparing multiple rolls of the same type of tape could be helpful in establishing the evidentiary value of a "match[6]."

Merrill and Bartick summarized the FBI's internal procedure for the analysis of black plastic tape in 1989. In this document they describe what to look for during the visual examination of tape, specifically any unique surface features or end marks, and what chemical analyses should be performed. They suggest analyzing the adhesive side, backing side, and a cast film of the plasticizer that has been extracted with chloroform by Internal Reflectance Spectroscopy (IRS) and then searching the library database for possible matches. From the adhesive side, the rubber component can be identified (most often as polyisoprene (PIR), polyisobutylene (PIB), styrene/isoprene copolymer (SIR), or styrene/butadiene copolymer (SBR)) and the type of plasticizer oil used can be classified as either aromatic or aliphatic. The type of backing can be confirmed as PVC, polyethylene (PE), or polypropylene (PP). The plasticizer can be typed as aromatic or aliphatic from the cast film. They also started analyzing the backing side by XRF but did not publish any of these results[7].

In addition to the above articles regarding the analysis of adhesive tapes, there have been numerous other studies done applying various forms of pyrolysis to the analysis of adhesives (polymers). In 1972, Wheals and Noble examined the forensic applications of Py-GC as it is used in the Metropolitan Police Forensic Science Laboratory in London, England. They describe the use and benefits of the method in analyzing paint, adhesives, plastics, synthetic fibers, and soil extracts. They provide sample pyrograms of an epoxy resin, a polyvinyl acetate copolymer, and a styrene/butadiene copolymer and conclude that the differences between brands are often such that a particular product can be identified. They also mention that even though filled adhesives (tiling cements, ceiling tile adhesives, etc.) often pose problems with IR spectroscopy, these products can be easily characterized by Py-GC[8].

Noble, Wheals, and Whitehouse completed an extensive study on the characterization of adhesives using Py-GC-MS and IR spectroscopy in 1974. They analyzed 179 commercially available adhesives in the form of dried films and their results indicate that all but fourteen of these could be assigned to one of twelve classes of adhesive, depending on the resin base used in the adhesive. They note that, if minor variations are examined, different products in the same class can be differentiated so that all products are discriminated and that the fourteen unclassified adhesives are all unique by both of the methods used[9].

In 1980, Wheals published a review of forensic applications, specifically polymer analysis, of pyrolysis and other thermal degradation techniques in which he assessed the merits and limitations of each. He describes the characteristics of the "ideal" technique for polymer analysis and then discusses the advantages and/or disadvantages of using various techniques to reach this "ideal." He includes Py-GC, Py-capillary-GC, multiple stage Py-GC, Py-MS, thermal gravimetry-MS, laser microprobe mass analysis, and different pyrolyzer types in this critique. Wheals then applies his ideas to the analysis of polymers such as paint (decorative gloss and vehicle), synthetic fibers, adhesives, and other miscellaneous substances. He refers to his previous work regarding Py-GC analysis, discussed above, and states that using Py-MS on the same sample set (179 tapes) can provide "rapid adhesive identification" where the mass pyrogram sometimes yields better qualitative information than the pyrogram or IR spectrum. (This was based on unpublished work at the Metropolitan Police Forensic Science Laboratory in London, England[10].)

Bakowski, Bender, and Munson analyzed ninety-one adhesives by Py-capillary column-GC-MS in 1985. The results obtained compared well with those of Noble et. al, discussed above, in that the adhesives fit into the general categories described therein with the exception of cyanoacrylic-based adhesives (super-glues) that are a newer group. Five IEDs were constructed using samples of twenty-four of the original ninety-one adhesives and nineteen residues were recovered post-blast. These represented thirteen of the twenty-four types of adhesives used in the construction of the devices. All of these residues could be properly associated to the original adhesive type after searching a database of composite spectra (correct adhesive was in the top five results of the search[11].)

Curry (1987) performed a study on ninety-four household adhesives in the U.K. using Py-MS to accommodate the forensic scientist's problem of small samples that are frequently contaminated and of unknown origin and composition. He discusses nine broad classes of basic polymer resins used in the adhesives and details certain compounding ingredients included in each. These include the plasticizers, tackifying resins, anti-oxidants, UV absorbers, processing aids, etc. that can be used to further discriminate within each group. Data from each group is presented[12].

Blackledge published a literature review in 1987 on the characterization of adhesive tapes in forensic science laboratories that included all of the articles previously mentioned. One technique that was not discussed in any of these works is fluorescence spectroscopy but it was examined in a previous study by Blackledge (1984). In it, the non-adhesive side of beige masking tape was examined and the method could distinguish between brands of tape and between lots of the same brand of tape. Dichloromethane extracts of the tapes were also examined and showed the same differentiation[13].

In a presentation given at the Midwestern Association of Forensic Scientists Fall Meeting in Minneapolis, Minnesota in 2001, Smith and Randle discuss the forensic examination of pressure sensitive tape. In addition to the history and background of pressure sensitive tapes in general, they detail that of black electrical tape and suggest a scheme for its analysis: a physical match (if possible), examination of physical characteristics (surface texture, adhesive color, width, and thickness), FTIR, Py-GC-MS, and elemental analysis, polarizing light microscopy (PLM), or fluorescence spectroscopy as a final step. They diagram the different layers of electrical tape, explain the composition of each, and give examples of the different components (plasticizers, fillers, stabilizers, etc.) Also, Smith and Randle suggest different plasticizer extractions for backings constructed of PVC, PP, or PE and provide a list of "useful IR bands to look for specific to electrical tape." Finally, they attach a reference "workbook" containing examples of IR spectra and pyrograms of plasticizers, backings, adhesives, and standards. They reference several articles dealing with pyrolysis and elemental analysis of electrical tape but do not include any detail or references regarding PLM or fluorescence spectroscopy[1].

1.3 Challenges Inherent to Population Studies-Our Approach

One of the biggest challenges in completing a population study is the need for a detailed understanding of the sample population in question. Before one can even begin to evaluate the ability of different methods to distinguish members of a population, the inherent heterogeneity of that population must be fully understood and accepted. In the case of electrical tape this heterogeneity arises from both chemical composition and product distribution. However, since most formulations of electrical tape are proprietary and the manufacturers are not willing to disclose the identity of some of the raw materials used, knowing the exact chemical makeup of some brands is very difficult, if not impossible, to do. With regards to distribution, a major tape manufacturer may produce a batch of tape and market some of the tape using their name. However, they may also sell part of that same batch to another company that then markets the tape under a different name. Chemically and physically the two tapes are considered to be the same, while commercially they are different. Therefore, trying to find a method that can distinguish these two tapes based on physical and/or chemical composition is not possible. Also, stating that a certain method is not useful in discriminating electrical tapes based on its inability to distinguish these two particular tapes is not accurate. This is why, prior to completing any population study, it is crucial to study the factors that affect population heterogeneity and monitor any changes in those factors over time. This last point ensures that any results obtained by the study retain their applicability.

Before the start of this project, the members of the research group took several steps to understand the production and distribution of electrical tape. In September of 2002, ATF representatives toured the 3M Hutchinson plant in Minnesota. Here, they were guided through the plant by engineers and were able to see the manufacture of vinyl electrical tapes first-hand. They attended a presentation and received a packet of information on everything from the history of the company to the manufacture and packaging of the final products. This particular plant produces both industrial and electrical vinyl tapes, including those brands that were later obtained as exemplars for this study (i.e., Temflex 1700, Super 88, and Super 33+). Information on the "global competitive situation" of vinyl electrical tape as of 2002 was also supplied to the group. This chart, showing the five major manufacturers of electrical tape in 2002, is shown in Figure 2. We were also informed that 3M discontinued the use of lead salts in its tapes as of 2001.[1]

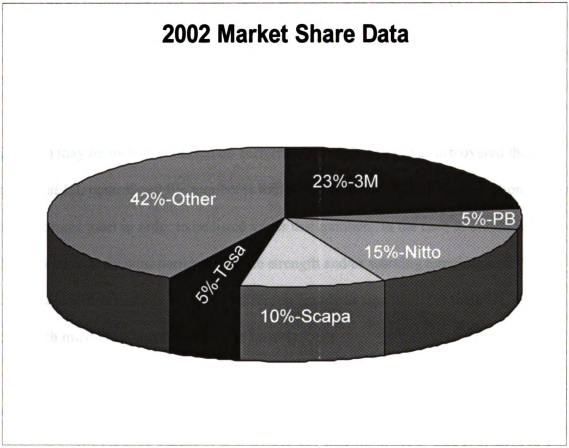

Figure 2: 2002 market share data for the five major manufacturers of electrical tape[4]

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In addition to making contacts with employees of one of the largest electrical tape manufacturers in the world, our group was interested in learning as much as possible about any product specifications unique to certain brands or manufacturers of electrical tape. The packaging of most brands shows the registered trademark of Underwriters Laboratories, Inc. (UL). Companies are permitted to use this symbol on products that have been evaluated by UL and are in compliance with UL's safety requirements. In addition, the alphanumeric UL control number (e.g., 206T) and file number (e.g., E62265) may be included. Based on conversations with UL, it was discovered that a particular UL control and file number is indicative of a certain set of specifications that a product must meet in order to be listed under that number. In the case of electrical tape this can include such properties as tensile strength and/or working temperatures. What this means to this study is that if two different brands of tape have the same UL number, they both must meet some of the same specifications.

An additional amount of insight into the presumptive manufacturer of a tape sample can be gained by looking at the Universal Product Code (UPC) found on the packaging. Where such a symbol is available, the first six digits can be converted to a manufacturer's code that can then be used to access a manufacturer listing kept by Uniform Codes Council (UCC); this is generally the true manufacturer, the former name of the manufacturer, or the distributor of the tape. Overall, by associating the UL and/or UPC markings in our sample set to a manufacturer of electrical tape, we can conclude that as of 2004 there were tapes from at least 7 manufacturers in circulation around the world: 3M, Nitto Denko Corp., Ningbo Sincere Adhesive Products Co., Ltd., Henkel Consumer Adhesives, Inc., ACHEM Technology Corp., Globe Industries, Corp., and

Symbio, Inc.

Once the requirement of understanding a population is met, the next logical step is acquiring a sample collection that is truly representative of the population as a whole. Table 3 lists the brand name and notation, quantity, year acquired, UPC, UL control and file numbers, and associated manufacturers of the thirty-eight rolls of electrical tape examined in this study. The nine exemplar rolls are listed first. Overall, this collection represents twenty different nominal brands and at least seven manufacturers of electrical tape. It is important to note that the tapes in this sample set were purchased in various years and stored in different environments; the effects of age and storage conditions on the physical and chemical composition of electrical tape have not yet been examined. Therefore, all conclusions and results of this work do not take these factors into account and should be interpreted as such.

Another problem that often arises when analyzing many samples is the tendency to focus on one or two particular methods of analysis. Sometimes, even when multiple methods are used, one does not provide any more useful information than another, i.e., every method used analyzes the same component of the sample or provides the same level of discrimination. Therefore, using multiple, orthogonal, methods of analysis is always important in trying to distinguish samples, especially when attempting to develop a complete analytical scheme. This study uses five distinct methods to analyze electrical tape. These include a cursory physical examination of adhesive color (black or clear), ATR-IR of the adhesive side, scanning electron microscopy-energy dispersive spectrometry (SEM-EDS) of the backing side, high temperature (HT)-GC-MS of plasticizer extracts, and Py-GC-MS of the entire tape.

Sample set data

Brand Name	Notation	Quantity	X

Table 3: Sample set data

Brand Name	Notation	Quantity	Year	UPC	UPC-Company	UL Control #	UL File #	UL-Manufacturer
Super 33+	A,B,C	3	2004	054007061328	3M	539H	E129200	3M
Super 88	A,B,C	3	2004	054007061434	3M	539H	E129200	3M
Temflex 1700	A	1	2004	054007697640	3M	539H	E129200	3M
	B	1	2004	054007495710	3M	539H	E129200	3M
	C	1	2004	054007697640	3M	539H	E129200	3M
667 Pro Series		1	N/A	N/A	N/A	590J	N/A	N/A
Bengal	A,B	2	N/A	N/A	N/A	327K	E34833	Nitto Denko Corp.
Commercial 700	A,B	2	N/A	054007042181	3M	539H	E129200	3M
Champion	A,B	2	2004	N/A	N/A	57RJ	E220094	Ningbo Sincere Adhesive Products Co., Ltd.
Duck		1	2004	075353043608	Manco, Inc.	362K	E49341	Henkel Consumer Adhesives, Inc.
Electro Tuff		1	2004	074508120614	Electro Tape Specialities, Inc.	362K	E52811	ACHEM Technology Corporation
Frost King	A	1	N/A	077578037776	Thermwell Products Co., Inc.	206T	N/A	N/A
	B	1	2001	077578037776	Thermwell Products Co., Inc.	206T	N/A	N/A
Globe		1	N/A	N/A	N/A	206T	E62265	Globe Industries Corp.
Intertape		1	2004	077922856800	Anchor Continental	362K	E52811	ACHEM Technology Corporation
Lepage		1	N/A	071980011300	LePages, Inc.	906B	N/A	N/A
Manco		1	N/A	N/A	N/A	590J	N/A	N/A
Powerworks		1	2004	076335026794	Barnett Brass and Copper, Inc.	590J	E50292	Symbio, Inc.
Scotch 33	A	1	1984	02120001044	3M	539H	E129200	3M
	B	1	1984	02120001044	3M	539H	E129200	3M
	C	1	1984	02120001044	3M	539H	E129200	3M
	D	1	1984	N/A	N/A	539H	E129200	3M
Shurtape		1	N/A	N/A	N/A	859X	N/A	N/A
Super 33+		1	2001	054007061328	3M	539H	E129200	3M
Super 88	A,B	2	2001	054007061434	3M	539H	E129200	3M
Tartan	A,B	2	N/A	054007496564	3M	539H	E129200	3M
Vanguard	A,B	2	N/A	N/A	N/A	521D	N/A	N/A
WUL		1	N/A	N/A	N/A	590J	E50292	Symbio, Inc.

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No matter what or how many methods are used in analyzing samples, one must always be sure to avoid sub-sampling a specimen. This means that the methods chosen to analyze a particular sample must do so at a level where the sample is homogeneous. Anything, electrical tape included, has a level of micro-heterogeneity. Therefore, anything, if analyzed at a small enough level, can be eliminated from itself. For example, when examining questioned and known paint chips, an analyst should not focus the examination on a single metallic flake in the color coat while ignoring all the other layers. In fact, this would undermine any attempt at comparing the questioned and known samples by not being able to associate the flake to anything other than another flake, let alone another layer or sample. It is more important in paint examinations to focus on the number, order, and overall composition of all the layers present. The five methods chosen for this particular study each analyze electrical tape at a homogeneous level.

Finally, after an appropriate sample set is acquired and analyzed, one more vital part of a successful population study is developing a way to compare the results. By using quantitative in addition to qualitative methods, some of the subjectivity involved in data analysis can be eliminated and seemingly unrelated data can be directly compared. For purposes of this study, a value "H" (heterogeneity) was defined to compare the ability of the methods to distinguish among the tapes in the sample set. This value is the ratio of the number of distinguishable classes found within a population (D) to the total number of items in the population (I). For the purposes of this study, I can be either rolls of tape or nominal brands of tape. The two most extreme examples of this "H" value are white cotton fibers and nuclear DNA. Since there is essentially only one distinguishable class of white cotton fibers and an infinite number of items, $H_{\text{white cotton fibers}}$ would be

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effectively 0. On the other hand, with just as many distinguishable classes of nuclear DNA as there are items, $H_{\text{nuclear DNA}}$ would be essentially 1. Presumably, the H values for the individual methods and combination of methods used in this study will lie between 0 and 1. The closer a method's H value is to 1, the better it is at distinguishing the tapes in a population.

Another form of quantitation that is used in this study is that of chemometrics, specifically factor analysis (FA). This technique is meant to reduce the original observed variables into fewer composite variables, or factors. These factors account for the inter-correlations among the observed variables and explain how certain variables are related to each other. FA is slightly different from principal component analysis; the purpose of the former is to explain relationships among variables, the purpose of the latter is to account for the maximum amount of variation in the original data set with the fewest number of factors, or components. FA is the method of choice if the observed variables are not error-free, as in the current study[14]. Specifically, in this study, the variables analyzed by this method are the weight percentages of various elements as determined by SEM-EDS; these include Pb, Sb, Ca, and Mg for tapes with black adhesive and Pb, Sb, Ca, Si, S, and Ti for tapes with clear adhesive.

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2. MATERIALS AND METHODS

2.1 Materials and Reagents

Nine exemplar rolls of black electrical tape were requested and received from the 3M Hutchinson plant in May of 2004. These consisted of three rolls of each of three different brands (Temflex 1700, Super 33+, and Super 88) of electrical tape from three different batches. Twenty- nine other rolls of black electrical tape were either purchased during the summer of 2004 or found in the exemplar room of the Bureau of Alcohol, Tobacco, Firearms, and Explosives National Laboratory Center (ATF-NLC). The complete list of all thirty-eight rolls is given in Table 3. The pentane for plasticizer extractions was supplied by Burdick and Jackson and used as received. Glass vials (4 mL) with PTFE-lined caps and 10 mL disposable syringes were received from Sigma-Aldrich and used as received. PTFE filters (0.45 μm) were supplied by Whatman and used as received.

2.2 Instrumental Parameters

The parameters for ATR-IR, SEM-EDS, HT-GC-MS, and Py-GC-MS are listed in Table 4.

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Table 4: Instrumental parameters

ATR-IR

- Spectrometer – SensIR Technologies, Travel IR
- Sampling – single-bounce horizontal diamond ATR
- Beam Splitter – zinc-selenide
- Detector – DTGS
- # Scans (sample and background) – 16
- Resolution – 4 cm^{-1}
- Software – OMNIC

SEM-EDS

- Scanning electron microscope – JEOL JSM-5910LV
- Source – tungsten
- Energy dispersive spectrometer
- Detector – EDAX SUTW
- Analyzer – EDAX Phoenix
- Software – EDAX Genesis
- Calibration settings:
  - Al peak Ká reference = 1.486 eV
  - Cu peak Ká reference = 8.040 eV

HT-GC-MS

- Gas Chromatograph:
  - Instrument – Perkin Elmer Clarus 500 GC
  - Column – HT5 (5% phenyl equiv. polycarborane-siloxane) aluminum-clad 25 m, 0.22 m i.d., 0.1 μm film
  - Temperature Program-
    - Initial temperature = 100°C hold for 1 min.
    - Rate = 15°C/min.
    - Final temperature = 370°C hold for 5 min.
  - Injector – Programmable temperature vaporizer: heated from 50-480°C, held for 2 min., reduced to 400°C, held for 3 min.
  - Split ratio– 30:1
  - Carrier gas – hydrogen, 1 ml/min.
- Mass Spectrometer:
  - Instrument – Perkin Elmer Clarus 500 MS
  - Analyzer – quadrupole
  - Ionization mode – EI
  - Solvent delay – off for 3 min., on from 3-20 min., off from 20-24 min.
  - Full scan – m/z 50-550
  - Scan time - .15 s
  - Inlet temperature – 300°C
  - Source temperature – 300°C
  - Electron energy – 70 eV
  - Software – Turbomass 5.0

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Table 4 (cont'd)

Py-GC-MS

Pyrolyzer – CDS Pyroprobe 2000
- Interface temperature = 200°C
- Pyrolysis conditions = 850°C for 15 s

Gas Chromatograph – Agilent Technologies 6890N
- Column – Supelco MDN-5S capillary column, 30 m x 0.25 mm i.d. x 0.25 µm
- Carrier gas – helium
- Split ratio – 20:1
- Temperature program-
    - Initial temperature = 40°C hold for 1 min.
    - Rate = 20°C/min.
    - Final temperature = 300°C hold for 6 min.
    - Inlet temperature = 300°C

Mass Spectrometer – Agilent Technologies 5973
- Analyzer – quadrupole
- Ionization mode – EI
- Full scan – m/z 10-700
- Scan time – .10 s
- Inlet temperature – 280°C
- Source temperature – 230°C
- Electron energy – 70eV

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2.3 Sample Preparation

For ATR-IR analysis of the adhesive side, pieces of tape measuring approximately 2"x ¾" were placed adhesive side down onto the diamond. Each piece was sampled in three different locations to test reproducibility. Backgrounds were run after each roll. For SEM-EDS analysis of the backing, pieces of tape measuring approximately ¾" x ¾" were placed adhesive side down onto aluminum stubs and secured in a stub holder. Three separate stubs were prepared for each roll of tape and each stub was analyzed one time.

For HT-GC-MS analysis of the plasticizer, pieces of tape measuring approximately ¾" x ¾" were folded in half and placed into 4 mL vials. Approximately 4 mL pentane was added to each vial and this was shaken vigorously by hand for approximately 10 seconds. The solutions were then filtered through 0.45 µm filters attached to disposable 10 mL syringes into labeled GC vials. Three pieces of tape from each roll were extracted in this manner. A process blank was also prepared using pentane in an empty 4 mL vial. Blanks of pentane were run between each sample. For Py-GC-MS analysis, 0.25 mm squares of tape were cut, adhesive side up, using a scalpel, tungsten probe, ruler, and stereoscope. These were then placed in a quartz tube containing a glass wool plug. Three pieces of each of the exemplar rolls were analyzed. The tube was cleaned after every sample by flash heating to 1000°C for 15 sec and the wool was changed between rolls. Blanks were run after every sample.

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3. RESULTS AND DISCUSSION

3.1 Physical Examination of Adhesive Color

Figure 3 shows the separation of the sample set based on adhesive color (black or clear) and ATR-IR analysis of the adhesive side. It is important to note that all the tapes with black adhesive are manufactured by 3M. Therefore, this characteristic could be an effective indicator of the manufacturer of a questioned or known fragment of tape if no labeling or packaging is available. Also, noting adhesive color can help focus the investigative collection of exemplars to only those tapes that share this feature with a questioned sample. Completing this exam takes only a few seconds and it should be one of the first steps in the analysis of electrical tape.

3.2 ATR-IR

The spectra shown in Figure 4 demonstrate the reproducibility of ATR-IR as it relates to the adhesive side of electrical tape. The top portion of Figure 4 contains the overlaid spectra from three rolls of Super 88 (2004) that were received directly from 3M and are known to have originated from three different batches of tape. The similarity in the spectra indicates there is little inter-batch variability with this component of the tape. The bottom portion of Figure 4 contains the overlaid spectra of three different positions on a single 2" x ¾" sample from a roll of WUL tape. Again, the lack of any significant differences in the spectra indicates little intra-roll variations in the adhesive. These spectra are shown as examples of the level of reproducibility achieved for all tapes in the sample set, both between and within rolls. (Samples from the beginning, middle, and end of three of the exemplar rolls were compared via ATR-IR and show the same level of reproducibility; however, this was not tested for all tapes in the sample set.)

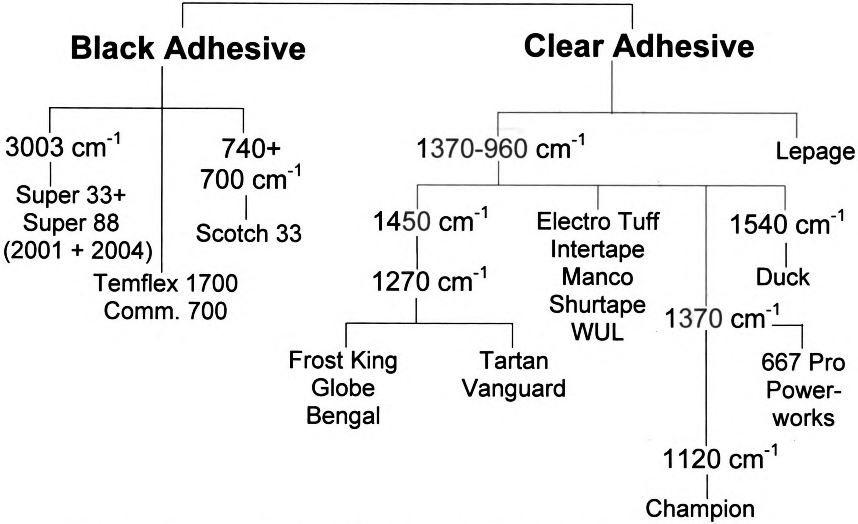

Figure 3: Overall differentiation based on adhesive color (black or clear) and ATR-IR analysis of adhesive side

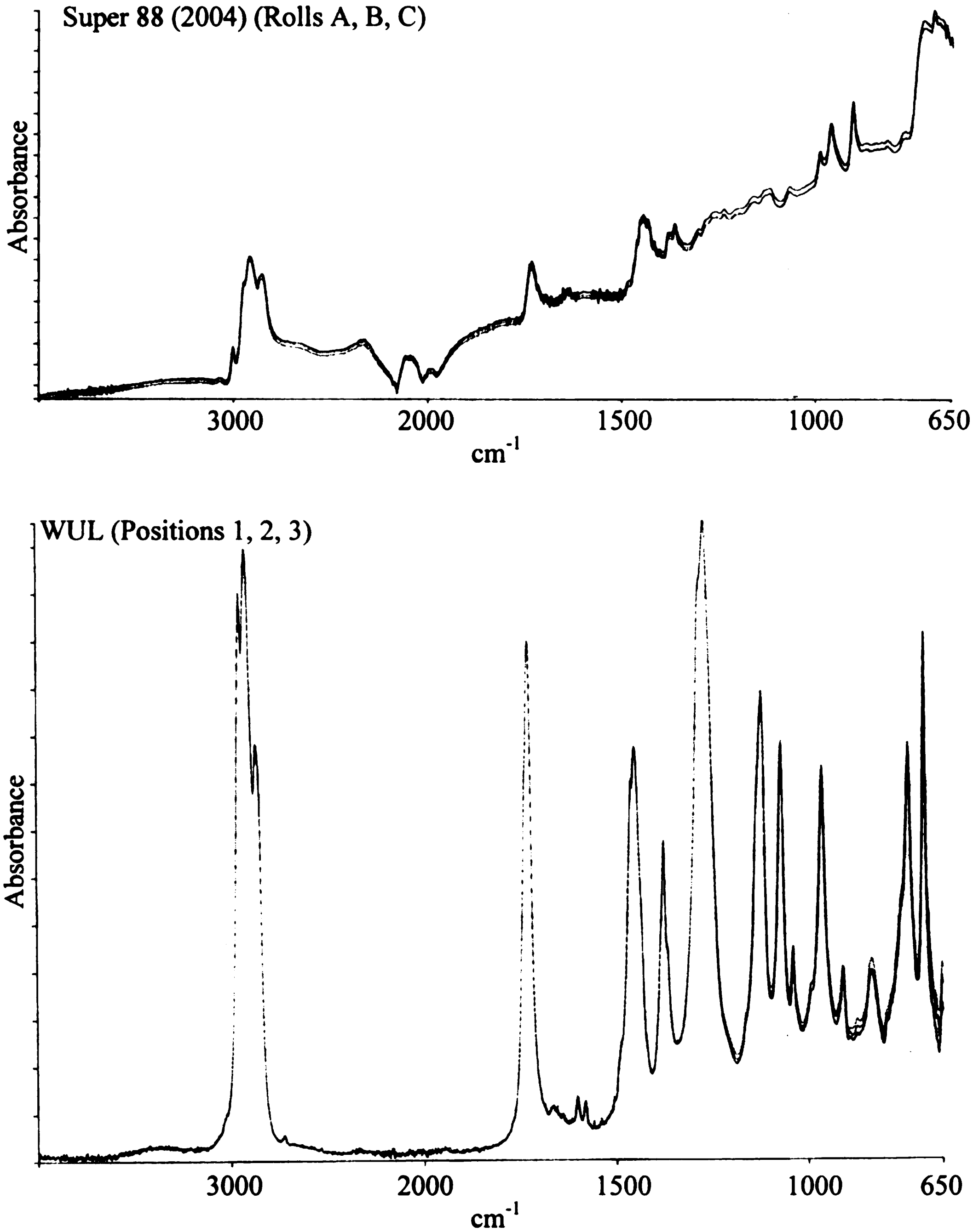

Figure 4: Examples of inter- and intra-roll reproducibility in ATR-IR of adhesive sides of black electrical tape (Super 88 (2004) and WUL)

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Figure 3 shows the overall differentiation of the sample set based on ATR-IR analysis of the adhesive side. The tapes are first grouped based on adhesive color, discussed previously, then by presence and/or absence of major absorbances, and finally by discernable and reproducible differences in the fingerprint region of the spectra. In this manner, the twenty nominal brands of tape are separated into ten distinct classes with four brands of tape being individualized. The goals of this particular analysis are to classify the general type(s) of plasticizer(s) used in the tape backing as either aliphatic or aromatic and to possibly gain information about the type of rubber polymer used in the adhesive. The results obtained relating to the plasticizer type will be compared to the HT-GC-MS data, as this method is expected to provide more definitive identification of the plasticizers used in the tapes. Also, since the polymer occurs in very small amounts relative to the plasticizer that leeches from the backing into the adhesive, any absorbances from the polymer are often swamped out by those from the plasticizer. When making decisions on the location of absorbances, the resolution of the instrument ($\pm$ 4 cm^{-1}) must be taken into account. Examples of characteristic IR bands common to electrical tape are listed in Table 5.

As previously stated, all tapes with black adhesive are brands manufactured by 3M and these can be divided into three distinct groups using ATR-IR. The spectra of Super 33+ (2004), Temflex 1700, and Scotch 33 are shown in Figure 5 to represent each of these three groups. Notice that all three tapes exhibit a large absorption from carbon black, which is added to the adhesive, resulting in the appearance of a rising baseline. This was a feature common to all tapes with black adhesive. Only Super 33+ (2004) has a small peak just above 3000 cm^{-1} corresponding to sp or sp^2 hybridized carbon-hydrogen bon

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bonds. Also, note that there are no strong absorbances at 699 cm^{-1} (styrene) or 740 cm^{-1} (phthalates) in the spectrum of Super 33+ (2004) or Temflex 1700, while these absorbances are very sharp in the Scotch 33 spectrum. Temflex 1700 and Scotch 33 have absorbances at 1270 cm^{-1}, 1120 cm^{-1}, and 1070 cm^{-1} (phthalates) that are lacking in the Super 33+ (2004) spectrum. Overall, the spectrum of Scotch 33 has a unique appearance in that the peaks are more defined (sharper) and more intense, relative to the baseline, than those of either Super 33+ (2004) or Temflex 1700. All three of these brands show characteristic absorbances for the C-C stretches of PBR at 910 cm^{-1} and 965 cm^{-1}. The spectra of the other brands of tapes in this group are presented in the appendix as Figures 25 and 26.

Table 5: Characteristic IR bands for electrical tape

Electrical Tape Component	**IR Bands (cm^{-1})**
Aromatic Plasticizer (phthalates)	1600, 1580, 650 1270, 1120, 1070 (ester stretches) 1570-1600 doublet (ring-stretching) 1730 (C=O) 705, 741 (sharp)
Aliphatic Plasticizer (adipates)	1375-1400 (hydrocarbons) 1170, 1140 (ester stretches) 1740 (C=O)
PVC	1427, 1251, 960, 690, 635, 615
PBR	965, 910, 775
Natural Rubber	1380, 835, 1660, 570, 1446, 1376
Styrene	699, 760

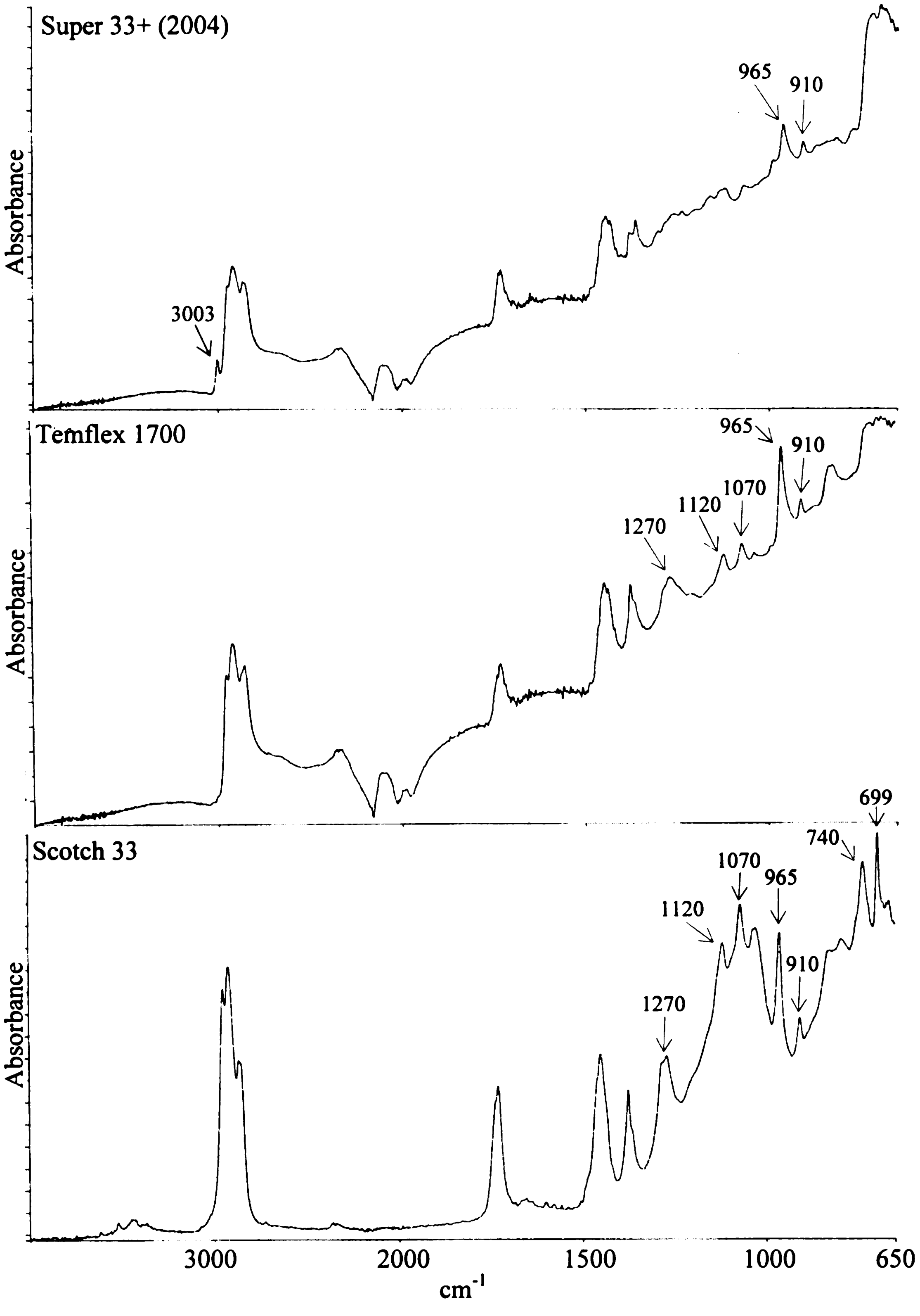


Figure 5: Examples of ATR-IR spectra of black electrical tapes with black adhesive (Super 33+ (2004), Temflex 1700, and Scotch 33)

Figure 6 shows the spectra of two tapes with clear adhesive that could be individualized by ATR-IR. The most important features in the Lepage spectrum are the complete lack of any significant peaks in the region from 1360 to 1000 cm^{-1}, the lack of a doublet at 1570 cm^{-1}, and the position of the C=O stretch at 1740 cm^{-1} (adipate). All the other tapes with clear adhesive have at least three significant peaks between 1360 and 1000 cm^{-1} (1270, 1120, and 1070 cm^{-1}), have a doublet at 1570 cm^{-1} from phthalate ring stretching, and the C=O stretch is located at 1730 cm^{-1} (phthalate). These characteristics tend to indicate that Lepage contains an adipate plasticizer while all the other tapes with clear adhesive contain a phthalate plasticizer. However, it is more likely that the spectrum of Lepage is actually representative of the rubber component(s) (i.e., broad 836 cm^{-1} absorbance and C-H bends at 1450 and 1375 cm^{-1} indicative of natural rubber, absorbances at 910 and 965 cm^{-1} from PBR, and a sharp absorbance at 699cm^{-1} indicative of styrene) in the adhesive rather than being dominated by absorbances from plasticizer. The dominant plasticizer absorbance in all of the tape spectra is that at 1730 or 1740 cm^{-1}; this particular absorbance in Lepage is one of the smallest. Therefore, it is possible that Lepage contains less plasticizer than all the other brands and/or the particular plasticizer used does not leech into the adhesive to the same extent. The spectrum of Duck brand tape exhibits a large absorbance at 1537 cm^{-1} that no other tape contains. This tape also contains several other absorbances that are indicative of phthalate plasticizer.

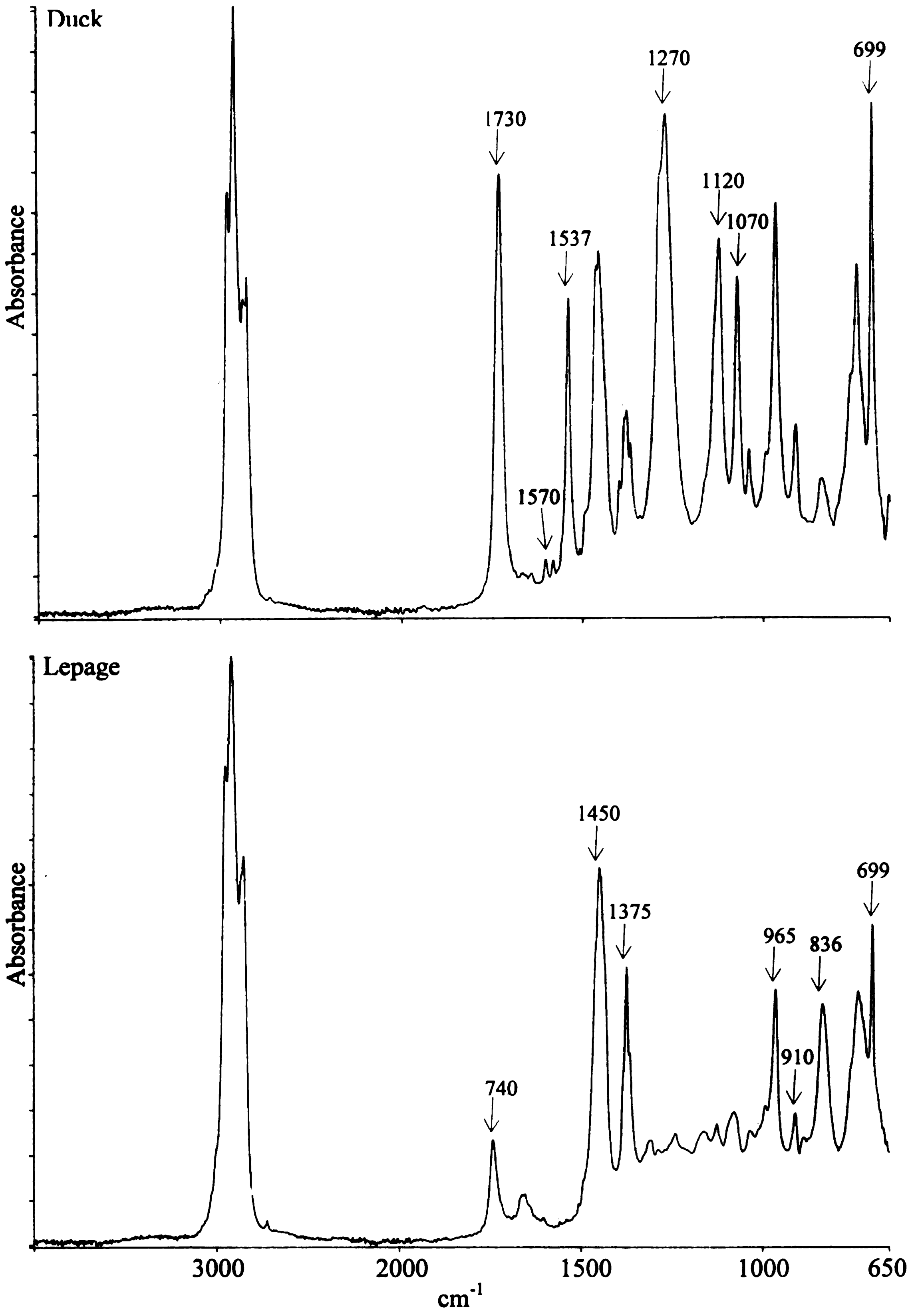


Figure 6: Examples of ATR-IR spectra of black electrical tapes with clear adhesive (Duck and Lepage)

The three spectra shown in Figure 7 are representatives of groups of tapes with clear adhesive that could be differentiated by small, yet reproducible, differences in the fingerprint region of the spectra. The most significant differences in peak shapes between Powerworks and Champion occur at 1120 and 740 cm^{-1}. Notice that there is a small, distinct, peak at a slightly higher frequency than 1120 cm^{-1} in the spectrum of Champion that is lacking in that of Powerworks. Also, the shoulder at a higher frequency than 740 cm^{-1} in Powerworks is not present in Champion's spectrum. There is also a difference between these two groups in the ratio of the 910 and 840 cm^{-1} absorbances. In Powerworks, the 910 cm^{-1} absorbance has a greater intensity than the 840 cm^{-1} peak; in Champion, this is reversed. The spectrum of the Manco adhesive differs from the other two in Figure 7 in the region of the 1370 cm^{-1} absorbance. Both Powerworks and Champion have multiple peaks in this region; Manco has only a single, sharp, peak. Manco lacks the shoulder on the 1120 cm^{-1} absorbance, differentiating it from Champion, and it has a single peak at 740 cm^{-1}, differentiating it from Powerworks. These three spectra also show that varying amounts of styrene (699 cm^{-1}) in the adhesive polymer can be an additional point of comparison between samples. The spectra of the other tapes in these groups are shown in the appendix as Figures 27-29.

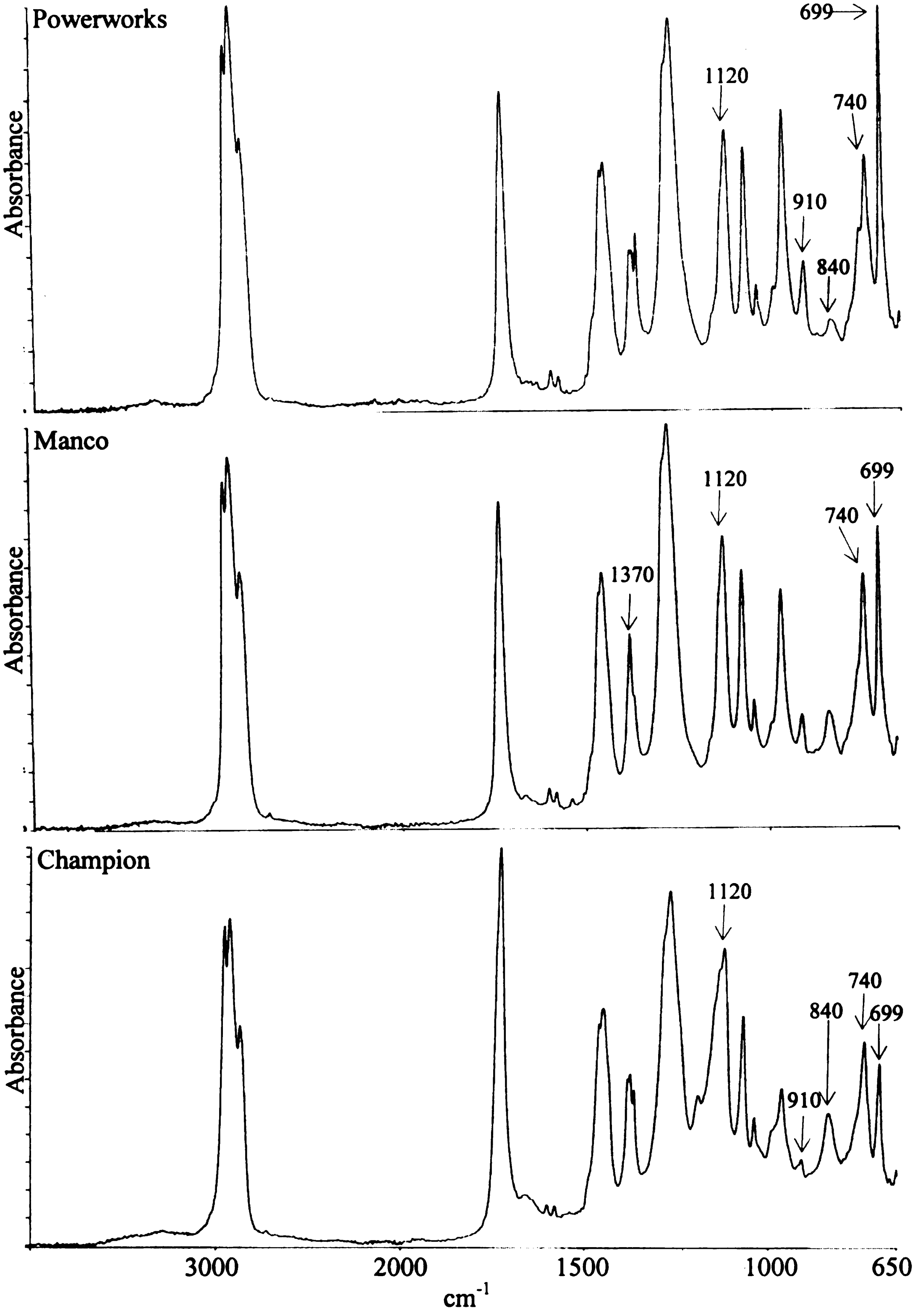


Figure 7: Examples of ATR-IR spectra of black electrical tapes with clear adhesive (Powerworks, Manco, and Champion)

The final two classes of tapes with clear adhesive are represented by the two spectra in Figure 8. The two features that separate these two groups from those previously mentioned are the small absorbance at a slightly higher frequency than 1450 cm^{-1} and the size and shape of the absorbance at 1040 cm^{-1}. The shoulder on the 1450 cm^{-1} peak is absent in all other tapes and the 1040 cm^{-1} peak is smaller and less defined in the spectra of Tartan and Frost King than in the rest of the sample set. The 1270 cm^{-1} absorbance is the distinguishing characteristic between these two groups in the position of the shoulder relative to the apex of the peak. In the Frost King group, the shoulder is at a higher frequency than 1270 cm^{-1} and in the Tartan group, the shoulder is at a lower frequency. Again, this particular absorbance is indicative of phthalate plasticizers and these two groups show that even small differences in type(s) and/or amount(s) of this component can be detected by ATR-IR. The spectra of the other tapes in these groups are shown in the appendix as Figures 29 and 30.

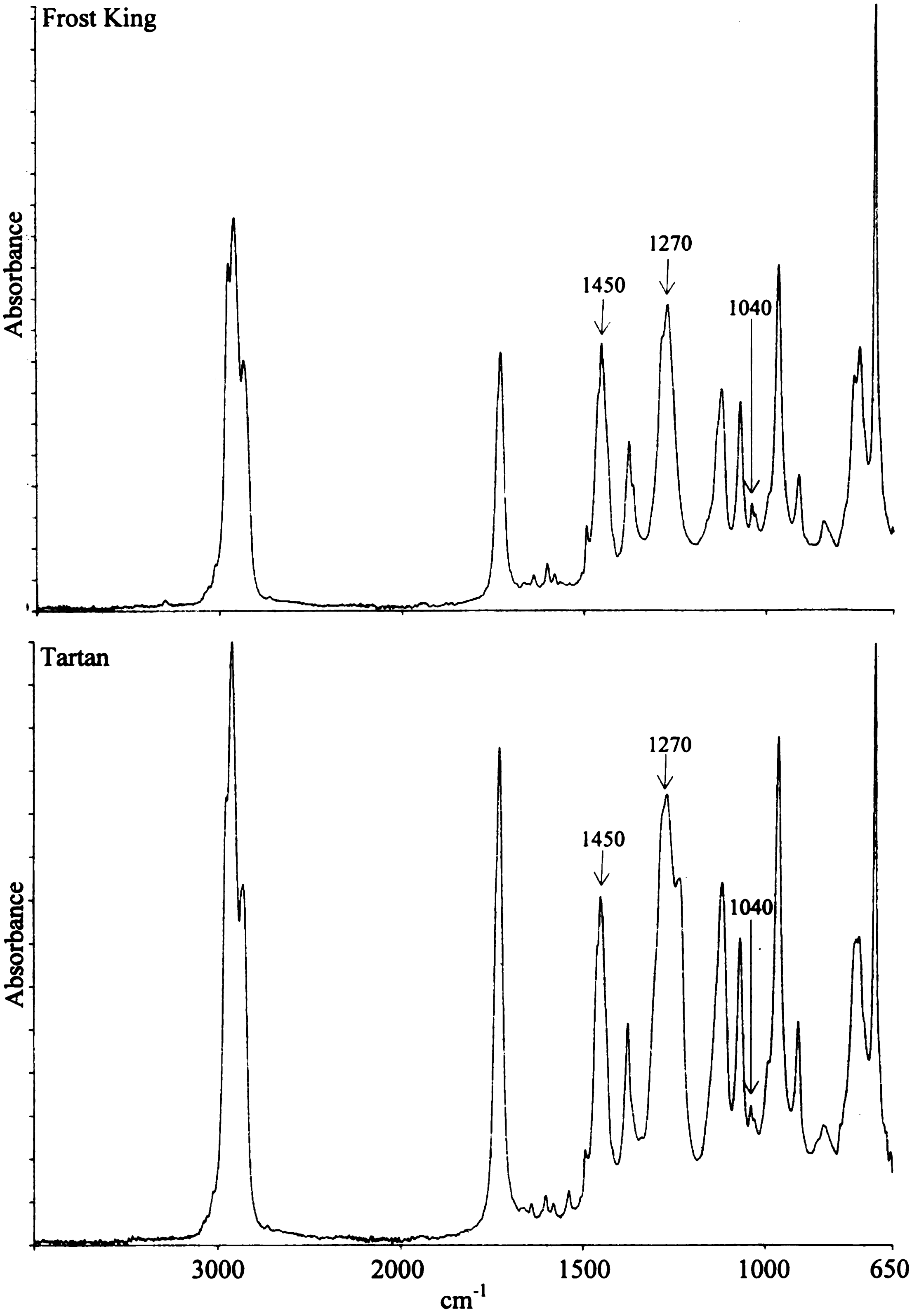


Figure 8: Examples of ATR-IR spectra of black electrical tapes with clear adhesive (Frost King and Tartan)

3.3 SEM-EDS

Figure 9 shows the overall differentiation of the sample set based on adhesive color and elemental composition. These two techniques divided the sample set into nine distinct classes, individualized four nominal brands (Bengal, Globe, Intertape, and Powerworks), and separated three nominal brands (Frost King, Super 33+, and Super 88) into two classes (one with lead (Pb) and/or sulfur (S) and one without). If these particular classes are now considered to be different nominal brands (the only difference being in the Pb/S content), the total number of brands in the sample set is increased from twenty to twenty-three. As in ATR-IR, there was no intra-roll variation observed in SEM-EDS.

All tapes contained carbon (C), oxygen (O), aluminum (Al) (from kaolin), and chlorine (Cl) (from PVC) and these elements were not included in the differentiation or later quantitative analysis. The presence and/or absence of Pb, S, magnesium (Mg), silicon (Si), calcium (Ca), antimony (Sb), and titanium (Ti) were the key characteristics used to differentiate the tapes. All of these elements are components in commonly used fillers, stabilizers, and fire retardants in electrical tape (Table 1).

The elements Pb and S, and Ca and Sb are included as pairs of elements, respectively, to account for the overlap of peaks in EDS. Since most of the inorganic fillers in electrical tape are present in trace amounts, it becomes increasingly difficult to discern if a peak is from one or both of the overlapping elements. Therefore, to reduce error, if a small, ambiguous, peak was present at these energies (2.3-2.4 eV for Pb and S, 3.6-4.0 eV for Ca and Sb) both elements were included in the differentiation. If it was a large peak and clearly identifiable, as is the case for Pb in the tapes with black adhesive, only a single element is listed. For purposes of factor analysis, both elements were included in all of the quantitation (wt%) and the software was able to determine whether there was Ca, Sb, Pb, and/or S present and, if so, how much of each.

The most important elements for distinguishing those tapes with black adhesive were Mg and Pb. The tapes either contained one or the other of these elements, never both. For example, the rolls of Super 88 and Super 33+ from 2001 contain Pb, while the rolls from 2004 do not. The reason for this was confirmed after speaking with a 3M representative, who stated that 3M discontinued the use of lead salts in its electrical tapes in 2001. For those tapes with clear adhesive the most distinguishing element was Si. The tapes were further divided based on presence/absence of Pb/S; the two rolls of Frost King were separated at this point in the differentiation. The presence/absence of Ca/Sb and Ti provided the final level of discrimination.

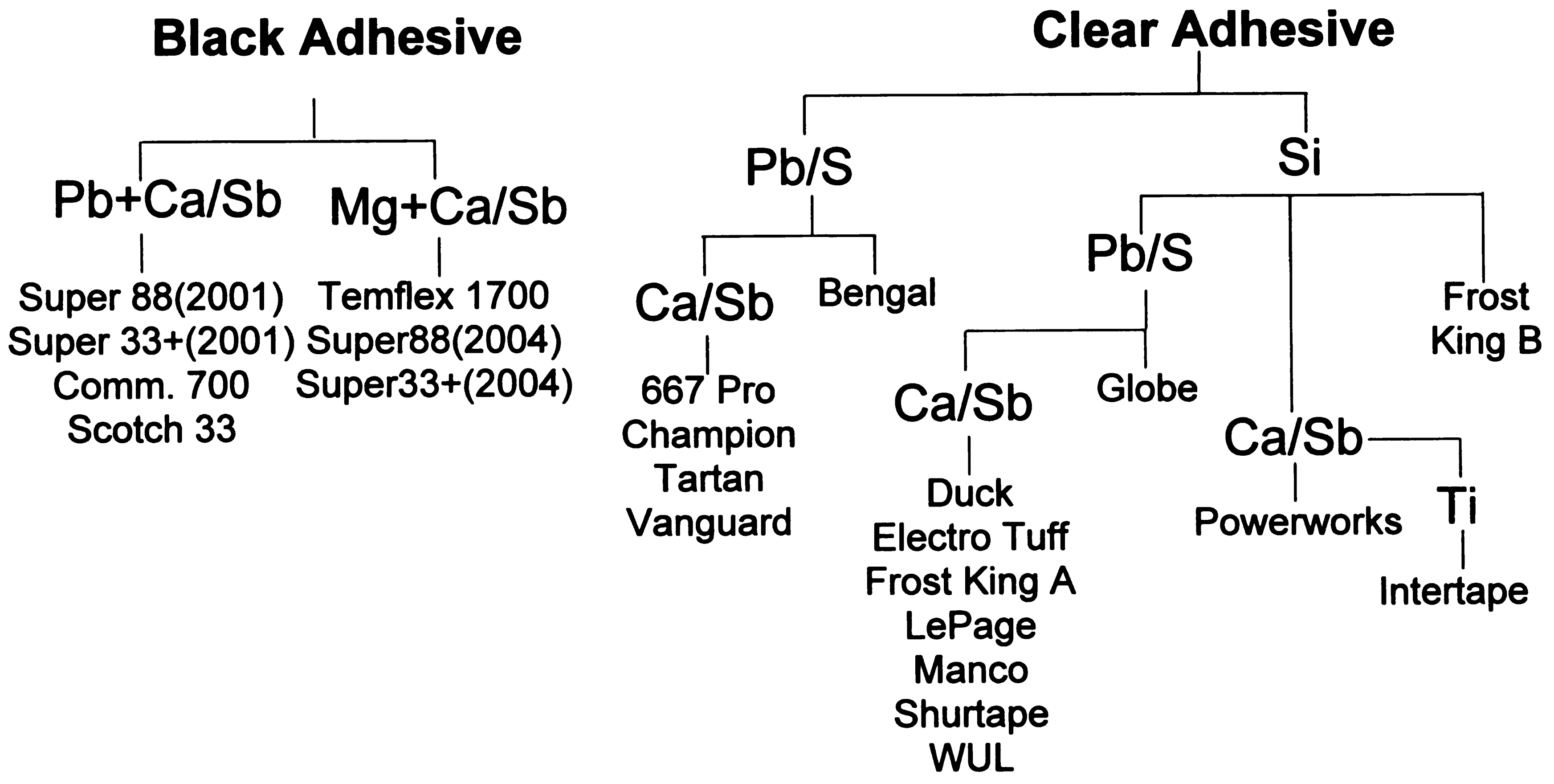
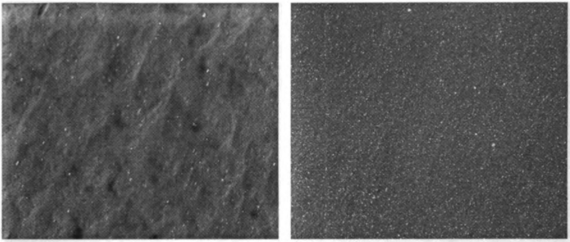


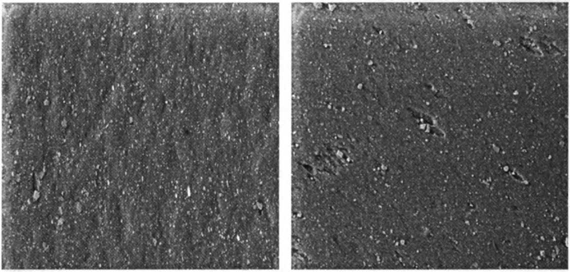
Figure 9: Overall differentiation based on adhesive color and SEM-EDS analysis of backing side

Not only was SEM-EDS used to analyze the elemental composition of the PVC backing, but this method also produced a magnified (200X) image of the texture of the backing. Some examples of the variance observed in the backing texture are shown in Figures 10 and 11. These are photos of the backing texture of samples of Bengal, Commercial 700, Electro Tuff, Frost King, Manco, Powerworks, and Shurtape and can be considered representative of these brands as there was no significant intra- or inter-roll variation in texture. Because this is such a subjective tool, it helps to separate the tapes into groups based on overall texture as smooth or rough, size and number and density of the inorganic particles, systematic defects, and the presence and/or amount of "bubbles," (oval indentations) or "craters" (jagged indentations).

As shown in Figures 10 and 11, these seven brands of tape can be separated into seven distinct texture groups. There will be no detailed discussion of all the textures observed (shown in the appendix as Figures 31-33) or exactly how many distinguishable groups this method could separate the population into, but it is important to note that this characteristic can provide some discrimination. Specifically, even though Duck, Electro Tuff, Frost King A, Lepage, Manco, Shurtape, and WUL are all in the same group based on elemental composition, they can be separated into four groups based on backing texture; these are Electro Tuff and Lepage, WUL, Shurtape, and Duck, Frost King A, and Manco. This feature can increase the distinguishing ability of the method, SEM-EDS, as a whole.

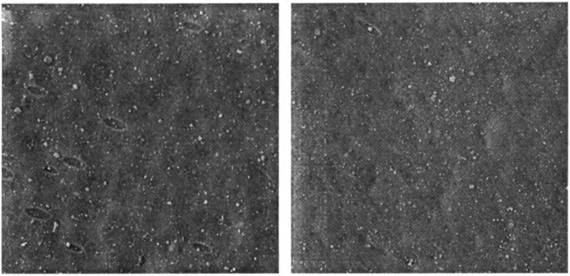


Bengal Commercial 700



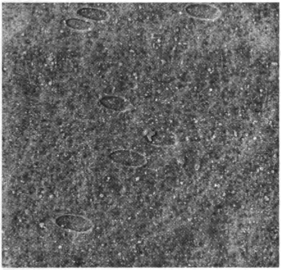
Electro Tuff Frost King

Figure 10: Examples of the backing texture of black electrical tapes under 200X magnification



Manco

Powerworks



Shurtape

Figure 11: Examples of the backing texture of black electrical tapes under 200X magnification

3.4 Factor Analysis of SEM-EDS Data

As discussed previously, the purpose of FA is to help explain the relationships between certain variables. In this study, the variables analyzed were the weight percentages of the elements detected by SEM-EDS in tapes with both black and clear adhesive. (The raw data is supplied in the appendix as Figures 34-38.) Since the tapes could quickly and easily be separated into those with black and those with clear adhesive, it makes sense to analyze each group separately rather than looking for minute chemical differences, when a clear visual differentiation is already possible. Therefore, the data presented is in two parts and each offers separate and valuable information.

Figure 12 is the FA plot for the elemental composition of seventeen rolls representing seven brands of 3M electrical tape (all with black adhesive.) The axes of this plot represent the underlying/latent factors that best describe the variance in the weight percentages (wt %) of Sb, Ca, Mg, and Pb. Factor 1 (F1) is plotted along the x-axis and represents 86.31% of the variation in the data while Factor 2 (F2) is plotted along the y-axis and represents another 13.69% of the variation. Together, these two factors account for 100% of the variation in the data as well as explain how certain variables are related to each other. Each point on the graph represents the average for the three replicates of each roll. The error bars represent the standard deviations in F1 and F2 and can be used to determine if two samples are differentiable; if the error bars overlap it is reasonable to assume that no differentiation can be made. Likewise, if the error bars do not overlap in either the x or y direction, it is assumed that there is enough variation in the elemental composition of the tapes to state that they are differentiable;

this criteria is the basis for the following discussion about the differentiation of the sample population based on FA.

In the development of the factors F1 and F2, the algorithm assigns different loadings, or coefficients, for each variable. Figure 13 shows the factor loadings for each variable relative to F1 and F2 for the tapes with black adhesive. The farther an element is displaced along an axis, the greater its effect on the overall value of that factor for each roll of tape. In this case, Mg and Pb are the two biggest contributors to F1, and Sb, Ca, Pb, and Mg all contribute to F2.

When comparing the differentiation based on visual inspection of the elements present or absent (nine groups) to the differentiation based on FA of the raw SEM-EDS data (eighteen groups), it becomes clear that FA can greatly improve the overall differentiation of an analytical scheme. Table 5 lists the twelve brands of tape that are individualized and the eleven brands that are part of six different groups after FA is incorporated into the SEM-EDS method. Using the nine original groups, the FA plot, and the raw data, detailed information about what differentiates certain brands of tapes can be gained; the same information could be surmised manually but would require more time and difficult manipulations.

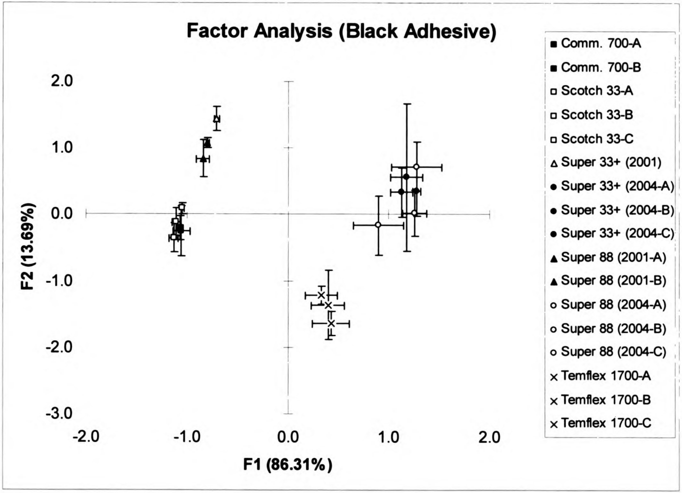


Figure 12: Factor analysis plot for tapes with black adhesive

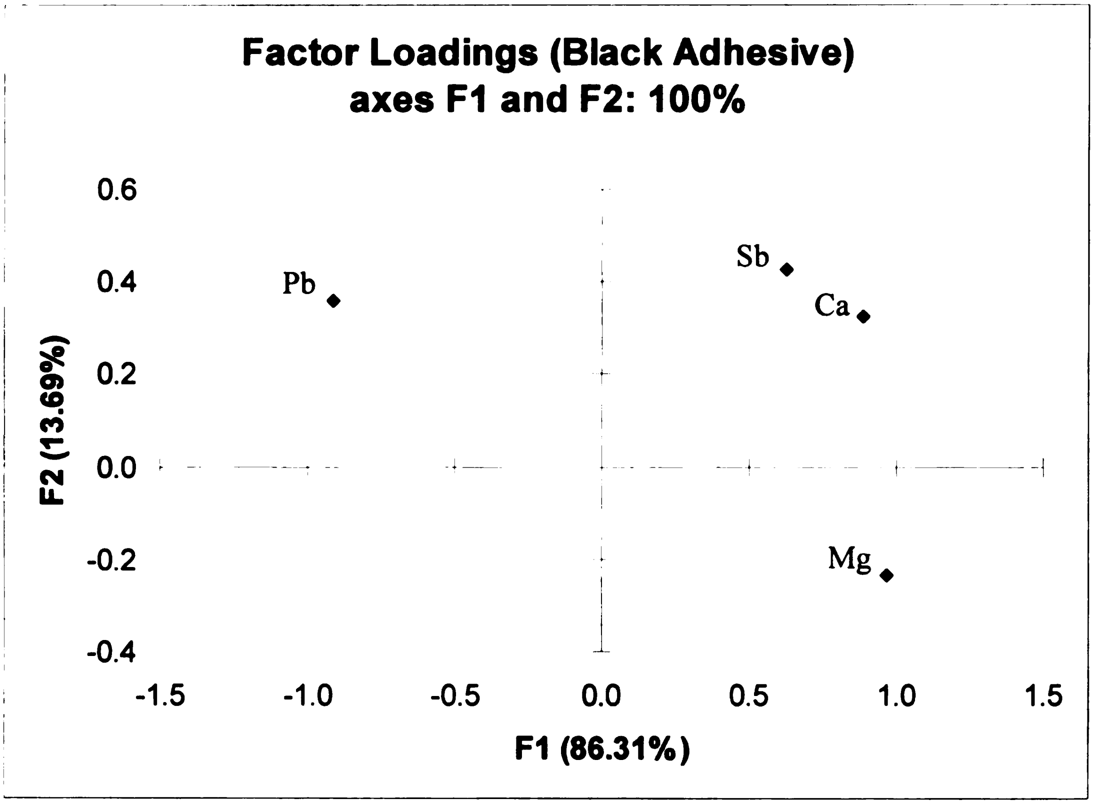


Figure 13: Factor loadings for tapes with black adhesive

Table 6: Overall differentiation based on factor analysis of SEM-EDS data

BRANDS INDIVIDUALIZED via SEM-EDS-FA

Bengal
Duck
Electro Tuff
Frost King B
Globe
Intertape
Lepage
Manco
Powerworks
Shurtape
Temflex 1700
Vanguard

BRANDS IN GROUPS via SEM-EDS-FA

Comm.700 / Scotch 33
Frost King A / WUL
Super 88 (2001) / Super 33+ (2001)
Super 88 (2004) / Super 33+ (2004)
Tartan / Champion
667 Pro Series / Champion

With this in mind, looking back at Figure 12, the relationship between certain elements can be examined. It is known from the raw EDS data that the four brands of tape to the left of the y-axis contain Pb, the three brands to the right of the y-axis contain Mg, and none of the brands contain both of these elements. However, the most important use for such a FA program would be to differentiate two samples that could not be easily differentiated by human interpretation of the results (i.e., in different groups in Figure 9.) In the FA plot, Temflex 1700 is clearly separated from Super 88 (2004) and Super 33+ (2004), and Scotch 33 and Commercial 700 are clearly separated from Super 88 (2001) and Super 33+ (2001). These differentiations appear to be based on differences in the amounts of Sb and/or Ca present in the tapes. Specifically, both Super 33+ (2004) and Super 88 (2004) contain approximately twice the amount of Sb as does Temflex 1700 and both Super 33+ (2001) and Super 88 (2001) contain slightly more Sb and Ca than do Commercial 700 and Scotch 33. Overall, FA of four elements in seven of the twenty-three brands of tape examined in this study was able to create two additional distinct classes of tapes. However, Super 33+ and Super 88 (2004 and 2001) are still not differentiable, within the same year, using elemental composition as a comparison.

Figures 14 and 15 show the factor loadings and FA plot for the sixteen brands of tape with clear adhesive, respectively. The factor loadings plot shows that Pb has the greatest effect on F1 while Si has the least effect. This plot also shows that Sb has the greatest effect on F2, Ca and Pb have the least effect, and Si, Ti, and S all contribute to F2. Looking at Figure 15, there are several important features to note; all tapes above the x-axis, except Champion, contain Sb while those below the x-axis do not, Champion is the only tape that contains Ca and not Si, Intertape is clearly separated from all other brands of tape based on the presence of Ti, and Powerworks and Frost King B are separated because they do not contain Pb or Ti.

Specifically, noticing that Vanguard is clearly separated from the other three brands in its original SEM-EDS group, an inspection of the raw data indicates that it does indeed contain about half the amount of Pb as do Tartan, Champion, and 667 Pro Series. Also, In the same manner, Shurtape and Lepage both contain Sb and are therefore separated from the other five brands in their group, but Lepage contains more than twice as much Pb as does Shurtape, placing it farther to the right on the FA plot. Also, Duck and Manco contain slightly less Pb than do either Frost King A or WUL, placing them farther to the left on the FA plot; Duck contains slightly more Pb than Manco. The other separations, involving Frost King A, WUL, Electro Tuff, Manco, Duck, Tartan and 667 Pros Series, can be attributed to differences in the amounts of Ca, Sb, S, Si, and Pb that are not readily noticeable in the raw data. (It is of interest to note that FA of the SEM-EDS data was able to separate the two different rolls of Bengal tape when initial inspection of the SEM-EDS data could not; this is another indication of the ability of FA to detect subtle differences in relative elemental compositions. This could be the first instance of variation in batch formulations that contain all of the same major ingredients in slightly different amounts that are only detectable by quantitation of the data.)

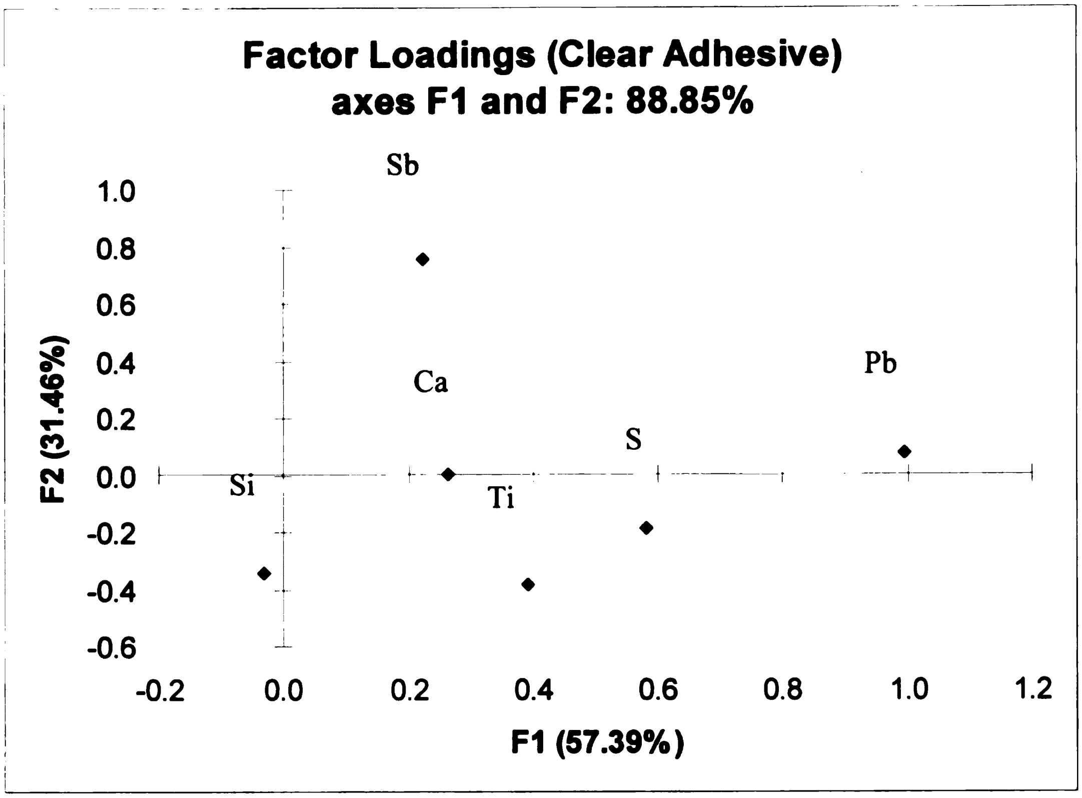


Figure 14: Factor loadings for tapes with clear adhesive

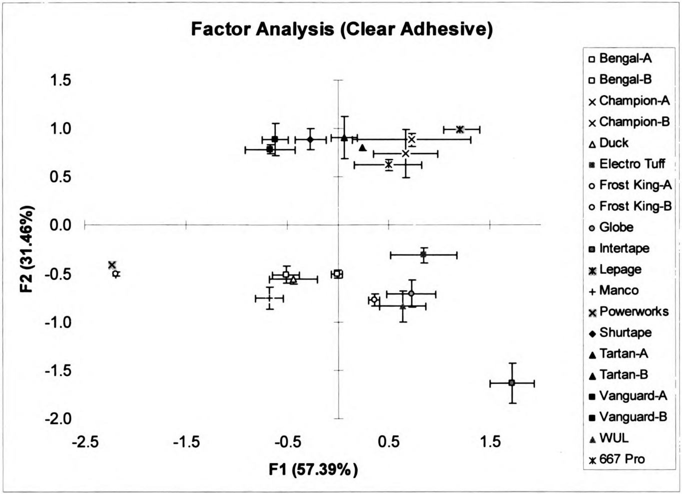


Figure 15: Factor analysis plot for tapes with clear adhesive

3.5 HT-GC-MS

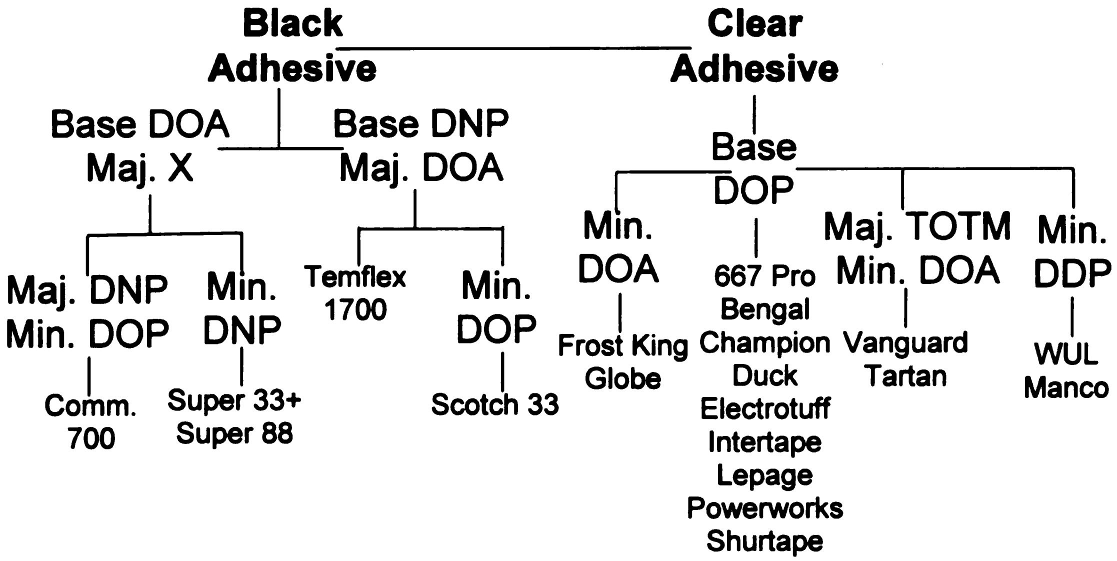
Figure 16 shows the overall differentiation of the sample set based on adhesive color and HT-GC-MS analysis of pentane plasticizer extracts. The definitions of the plasticizer abbreviations and what defines a "base," "major," and "minor" component are listed at the bottom of the figure along with the extracted ion associated with each plasticizer type. All plasticizers were identified based on standards run under the same conditions as the samples or a combination of the mass spectrum of the extracted ion and retention time relative to that of known standards. As Figure 16 shows, the differentiation of the tapes is based on the relative concentrations of the various plasticizers present.

Some trace level components were also detected at less than 1% of the base peak height. Examples included DOA in Duck and DEP and DOP in Super 33+ (2001 and 2004). These components were not included in the differentiation as they may not be reliably detected in all samples from the same source. These differences could arise from slight variations in sample preparation, contamination, or the sample size. With case samples, trace level components can be transferred to electrical tape in any number of ways and, if the source cannot be determined, this may lead to inconclusive association/elimination results.

It is important to note that all tapes that contain DOA as either the base or major component or that contain a major amount of an unknown adipate at 15.4 minutes are known to be manufactured by 3M and have black adhesive. This indicates that 3M is the only manufacturer of those represented by the sample set that uses adipates as the main type(s) of plasticizer. All tapes with clear adhesive have DOP as their base plasticizer and Tartan and Vanguard are the only two tapes in the sample set that show TOTM in the 149, 305 extracted ion chromatogram.

With regards to the ATR-IR data discussed previously, the class represented by Super 33+ (2004) lacks the absorbances at 1270, 1120, 1070, and 740 cm^{-1} that are indicative of phthalate plasticizers; however, these tapes also lack any significant IR absorbances that would be considered indicative of the adipate plasticizers shown in the HT-GC-MS results. The Scotch 33 brand and the group represented by Temflex 1700 in ATR-IR have DOP as the base plasticizer by HT-GC-MS and contain most of the corresponding absorbances by ATR-IR. Most tapes with clear adhesive (all except Lepage) contain IR absorbances characteristic of aromatic plasticizers and Tartan and Vanguard are in a separate group by both ATR-IR and HT-GC-MS. However, given that (according to HT-GC-MS) most of these tapes contain multiple plasticizers of both types, it makes sense that the ATR-IR data would not be explicitly indicative of one plasticizer type over another. In this case, HT-GC-MS would be the better choice to assess plasticizer type as the results are mutually exclusive of each other and not complicated by multiple components.

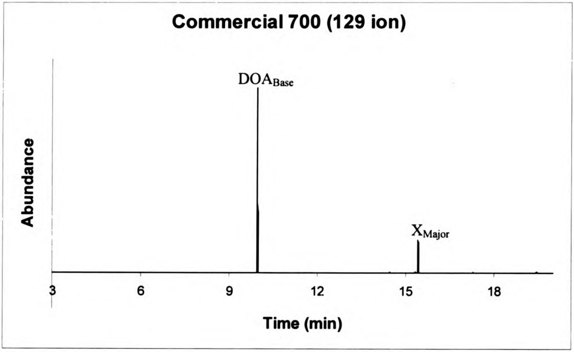
Figures 17-23 show the HT-GC-MS extracted ion chromatograms (m/z 129, 149, and 305) of selected tapes from the groups shown in Figure 16. These are representative of the replicate samples; the only differences that occurred within brands were at the trace levels and not considered in the overall differentiation. Each chromatogram is labeled with the plasticizer abbreviation defined in Figure 16 and the relative amount of each plasticizer as base, major, minor, or trace. The extracted ion chromatograms for the other tape brands are shown in the appendix as Figures 39-46.



Extracted Ion 129
    DOA – bis(2-ethylhexyl)adipate
    X – unidentified adipate at 15.4 min.
Extracted Ion 149
    DEP – diethylphthalate
    DOP – bis(2-ethylhexyl)phthalate
    DNP – diisononylphthalate
    DDP – diisodecylphthalate
Extracted Ion 305
    TOTM – tri(2-ethylhexyl)trimelitate

Base = largest peak height in extracted ion chromatogram
Maj. (major) = less than 100%, greater than 10% of base peak height
Min. (minor) = less than 10%, greater than 1% of base peak height

Figure 16: Overall differentiation based on adhesive color and HT-GC-MS analysis of plasticizer extracts



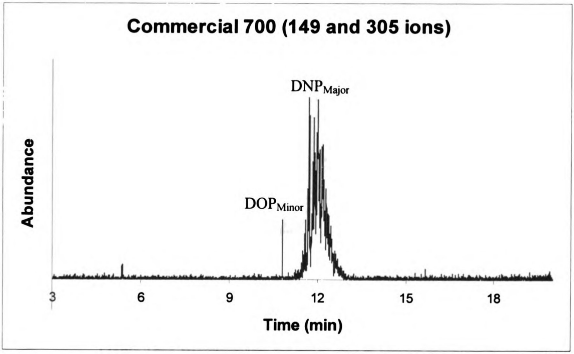
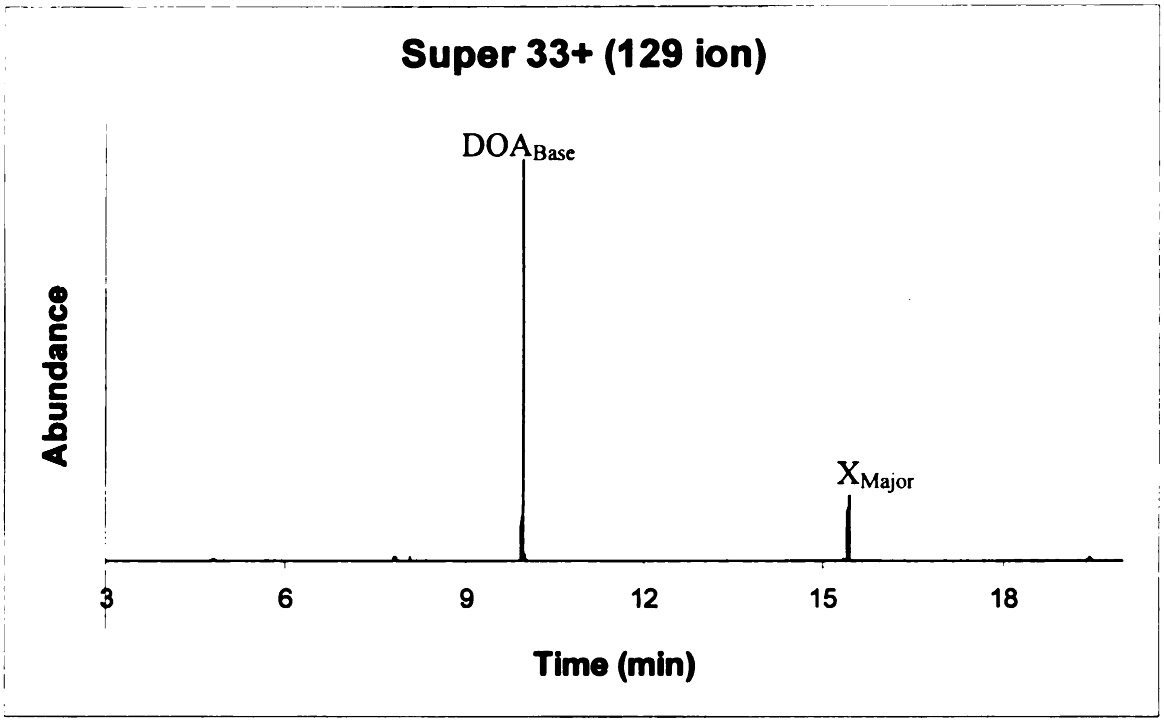


Figure 17: Extracted ion chromatograms of Commercial 700 tape



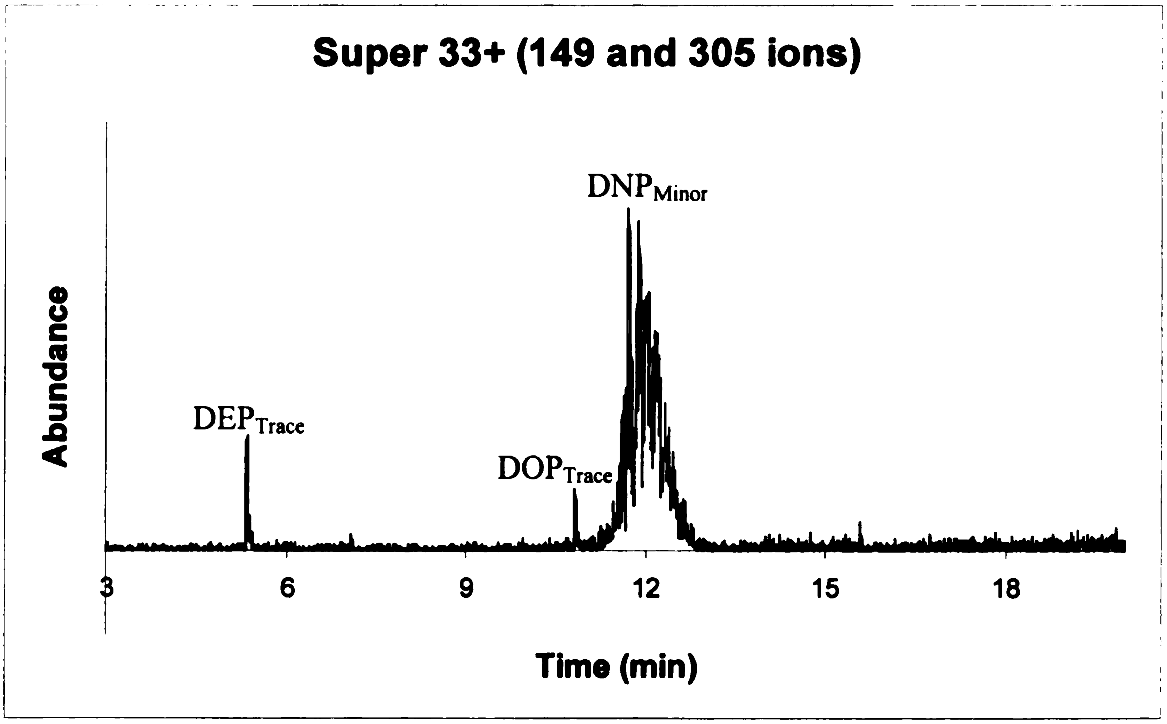
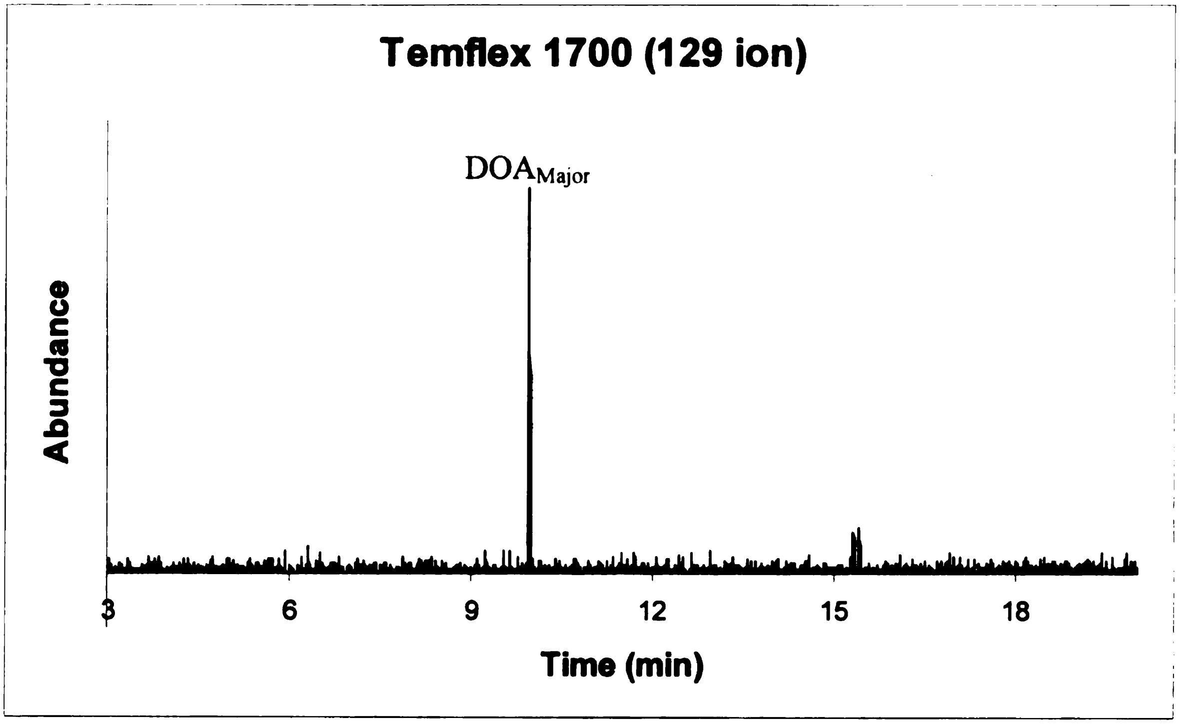


Figure 18: Extracted ion chromatograms of Super 33+ tape



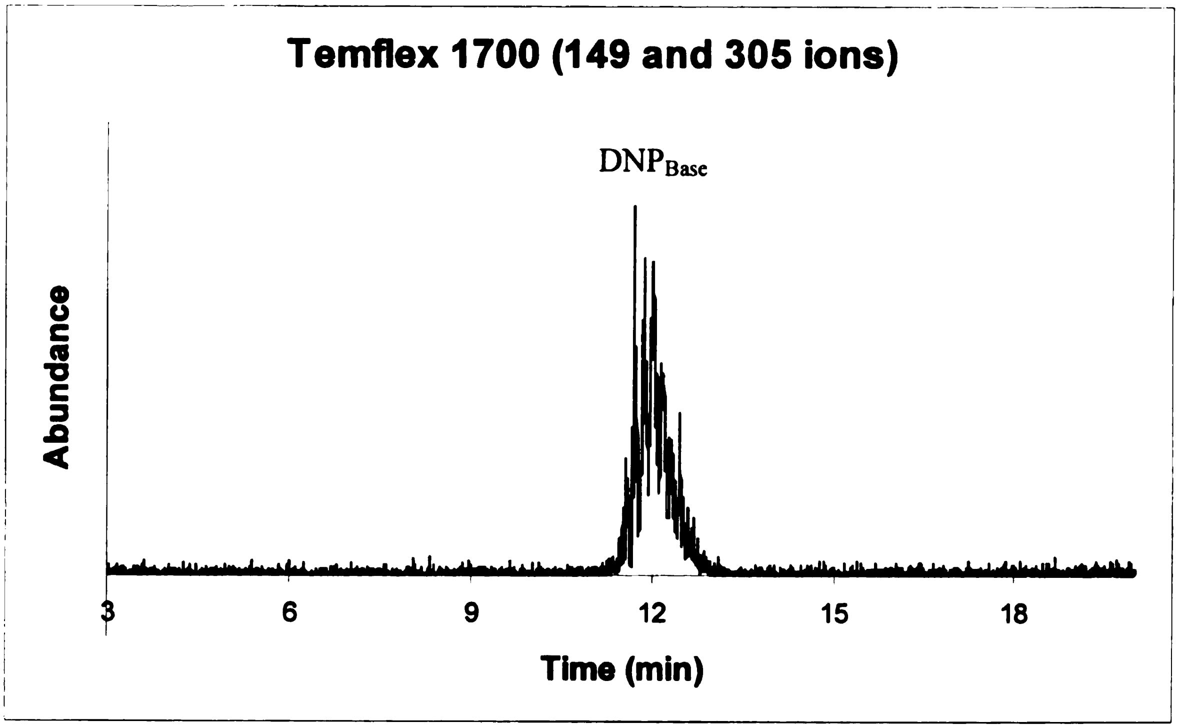
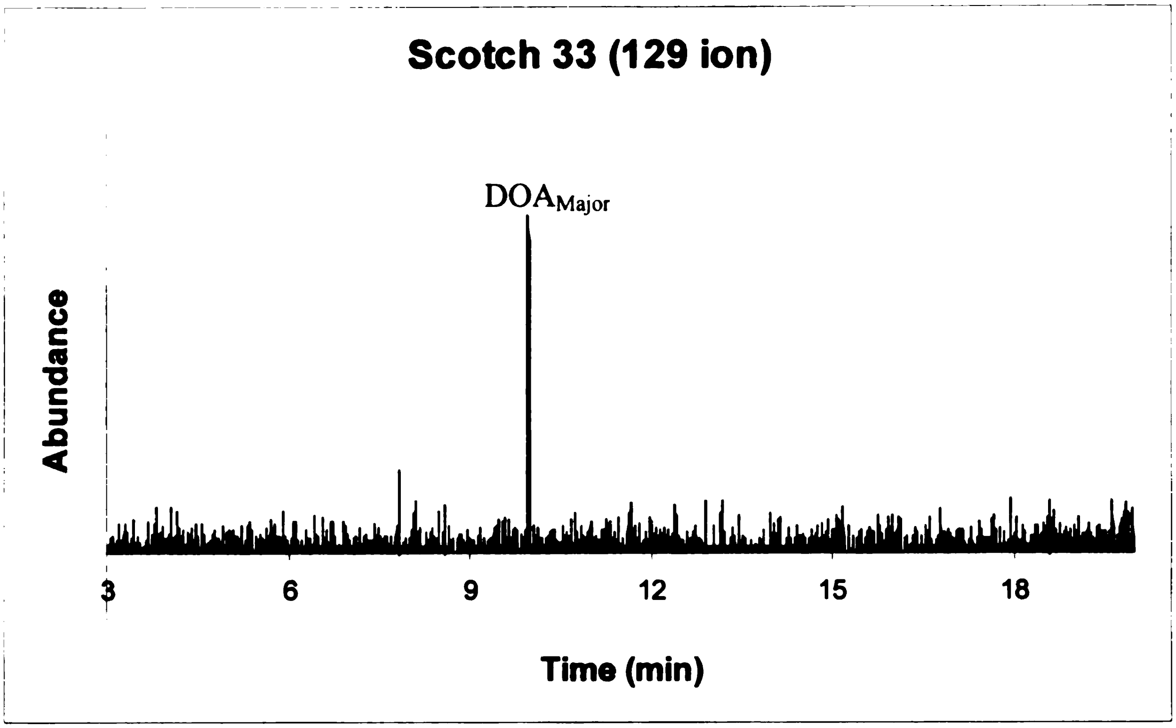


Figure 19: Extracted ion chromatograms of Temflex 1700 tape



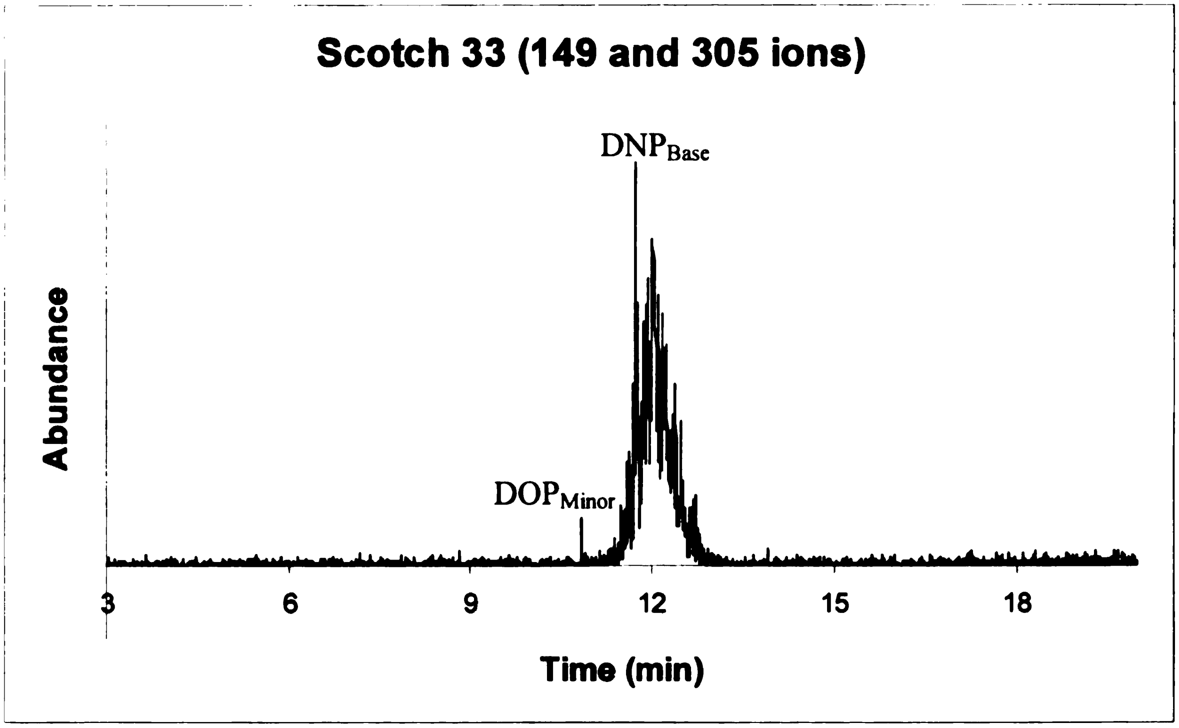
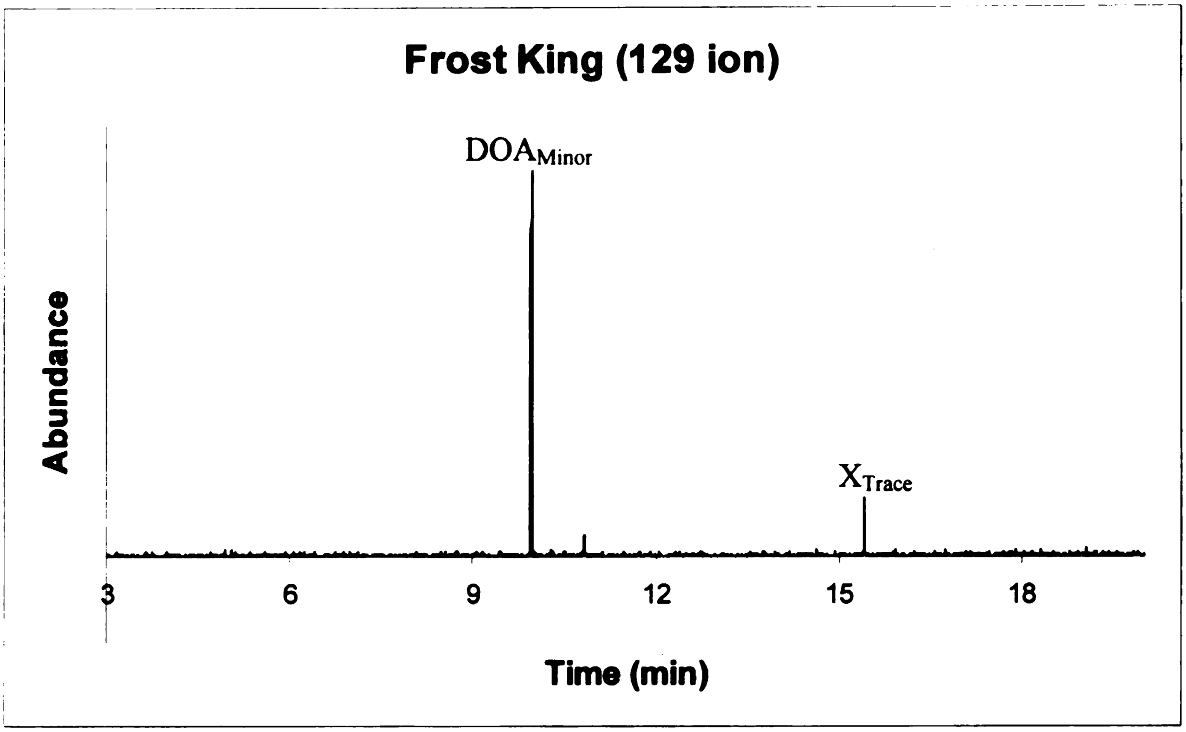


Figure 20: Extracted ion chromatograms of Scotch 33 tape



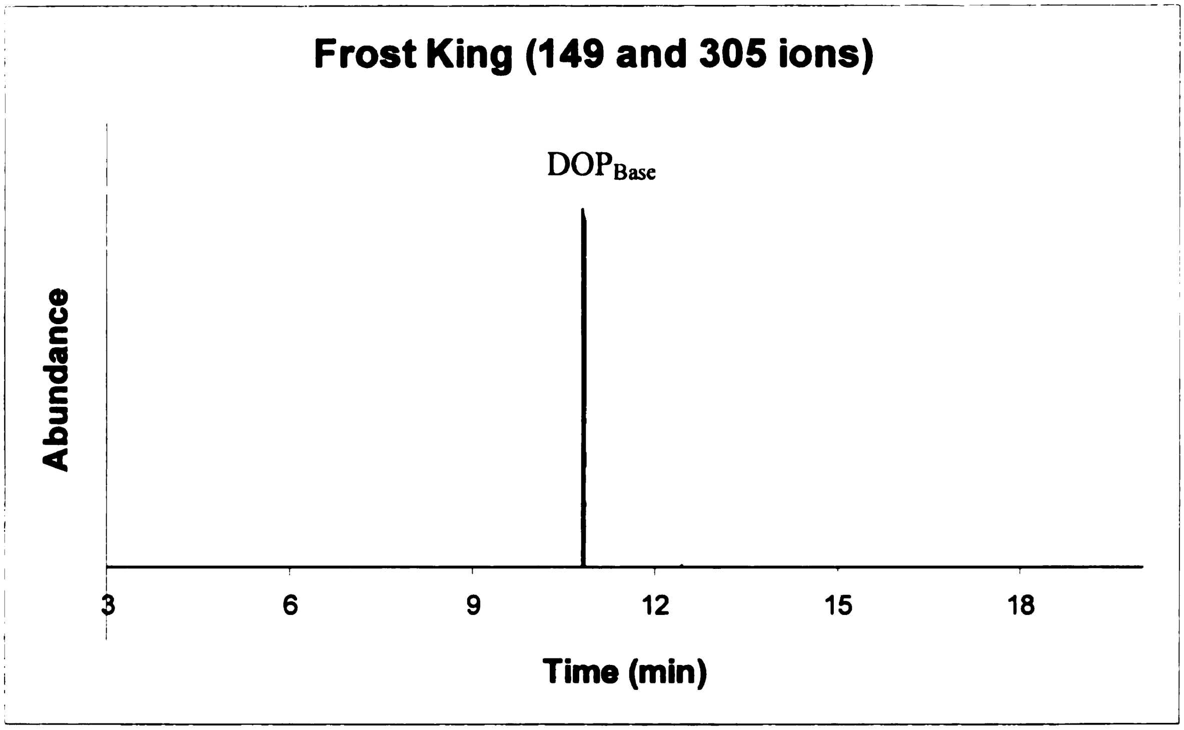
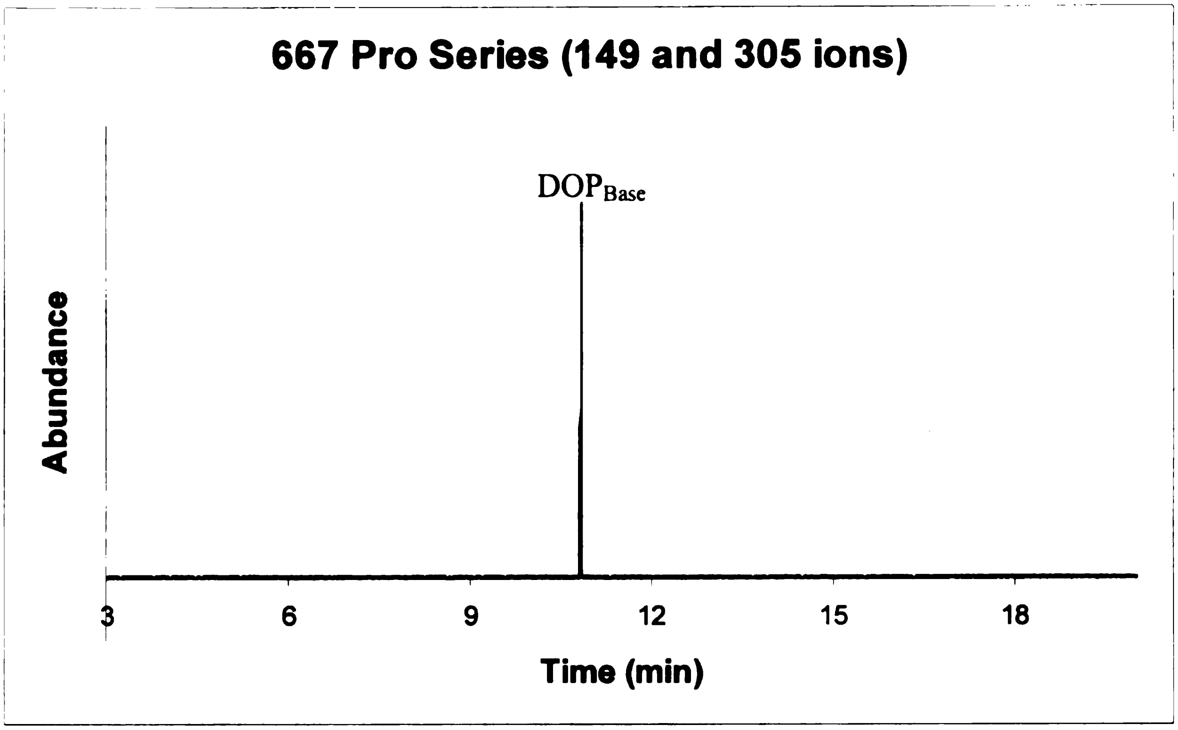


Figure 21: Extracted ion chromatograms of Frost King tape



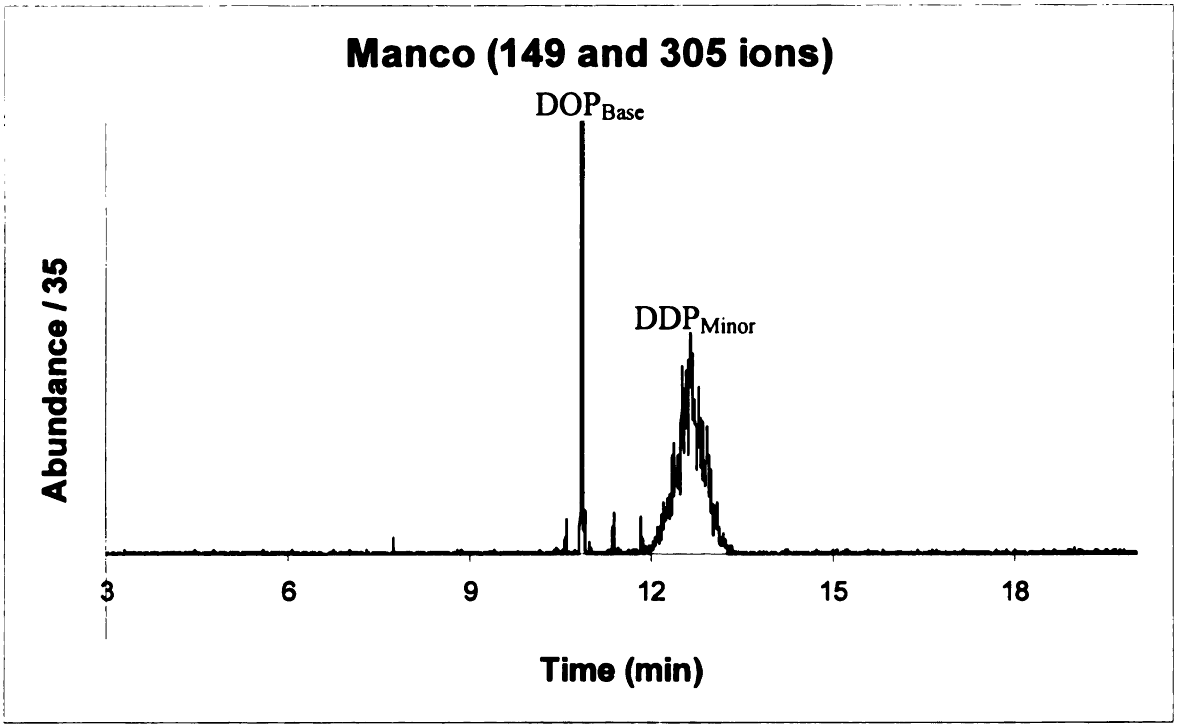
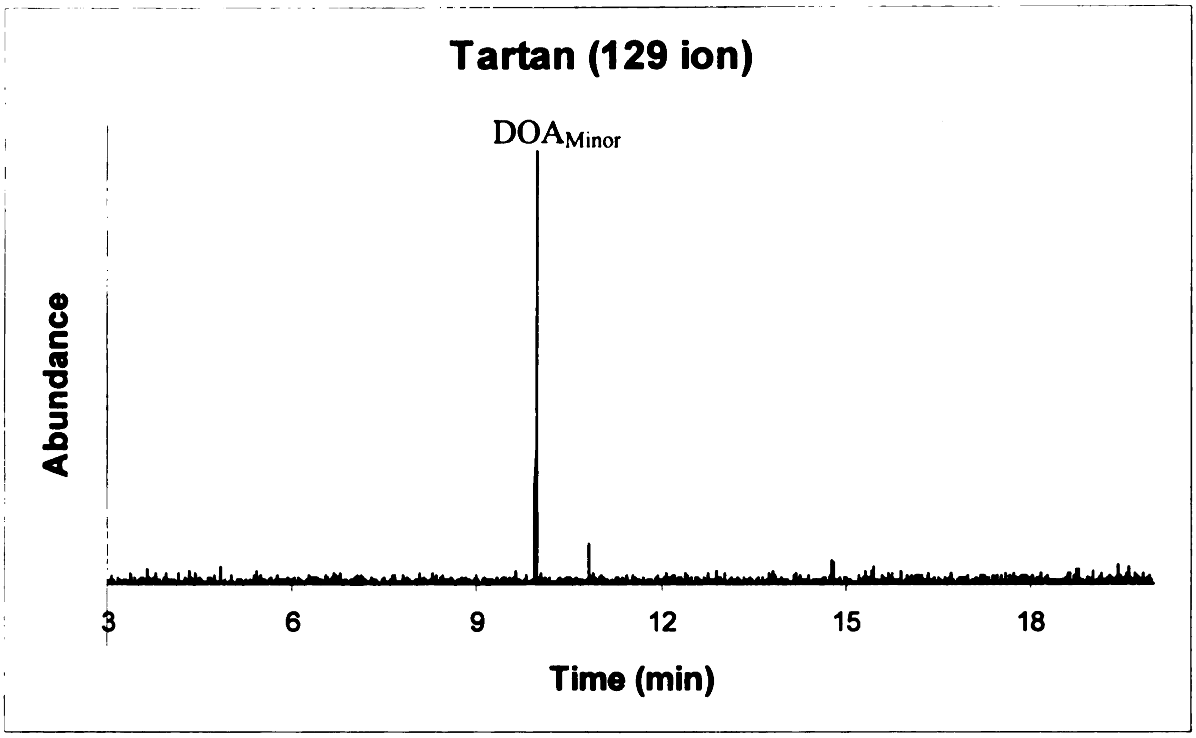


Figure 22: Extracted ion chromatograms of 667 Pro Series (top) and Manco (bottom) tapes



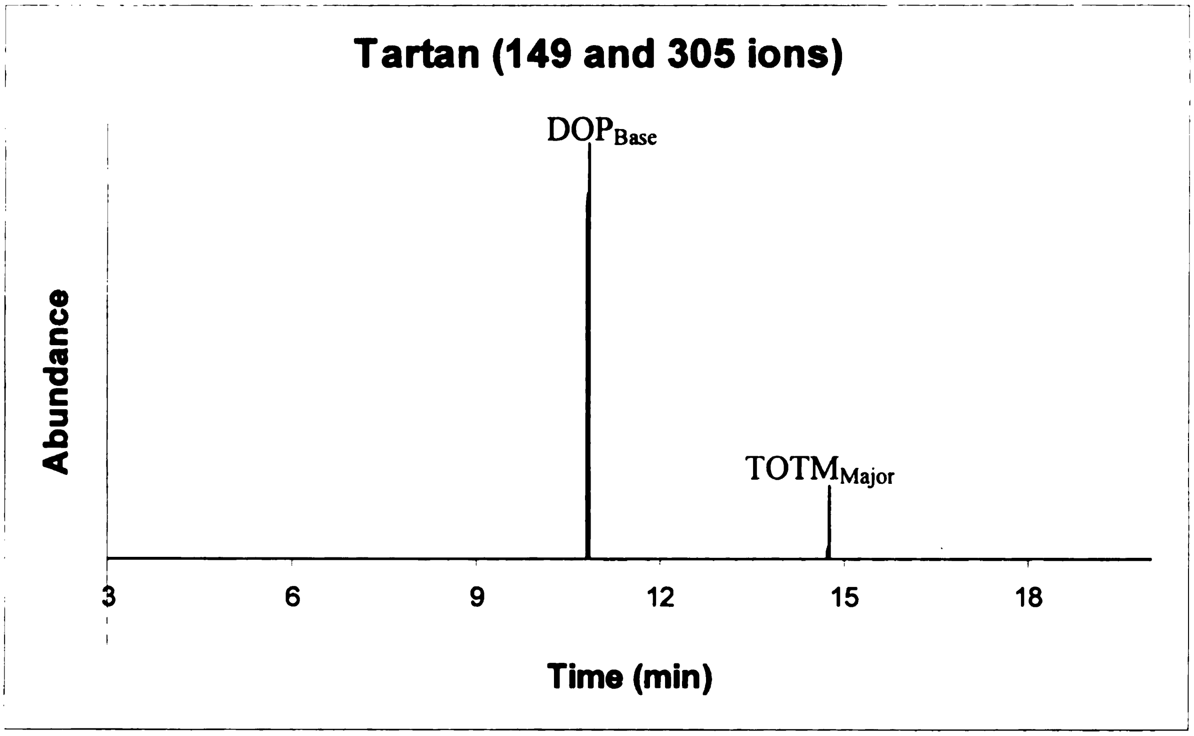


Figure 23: Extracted ion chromatograms of Tartan tape

Overall, HT-GC-MS has proven to be a very effective method for both comparison and identification of electrical tape plasticizers. Its high operating temperatures allow for better chromatography of high molecular weight species, such as plasticizers. In fact, the normal maximum temperature of a typical GC column is 325°C; the HT model reaches this temperature at 16 min of a 20 min program. As can be seen by the HT chromatograms presented here, there are at least two plasticizers that elute very close to this temperature (TOTM and the unidentified adipate). Also, with the injector at temperatures of 400°C and above, the initial vaporization of the plasticizers is improved from that in typical GC-MS.

3.6 Py-GC-MS

Pyrolysis is a well-known and established technique for the analysis of polymers.[6,8,9,10,11,12] However, the results of this study raise questions about its suitability for electrical tape analysis. Given that this particular product is a heterogeneous mixture of many different components, it is not surprising that any results obtained from the pyrolysis of electrical tape are difficult to interpret; in this case, inconsistent to the point of not being able to associate a fragment of tape to the roll from which it is known to have originated.

Figure 24 shows the pyrograms of two samples of electrical tape taken from the same roll of tape (Super 88 (2004)) and run sequentially on the same day. The labeled peaks represent cyclopentanone, a known pyrolysis product of adipates, and various forms of the intact adipates from the plasticizers in the tape. As is easily observed from the chromatograms, the ratio of these components to one another is not consistent, even in samples run sequentially. Also, the abundance of the adipates varies from one sample to another (different ratios of three large peaks labeled). This indicates that the pyrolysis of these specific components (i.e., the plasticizers) is not reproducible.

The cause for this variation is not yet completely understood but is believed to be the result of the plasticizers thermally desorbing from the tape at relatively low temperatures during instrument equilibration and initial pyrolysis. If this is the case, unless this process can be prevented or controlled, the pyrolysis of an electrical tape sample will never occur in exactly the same manner and/or produce the same components in the same abundances. With any method, the more variation observed within a single source, the less valuable the information obtained in making comparisons between questioned and known samples. Therefore, Py-GC-MS will not be considered in the remainder of this work for purposes of electrical tape differentiation.

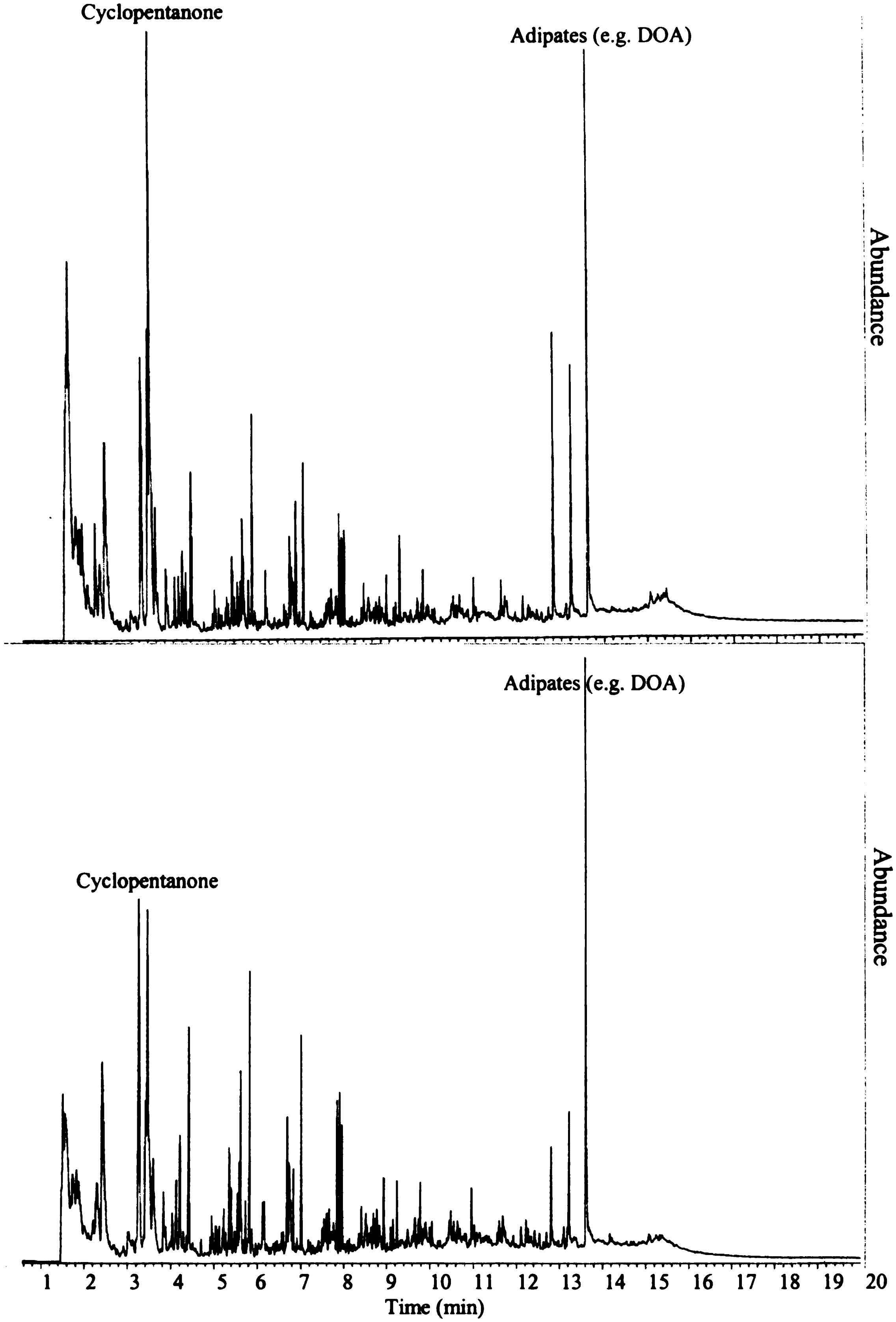


Figure 24: Pyrograms of two samples from the same roll of tape (Super 88 (2004)) analyzed on the same day

3.7 Comparison of Methods Using "H" Values

For this study, H can be computed for each individual method (ATR-IR, SEM-EDS (with and without FA), and HT-GC-MS) as well as for the combination of all three methods. All that is needed is the number of distinct classes generated by a method, D, as well as the total number of items in the population, I. Since several nominal brands are represented by multiple rolls, it is more applicable to define I in terms of brands of tape, 23 (including the "brands" that were created based on presence/absence of Pb in SEM-EDS), than in terms of rolls of tape. Therefore, if ATR-IR, SEM-EDS, and HT-GC-MS have D values of 10, 9, and 8, respectively, and I is set at 23 brands of tape, the H values for these methods are 0.43, 0.39, and 0.35, respectively; combining the three methods results in 18 distinguishable groups and an H value of 0.78. However, if FA is incorporated into SEM-EDS, the D value for this method increases to 18 and H for the method is now .78; H for the three methods (ATR-IR, SEM-EDS-FA, and HT-GC-MS) increases to .91 (D=21). It is apparent that using multiple, orthogonal, methods, including quantitation, provides greater differentiation of this sample population than any single method.

4. CONCLUSIONS AND FUTURE WORK

4.1 Conclusions

Ideally, the techniques employed in a forensic comparison scheme will be able to differentiate any two samples of a population that are truly different. In order to evaluate the success of a scheme at achieving this goal several issues must first be addressed. This study focuses on the forensic analysis of PVC electrical tape and attempts to determine the set of techniques that is most effective at distinguishing members of the electrical tape population. This is accomplished by researching and monitoring the electrical tape market, analyzing a representative population with multiple, orthogonal, methods, and quantitatively comparing the results.

By making contacts with an electrical tape manufacturer (3M) and product code companies (UL and UCC), valuable information about the past and current electrical tape market is obtained. This is that there were five major manufacturers of electrical tape in 2002, 3M discontinued the use of lead salts in its tapes in 2001, and that UL control, UL file, and UCC numbers can be used to indicate the manufacturer of a roll of electrical tape. All of this information helped in the collection of a population (thirty-eight rolls) representing at least seven manufacturers and twenty nominal brands of electrical tape. The sample collection also contained exemplar samples in order to study intra- and inter-batch variation.

A cursory physical examination of adhesive color was able to separate the population into those tapes with clear adhesive and those with black adhesive. ATR-IR analysis of the adhesive side of the tapes was able to differentiate each of these two groups and separate the population into ten distinct spectral classes with four brands of tapes individualized (Scotch 33, Champion, Lepage, and Duck). SEM-EDS analysis of the backing side divided the population into nine classes of different elemental composition with four nominal brands individualized (Bengal, Globe, Intertape, and Powerworks) and three nominal brands separated into those containing lead and those not (Frost King, Super 33+, and Super 88). Incorporating FA into the SEM-EDS analysis was able to separate the population into eighteen groups and individualize twelve brands (listed in Table 5). HT-GC-MS analysis of pentane plasticizer extracts resulted in eight classes based on the types and relative amounts of plasticizers present with three brands individualized (Commercial 700, Temflex 1700, and Scotch 33). Unlike some previous works, this study found that Py-GC-MS is not a very effective method for electrical tape analysis. The results obtained indicate that one of the most abundant components of electrical tape, plasticizers, can not be reproducibly pyrolyzed to the point of making confident associations between questioned and known fragments of electrical tape. The sample-to-sample and day-to-day variations are such that use of this method should be a last resort in electrical tape analysis.

Quantitation of the relative abilities of ATR-IR, SEM-EDS-FA, and HT-GC-MS to distinguish the population resulted in H values of 0.43, 0.78, and 0.35, respectively. The combination of all three of these methods increased this value to 0.91, supporting the idea that using multiple methods of analysis can increase differentiation within a population. Also, these values can help in developing an overall scheme for electrical tape analysis by using the methods with the highest H values (most discriminating) in the beginning of an analysis and the methods with the lowest H values (least discriminating) at the end of an analysis. However, the amount of sample preparation, time of analysis, and relative destructiveness of each method must also be taken into account. ATR-IR and SEM-EDS require very little, if any, sample preparation and are relatively quick analyses while HT-GC-MS necessitates a pentane extraction of the plasticizer and a twenty minute run on the instrument. With regards to destructiveness, HT-GC-MS causes the most damage to a sample, SEM-EDS causes limited changes, and ATR-IR is completely non-destructive.

Therefore, taking all of this into consideration, the results of this study indicate that although SEM-EDS-FA does provide the most discrimination, the most appropriate scheme for electrical tape analysis would start with ATR-IR analysis of the adhesive side due to its lack of sample destruction. After ATR-IR, SEM-EDS-FA analysis of the backing and HT-GC-MS analysis of plasticizer extracts, the most destructive method, can provide further differentiation if needed. A more detailed physical examination than was done in this study, which used only adhesive color, should be completed prior to any instrumental analyses. This would include width and thickness measurements and microscopic texture of the backing and adhesive.

Based on the FA results obtained with SEM-EDS data, it is probable that incorporating such an analysis into other methods can provide greater discrimination as well as allow for the correlation of data that was previously very difficult, it not impossible, to relate. Therefore, a more in depth look at factor analysis as a way to differentiate the electrical tape population is necessary.

Overall, both intra- and inter-brand variation in electrical tape composition was observed in this study while neither intra-batch (intra-roll) nor inter-batch variation was observed (excluding the initial Py-GC-MS results). However, given the large error bars for some of the exemplar rolls of tape in FA, there is the possibility that there is a small amount of intra- and inter-batch variation that could be detected under different parameters. If this is the case, once detected, the heterogeneity of the population is increased as is the significance of any association. Problems with this level of detection arise when different batches from different nominal brands start to overlap (error bars) and the observed variation would need to be tested using manufacturer exemplars.

Nineteen of twenty-three nominal brands of electrical tape were differentiated using instrumental and chemometric methods. Three nominal brands (Super 88, Super 33+, and Frost King) were separated into two groups based on the presence/absence of Pb in SEM-EDS. Super 33+ (2001) and Super 88 (2001) and Super 33+ (2004) and Super 88 (2004) are the only brands of tape that could not be distinguished by the methods used in this study.

Both of the Super 33+ brands and both of the Super 88 brands were manufactured by 3M (UL control #539H) and both have the same specifications (Table 2). However, the overall thickness of the tapes, as labeled, differs by 1.5mil (1mil = .001 inch) indicating that these two brands of tape are most likely composed of the same raw materials used in different amounts. Therefore, it is not necessarily a downfall of the analytical scheme that these two brands of tape could not be differentiated. In fact, even if thickness measurements were completed prior to any instrumental analyses, the results obtained would not be heavily relied on when making conclusions about possible origins of a questioned sample, given the nature of PVC (stretches and changes shape when used). So, stating that these two brands of tape are indistinguishable is a reasonable conclusion.

4.2 Future Work

The goals of this study were to develop an overall analytical scheme for the forensic analysis of electrical tape while addressing issues that are often ignored when completing population studies such as the one at hand. To date, a preliminary analytical scheme has been developed with instrumental techniques as its base and some important work has been done regarding an assessment of product codes and the manufacture and distribution of electrical tape. However, there is more work that can be done in addressing some issues that were not included in the original project because of either time constraints or having arisen during the course of the research.

Each step in the proposed scheme has the potential for improvement. Including width and thickness measurements and a more detailed examination of the backing and adhesive textures by both stereomicroscopy and SEM-EDS would ensure a complete physical examination prior to employing any instrumental techniques. Devising a method to isolate the adhesive and analyzing this portion via ATR-IR would elicit additional, possible very useful, information on the types of rubbers and tackifiers used in electrical tape. Adjusting the SEM-EDS parameters so as to increase detection of trace elements would reduce the ambiguity of elements with overlapping energies and provide more definitive results in this step of the analysis. Also, decreasing the magnification in SEM-EDS would analyze larger sections of the tape at once and perhaps eliminate some of the variation (micro-heterogeneity) in the elemental data obtained (error bars in FA.) Obtaining and running additional plasticizer standards on HT-GC-MS could aid in the identification of the adipate that elutes at 15.4 minutes in the 3M tapes. Also, continued chemometric analysis of the data obtained from each individual method can determine if such analysis is useful for further differentiation of the sample population. Comparing data from different techniques via chemometrics could be a way to objectively evaluate relationships within data that were previously undetected.

Just as important as improving the analytical methods used in this project is continuing to monitor the sample population for changes in composition and/or distribution so the developed scheme retains its usefulness. This involves making and maintaining contacts with companies that control these aspects of the electrical tape population. Also, through these relationships additional exemplars can be obtained and a more in depth study of inter- and intra- batch variation can be completed. One vital aspect of forensic analyses that was not examined in this work is that of the applicability of the scheme to case samples, not just samples collected for the study. To do this, both pre- and post-use samples of electrical tape must be analyzed so as not to assume that the chosen scheme is able to differentiate the used samples to the same extent as pristine samples. Changes in the structural and/or chemical integrity of the tape can occur from stretching and/or use in an exploding IED; studies analyzing these effects are warranted. Finally, due to known plasticizer leeching, it is of concern to the researchers of this project that storage conditions of electrical tape samples may affect the chemical composition of the samples and hinder forensic comparisons. Because of this, until further work is done to determine if this is indeed an issue, samples of electrical tape should not be stored in items made from materials containing large amounts of plasticizers (i.e., plastics).

APPENDIX

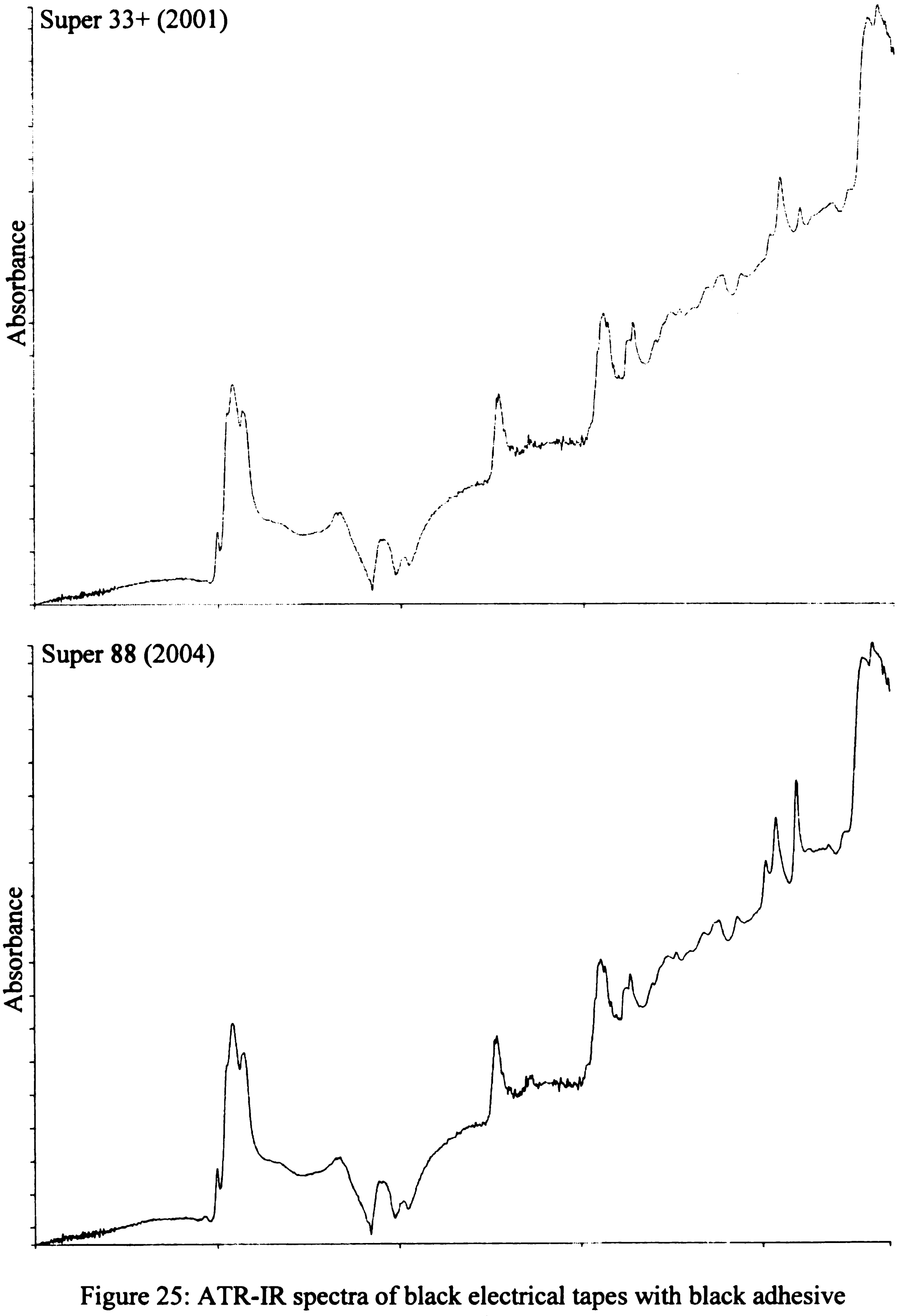


Figure 25: ATR-IR spectra of black electrical tapes with black adhesive (Super 33+ (2001) and Super 88 (2004))

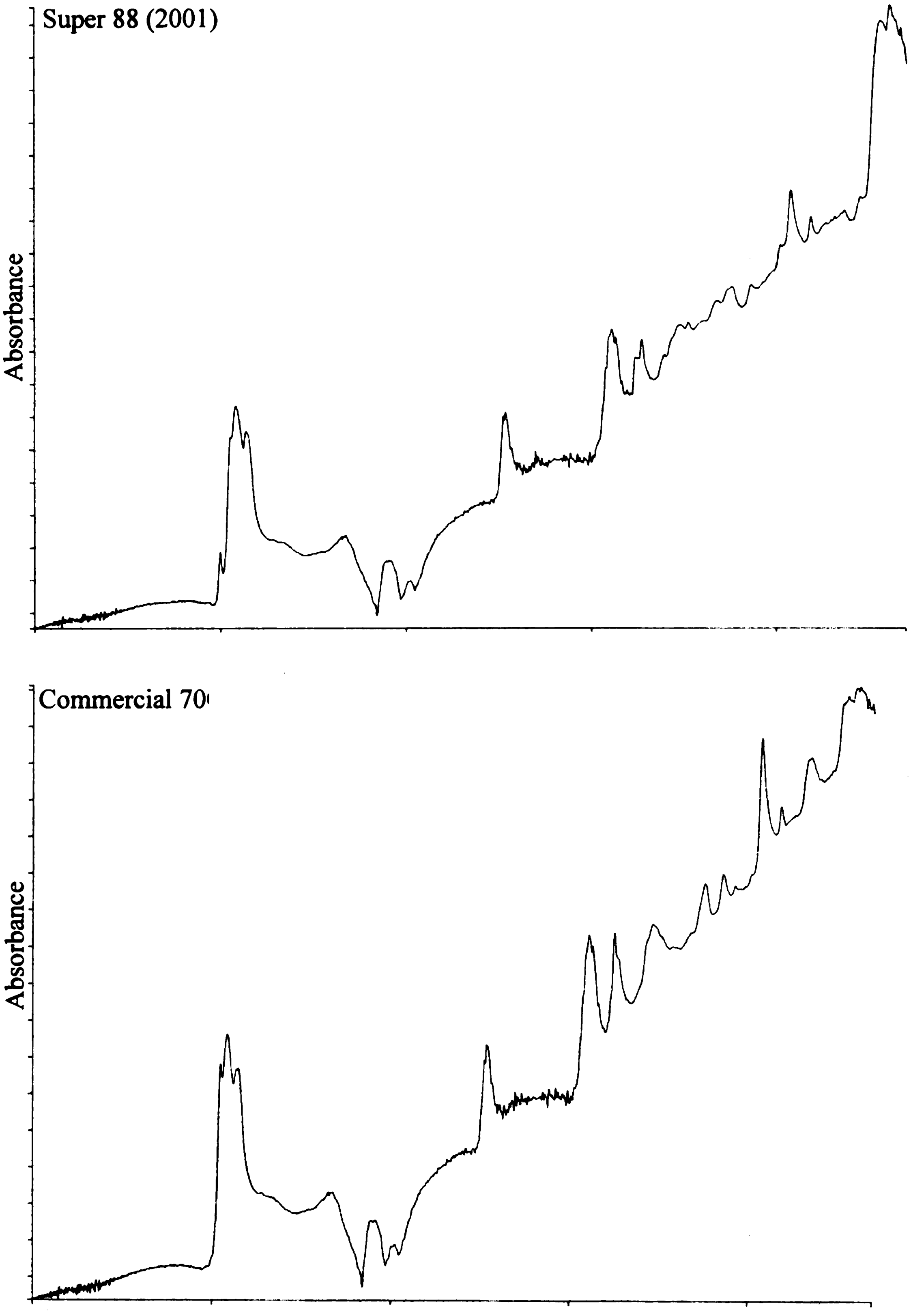


Figure 26: ATR-IR spectra of black electrical tapes with black adhesive (Super 88 (2001) and Commercial 700)

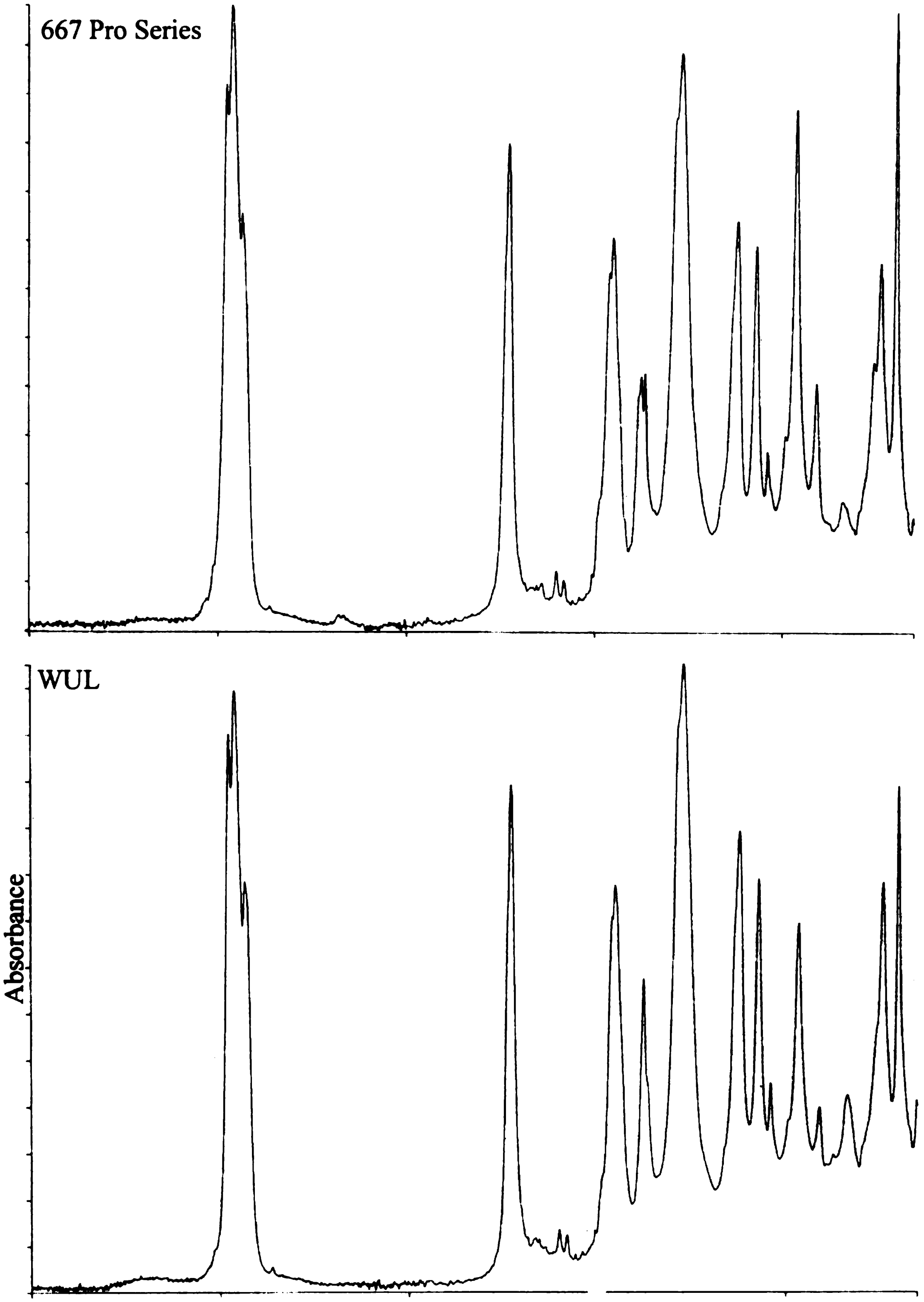


Figure 27: ATR-IR spectra of black electrical tapes with clear adhesive
(667 Pro Series and WUL)

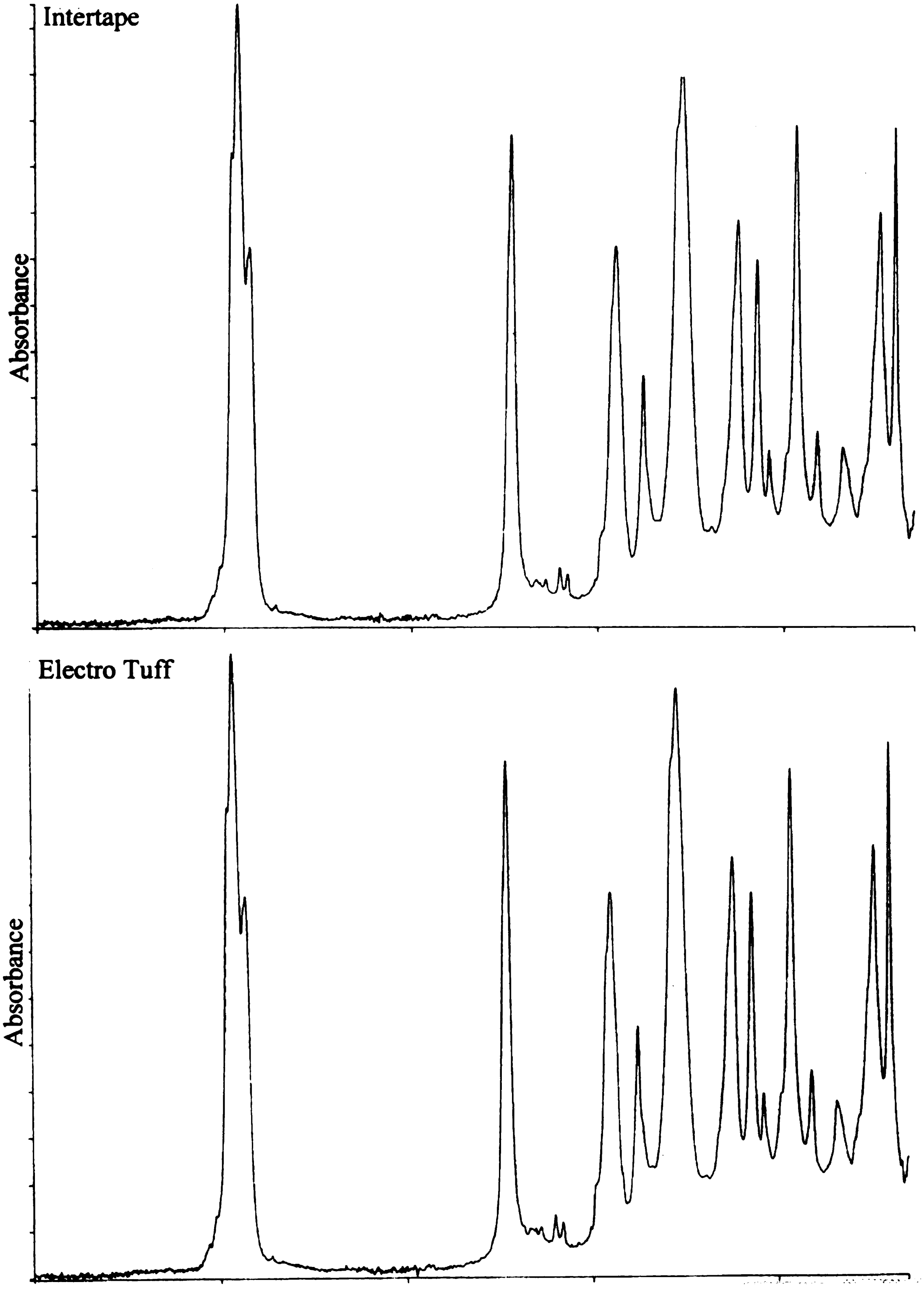


Figure 28: ATR-IR spectra of black electrical tapes with clear adhesive (Intertape and Electro Tuff)

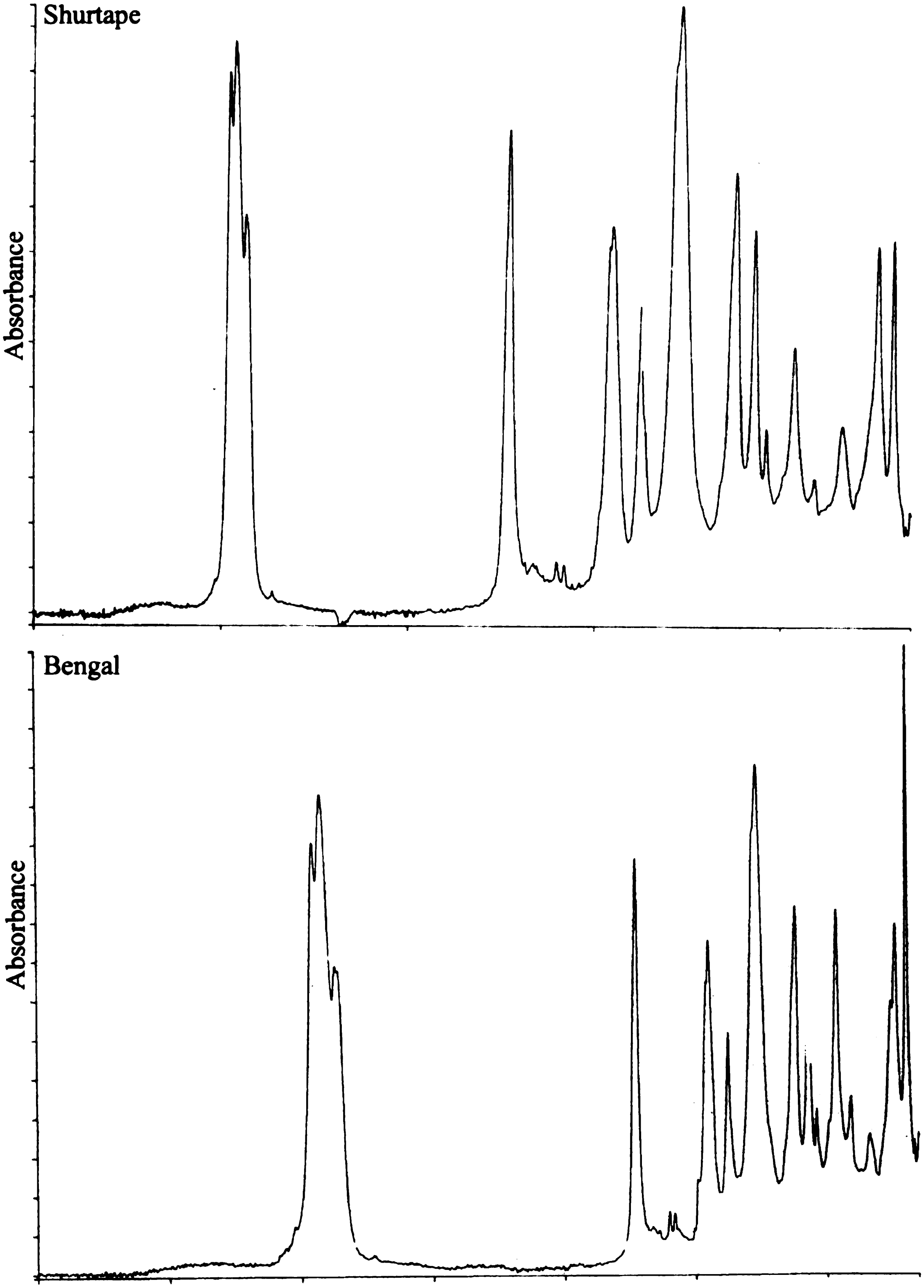


Figure 29: ATR-IR spectra of black electrical tapes with clear adhesive (Shurtape and Bengal)

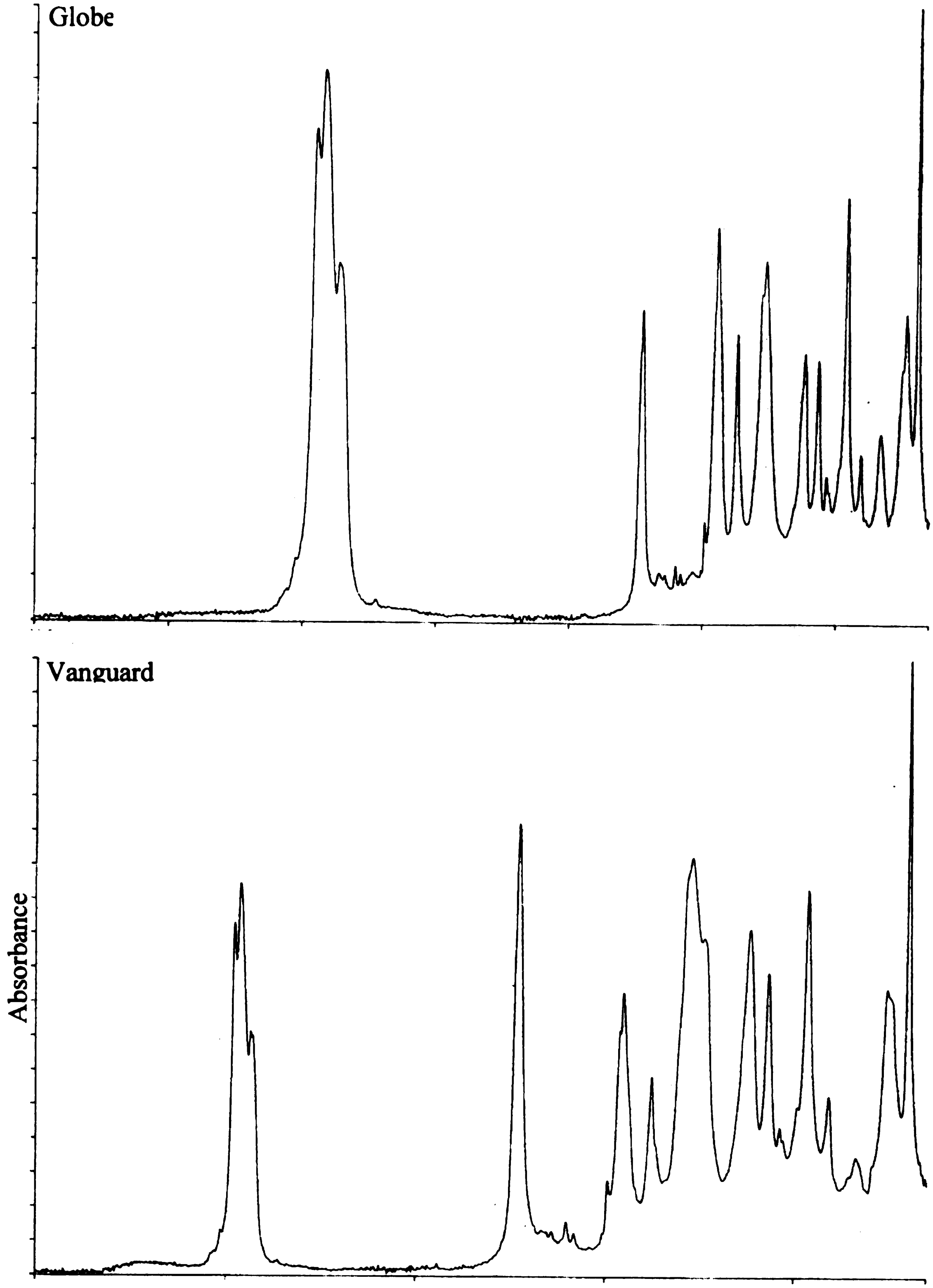
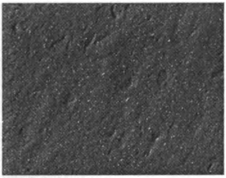
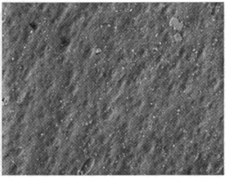
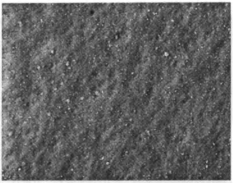


Figure 30: ATR-IR spectra of black electrical tapes with clear adhesive (Globe and Vanguard)


667 Pro Series


Champion


Duck


Globe

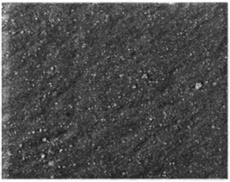
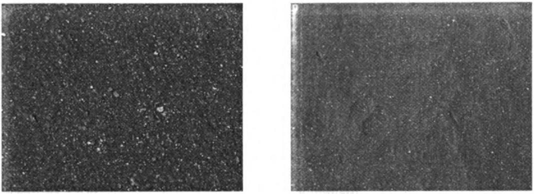
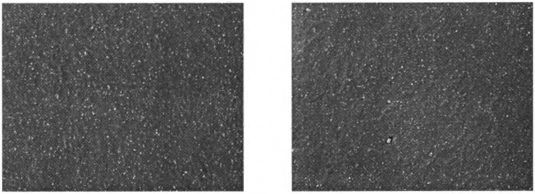

Intertape

Figure 31: Examples of the backing textures of black electrical tapes under 200X magnification



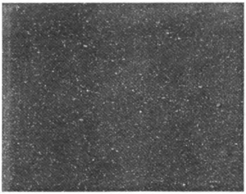
Lepage

Scotch 33



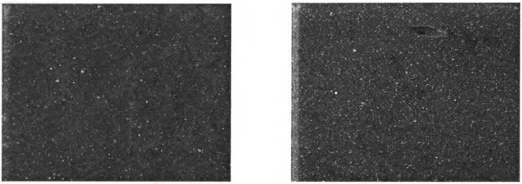
Super 33+

Super 88



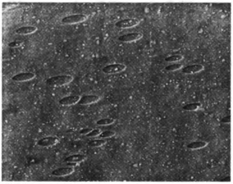
Tartan

Figure 32: Examples of backing textures of black electrical tapes under 200X magnification



Temflex 1700

Vanguard



WUL

Figure 33: Examples of the backing textures of black electrical tapes under 200X magnification

Sample	Element		
Temflex	**Mg**	**Sb**	**Ca**
A1	0.20	0.76	0.26
A2	0.16	0.76	0.28
A3	0.19	0.69	0.29
B1	0.23	0.64	0.26
B2	0.18	0.67	0.22
B3	0.17	0.49	0.22
C1	0.21	0.76	0.35
C2	0.21	0.72	0.19
C3	0.20	0.68	0.23
Average	0.19	0.69	0.26
Super 88 (2004)			
A1	0.25	1.52	0.52
A2	0.27	1.50	0.44
A3	0.28	1.69	0.38
B1	0.24	1.52	0.34
B2	0.21	1.53	0.33
B3	0.23	1.61	0.51
C1	0.23	1.62	0.48
C2	0.22	1.81	0.51
C3	0.24	1.82	0.60
Average	0.24	1.62	0.46
Super 33 + (2004)			
A1	0.28	1.13	0.25
A2	0.29	1.75	0.52
A3	0.26	1.72	0.44
B1	0.26	1.64	0.46
B2	0.25	1.55	0.38
B3	0.23	1.59	0.46
C1	0.26	1.46	0.42
C2	0.21	1.58	0.40
C3	0.27	1.47	0.74
Average	0.26	1.54	0.45

Figure 34: Weight percentage values of elements for electrical tapes with black adhesive

Sample	Element		
Super 33+ (2001)	**Sb**	**Ca**	**Pb**
1	1.31		1.56
2	1.51		2.14
3	1.50		1.85
Average	1.44		1.85
Super 88 (2001)			
A1	0.96	0.27	1.57
A2	0.82	0.22	1.58
A3	1.05	0.27	1.77
B1	1.06	0.26	1.80
B2	0.93	0.25	1.61
B3	1.09	0.25	1.76
Average	0.99	0.25	1.68
Scotch 33			
A1	0.62	0.10	1.74
A2	0.44	0.08	1.76
A3	0.69	0.10	1.85
B1	0.53	0.08	1.33
B2	0.50	0.09	1.50
B3	0.67	0.10	1.71
C1	0.74	0.07	1.74
C2	0.70	0.13	1.75
C3	0.81	0.09	1.69
D1	0.55	0.09	1.83
D2	0.64	0.09	1.90
D3	0.65	0.07	1.81
Average	0.62	0.09	1.76
700 Comm			
A1	1.49		1.12
A2	1.43		1.31
A3	1.23		1.13
B1	1.34		1.28
B2	1.13		1.23
B3	1.06		1.27
Average	1.28		1.22

Figure 35: Weight percentage values of elements for electrical tapes with black adhesive

Sample	Element				
Tartan1710	**Sb**	**Ca**	**Pb**	**S**	**Si**
A1	1.04		0.88		
A2	1.15		0.89		
A3	1.28		0.97		
B1	0.97		0.74		
B2	1.03		0.81		
B3	1.03		0.61		
Overall	1.08		0.82		
667 Pro					
1	1.07		0.69	0.14	
2	1.07		0.85	0.12	
3	1.07		0.91	0.14	
Average	1.07		0.82	0.13	
Bengal					
A1			0.44	0.10	
A2			0.50	0.12	
A3			0.30	0.12	
B1			0.61	0.15	
B2			0.60	0.11	
B3			0.58	0.10	
Overall			0.51	0.12	
Champion					
A1		2.82	1.38		
A2		2.94	1.18		
A3		2.77	1.09		
B1		2.70	1.06		
B2		2.64	1.33		
B3		2.83	1.03		
Overall		2.78	1.18		
Frost King					
A1		1.80	0.78		1.61
A2		1.67	1.11		1.61
A3		1.68	1.32		1.62
B1					1.76
B2					1.65
B3					1.68
Overall		1.72	1.07		1.66

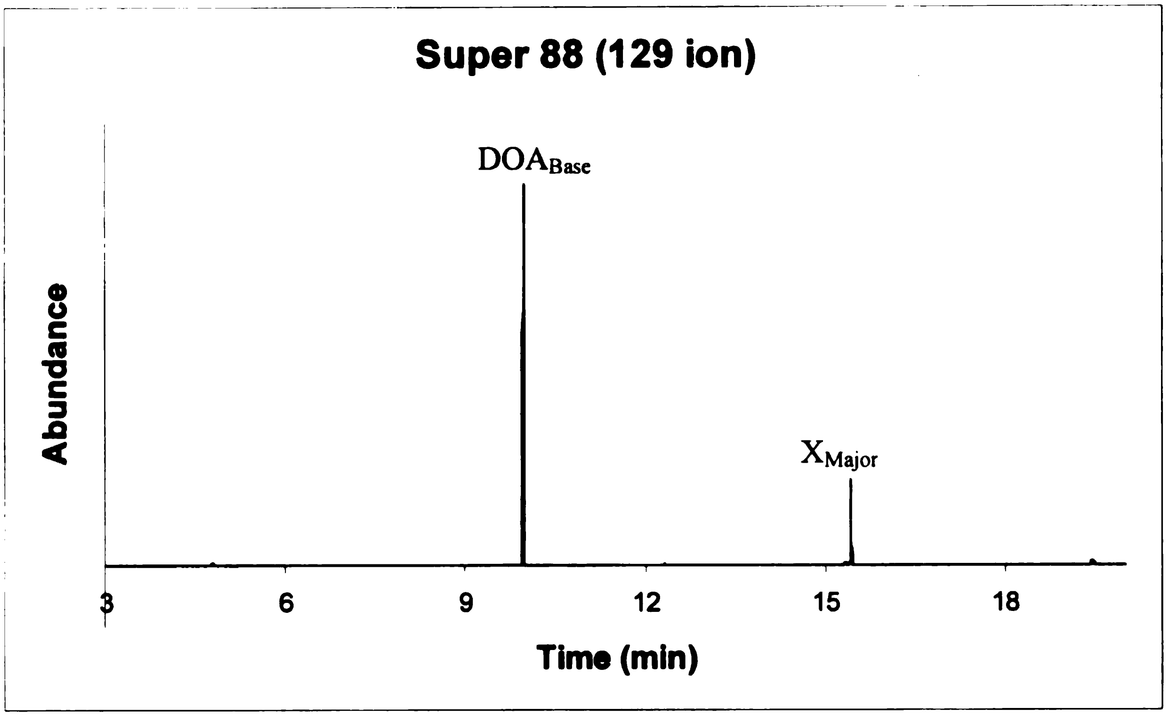
Figure 36: Weight percentage values of elements for electrical tapes with clear adhesive

Sample	Element					
Globe	Sb	Ca	Pb	S	Si	Ti
1			1.09		1.22	
2			1.11		1.28	
3			1.16		1.13	
Average			1.12		1.21	
Duck						
1		0.69	0.77		1.02	
2		0.81	0.77		1.03	
3		0.72	0.64		0.88	
Average		0.74	0.73		0.98	
InterBlack						
1		0.95			1.18	0.26
2		1.03			1.13	0.22
3		0.96			1.10	0.21
Average		0.98			1.14	0.23
LePage						
1	1.50		1.21		2.16	
2	1.80		1.55		2.30	
3	1.72		1.52		2.04	
Average	1.67		1.43		2.17	
Manco						
1		1.22			1.46	
2		1.06	0.53	0.13	1.38	
3		1.02	0.47	0.07	1.36	
Average		1.10	0.50	0.10	1.40	
PowerW						
1		0.82			1.15	
2		0.76			1.19	
3		0.81			1.15	
Average		0.80			1.16	
ShurTape						
1	1.54	1.15	0.56	0.11	1.41	
2	1.43	1.16	0.69	0.06	1.43	
3	1.31	1.22	0.49	0.07	1.36	
Average	1.43	1.18	0.58	0.08	1.40	
TuffTape						
1		0.89	1.29		1.02	
2		0.97	0.92		1.01	
3		0.88	1.14		1.03	
Average		0.91	1.12		1.02	

Figure 37: Weight percentage values of elements for electrical tapes with clear adhesive

Sample	**Element**				
Vanguard	**Sb**	**Ca**	**Pb**	**S**	**Si**
A1	1.17				
A2	1.19				
A3	1.04				
B1	1.14		0.42	0.11	
B2	1.01		0.49	0.06	
B3	1.00		0.39	0.07	
Overall	1.09		0.43	0.08	
WUL					
1		0.97	0.85	0.14	1.21
2		0.96	0.90	0.12	1.15
3		1.18	0.68	0.17	1.24
Average		1.04	0.81	0.14	1.20

Figure 38: Weight percentage values of elements for electrical tapes with clear adhesive



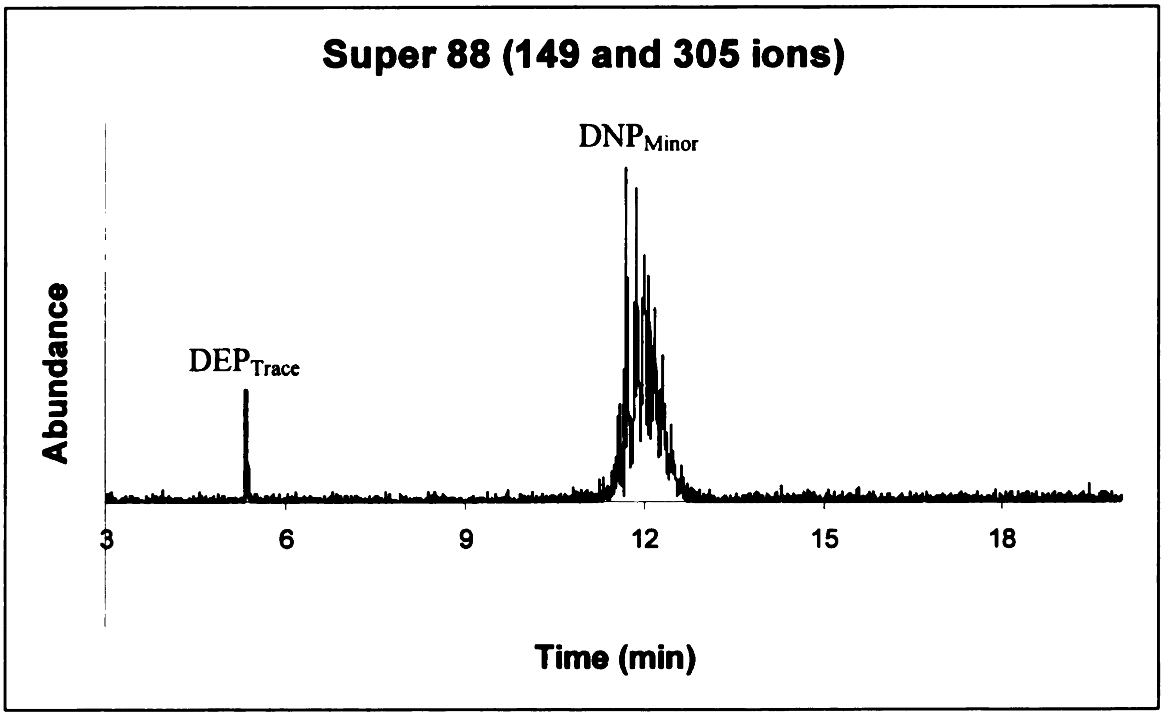


Figure 39: Extracted ion chromatograms of Super 88 tape

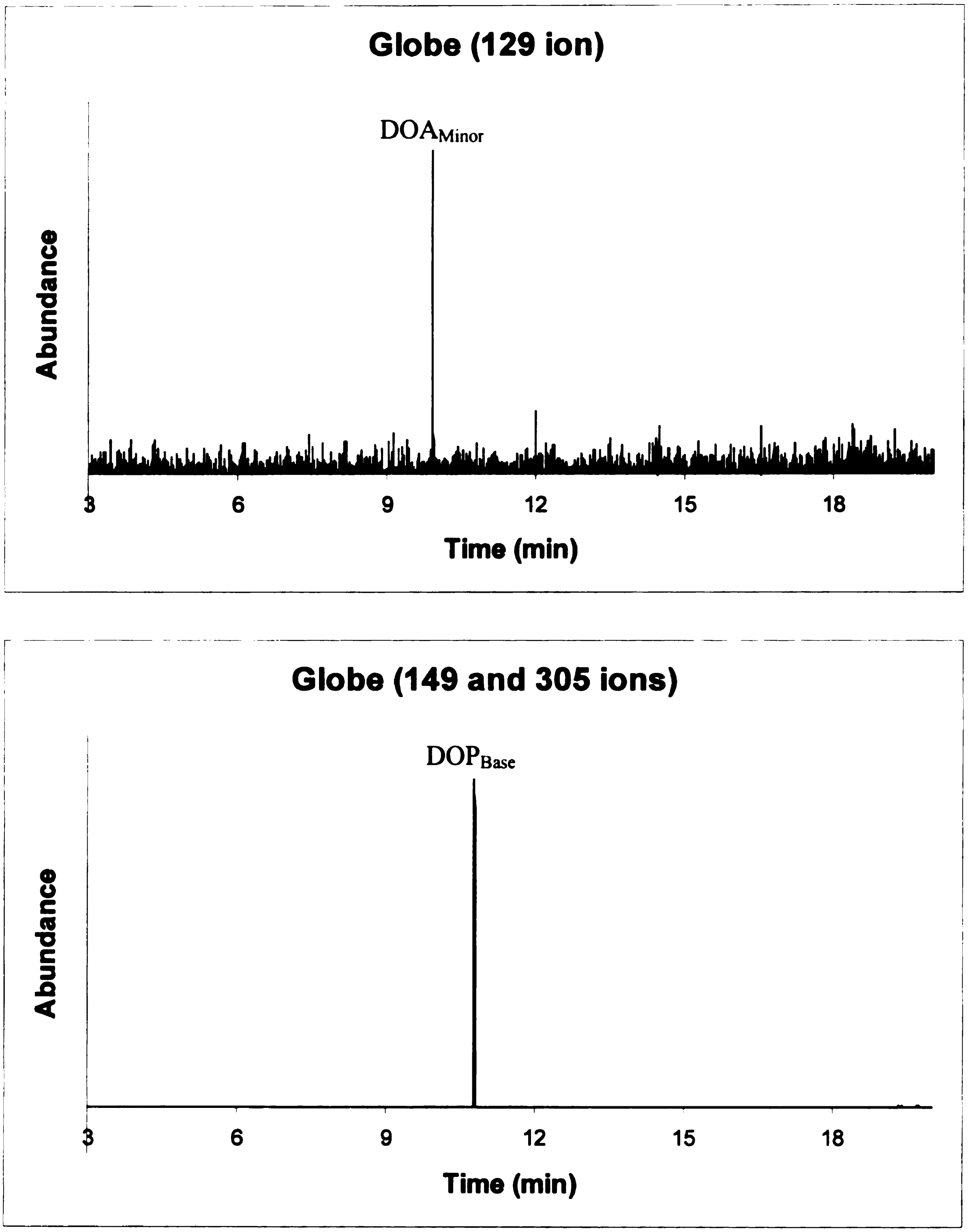


Figure 40: Extracted ion chromatograms of Globe tape

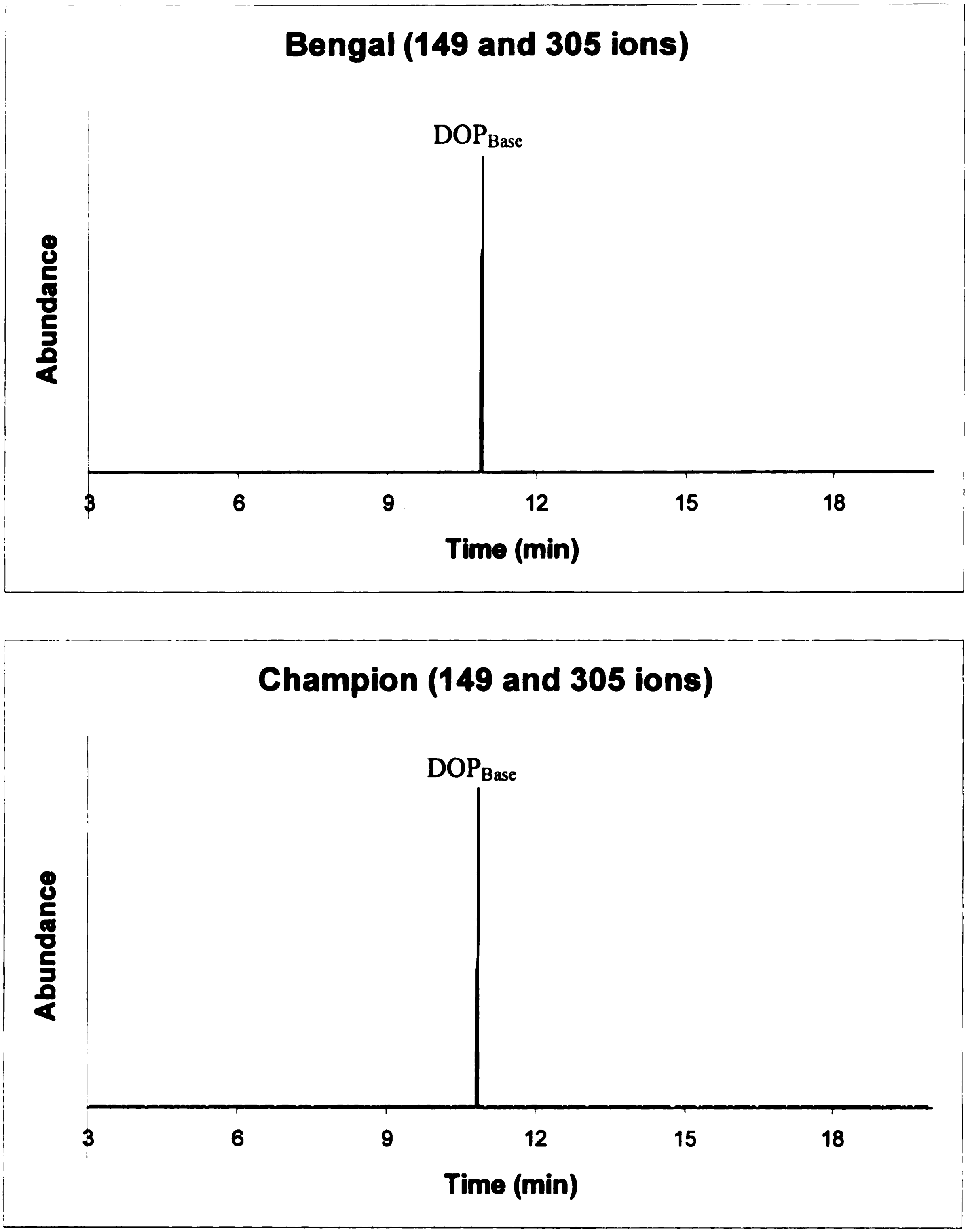


Figure 41: Extracted ion chromatograms of Bengal (top) and Champion (bottom) tapes

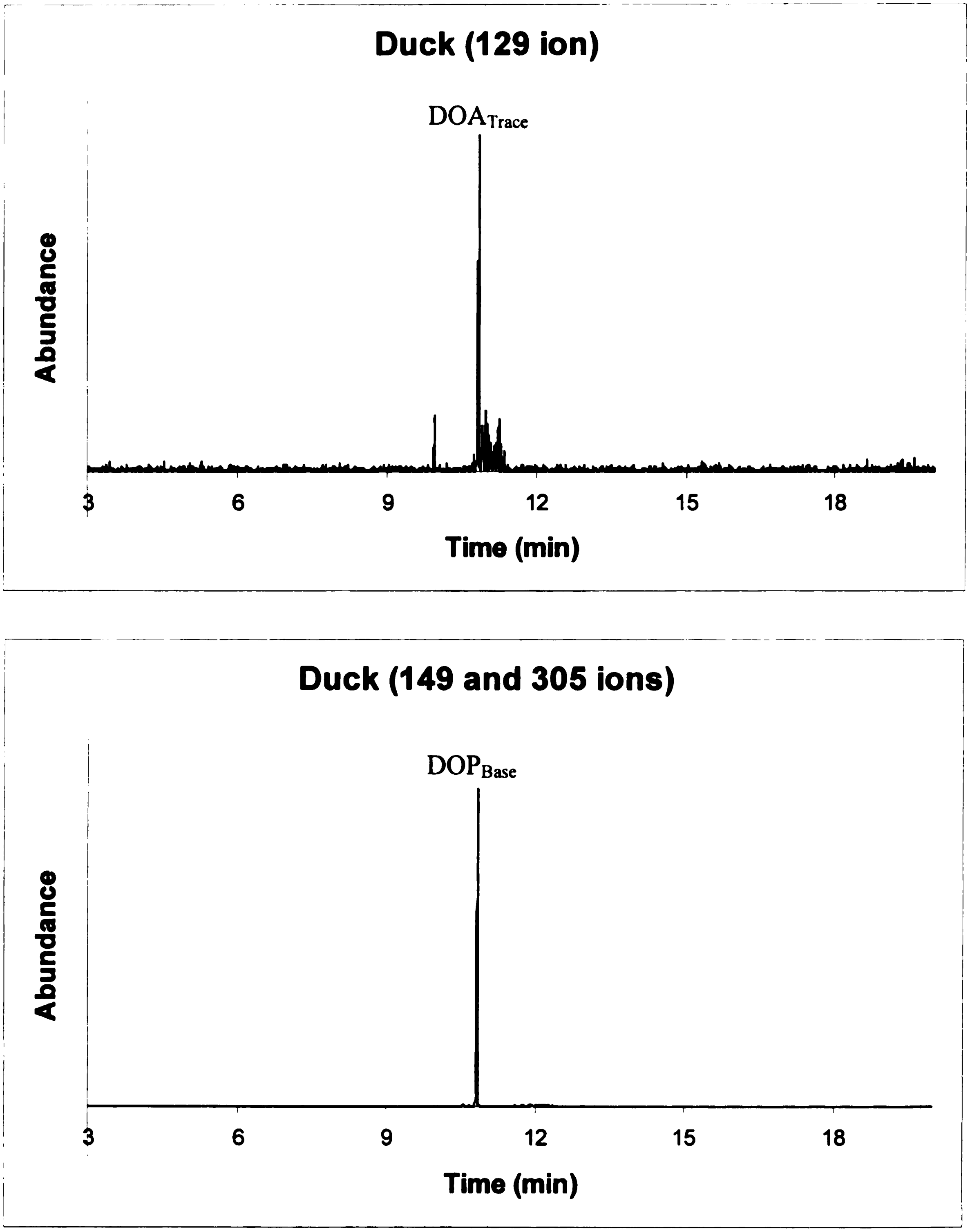
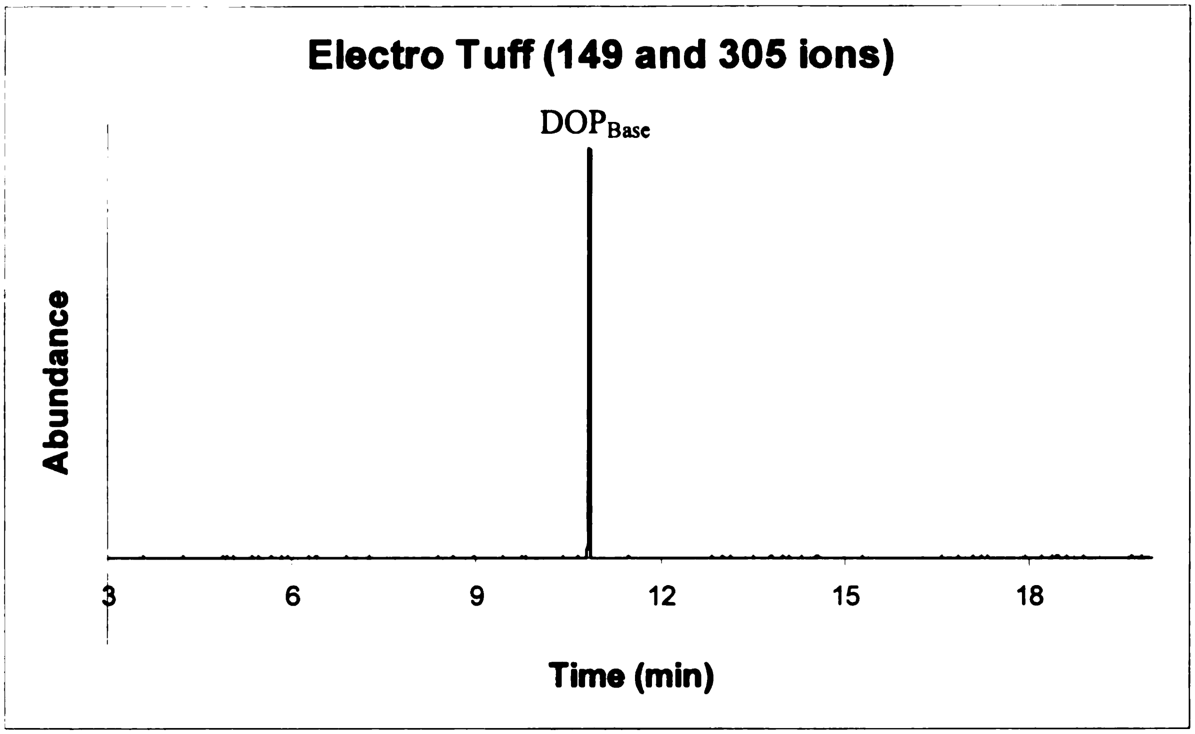


Figure 42: Extracted ion chromatograms of Duck tape



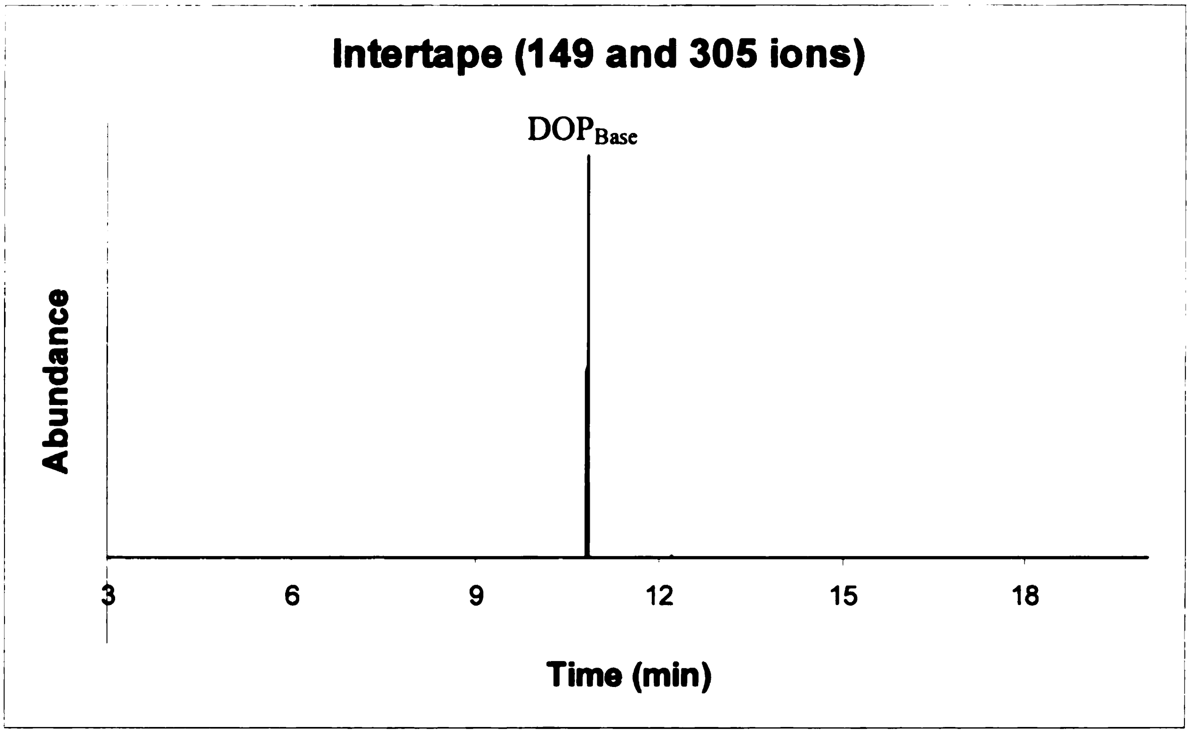
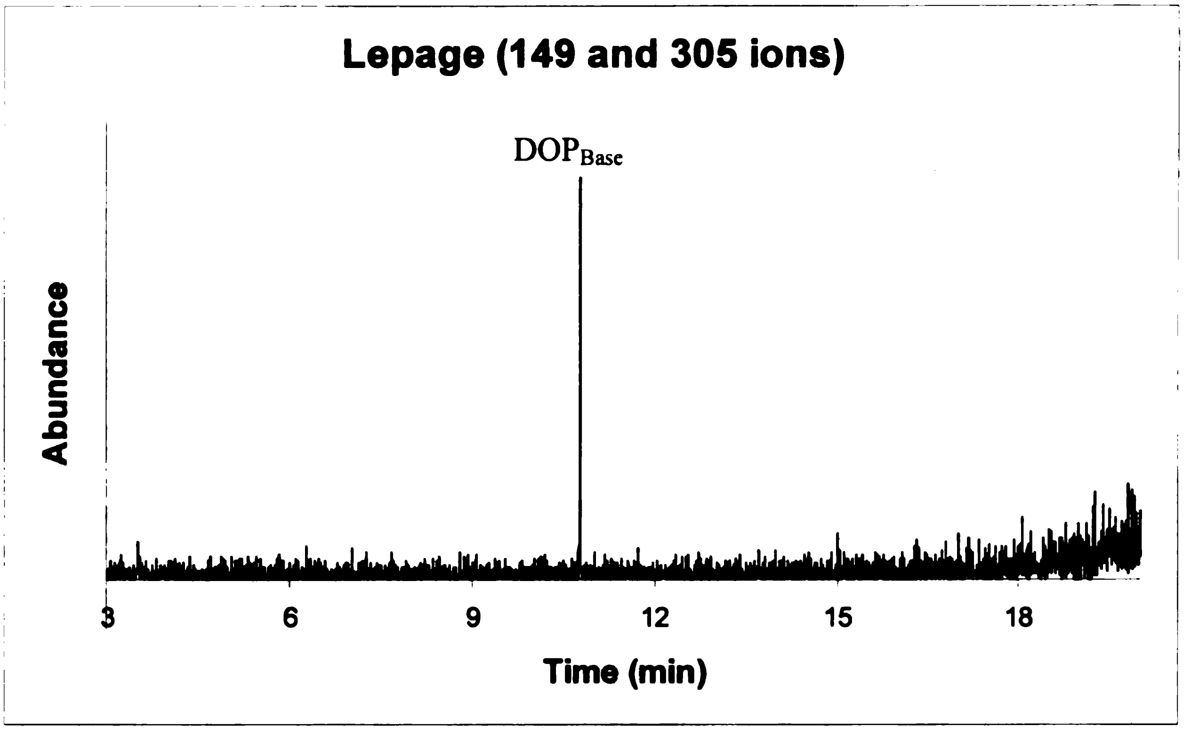


Figure 43: Extracted ion chromatograms of Electro Tuff (top) and Intertape (bottom) tapes



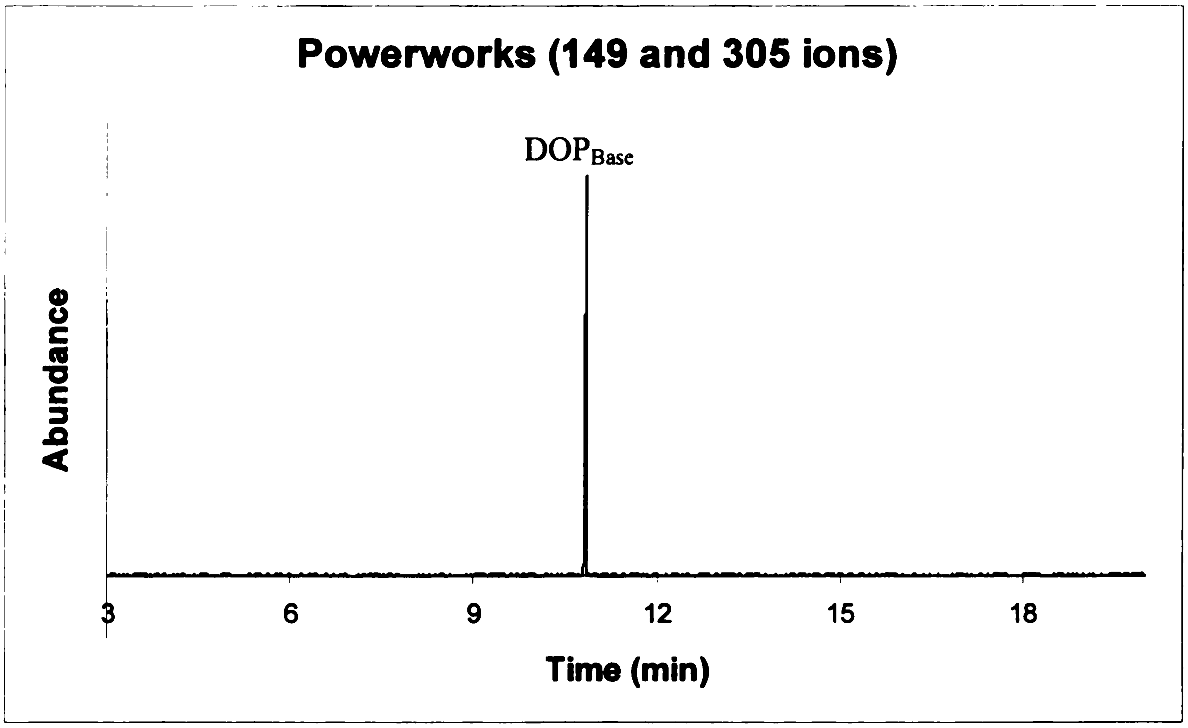
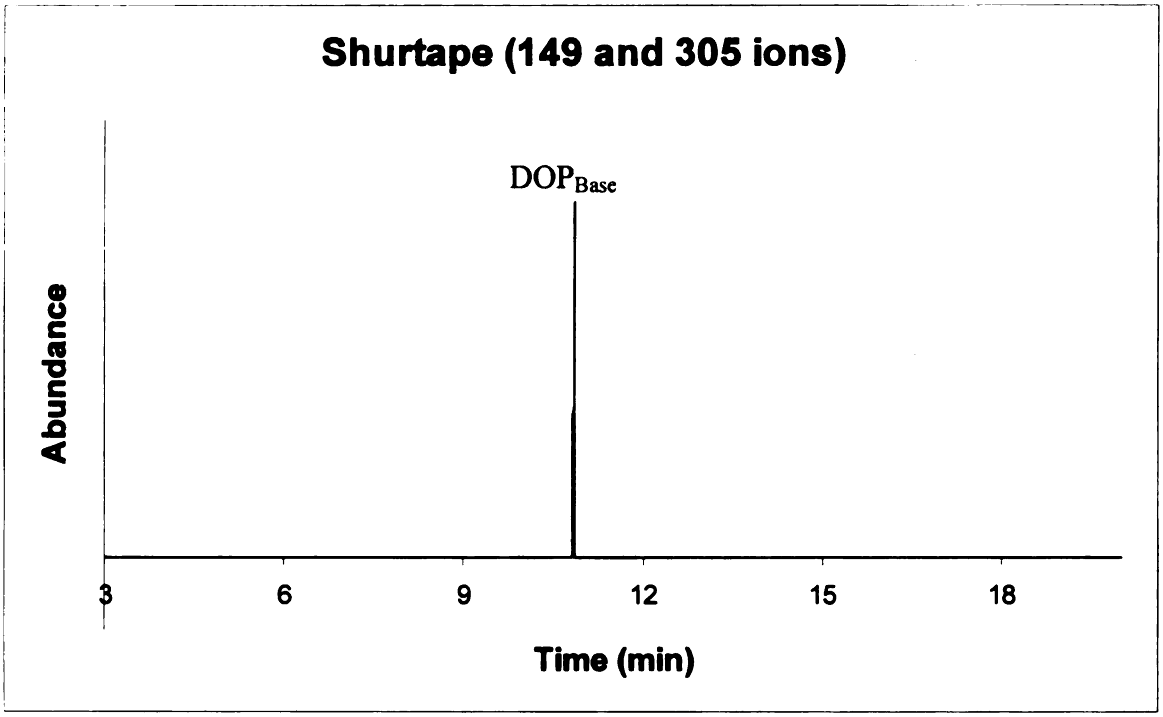


Figure 44: Extracted ion chromatograms of Lepage (top) and Powerworks (bottom) tapes



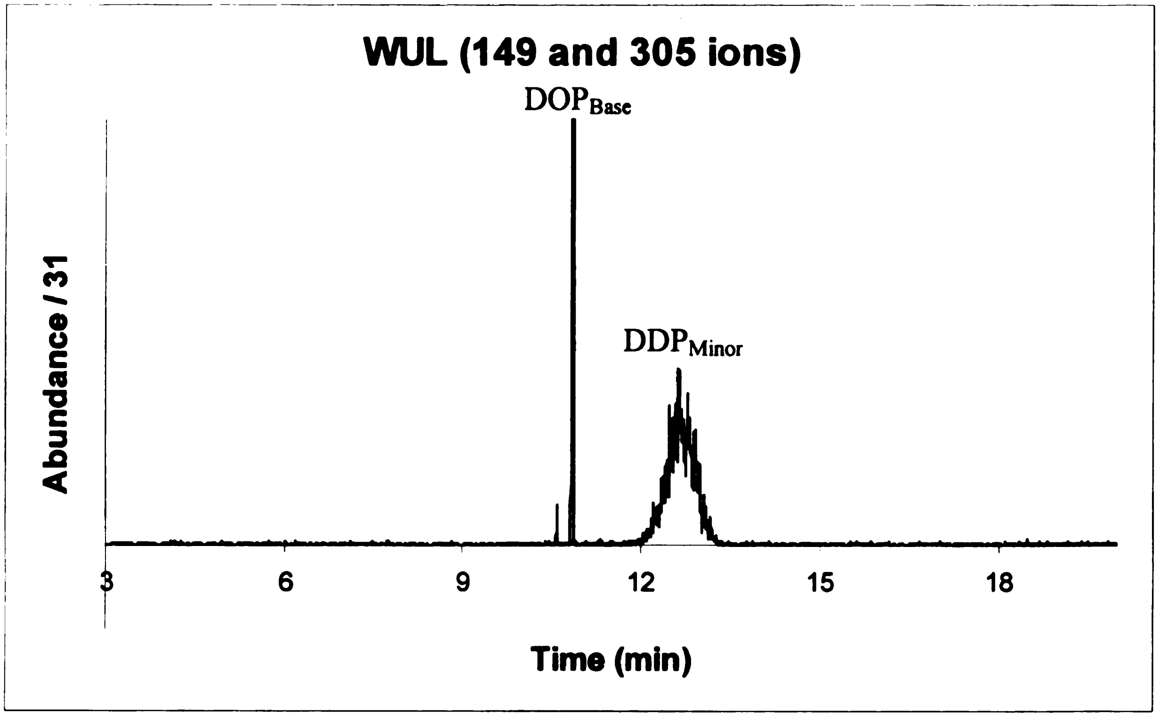
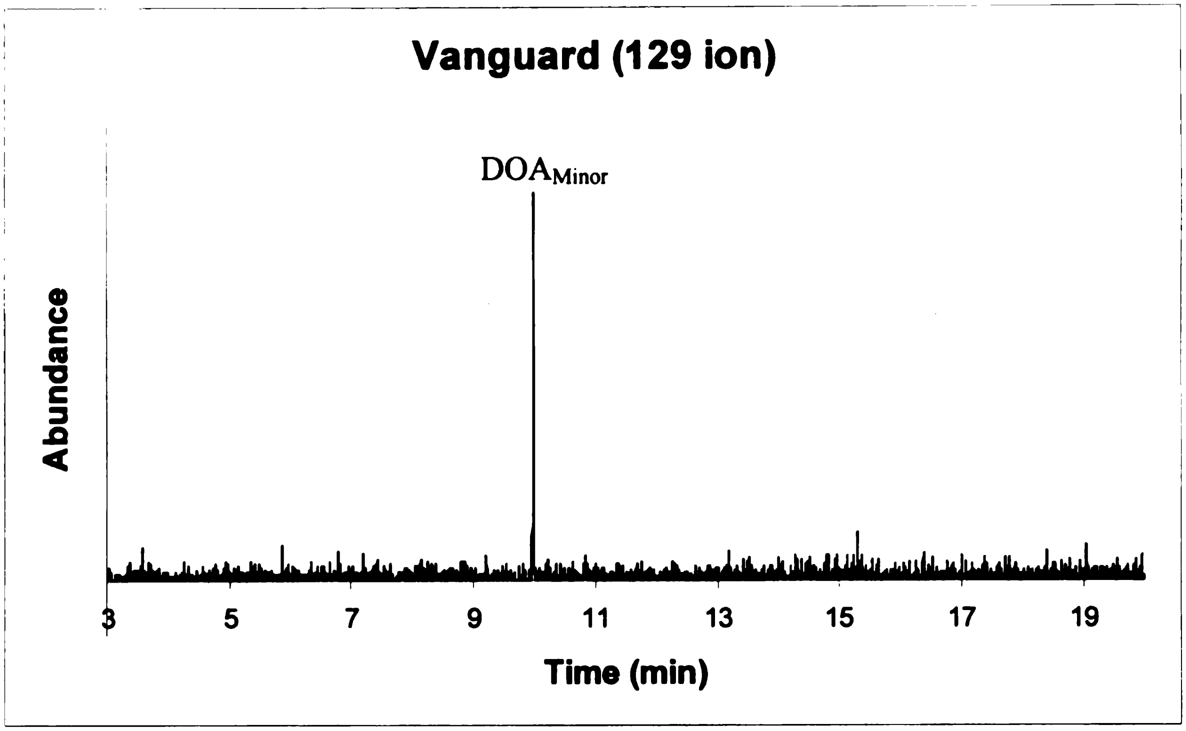


Figure 45: Extracted ion chromatograms of Shurtape (top) and WUL (bottom) tapes



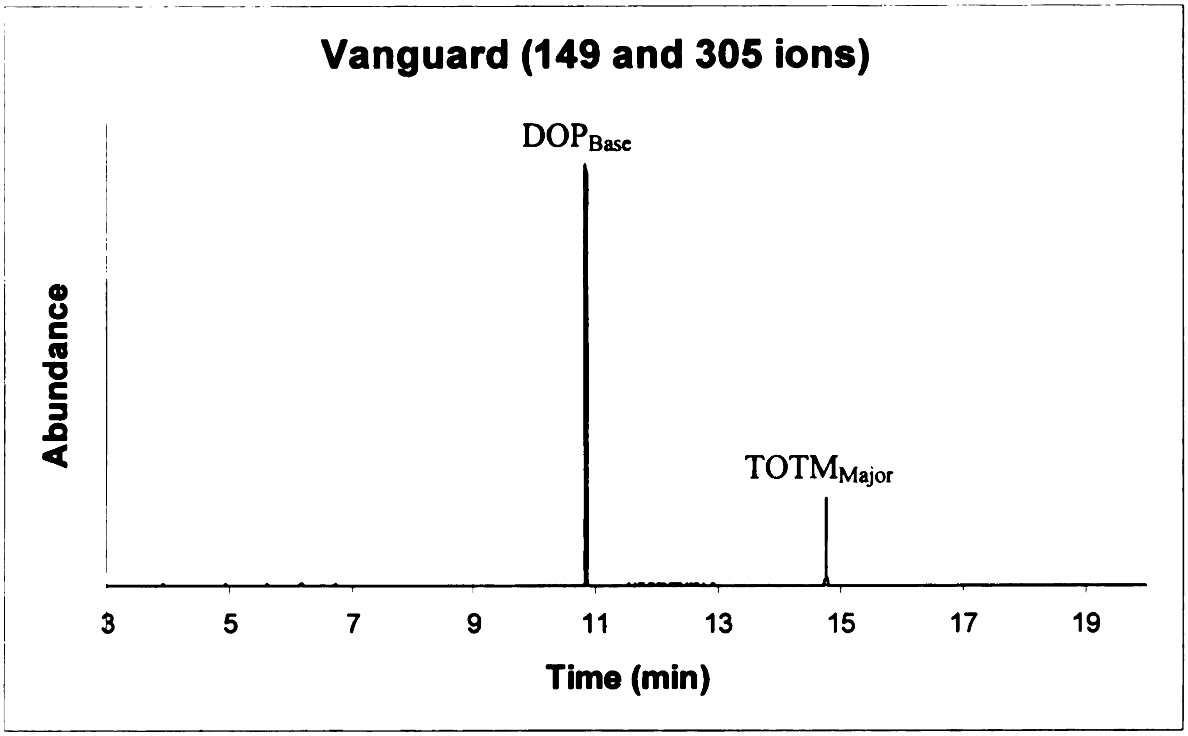


Figure 46: Extracted ion chromatograms of Vanguard tape

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6. REFERENCES

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