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DIFFERENTIATION OF SMOKELESS POWDERS USING FOURIER TRANSFORM INFRARED SPECTROPHOTOMETRY AND MORPHOLOGY

presented by

Melissa Dawn Felton

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DIFFERENTIATION OF SMOKELESS POWDERS USING FOURIER TRANSFORM INFRARED SPECTROPHOTOMETRY AND MORPHOLOGY

Ву

Melissa Dawn Felton

A THESIS

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

School of Criminal Justice

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ABSTRACT

DIFFERENTIATION OF SMOKELESS POWDERS USING FOURIER TRANSFORM INFRARED SPECTROPHOTOMETRY AND MORPHOLOGY

Ву

Melissa Dawn Felton

In this study, a combination of Fourier transform infrared spectrophotometry (FTIR) and morphology was used to differentiate between brands of smokeless powders. spectra and morphological characteristics such as shape, color, perforation, and size were obtained for each of 50 smokeless powders. In addition, a digital micrograph of each powder was taken using a scanning electron microscope. After analyzing all of the powders, the FTIR spectra, morphological data, and micrographs were compiled to create a reference manual. Three of the 50 smokeless powders examined were randomly selected by Christopher Bommarito, a forensic scientist with the Michigan State Police, and reanalyzed. Based on the results of these analyses, each of the three questioned powders was identified correctly. Because these questioned powders were randomly selected from the powders in the reference manual, the results are consistent within this database only. Therefore, the reference manual can be used for identification purposes as long as the questioned powder is one of the smokeless powders in the database.

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FTIR

GC —

GC-MS

GIN -

HPLC

IR —

КВr —

MCT -

NC —

NG —

PYR _

SEM _

UV/TEA

LIST OF ABBREVIATIONS

FTIR — Fourier transform infrared spectrophotometry

GC — gas chromatography

GC-MS — gas chromatography-mass spectrometry

GTN — glycerol trinitrate

HPLC — high performance liquid chromatography

IR — infrared

KBr — potassium bromide

MCT — mercury cadmium telluride

NC — nitrocellulose

NG — nitroglycerin

PMR — proton magnetic resonance

SEM — scanning electron microscope

UV/TEA — ultraviolet/thermal energy analyzer

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INTRODUCTION

Smokeless powders are propellants formulated and manufactured for use in firearms [7]. Specifically, they are used by firearms enthusiasts in reloading cartridge casings and shotgun shells. For this reason, smokeless powders are readily available for purchase at sporting goods stores.

Aside from their legitimate use in firearms, smokeless powders are sometimes used in the production of pipe bombs and other improvised explosive devices. Following an explosion, unburned particles of the smokeless powder may remain. Since forensic scientists occasionally encounter such particles, a method capable of differentiating between manufacturers of smokeless powders is desirable.

For instance, if a single brand of smokeless powder was used and the brand could be identified, then investigative efforts could be focused on suspects with that particular brand in their possession. Even if the brand could not be identified, the smokeless powder found at the crime scene could still be compared to smokeless powder(s) found in a suspect's possession.

In this study, FTIR spectra and morphological characteristics such as shape, color, perforation, and size were obtained for a variety of smokeless powders. In addition, a digital micrograph of each powder was taken using a scanning electron microscope. The spectra,

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morphological data, and micrographs were then compiled to create a reference manual.

Although analytical techniques such as morphology, pyrolysis gas chromatography, proton magnetic resonance, gas chromatography, gas chromatography-mass spectrometry, high performance liquid chromatography, and infrared spectrophotometry have been used to differentiate between brands of smokeless powders, no studies have combined FTIR with morphology. Furthermore, none of the previous studies on the differentiation of smokeless powders compiled the results into a reference manual.

For these reasons, research on the differentiation of smokeless powders by FTIR and morphology would benefit the forensic science community. Specifically, this research method would provide forensic scientists with yet another technique to use. Depending on the instrumentation available at a particular laboratory, this method might be more readily available than other methods.

In addition, the reference manual allows the forensic scientist to compare a questioned sample's FTIR spectrum and morphological characteristics to known samples in order to determine the brand of smokeless powder. Without this reference material for comparison, identification would not be possible.

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Smokeless Powders

Smokeless powders, the safest and most powerful of the low explosives, are grouped into three general categories including single, double, and triple-base [14]. Single and double-base smokeless powders are used in rifle and pistol cartridges, respectively [4, 10]. Triple-base powders are used in rockets and military ordinance [1]. For these reasons, single and double-base powders are encountered in the forensic science laboratory more often than triple-base powders.

The main constituent of single-base powders is nitrocellulose (NC) [1, 6, 12]. In addition to NC, double-base powders also contain nitroglycerin (NG) [14, 18]. The NG present in smokeless powders is known as glycerol trinitrate (GTN), whereas the NG in dynamite is a mixture of GTN and ethylene glycol dinitrate [4]. Triple-base powders are composed of NC, NG, and nitroguanidine [3].

In order to enhance the performance and/or add stability to the powder, the primary ingredients are usually combined with a variety of additional organic compounds [1, 4]. Specifically, these additives act as stabilizers, plasticizers, and flash inhibitors.

Stabilizers are added to prolong the storage life of the smokeless powders [1, 6]. Common stabilizers used are diphenylamine, ethyl centralite, and methyl centralite [4, 6-7]. Plasticizers, which are used to provide strengthened flexibility, include dibutyl phthalate, diethyl phthalate,

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dimethyl phthalate, and triacetin [10]. Dinitrotoluene and potassium sulfate are commonly found in smokeless powders as flash inhibitors, also known as burning modifiers [7, 10].

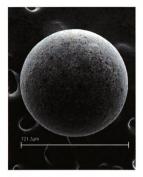
According to Meng and Caddy, graphite is also found in some propellants [10]. Used as a coating, the graphite prevents the accumulation of static electricity and acts as a surface lubricant to improve the flow properties of the powders.

Besides these commonly encountered additives, other organic compounds may be found in smokeless powders. A list of 23 such compounds was compiled by the FBI Laboratory [8]. These components are listed in Appendix A.

In addition to the different chemical compositions of smokeless powders, propellants can also be divided into categories based on physical characteristics [20]. In particular, physical characteristics such as size, shape, color, and perforation, or lack thereof, differ for various powders [5, 11]. By varying size, shape, and perforation, the burning rate of the smokeless powders can be adjusted.

Based on shape, smokeless powders can be divided into five basic categories including ball, cylinder, disk, flake, and flat ball [11]. An example of each shape is displayed in Figure 1. Color ranges from gray to black with some smokeless powders having central colored spots as well. For example, red and green central spots are present on Hercules Red Dot and Green Dot powders, respectively [20].

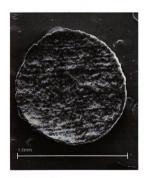
Perforation, or lack thereof, can further distinguish



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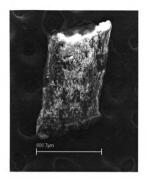
Ball powder.

Cylinder powder.



Disk powder.

Figure 1 — Five basic shapes of smokeless powders.



Flake powder.



Flat ball powder.

Figure 1 (cont'd).

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between different brands of disk and cylinder powders [3].

Figure 2 illustrates the difference between a perforated and non-perforated disk powder.

Due to the variety of chemical and physical characteristics present in propellants today, analysis of these characteristics can be used to differentiate between smokeless powders.

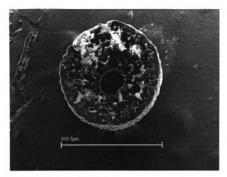
Fourier Transform Infrared Spectrophotometry

Fourier transform infrared spectrophotometry (FTIR) is an analytical technique used to measure the absorption of infrared (IR) light by a chemical substance [18]. Radiation in this region corresponds to the vibrational frequencies of molecules and includes the wavenumbers from 4000 to 690 cm⁻¹, where cm⁻¹ is the inverse of wavelength [15]. The relatively high cutoff at 690 cm⁻¹ is due to the detector used in this research, a narrow-band mercury cadmium telluride (MCT) detector.

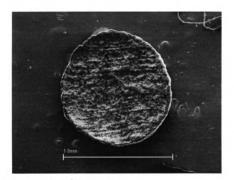
Instead of a diffraction grating, which is used in dispersive instruments, FTIR instruments utilize a Michelson interferometer. As seen in Figure 3, the basic components of a Michelson interferometer are an IR source, a beam splitter, a fixed and a moving mirror, and a detector [15].

A collimated beam from the IR source is directed into the interferometer where it strikes the beam splitter. This beam is then split into two collimated beams, with one beam travelling to a fixed mirror and the other beam travelling

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Perforated disk powder.



Non-perforated disk powder.

Figure 2 — Perforated and non-perforated disk powders.

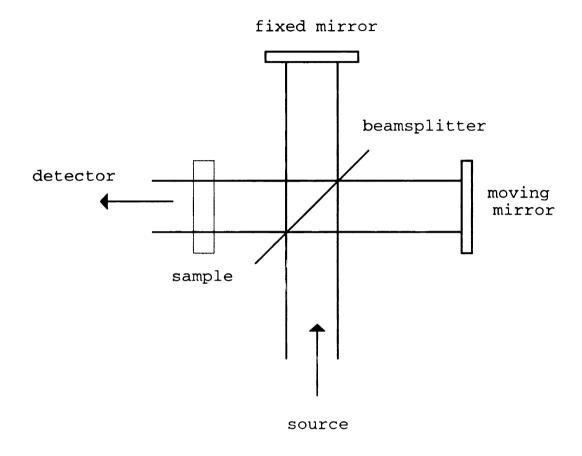


Figure 3 — Basic components of a Michelson interferometer.

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to a moving mirror. The two beams reflect off of the two mirrors and recombine at the beam splitter. The resulting beam then passes through the sample and on to the MCT detector. The signal from the detector is then processed by the computer and an FTIR spectrum, a plot of percentage transmittance versus wavenumber, results.

In addition to these basic components, some instruments also have an IR microscope attached in order to analyze extremely small samples. The sample to be analyzed is placed on a potassium bromide (KBr) window, which does not absorb in the mid-IR region [15]. The microscope is then used to view the sample and direct the IR beam through the specific area of the substance to be analyzed [2].

Using the FTIR instrument's data system, multiple spectral scans of the background are collected and averaged [17]. By averaging the scans, the signal to noise ratio is increased. Once the composite spectrum for the background is obtained, multiple scans of the background plus sample are collected and averaged. The Fourier transform function then subtracts the background's average transmittance, resulting in the transmittance spectrum of the sample [15]. Transmittance is the preferred method of sampling since spectra obtained using reflectance usually suffer poor reproducibility [2].

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REVIEW OF THE LITERATURE

A variety of analytical techniques have been used in an attempt to differentiate between manufacturers of smokeless powders. Specifically, past studies utilized methods including morphology, pyrolysis gas chromatography, proton magnetic resonance (PMR), gas chromatography (GC), gas chromatography-mass spectrometry (GC-MS), high performance liquid chromatography (HPLC), and infrared spectrophotometry to differentiate between brands of smokeless powders.

Although several studies used a combination of these methods to analyze smokeless powders, no studies were found that combined FTIR with morphology, as is being done in this research.

Physical characteristics alone were used to analyze 12 smokeless powders [20]. Although only a small number of samples were examined, particle size and shape positively identified each of the smokeless powders. Because such characteristics differed for the brands of propellants examined, morphology was sufficient for identification purposes.

Pyrolysis gas chromatography was used by Newlon and Booker in an effort to differentiate between smokeless powders [13]. Of the 40 different smokeless powders analyzed, each chromatogram could be distinguished from the others. In particular, significant detail such as the

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presence of peaks or the variation in relative peak areas of two or more peaks characterized each individual powder.

In 1989, Keto attempted to distinguish between smokeless powders using pyrolysis capillary gas chromatography [7]. Unlike Newlon and Booker's previous study, this research utilized statistical analysis to determine if pyrolysis gas chromatography is a reliable method for identification purposes. Following the analysis of four smokeless powders from each of three manufacturers, variations in relative peak areas between the different brands were seen. However, statistical analysis of the peak areas revealed that these differences were not significant. For this reason, Keto concluded that this technique has limited value for identifying the source of powder.

Meyers and Meyers achieved discrimination between smokeless powders through a combination of proton magnetic resonance and gas chromatography [11]. Although the use of PMR alone permitted discrimination between powders from different manufacturers, GC analysis also permitted discrimination between powders within a single manufacturer. Therefore, a combination of the two techniques provided the best method for differentiating between smokeless powders.

Capillary column gas chromatography-mass spectrometry enabled Martz and Lasswell to compare and identify smokeless powders [9]. Approximately 100 smokeless powder extracts were resolved into their organic components and identified by their mass spectra. By merging the spectra of the major

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peaks found in each extract, a composite spectrum for each powder was generated and used to build a spectral library. Because each of the spectra differed, identification of a smokeless powder could be accomplished by comparing a questioned powder's spectrum to those in the library through a computer search. A direct comparison of the spectra and physical properties of the smokeless powders could then confirm the computer's identification.

Bender analyzed 17 smokeless powder extracts using high performance liquid chromatography with ultraviolet/thermal energy analyzer (UV/TEA) detection [3]. Using this technique, seven major components of smokeless powders were separated and identified. Bender concluded that the relative quantities of these major components and the presence or absence of minor components could be used to discriminate between the powders. For this reason, HPLC with UV/TEA detection proved to be a valuable method for identifying brands of smokeless powders.

Infrared spectrophotometry followed by gas chromatography-mass spectrometry was capable of detecting all of the main constituents of single particles of smokeless powders [6]. The preliminary IR examination was used to identify nitrocellulose. Following this analysis, GC-MS was used to identify the additional components of the particles such as nitroglycerin as well as stabilizers, plasticizers, and burning modifiers. By combining these two

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techniques, individual particles of smokeless powders were positively identified.

In 1992, Andrasko used GC and HPLC to compare smokeless powder flakes recovered from around bullet holes on clothing to those from a particular cartridge or ammunition box found in a suspect's possession [1]. After analyzing more than 20 propellant samples from various manufacturers, Andrasko concluded that both GC and HPLC analyses could distinguish between smokeless powders from different manufacturers.

Because different powders showed qualitative differences in composition, single flakes were sufficient for distinguishing between different manufacturers.

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MATERIALS AND METHODS

Approximately 4 to 6 particles of smokeless powder were placed in a small piece of filter paper. About 8 to 10 drops of acetone were then added to the powder. Due to the high solubility of smokeless powders in acetone, this solvent successfully extracts the organic components from the particles [4, 16, 19].

The resulting liquid extract was collected on a microscope slide and allowed to evaporate to dryness. Following evaporation, the residue was scraped off with a scalpel and placed on the surface of a KBr window. The residue was then analyzed using FTIR with a microscope attachment.

The above procedure was used to analyze 50 smokeless powders that were obtained from the Michigan State Police Forensic Laboratory in East Lansing, Michigan. An alphabetical list of the powders analyzed is given in Table 1. The laboratory's FTIR instrument, a Perkin-Elmer Spectrum 1000 with AutoImage Microscope System (Version 3.1) was used to analyze all 50 powders.

Prior to each day's analyses, liquid nitrogen was added to cool the MCT detector. As previously noted, the lower wavenumber limit for the MCT detector was 690 cm⁻¹. Thirty minutes after the addition of liquid nitrogen, the energy levels of the instrument were monitored. The microscope's stage was then initialized and the aperture was calibrated.

Table 1 — Alphabetical list of smokeless powders analyzed.

```
Alcan AL-5
Alcan AL-7
Alcan AL-8
Alcan AL-120
Dupont #5 Pistol
Dupont #6 Pistol
Dupont 700X
Dupont 800X
Dupont Hi-Skor
Dupont IMR-3031
Dupont IMR-4064
Dupont IMR-4198
Dupont IMR-4227
Dupont IMR-4759
Dupont PB
Dupont SR-4756
Dupont SR-7625
Hercules 2400
Hercules Blue Dot
Hercules Bullseve
Hercules Green Dot
Hercules Herco
Hercules Hi-Vel-2
Hercules Red Dot
Hercules RL-11
Hodgdon H-110
Hodgdon H-322
Hodgdon H-335
Hodgdon H-375
Hodgdon H-380
Hodgdon H-4198
Hodgdon H-450
Hodgdon H-4831
Hodgdon H-4895
Hodgdon H-570
Hodgdon H-870
Hodgdon HS-5
Hodgdon HS-7
Hodgdon X-58
Norma N-200
Norma N-201
Norma N-204
Norma N-205
Winchester 296
Winchester 450-LS
Winchester 473-AA
Winchester 500-HS
Winchester 571
Winchester 760
Winchester 785
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Once calibration was completed, the smokeless powder residue was located in the field of view and the aperture was adjusted. By adjusting the aperture, the exact area of the sample to be analyzed was isolated [2].

After the area of the sample to be analyzed was selected, the transmittance mode was used to collect 32 scans of the background followed by 32 scans of the background plus the smokeless powder residue. The computer then ratioed the spectra in order to obtain a spectrum of the smokeless powder. Following analysis, each of the spectra was saved to the hard drive. The FTIR spectra were then compiled into a spectral library of smokeless powders.

A digital micrograph of one particle of each smokeless powder was then taken using a LEO 435VP scanning electron microscope (SEM). After each image was captured, it was annotated with the particle's size. This was done using the software's measurement tools. Once annotated, the images were saved to the hard drive.

Besides measuring one particle of each smokeless powder using the SEM's measurement tools, ten individual particles of a randomly selected powder (Hercules Bullseye) were also measured (Table 2). In addition, the standard deviation of the particle size data was calculated. The purpose of such measurements was to determine whether the size of a smokeless powder varies considerably within a batch.

In addition to the size characteristics, other morphological characteristics for each smokeless powder were

Table 2 — Particle size data for Hercules Bullseye.

PARTICLE	SIZE
1	838 µm
2	829 µm
3	830 µm
4	848 µm
5	834 μm
6	824 µm
7	844 µm
8	826 µm
9	815 µm
10	852 μm

The population standard deviation is calculated by taking the square root of the mean of the squares of the deviations from the arithmetic mean of the distribution.

$$\sigma = \sqrt{\sigma^2} = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \mu)^2}{N}} = \sqrt{\frac{1202}{10}} = 10.964 \ \mu \text{m}$$

 μ = population mean (834 μ m) N = number of samples (10)

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noted and the observations recorded. Specifically, the shape (ball, cylinder, disk, flake, or flat ball), color, and perforation, or lack thereof, were recorded. A complete list of all of the morphological characteristics is given in Table 3.

Using a series of cutting and pasting techniques, the micrograph and FTIR spectrum of each powder were printed along with the corresponding morphological characteristics. The data were then compiled to create a reference manual (Appendix B).

Once the reference manual was created, three of the 50 smokeless powders were randomly selected and reanalyzed. The actual results of the three powders, which were randomly selected by Christopher Bommarito, a forensic scientist with the Michigan State Police, were not revealed until the powders had been examined and identified.

The powders were analyzed using FTIR and morphology, as discussed previously. The results were then compared to those in the reference manual in an effort to identify the possible manufacturer(s) of the questioned smokeless powders. A diagram of the analytical scheme used for identifying smokeless powders is shown in Figure 4.

Table 3 — Morphological characteristics.

Table 3 — Morphological characteristics.					
NAME	SHAPE	COLOR	PERFORATED	SIZE	
Alcan AL-5	Flake	Black	No	607 μm	
Alcan AL-7	Flake	Black	No	611 µm	
Alcan AL-8	Flake	Gray	No	1.3 mm	
Alcan AL-120	Disk	Black	Yes	820 µm	
Dupont #5 Pistol	Disk	Gray	No	920 µm	
Dupont #6 Pistol	Flake	Black	No	1.1 mm	
Dupont 700X	Disk	Black	Yes	1.5 mm	
Dupont 800X	Disk	Black	No	1.8 mm	
Dupont Hi-Skor	Disk	Gray	Yes	849 µm	
Dupont IMR-3031	Cylinder	Gray	Yes	2.0 mm	
Dupont IMR-4064	Cylinder	Gray	Yes	2.1 mm	
Dupont IMR-4198	Cylinder	Gray	Yes	2.2 mm	
Dupont IMR-4227	Cylinder	Black	Yes	570 μm	
Dupont IMR-4759	Cylinder	Gray	Yes	1.4 mm	
Dupont PB	Disk	Black	Yes	805 μm	
Dupont SR-4756	Disk	Gray	No	1.1 mm	
Dupont SR-7625	Disk	Black	No	729 µm	
Hercules 2400	Disk	Gray	No	741 µm	
Hercules Blue Dot	Disk	Black	No	1.3 mm	
Hercules Bullseye	Disk	Gray	No	838 µm	
Hercules Green Dot	Disk	Black	Yes	1.3 mm	
Hercules Herco	Disk	Black	No	1.6 mm	
Hercules Hi-Vel-2	Cylinder	Gray	Yes	2.3 mm	
Hercules Red Dot Hercules RL-11	Disk Cylinder	Black	No Yes	1.6 mm	
Hodgdon H-110	Flat Ball	Gray Black	No	1.2 mm	
Hodgdon H-322	Cylinder	Black	Yes	566 μm	
Hodgdon H-335	Flat Ball		No	810 µm	
		Gray		653 μm	
Hodgdon H-375	Flat Ball	Gray	No	585 μm	
Hodgdon H-380	Ball	Black	No	636 µm	
Hodgdon H-4198	Cylinder	Gray	Yes	2.2 mm	
Hodgdon H-450	Flat Ball	Gray	No	929 µm	
Hodgdon H-4831	Cylinder	Gray	Yes	2.1 mm	
Hodgdon H-4895	Cylinder	Gray	Yes	1.2 mm	
Hodgdon H-570 Hodgdon H-870	Cylinder	Gray	Yes	2.1 mm	
	Ball	Black	No	721 µm	
Hodgdon HS-5	Flat Ball	Gray	No	529 μm	
Hodgdon HS-7	Flat Ball	Gray	No	917 μm	
Hodgdon X-58	Flat Ball	Gray	No	1.2 mm	
Norma N-200	Cylinder	Gray	No	1.0 mm	
Norma N-201	Cylinder	Gray	No	1.1 mm	
Norma N-204 Norma N-205	Cylinder	Gray	No	1.4 mm	
Winchester 296	Cylinder Flat Ball	Gray Black	No No	1.5 mm	
				573 μm	
Winchester 450-LS	Flat Ball	Gray	No	934 μm	
Winchester 473-AA	Flat Ball	Gray	No	594 µm	
Winchester 500-HS	Flat Ball	Gray	No	724 µm	
Winchester 571	Flat Ball	Gray	No	899 µm	
Winchester 760	Flat Ball	Black	No	809 µm	
Winchester 785	Flat Ball	Gray	No	959 µm	
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Figure

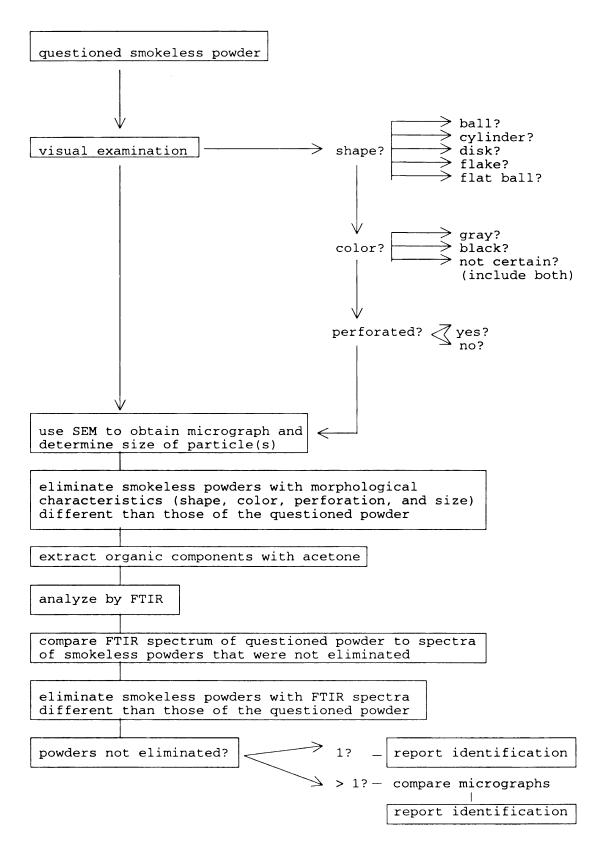


Figure 4 — Analytical scheme for identifying smokeless powders.

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RESULTS AND DISCUSSION

Because the shape of a smokeless powder is easy to discern and because the shape falls into one of five groups, this morphological characteristic was used initially to categorize the 50 smokeless powders. Based on shape, the 50 smokeless powders were divided into five groups — ball, cylinder, disk, flake, and flat ball (Table 4). Specifically, there were 2 ball powders, 16 cylinder powders, 14 disk powders, 4 flake powders, and 14 flat ball powders.

Each of the FTIR spectra within the groups was then analyzed and categorized. All 50 FTIR spectra displayed peaks at 1650, 1380, 1280, 1065, 835, and 750 cm⁻¹. For this reason, these peaks were not used to differentiate between the smokeless powders. It is important to note that variations in wavenumbers (cm⁻¹) may occur due to error margins for the peaks. Specifically, peaks may vary by as many as six wavenumbers.

Ball Powders

Both of the ball powders shared peaks at 1718, 1456, 1125, and 1001 cm⁻¹ (Figure 5). Hodgdon H-870 had additional peaks at 1024 and 915 cm⁻¹, whereas Hodgdon H-380 did not.

Tabl-SHAE Ball Cyl: Disk Flake Flat

Table 4 — Smokeless powders grouped according to shape.

	keless powders grouped according to shape.				
SHAPE	NAME				
Ball	Hodgdon H-380				
	Hodgdon H-870				
Cylinder	Dupont IMR-3031				
	Dupont IMR-4064				
	Dupont IMR-4198				
	Dupont IMR-4227				
	Dupont IMR-4759				
	Hercules Hi-Vel-2				
	Hercules RL-11				
	Hodgdon H-322				
	Hodgdon H-4198				
	Hodgdon H-4831				
	Hodgdon H-4895				
	Hodgdon H-570				
	Norma N-200				
	Norma N-201				
	Norma N-204				
	Norma N-205				
Disk	Alcan AL-120				
	Dupont #5 Pistol				
	Dupont 700X				
	Dupont 800X				
	Dupont Hi-Skor				
	Dupont PB				
	Dupont SR-4756				
	Dupont SR-7625				
	Hercules 2400				
· · · · · · · · · · · · · · · · · · ·	Hercules Blue Dot				
	Hercules Bullseye				
	Hercules Green Dot				
	Hercules Herco				
	Hercules Red Dot				
Flake	Alcan AL-5				
1 1 4 1 1 1	Alcan AL-7				
	Alcan AL-8				
	Dupont #6 Pistol				
Flat Ball	Hodgdon H-110				
	Hodgdon H-335				
	Hodgdon H-375				
	Hodgdon H-450				
	Hodgdon HS-5				
	Hodgdon HS-7				
	Hodgdon X-58				
	Winchester 296				
	Winchester 450-LS				
	Winchester 473-AA				
	Winchester 500-HS				
	Winchester 571				
	Winchester 760				
	Winchester 785				
	WINCHESCEL 703				

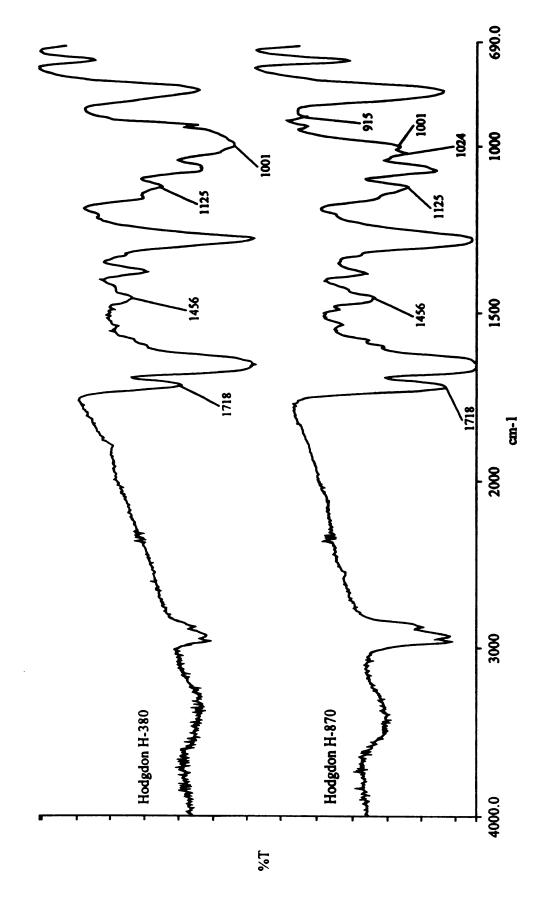


Figure 5 -- Ball powders.

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Cylinder Powders

Twelve of the sixteen cylinder powders displayed a peak at 1718 cm⁻¹ (Figures 6-9). Of these powders, three spectra had an additional peak at 1603 cm⁻¹ (Figure 6). Dupont IMR-4064 and IMR-4198 exhibited similar spectra and shared prominent peaks at 1531, 1348, 1204, 915, 791, and 732 cm⁻¹. Norma N-204 also had peaks in these regions. However, the peaks at 915, 791, and 732 cm⁻¹ were not as prominent as those in Dupont IMR-4064 and IMR-4198.

Three cylinder powders shared a peak at 1544 cm⁻¹ and displayed similar spectral characteristics (Figure 7). The remaining six powders with a peak at 1718 cm⁻¹ can be seen in Figures 8 and 9. Three of these powders had prominent peaks at 1593 and 1495 cm⁻¹ (Figure 8), whereas the other three powders did not (Figure 9).

Only four of the cylinder powders lacked a peak at 1718 cm⁻¹ (Figures 10 and 11). Of these four powders, Hodgdon H-4831 and H-570 exhibited similar spectra with peaks at 1603 and 1204 cm⁻¹ (Figure 10). In addition, these powders displayed the same prominent peaks as those in Dupont IMR-4064 and IMR-4198 (Figure 6). These peaks were not present in Dupont IMR-4759 and Hodgdon H-4895 (Figure 11). However, the spectra were similar to one another.

Disk Powders

All of the disk powders shared peaks at 1456, 1158, and 1119 cm⁻¹ (Figures 12-16). In addition, five powders had an

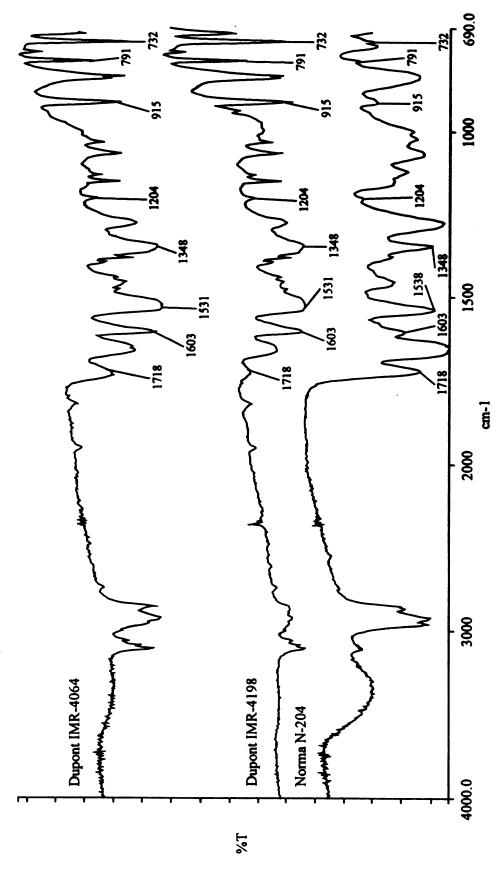


Figure 6 — Cylinder powders (A).

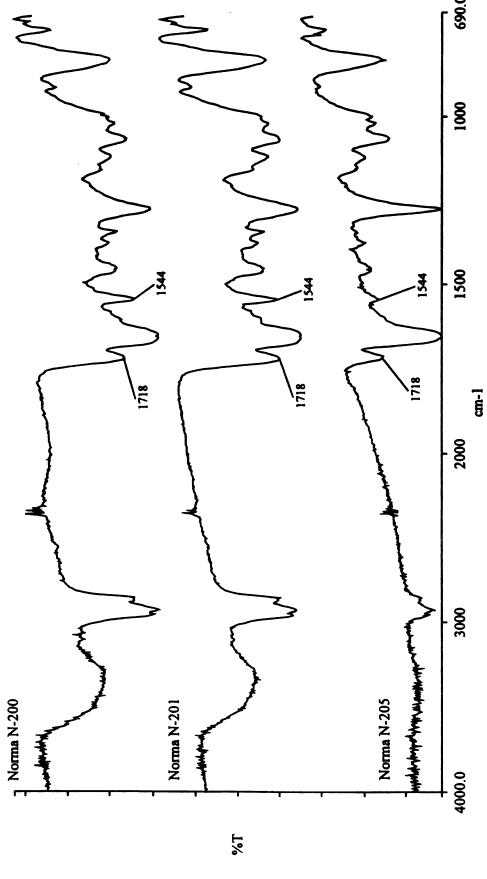


Figure 7 — Cylinder powders (B).

7 Hercules Hi. Vel. 7

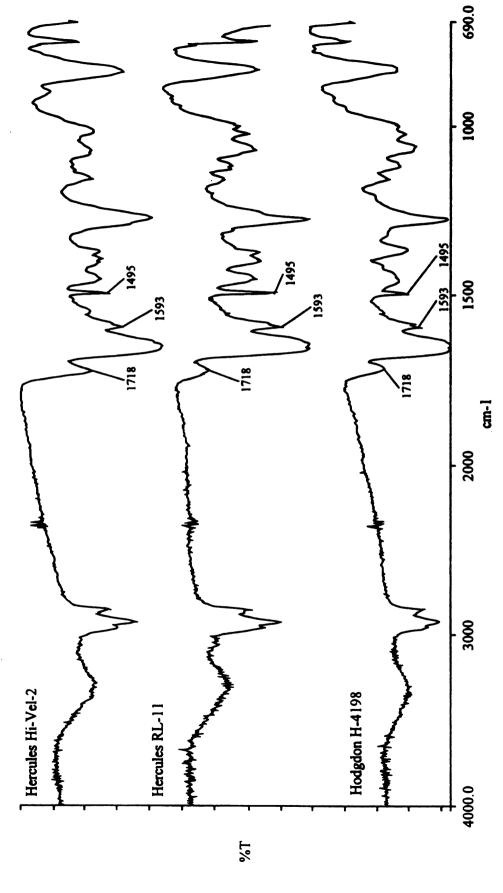


Figure 8 — Cylinder powders (C).

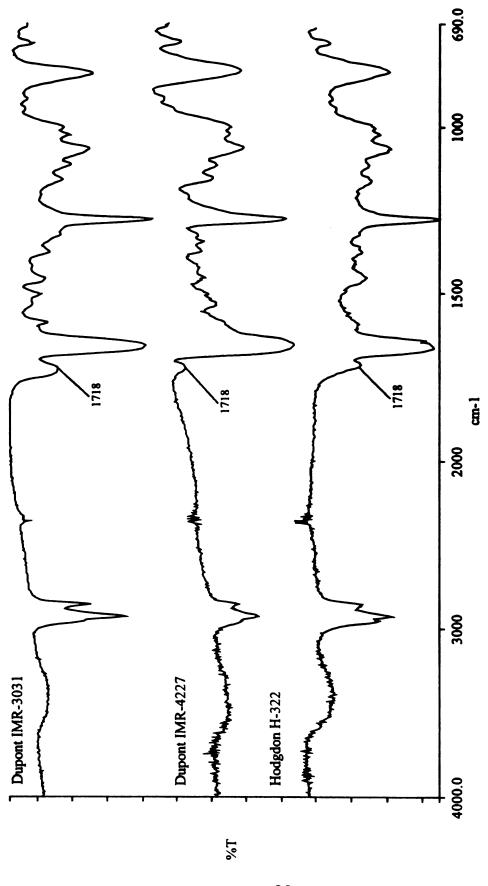


Figure 9 — Cylinder powders (D).

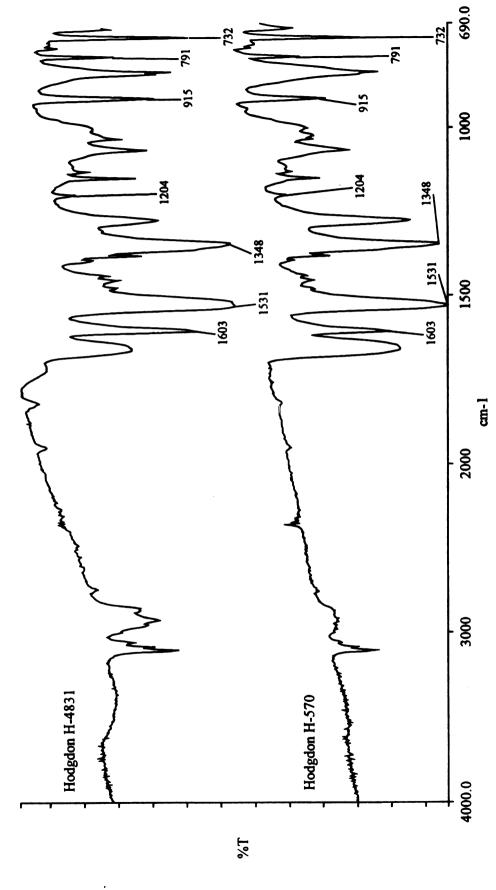


Figure 10 — Cylinder powders (E).

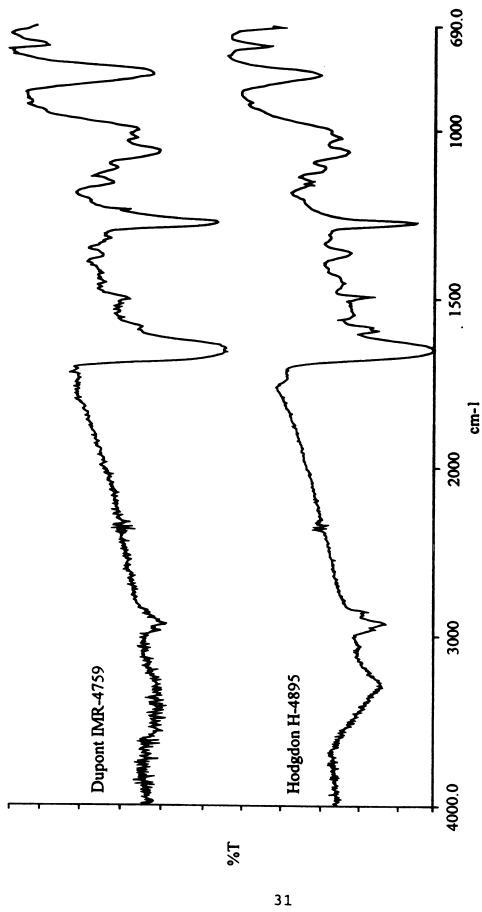


Figure 11 — Cylinder powders (F).

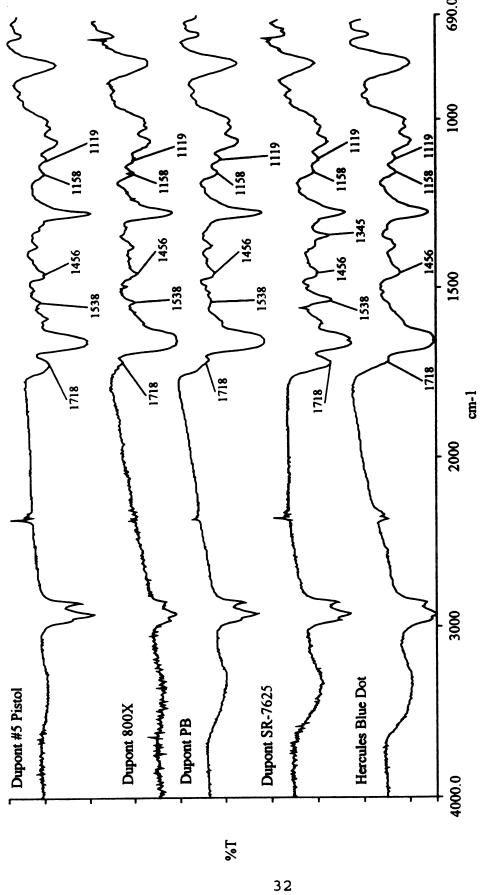


Figure 12 — Disk powders (A).

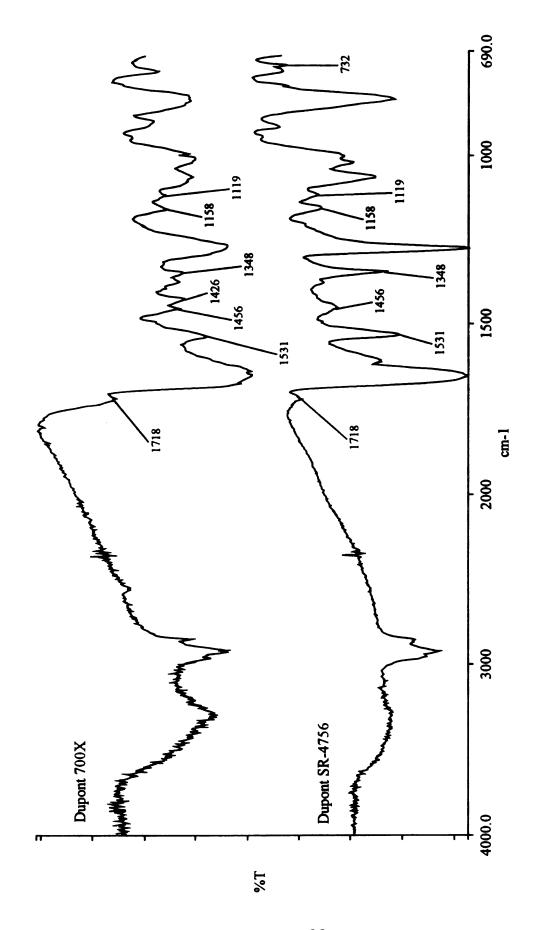


Figure 13 — Disk powders (B).

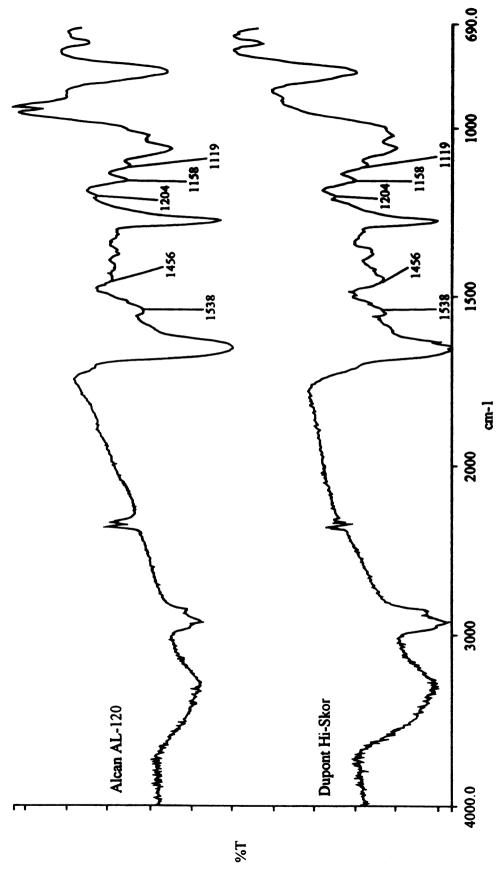


Figure 14 — Disk powders (C).

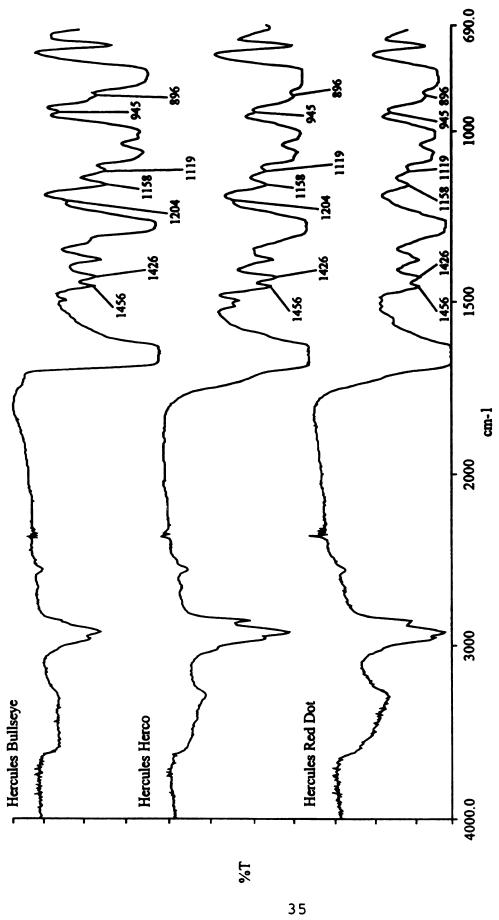


Figure 15 — Disk powders (D).

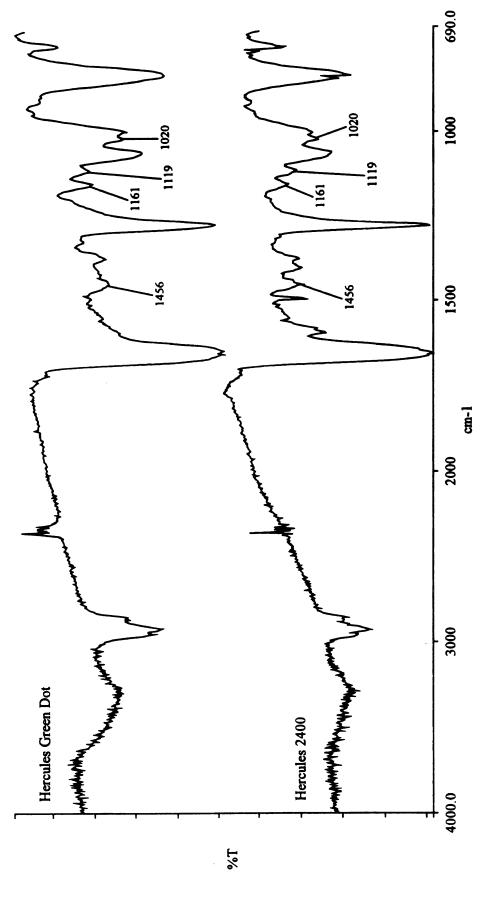


Figure 16 — Disk powders (E).

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extra peak at 1718 cm⁻¹ (Figure 12). Dupont #5 Pistol, 800X, PB, and SR-7625 also displayed a peak at 1538 cm⁻¹, whereas Hercules Blue Dot did not. Furthermore, Dupont SR-7625 had a prominent peak occurring at 1345 cm⁻¹, which was not seen in the other spectra.

Both Dupont 700X and SR-4756 showed a doublet at 1718 and peaks at 1531 and 1348 cm $^{-1}$ (Figure 13). SR-4756 also had an extra peak at 732 cm $^{-1}$, whereas Dupont 700X had an extra peak at 1426 cm $^{-1}$.

Seven disk powders lacked a peak at 1718 cm⁻¹ (Figures 14-16). Of these powders, Alcan AL-120 and Dupont Hi-Skor shared peaks at 1538 and 1204 cm⁻¹ (Figure 14). Hercules Bullseye and Herco also had a peak at 1204 cm⁻¹ but lacked a peak at 1538 cm⁻¹ (Figure 15). In addition, these two spectra displayed similar spectral characteristics with peaks at 1426, 945, and 896 cm⁻¹. Hercules Red Dot also had these peaks present but lacked peaks at 1538 and 1204 cm⁻¹ (Figure 15).

Hercules Green Dot and 2400 lacked prominent peaks at 1538, 1426, 1204, 945, and 896 cm⁻¹ (Figure 16). However, these two spectra did share a prominent peak at 1020 cm⁻¹ that was absent from the spectra in Figure 15.

Flake Powders

All four flake powders shared peaks at 1456, 1119, and 1020 cm⁻¹ (Figure 17). Dupont #6 Pistol had a doublet occurring at 1731 and an extra peak at 896 cm⁻¹. Alcan AL-5,

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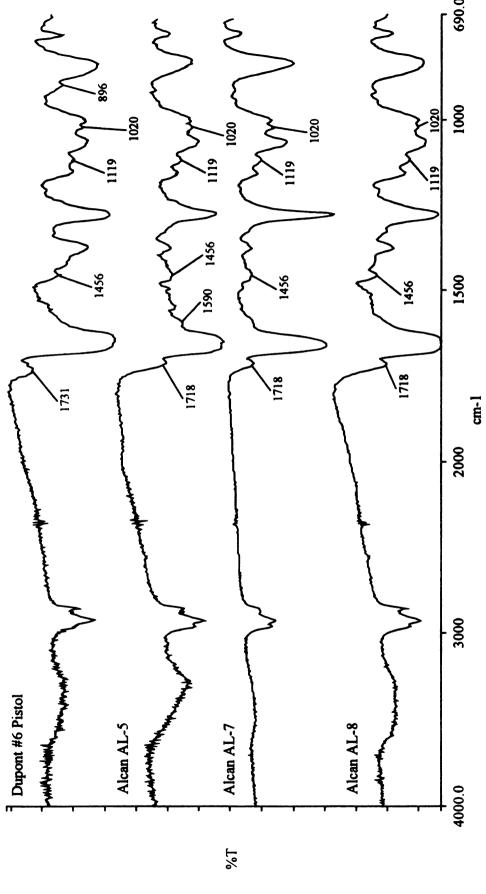


Figure 17 — Flake powders.

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AL-7, and AL-8 had a peak present at 1718 cm⁻¹. Alcan AL-5 also displayed a peak at 1590 cm⁻¹, which was not present in the other flake powders.

Flat Ball Powders

Ten of the fourteen flat ball powders had a peak present at 1718 cm⁻¹ (Figures 18-20). In addition, these ten powders also displayed a peak at 1456 cm⁻¹.

Three flat ball powders displayed prominent peaks at 1597 and 1119 cm⁻¹ (Figure 18). Of these powders, Winchester 760 had an additional peak at 1534 cm⁻¹.

Seven flat ball powders also had a peak present at 1119 but lacked a prominent peak at 1597 cm⁻¹ (Figures 19 and 20). All of these spectra also had a peak at either 1161 or 1158 cm⁻¹ that was absent from the spectra in Figure 18.

Hodgdon HS-5 had peaks at both 1426 and 1191 cm⁻¹ (Figure 20). None of the other flat ball powders showed the presence of both of these peaks. The four remaining flat ball powders displayed similar spectra and lacked a prominent peak at 1718 cm⁻¹ (Figure 21).

A clearer representation of the FTIR spectra results (Figures 5-21) is given in Appendix C.

Validation of Reference Manual

The morphological characteristics (shape, color, perforation, and size) and FTIR spectra for the three

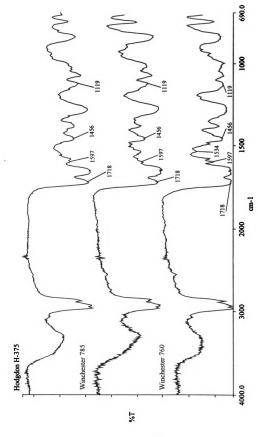


Figure 18 — Flat ball powders (A).

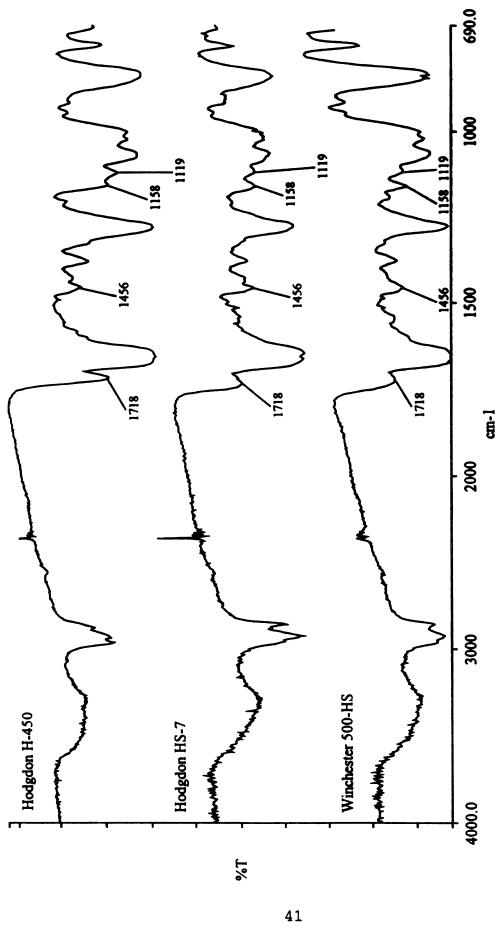


Figure 19 — Flat ball powders (B).

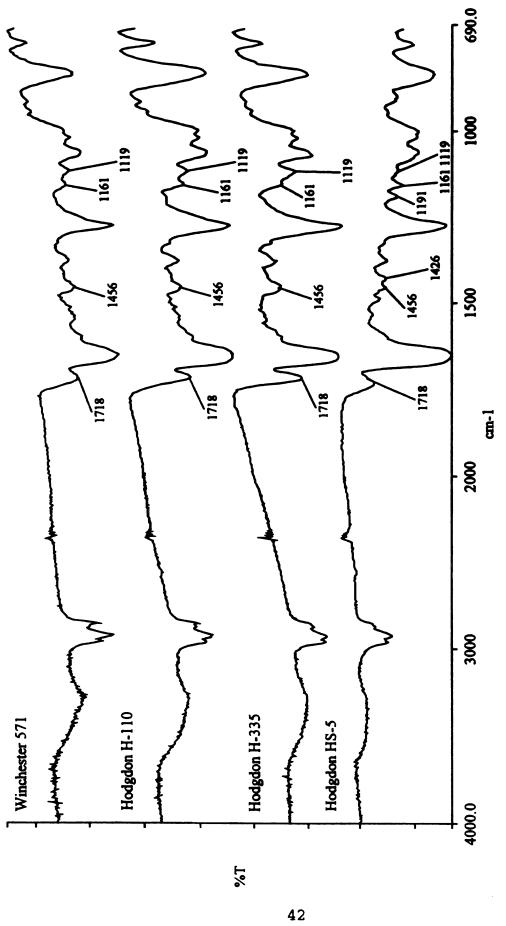


Figure 20 — Flat ball powders (C).

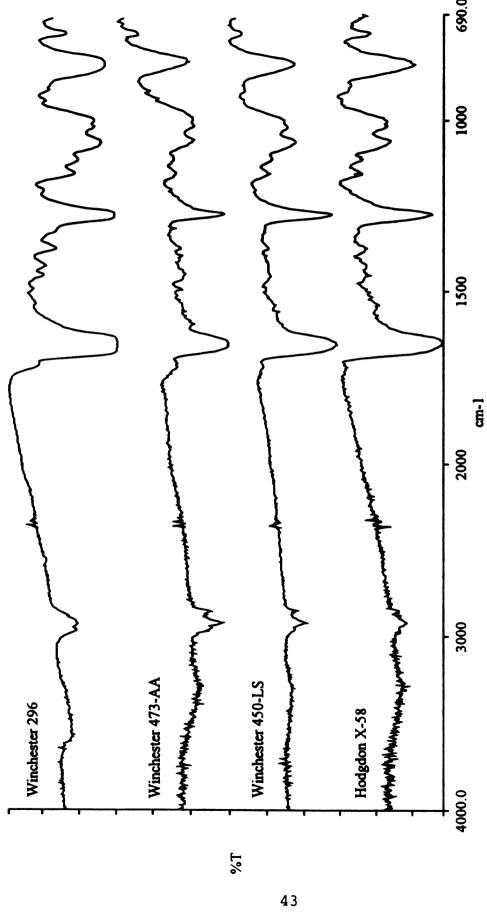


Figure 21 — Flat ball powders (D).

randomly selected smokeless powders can be found in Table 5 and Figures 22-24, respectively. According to the data shown previously in Table 2, the particle size of a smokeless powder does not vary considerably within a batch. For this reason, size is a useful morphological characteristic for differentiating between smokeless powders.

Based on morphological characteristics alone, a number of smokeless powders in the reference manual were eliminated as the possible brands of the questioned powders.

Specifically, only seven of the fifty powders had similar shape, color, perforation, and size characteristics as Questioned Smokeless Powder #1 (Table 6). Only one of the fifty powders shared similar morphology to Questioned

Smokeless Powder #2 (Table 7). Furthermore, three powders shared similar morphology to Questioned Smokeless Powder #3 (Table 8).

Using the FTIR spectra, the possible brands of the questioned powders were narrowed further. Of the seven powders sharing similar morphological characteristics to Questioned Smokeless Powder #1, two of the powders (Hodgdon H-4831 and Hodgdon H-570) shared similar spectral characteristics (Figure 25). Because more than one powder shared similar spectral characteristics to the unknown, a micrograph comparison was conducted (Figure 26). Based on this comparison, Hodgdon H-4831 could not be eliminated as a possible source for this unknown powder.

Table 5 — Morphological characteristics of questioned smokeless powders.

NAME	SHAPE	COLOR	PERFORATED	SIZE
Questioned Smokeless Powder #1	Cylinder	Gray	Yes	2.1 mm
Questioned Smokeless Powder #2	Disk	Black	No	734 µm
Questioned Smokeless Powder #3	Disk	Black	No	1.6 mm



Figure 22 - FTIR spectrum of Questioned Smokeless Powder #1.

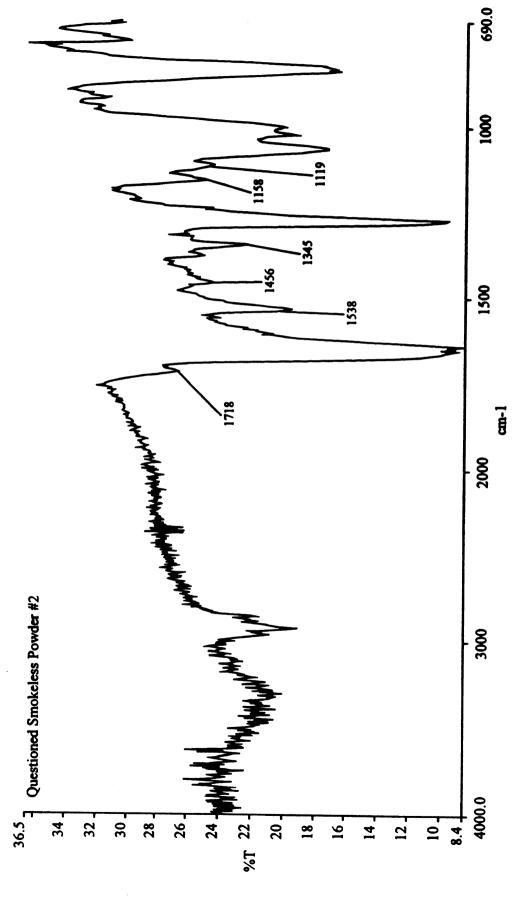


Figure 23 — FTIR spectrum of Questioned Smokeless Powder #2.

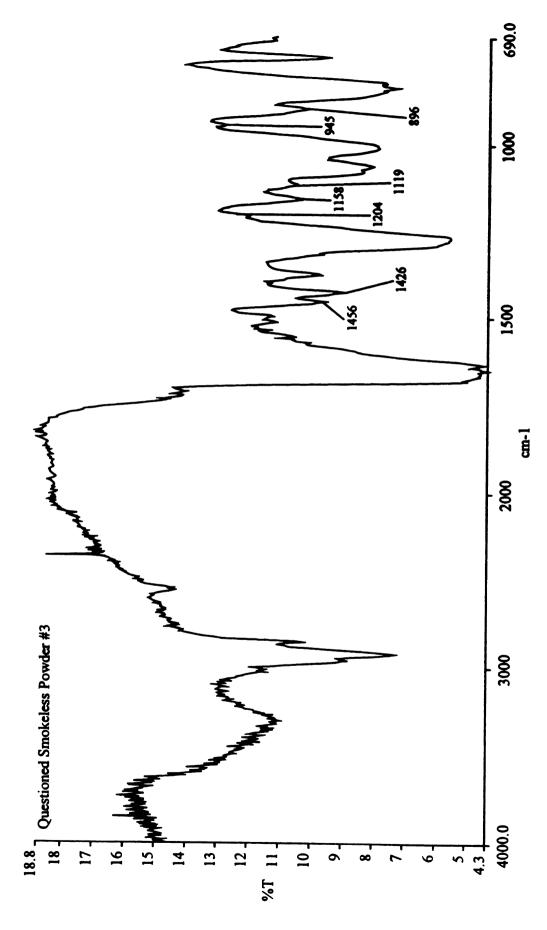


Figure 24 -- FIIR spectrum of Questioned Smokeless Powder #3.

Table 6 — Powders with similar morphology to Questioned Smokeless Powder #1.

NAME	SHAPE	COLOR	PERFORATED	SIZE
Questioned	Cylinder	Gray	Yes	2.1 mm
Smokeless Powder #1				
Dupont IMR-3031	Cylinder	Gray	Yes	2.0 mm
Dupont IMR-4064	Cylinder	Gray	Yes	2.1 mm
Dupont IMR-4198	Cylinder	Gray	Yes	2.2 mm
Hercules Hi-Vel-2	Cylinder	Gray	Yes	2.3 mm
Hodgdon H-4198	Cylinder	Gray	Yes	2.2 mm
Hodgdon H-4831	Cylinder	Gray	Yes	2.1 mm
Hodgdon H-570	Cylinder	Gray	Yes	2.1 mm

Table 7 — Powder with similar morphology to Questioned Smokeless Powder #2.

NAME	SHAPE	COLOR	PERFORATED	SIZE
Questioned Smokeless Powder #2	Disk	Black	No	734 µm
Dupont SR-7625	Disk	Black	No	729 µm

Table 8 — Powders with similar morphology to Questioned Smokeless Powder #3.

NAME	SHAPE	COLOR	PERFORATED	SIZE
Questioned	Disk	Black	No	1.6 mm
Smokeless Powder #3				
Dupont 800X	Disk	Black	No	1.8 mm
Hercules Herco	Disk	Black	No	1.6 mm
Hercules Red Dot	Disk	Black	No	1.6 mm

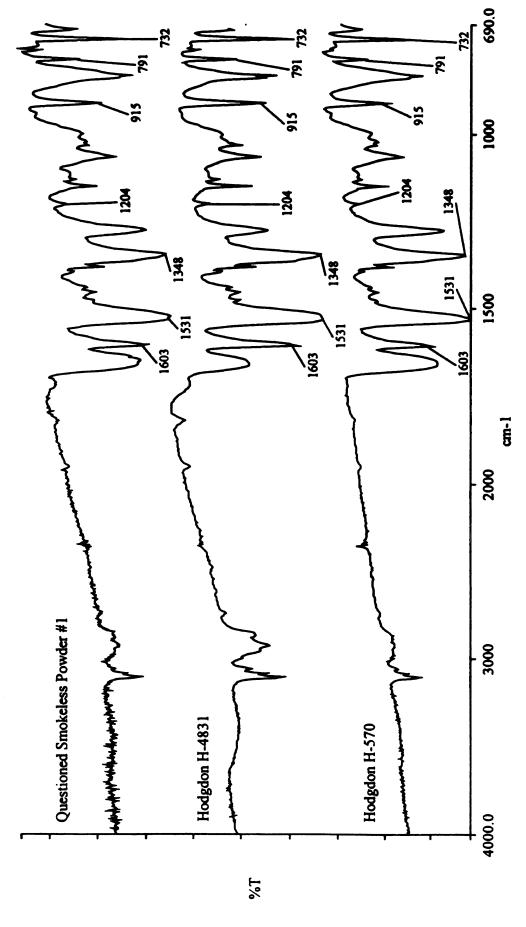
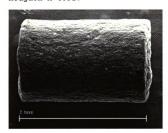


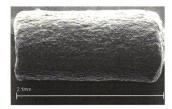
Figure 25 — Comparison of spectra similar to Questioned Smokeless Powder #1.



Hodgdon H-4831.



Hodgdon H-570.



Questioned Smokeless Powder #1.

Figure 26 — Micrograph comparison of Hodgdon H-4831 and H-570 to Questioned Smokeless Powder #1.

Only Dupont SR-7625 displayed similar morphological characteristics to Questioned Smokeless Powder #2. In addition, this powder displayed similar spectral characteristics to the questioned powder (Figure 27). For this reason, Dupont SR-7625 could not be eliminated as a possible source for Questioned Smokeless Powder #2.

After comparing Questioned Smokeless Powder #3 to the three powders sharing similar morphological characteristics, only Hercules Herco displayed similar spectral characteristics as those in Questioned Smokeless Powder #3 (Figure 28). Therefore, Hercules Herco could not be eliminated as a source for this unknown powder.

Following the comparison of the morphology and FTIR spectra of the questioned powders to those in the reference manual, the actual unknowns used were revealed. Questioned Smokeless Powder #1 was Hodgdon H-4831, #2 was Dupont SR-7625, and #3 was Hercules Herco.

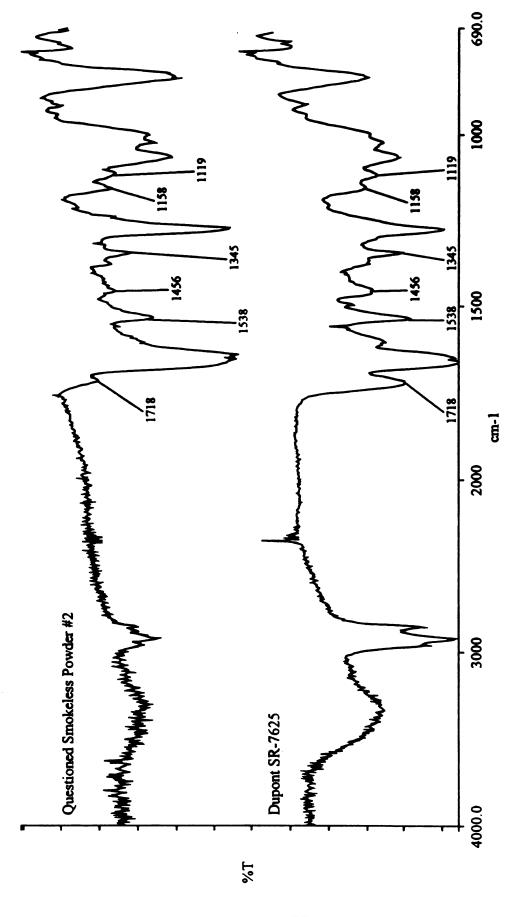


Figure 27 — Comparison of spectra similar to Questioned Smokeless Powder #2.

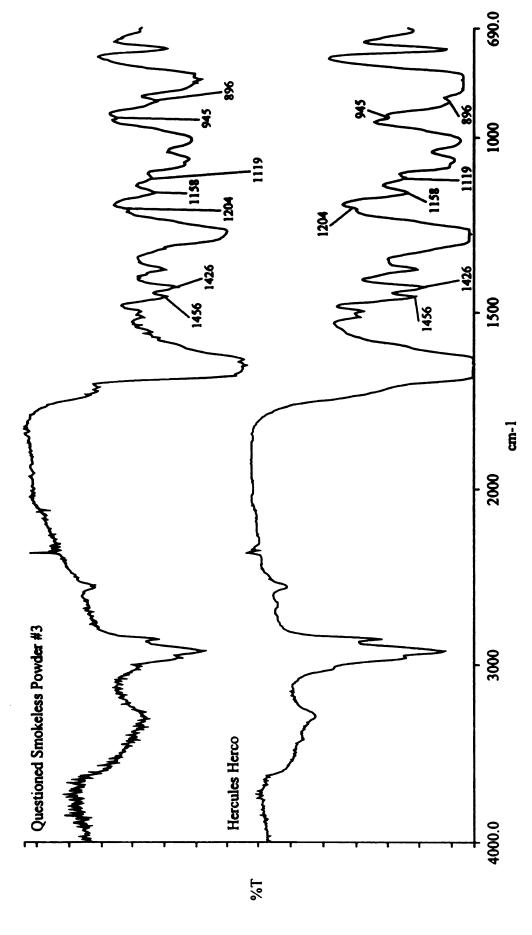


Figure 28 — Comparison of spectra similar to Questioned Smokeless Powder #3.

CONCLUSION

As seen in this research, differentiation between smokeless powders was achieved using a combination of FTIR and morphology. Of the fifty smokeless powders analyzed, some shared similar morphological characteristics and some shared similar spectral characteristics. However, none of the powders analyzed shared both similar morphological and spectral characteristics.

Since the smokeless powders analyzed in this study did not share similar morphology and FTIR spectra, identification of questioned smokeless powders could be achieved. For example, each of the three randomly selected questioned powders was identified correctly by following the analytical scheme shown earlier in Figure 4.

Because these three questioned powders were randomly selected from the powders in the reference manual, the results are consistent within this database only.

Therefore, the reference manual can be used for identification purposes as long as the questioned powder is one of the smokeless powders in the database.

Identification of the brand of a smokeless powder will be extremely beneficial to the forensic science community.

If a forensic scientist can determine the brand of smokeless powder used in an explosion, then investigative efforts could be focused on suspects with that particular brand of powder in their possession. Furthermore, suspects who do

not have that powder in their possession could be experated.

Even if the brand of smokeless powder cannot be identified, comparison of the smokeless powder found at the crime scene to powder found in a suspect's possession will still have strong probative value. Since the smokeless powders in this study did not share similar morphological and spectral characteristics, two powders displaying such similarities could have originated from a common source.

While 4 to 6 particles of each smokeless powder were used in this study, identification is still possible if fewer particles are recovered from a crime scene. While conducting my research at the Michigan State Police Forensic Laboratory in East Lansing, Michigan, a case arose involving one particle of what appeared to be smokeless powder.

Upon extraction of the particle's organic components with acetone, the resulting residue was analyzed using FTIR with a microscope attachment. Once the spectrum was obtained, a search of the smokeless powder spectral library was conducted. The single particle's FTIR spectrum displayed all of the same peaks as one of the smokeless powders in the library.

Although the morphological characteristics of this questioned particle were not analyzed, these results still demonstrate the usefulness that FTIR has for differentiating between smokeless powders. Moreover, these results indicate

that brand identification can be achieved in cases where only one particle of smokeless powder is recovered.

It should be noted that spectra obtained on one FTIR instrument should appear similar to spectra obtained on another FTIR instrument. Therefore, the spectra obtained on a different FTIR instrument should appear essentially the same as those displayed in the reference manual. The reason FTIR spectra obtained on one instrument are similar to spectra obtained on another instrument is because FTIR does not depend on the instrument's settings.

In order to continue advancement of the forensic sciences, future research is necessary. Specifically, additional research on the differentiation of smokeless powders should be conducted. Although 50 of the 80 smokeless powders available at the Michigan State Police Forensic Laboratory were analyzed, additional brands of smokeless powders should be obtained and analyzed. The FTIR spectra and morphological characteristics of the powders could then be added to the reference manual.

Such research should be pursued until all available smokeless powders are displayed in the reference manual. According to the Arson and Explosives Section of the Alcohol, Tobacco, and Firearms Forensic Science Laboratory in Rockville, Maryland, 134 powders are currently available in the United States.

Besides extending the reference manual, the spectral library of smokeless powders should be expanded as well.

Although creating a spectral library was not the ultimate goal of this research, an FTIR search program was generated in the process. Because FTIR search programs allow the rapid comparison of a questioned sample's spectrum to those in the library, such programs are extremely beneficial to the forensic science community.

In addition to expanding the spectral library, another library could be created as well. Specifically, the morphological data and FTIR spectra could be compiled into a single searchable program. Such a program would provide similar results to those obtained using the reference manual but in a shorter period of time.

Additional research studies could focus on using other methods to differentiate between smokeless powders. In particular, diffuse reflectance infrared Fourier transform spectrophotometry could be used in an effort to discriminate between propellants. Using this technique, the smokeless powders could either be mixed with KBr or ran neat. The spectra obtained using this method could then be compared to those obtained using FTIR to determine which technique yields more information.

A final topic for future research could focus on differences in smokeless powders over time. Various smokeless powders could be analyzed using FTIR and morphology over a certain time period. The results from the different times could then be compared to determine whether

the FTIR spectra and/or morphology of propellants change with age.

Due to the increase in violence involving homemade explosives, it is likely that smokeless powders will continue to be encountered in the forensic science laboratory.

Once smokeless powders are recovered from the crime scene, the particles can be analyzed using FTIR and morphology. The results can then be compared to those in the reference manual in order to identify the brand of smokeless powder used, assuming the powder is in the database.

Furthermore, the results can be compared to smokeless powders found in the possession of a suspect in order to determine whether the two powders could have originated from a common source. This piece of information could be the ultimate link to connect a suspect with an explosive device.

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APPENDICES

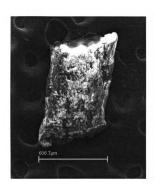
APPENDIX A

Organic compounds that may be found in smokeless powders.

cresol resorcinol carbazole diphenylamine dimethyl phthalate N-nitrosodiphenylamine dinitrocresol carbanilide nitrodiphenylamine triacetin nitrocellulose dinitrotoluene RDX (cyclonite) diethyl phthalate nitroglycerin trinitrotoluene dimethylsebacate N, N-dimethylcarbanilide (methyl centralite) 2, 4-dinitrodiphenylamine N, N-diethylcarbanilide (ethyl centralite) dibutyl phthalate PETN (pentaerythritol tetranitrate) N, N-dibutylcarbanilide (butyl centralite)

APPENDIX B

Reference manual of smokeless powders.

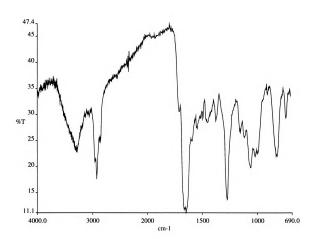


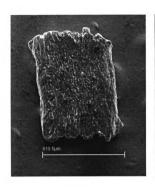
Shape: Flake

Color: Black

Perforated: No

Size: 607 μm



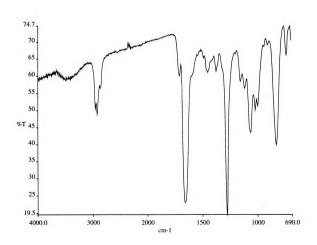


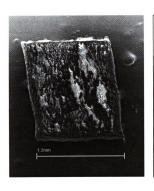
Shape: Flake

Color: Black

Perforated: No

Size: 611 μm



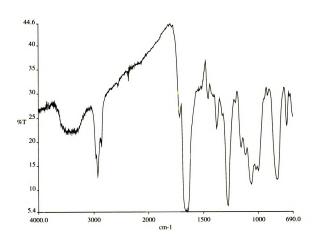


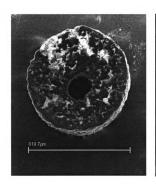
Shape: Flake

Color: Gray

Perforated: No

Size: 1.3 mm



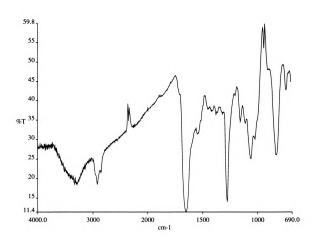


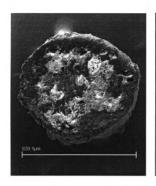
Shape: Disk

Color: Black

Perforated: Yes

Size: 820 μm





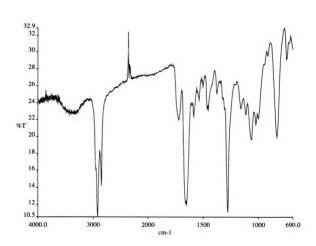
Dupont #5 Pistol

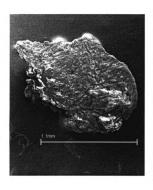
Shape: Disk

Color: Gray

Perforated: No

Size: 920 μm





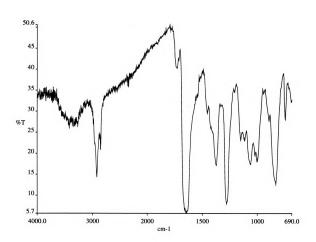
Dupont #6 Pistol

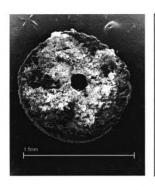
Shape: Flake

Color: Black

Perforated: No

Size: 1.1 mm





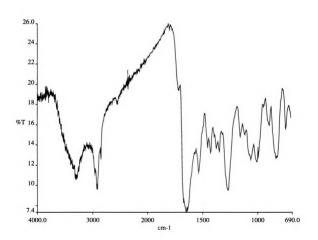
Dupont 700X

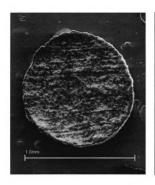
Shape: Disk

Color: Black

Perforated: Yes

Size: 1.5 mm





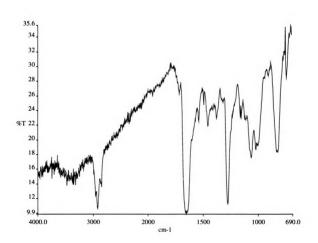
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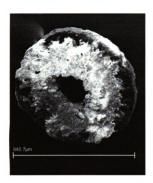
Shape: Disk

Color: Black

Perforated: No

Size: 1.8 mm





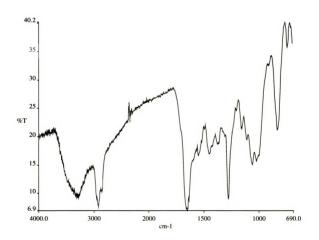
Dupont Hi-Skor

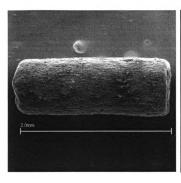
Shape: Disk

Color: Gray

Perforated: Yes

Size: 849 μm





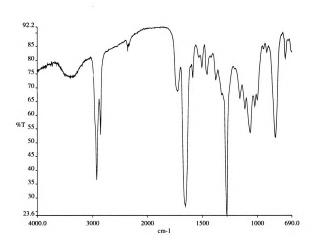
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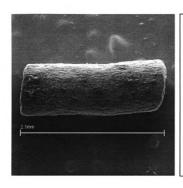
Color: Gray

Perforated:

Yes

Size: 2.0 mm



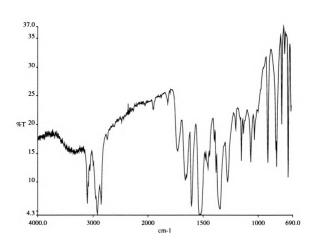


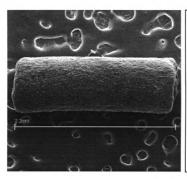
Shape: Cylinder

Color: Gray

Perforated: Yes

Size: 2.1 mm





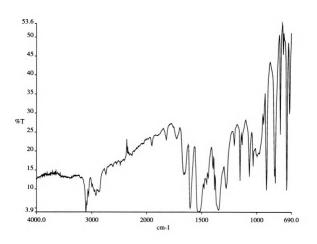
Shape: Cylinder

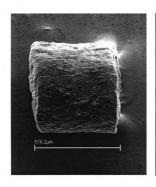
Color: Gray

Perforated: Yes

Les

Size: 2.2 mm



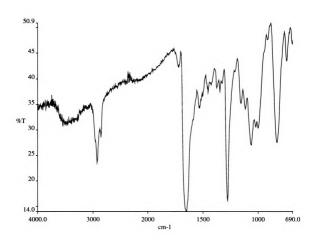


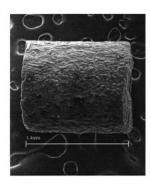
Shape: Cylinder

Color: Black

Perforated: Yes

Size: 570 μm



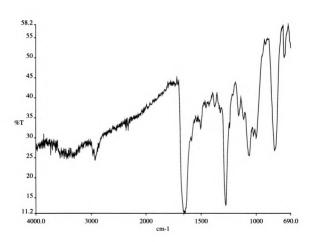


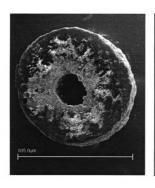
Shape: Cylinder

Color: Gray

Perforated: Yes

Size: 1.4 mm





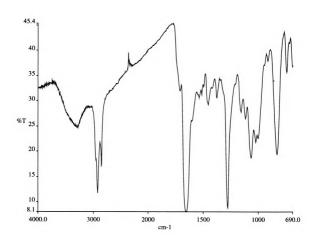
Dupont PB

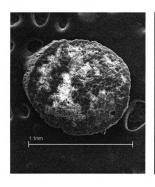
Shape: Disk

Color: Black

Perforated: Yes

Size: 805 μm





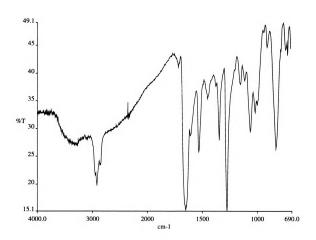
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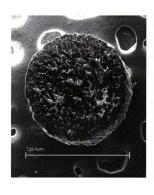
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Color: Gray

Perforated: No

Size: 1.1 mm





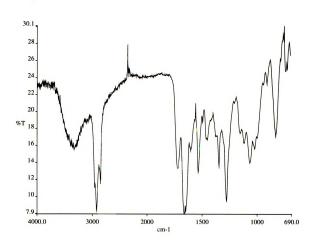
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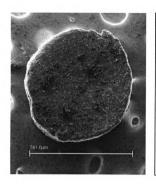
Shape: Disk

Color: Black

Perforated: No

Size: 729 µm





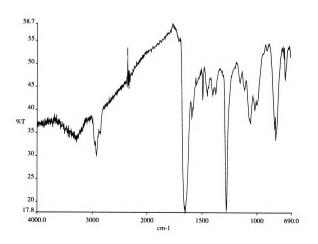
Hercules 2400

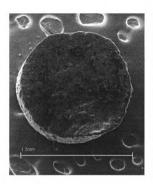
Shape: Disk

Color: Gray

Perforated: No

Size: 741 μm





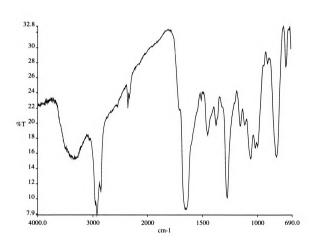
Hercules Blue Dot

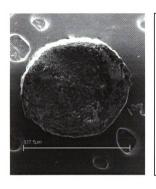
Shape: Disk

Color: Black

Perforated: No

Size: 1.3 mm





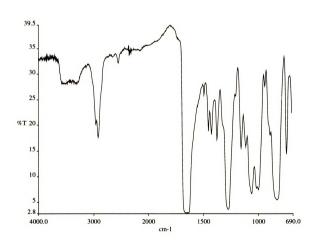
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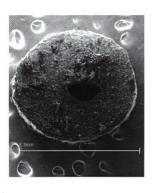
Shape: Disk

Color: Gray

Perforated: No

Size: 838 μm





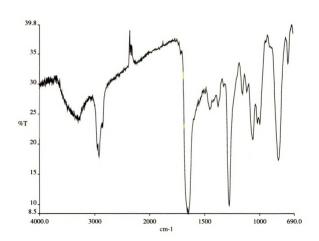
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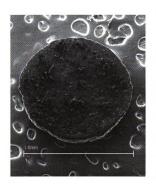
Shape: Disk

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Perforated: Yes

Size: 1.3 mm





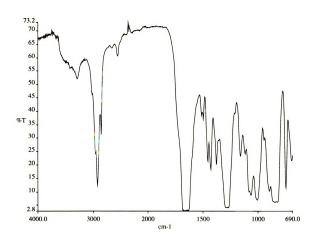
Hercules Herco

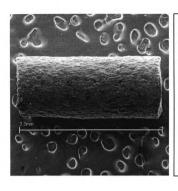
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Perforated: No

Size: 1.6 mm





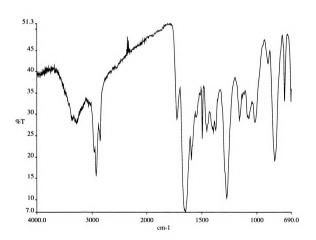
Hercules Hi-Vel-2

Shape: Cylinder

Color: Gray

Perforated: Yes

Size: 2.3 mm





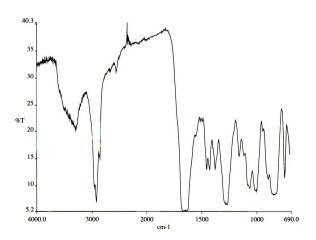
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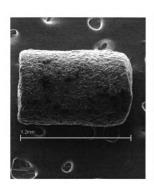
Shape: Disk

Color: Black

Perforated: No

Size: 1.6 mm





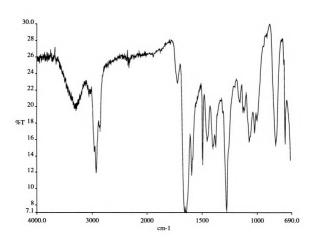
Hercules RL-11

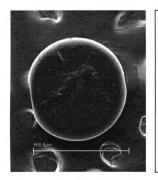
Shape: Cylinder

Color: Gray

Perforated: Yes

Size: 1.2 mm





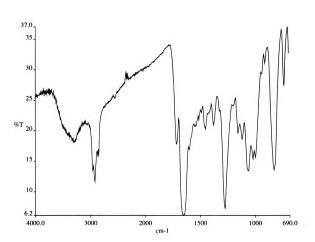
Hodgdon H-110

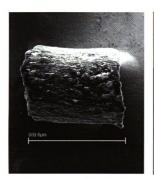
Shape: Flat Ball

Color: Black

Perforated: No

Size: 566 µm



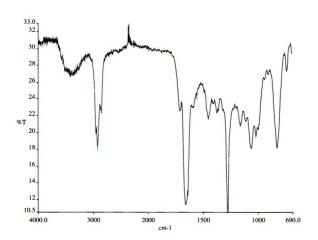


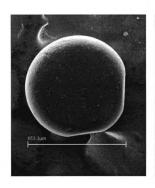
Shape: Cylinder

Color: Black

Perforated: Yes

Size: 810 μm



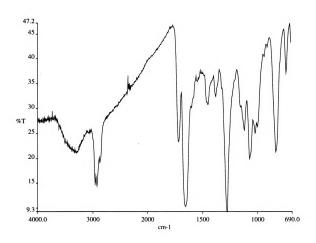


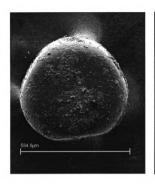
Shape: Flat Ball

Color: Gray

Perforated: No

Size: 653 μm



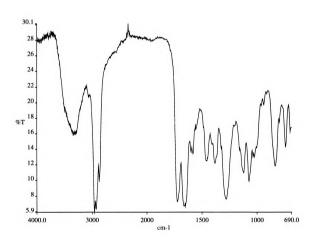


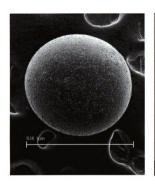
Shape: Flat Ball

Color: Gray

Perforated: No

Size: 585 µm



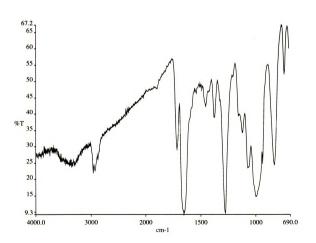


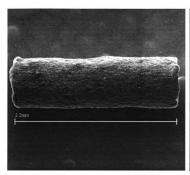
Shape: Ball

Color: Black

Perforated: No

Size: 636 µm





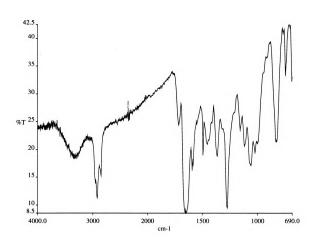
Shape: Cylinder

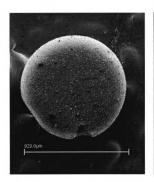
Color: Gray

Perforated:

Yes

Size: 2.2 mm



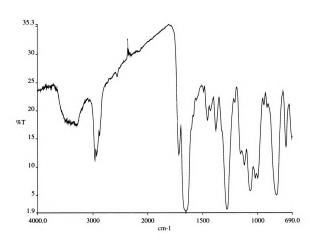


Shape: Flat Ball

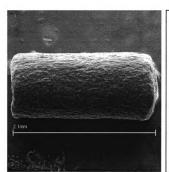
Color: Gray

Perforated: No

Size: 929 μm





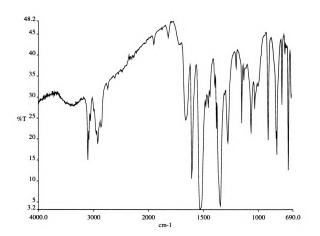


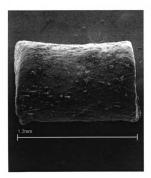
Shape: Cylinder

Color: Gray

Perforated: Yes

Size: 2.1 mm



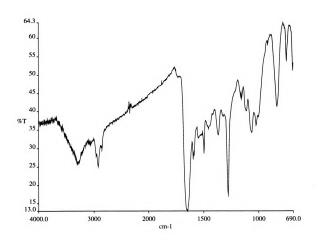


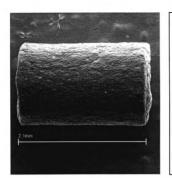
Shape: Cylinder

Color: Gray

Perforated: Yes

Size: 1.2 mm



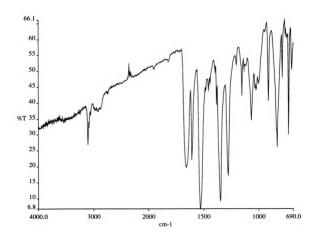


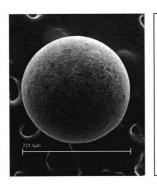
Shape: Cylinder

Color: Gray

Perforated: Yes

Size: 2.1 mm



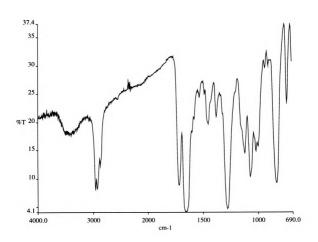


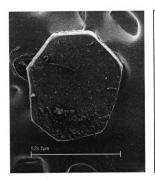
Shape: Ball

Color: Black

Perforated: No

Size: 721 μm



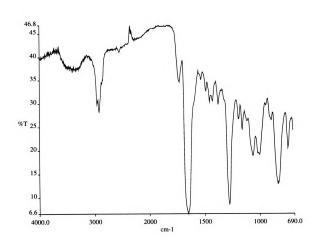


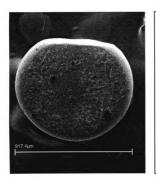
Shape: Flat Ball

Color: Gray

Perforated: No

Size: 529 µm



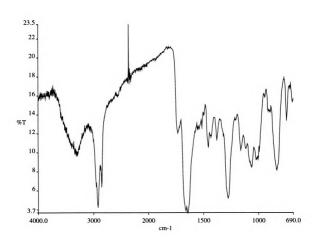


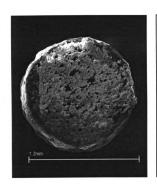
Shape: Flat Ball

Color: Gray

Perforated: No

Size: 917 μm





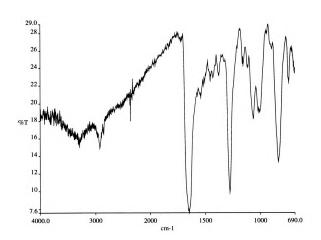
Hodgdon X-58

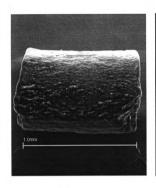
Shape: Flat Ball

Color: Gray

Perforated: No

Size: 1.2 mm



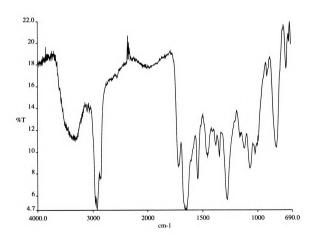


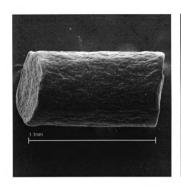
Shape: Cylinder

Color: Gray

Perforated: No

Size: 1.0 mm



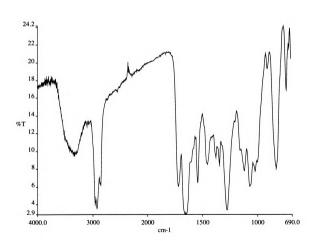


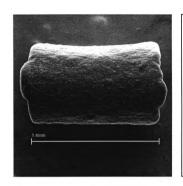
Shape: Cylinder

Color: Gray

Perforated: No

Size: 1.1 mm



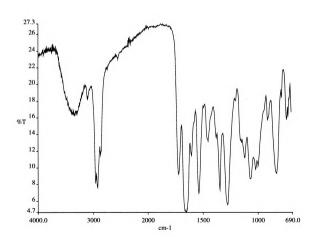


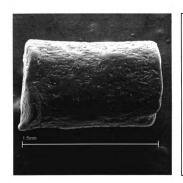
Shape: Cylinder

Color: Gray

Perforated: No

Size: 1.4 mm



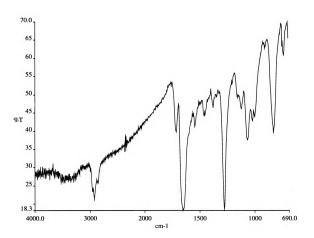


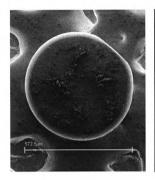
Shape: Cylinder

Color: Gray

Perforated: No

Size: 1.5 mm



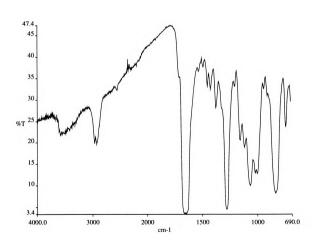


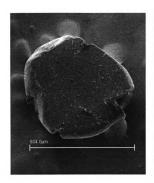
Shape: Flat Ball

Color: Black

Perforated: No

Size: 573 µm





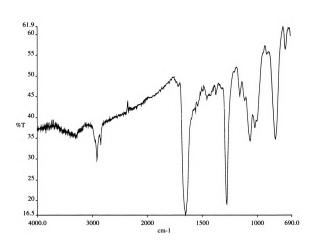
Winchester 450-LS

Shape: Flat Ball

Color: Gray

Perforated: No

Size: 934 µm





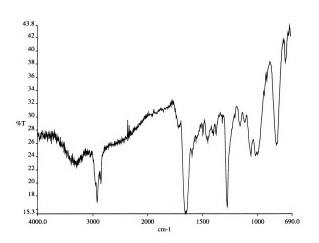
Winchester 473-AA

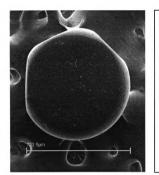
Shape: Flat Ball

Color: Gray

Perforated: No

Size: 594 µm





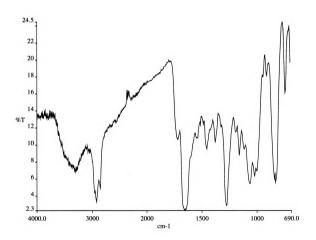
Winchester 500-HS

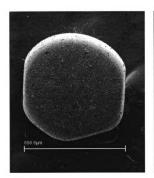
Shape: Flat Ball

Color: Gray

Perforated: No

Size: 724 μm



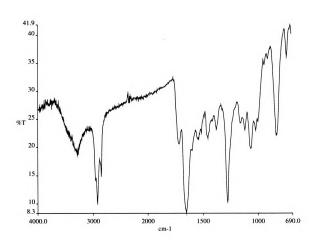


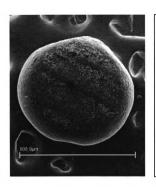
Shape: Flat Ball

Color: Gray

Perforated: No

Size: 899 µm



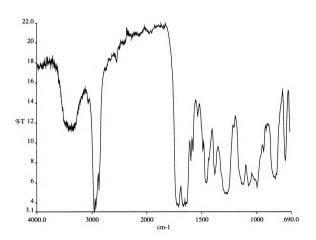


Shape: Flat Ball

Color: Black

Perforated: No

Size: 809 µm



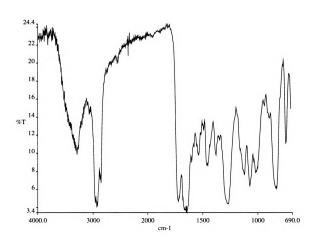


Shape: Flat Ball

Color: Gray

Perforated: No

Size: 959 µm



APPENDIX C

Distinguishing peaks present in the FTIR spectra of each of the 50 smokeless powders analyzed.

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SHAPE

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	Alcan AL-8		×					~	×					×	×						
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	Winchester 473-AA														 					
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