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The Characterization of Match Head Based Improvised
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Major Professor's Signature

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THE CHARACTERIZATION OF MATCH HEAD BASED IMPROVISED EXPLOSIVE DEVICES

By

Jeffrey Harrison Dake

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ABSTRACT

THE CHARACTERIZATION OF MATCH HEAD BASED IMPROVISED EXPLOSIVE DEVICES

By

Jeffrey Harrison Dake

An important aspect in the analysis of improvised explosive devices (IEDs) is the identification of the explosive load used. Currently, very little information has been published on the characterization of IEDs employing matches as the primary explosive load. Many analytical methods have been shown to be effective in the characterization of the various components found in matches. Little has been published on the identification of these components in the complex deflagration products often encountered in post blast residues. Detection of unconsumed material has been demonstrated by scanning electron microscopy with various elements being identified (Glattstein et al, 1991). Using established methods of explosive residue analysis, a system was implemented to analyze the materials recovered from post blast debris of match head based IEDs. The combined physical and chemical properties were used to create a classification scheme for match head based explosive IEDs, which would also eliminate most other low explosive mixtures as possible explosive loads in the recovered debris from an IED. In addition, an examination of fragmentation patterns was performed on the recovered PVC device fragments. Physical characteristics and size groupings of the fragments were observed in an attempt to correlate fragmentation patterns with preparation methods of the match heads used in the IEDs. The results of this study can ultimately be used to compile more specific class information from exploded bomb fragments characteristic to match head devices.

For Amber

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I would like to thank Jamie Crippin for allowing me to perform this research for him, as well as supporting the preliminary work on this project. Thanks are also in order to Dr. Jay Siegel for his direction and support through all of my graduate studies. I would also like to thank Guy Nutter and Dr. Vincent Hoffman for serving on my thesis committee. Thanks to the WFLETC, Pueblo Police Department Bomb Squad, and the Michigan State Police Crime Laboratory for the use of their facilities and expertise.

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Thanks to my friends who have supported me in my endeavors. Most importantly I would like to thank my parents, who have shown an unshakable faith in my abilities and potential.

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INTRODUCTION

Very little research has been done on improvised explosive devices, or IEDs, utilizing match heads as an explosive load. As matches are readily available to virtually any individual, they present a convenient explosive for IEDs. Therefore, it is important for characterization information to be made available to forensic scientists who may receive these types of samples in casework.

Robert Boyle first developed matches when he discovered that mechanical mixtures of sulfur and phosphorus would ignite when subjected to friction. Early matches used a form of white phosphorus that proved highly toxic, and in 1845 it was replaced with the safer red phosphorus, or phosphorus sesquisulfide. Early matches contained the sulfur in a binder with a small tip of red phosphorus, but these were prone to unexpected combustion. This problem was alleviated by Carl Lundstrom who placed the red phosphorus on a friction strip, creating the first "safety" matches. Presently, the formula for matches has become relatively uniform with phosphorus sesqisulfide and sulfur being the primary elements. The other components used in the binder and coloring include: animal glue, starch, dextrin, potassium chlorate, zinc oxide, calcium carbonate, diatomaceous earth, siliceous filler, a potassium chromate or lead burning rate catalyst, powdered glass, dammar gum, and paraffin. "Safety" match tips contain the sulfur compound in a binder. The phosphorus is contained in the striking surface on the package to prevent accidental combustion of the match, and "safety" matches require friction between the match end and the striker for ignition. "Strike anywhere" matches consist of sulfur contained in a binder on the match end, with a small tip of phosphorus sesquisulfide contained in a binder. These matches contain both of the chemical elements for combustion on the tip, and require only friction for ignition. Currently, "strike anywhere" matches are no longer being produced in the United States; however, they are still imported and can easily be obtained.

It has been demonstrated that scanning electron microscopy–energy dispersive x-ray spectroscopy can detect and characterize the residues of "safety" matches on the interiors of pipe fragments from exploded IED's (Glattstein, Landau, and Zeichner, 1991). Their analysis was restricted to elemental analysis by SEM-EDS, in which the following elements were successfully identified: chlorine, potassium, sulfur, silicon, calcium, titanium, magnesium, zinc, chromium, aluminum, and iron. This experiment has demonstrated that it is possible to detect residues from match heads on exploded bomb fragments. The major drawback to this approach is that SEM-EDS is a method of particle analysis. Often in the analysis of explosive debris, the explosive residues are extracted from debris in a matrix. In light of this, it would be preferable to incorporate a chromatographic technique which would be able to separate the match head components from the matrix.

Beveridge et al. developed a system for the analysis of explosive residues which utilizes a combination of techniques to characterize the explosive loads used in various IEDs (Beveridge et al., 1975). The analytical scheme employs morphological examinations, crystal structure examinations, chromatographic techniques, and several instrumental methods for the identification of components in the residues. More recently, the Technical Working Group for Fire and Explosions (TWGFEX) has created guidelines for the identification of intact explosives. TWGFEX identifies four categories of techniques for the analysis of explosives; category 1 techniques provide significant structural and/or elemental composition, category 2 techniques provide limited structural and/or elemental composition, category 3 techniques provide a high degree of selectivity, and category 4 techniques are useful but do not fall into one of the other categories (TWGFEX, pg 1-2). In its guidelines, TWGFEX states that a category 1 technique is sufficient for identification, while a category 2 technique requires one additional technique and a category 3 technique requires two additional techniques for identification. In addition to the listed techniques, the guidelines emphasize the morphological examination of recovered explosives as a means for suggesting further analysis. In a separate document by TWGFEX, methods for the training of explosives analysts are outlined. The importance of a physical examination of the debris recovered from pyrotechnic devices is discussed as an important means of directing further analytical methods (TWGFEX, pg 23-24). Physical characteristics such as visual observations, size, and odor are identified as being of probative value in the analysis of unknowns.

Using the systems presented by Beveridge et al., an analytical scheme was designed to detect the various components of commercial matches and their deflagration products, with particular emphasis on the characterization of sulfur, phosphorus sesquisulfide, and potassium chlorate. TWGFEX guidelines for the analysis of intact particles were adhered to for sufficient identification of explosive materials. The techniques selected were the following; Fourier transform infrared spectrophotometry (FTIR) a category 1 technique, ion chromatography (IC) a category 3 technique, and polarizing light microscopy (PLM) a category 3 technique. The TWGFEX guidelines state that if two different microscopy examinations (such as microchemical tests and microcrystal tests) are employed, they may count as two of the three required techniques for category 3 identification. In addition, a morphological examination was included in the analytical scheme.

The initial visual examination is of great importance in trace evidence analyses, and the examination of explosive debris is no exception. Washington and Midkiff reported characterization of explosives by morphological examination of unconsumed material in 98% of the samples that were identified in their analytical scheme (Washington and Midkiff, 1972). As the combustion material found in matches often has a color imparted to the mixture, morphological examinations may be an effective presumptive method that will provide valuable probative information.

The detection of potassium chlorate by aqueous recrystalization is possible utilizing polarized light microscopy to identify diamond shaped crystals of low order birefringence (Hopen and Kilbourn, 1985). The detection of potassium chlorate is significant in the analysis of match head based IEDs, as potassium chlorate has been banned in most commercial propellants and pyrotechnic mixtures produced in the United States. Because of this ban, no commercial low explosives or pyrotechnic mixtures produced in the United States, with the exception of matches, should contain potassium chlorate. IR has also been shown to be an effective method of characterizing potassium chlorate (Miller and Wilkins, 1952). In addition, FTIR may be useful in detecting organic components used in the binder material of commercial matches.

A separation technique was added to the analytical scheme to account for the complex mixtures often encountered in explosive loads and residues. Ion chromatography was selected for its low limits of detection, and its ability to separate and detect various anionic compounds. The detection of chlorates supports microcrystal and FTIR results, as well as complement microchemical results in the detection of potassium chlorate. In addition, ionic reaction products from the deflagration process are expected and may be detectable by IC.

The first goal of this study is to attempt to identify characteristic fragmentation patterns related to the various preparations of match head based explosive loads. There is a possibility that various load types and the methods with which the filler is prepared will alter the brisance, or shattering power, of the IEDs. While there is no direct method for measuring brisance, it is expected that visible differences will be identified in both the fragment sizes and the fragmentation patterns of the devices with the varying brisances of the different explosive loads (Manon, 1976). Devices that employ cut match heads will have less actual explosive material than other devices, due to the presence of the wood or paper match end within the head. Also, the "whole" nature of the cut match heads will create air pockets within the devices, which should lower their ultimate brisance. Devices that employ match heads where the material has been chipped or scraped from the wooden body will contain greater amounts of explosive load, as the wood or paper match body will not be present. Due to the shape of the chipped fragments air pockets will be present thus decreasing the amount of space occupied within the device, and lowering the overall brisance. Devices containing ground match heads will utilize the ideal amount of space within the device, and should result in the highest brisance devices. Also, there is a possibility of change in brisance, either an increase or decrease, with the variation of the type of match used in device construction. The presence of phosphorus in the "strike anywhere" match heads will decrease the amount of sulfur in the device, which may decrease the brisance. Conversely, the mixtures of sulfur and phosphorus have a much lower ignition temperature than either individual element, and may lead to more complete deflagration in the "strike anywhere" devices, and thus a higher brisance charge. With greater brisance, more fragmentation is expected, and the type of fragmentation may be indicative of the load type or method used in device construction.

The second goal of this study is to develop a system of analysis that will identify morphological and chemical characteristics of match head based IEDs, which will ultimately allow forensic analysts to identify a device as having employed matches as the primary explosive load. Detection of two main components used in matches, sulfur and potassium chlorate, will allow for differentiation of match head material from other commercial and improvised low explosives. Detection of phosphorus sesquisulfide, or a degradation product thereof, may allow for differentiation between "strike anywhere" and "safety" matches.

EXPERIMENTAL

Instrumentation and Materials

Samples were constructed and detonated at the Western Forensic Law Enforcement Training Center in Pueblo, Colorado. Materials for sample construction were purchased at hardware stores. The three different load types were prepared in the following manners: The "cut" devices had the head of the match cut from the match stick using wire cutters. The "chipped" devices had the match head material physically removed from the stick using a combination of crushing the head and subsequent removal with jeweler's tweezers. The "ground" devices were prepared in the same manner as the "chipped" devices with the match head material then being collected and placed in a glass mortar and pestle and ground with methanol to decrease friction, the sample was then dried, further ground, and sieved to remove gross wood particles. All devices were filled to approximately three quarters of their total volume to facilitate deflagration.

Four inch steel pipes with 1.5 inch diameters and threaded ends were used for the steel devices. The ends caps were 1.5 in diameter steel caps. A hole was drilled in one end cap to allow for the insertion of the fuse. A four foot polyvinyl chloride (PVC) pipe was cut into four inch long sections with a hacksaw for the PVC devices. An end cap was glued to one end using PVC cement, and a threaded cap was glued to the other end of each device. A threaded plug was then screwed into the threaded end, with a hole drilled in the plug to allow for the insertion of the fuse.

The complete sample set represents both steel and PVC based devices utilizing both safety and strike anywhere matches, as well as all three preparation types (Table 1).

Sample Designation	Pipe Type	Match Type	Preparation Method
P1, P1B	PVC	Wooden Safety	Cut
P2, P2B	PVC	Paper Safety	Cut
P3, P3B	PVC	Wooden Safety	Chipped
P4, P4B	PVC	Strike Anywhere	Chipped
P5, P5B	PVC	Wooden Safety	Ground
P6, P6B	PVC	Strike Anywhere	Ground
P7, P7B	PVC	Strike Anywhere	Cut
S1, S1B	Steel	Wooden Safety	Cut
S2, S2B	Steel	Paper Safety	Cut
S3, S3B	Steel	Wooden Safety	Chipped
S4, S4B	Steel	Strike Anywhere	Chipped
S5, S5B	Steel	Wooden Safety	Ground
S6, S6B	Steel	Strike Anywhere	Ground
S7, S7B	Steel	Strike Anywhere	Cut

Table 1. List of all samples treated as unknowns in this study, their sample designations, match type used, and preparation method used

Reference samples of the wooden safety matches, paper safety matches, and wooden strike anywhere matches were taken for analytical comparisons. Samples P1, P2, S1, and S2 were all detonated at the Pueblo Police Department's bomb range. Samples of soil were collected prior to detonation to test for contamination. The samples were buried in one foot of soil and detonated using an electric match. All of the other samples were detonated in an open desert area on the edge of the Colorado State University Pueblo campus. Soil samples were collected prior to detonation to test for contamination. Samples were placed in one foot deep holes and covered with wooden planks and sandbags, and were detonated with electric matches. All fragments were collected by manually sifting and placed in plastic bags for storage and transport. Samples S5 and S5B were never received from WFLETC, and thus were not analyzed.

The microscopy was performed on an Olympus BX41 polarizing light microscope (PLM), with a 530 nm full wave plate. Photomicrographs were taken with a Nikon digital camera model DXM1200F attached to a Nikon SMZ800 microscope, using the Nikon Act 1 software in fine mode. Recrystalized materials from aqueous extractions were run on a Perkin Elmer Spectrum One (FTIR) instrument in transmittance mode, using scan averaging over 16 scans from 4000-650 cm^{-1}. Aqueous extractions were also run on a Dionex Dx-120 ion chromatograph (IC) equipped with an IonPac AS9-HC 4x250 mm Analytical column and an electrochemical detector. A 9 mM solution of Na_2CO_3 was used as the mobile phase. A sample amount of 25 μL was injected and the sample was allowed to run for 25 minutes.

Experimental Design

In the first phase of the project, a system for the analysis of the reference samples and soil samples was designed. Approximately 0.02 grams of each sample was placed in a clean glass test tube, to which approximately a milliliter of chloroform was added. The sample was then vortexed and allowed to settle. A drop of the chloroform solution was placed on a microscope slide for examination by PLM. The edges of the drop were observed as it evaporated and any crystal formations were documented. This process was repeated up to three times to allow the sample to concentrate sufficiently, facilitating crystal formation. The test tube was then placed on a hot plate to dry the sample. A small amount of deionized water was placed in the dried test tube and the sample was vortexed and allowed to settle. A drop of the water solution was placed on a slide for examination by PLM. The edges of the drop were observed for any recrystalization as the drop evaporated. The process was repeated up to three times to allow the sample to concentrate, facilitating recrystalization. Representative crystals from the positive controls were photographed using the stereomicroscope. Approximately half a milliliter of the aqueous solution from the test tube was then placed in a vial to be analyzed by IC. The samples were placed into sample vials with filter caps and loaded into cassettes for auto sampling. An external standard was run at the beginning and end of each sample set and a blank of deionized water was run in between each sample. The remainder of the aqueous sample from the test tube was filtered using a syringe filter with a .45 μm pore size PETN filter to remove gross particulates. The filtrate was collected in a clean Petri dish and dried on a hot plate. The dried crystals were then removed, placed in a mortar with potassium bromide and ground. The sample was then pressed into a pellet and a transmittance infrared spectrum was taken.

In the second phase of the experiment, the PVC and steel IED samples were analyzed. The fragments were first visually examined both macroscopically and microscopically for characteristic features. The fragments for the PVC samples were grouped into three size classes in an attempt to classify different filler types. All measurements were taken with a ruler. The measurements were based on the maximum length and width of the fragments, and were labeled as: small (both sides smaller than .5 cm), medium (both sides between .5 cm and less than 1.5 cm), and large (at least one side greater than or equal to 1.5 cm).

During the physical examination, any consumed or unconsumed material was removed from the fragments physically and collected for chemical analysis. To obtain the residue material from the steel pipes, the interior of the pipe was rinsed with chloroform and collected in a test tube, and the pipe was allowed to dry. The interior was then rinsed with warm deionized water to facilitate the extraction of the potassium chlorate, and the rinse was collected in a test tube. To obtain the residue material from the PVC fragments, the interior of the fragments was first washed with methanol, as chloroform may extract plasticizers from the PVC surface. The methanol extraction was then dried on a hotplate and the sample was reconstituted with a small amount of chloroform. Aqueous extractions from the PVC fragments were performed in the same manner as the steel fragments.

The consumed, unconsumed, and residue material from each sample was analyzed using the methods developed in the first phase of the experiment. The purpose of these experiments was to determine the effectiveness of the various methods in characterizing the components from the explosive load present in match head based IEDs.

RESULTS

Images in this thesis are presented in color. All chromatograms presented in this thesis may be compared with the standard in figure 10.

Soil Samples and Match Head Reference Standards

The results from the microchemical and microcrystal tests performed on the match reference standards and the soil background samples are included in Table 2. The water recrystalization from all of the match standards, both unconsumed and consumed, resulted in diamond shapes with low order birefringence indicative of potassium chlorate. The platinum chloride microchemical tests resulted in the formation of hexagons with three short sides and three long sides for all of the match references standards, indicating the presence of potassium. Only 66% of the chloroform recrystalizations from the match reference standards resulted in the formation of saw shaped crystals indicative of elemental sulfur, while the consumed wooden "safety" match and the consumed "strike anywhere" match standards resulted in unidentifiable crystal structures. The soil background samples from the CSU Pueblo site (referred to as School samples) gave no appreciable crystals in either the water recrystalization or the chloroform recrystalization. Some hexagons were identified in the platinum chloride microchemical tests, but they were in a much smaller amount than those observed in the reference standards. The soil background samples from the Pueblo Police bomb range (referred to as Range samples) displayed a multitude of crystal structures in the water recrystalizations, with the predominant structures observed being needles, clusters of needles, and crossed plates indicative of ammonium nitrate. No diamond shapes were observed. The chloroform recrystalizations from the Range samples gave no appreciable crystal structures. Some

Sample Name	H2O Recrystalization	PTCL Microchemical	CHCL3 Recrystalization
Wood Safety Whole	Positive	Positive	Positive
Wood Safety Burnt	Positive	Positive	Inconclusive
Paper Safety Whole	Positive	Positive	Positive
Paper Safety Burnt	Positive	Positive	Positive
Strike Anywhere Whole	Positive	Positive	Positive
Strike Anywhere Burnt	Positive	Positive	Inconclusive
School 1	Negative	Slight Positive	Negative
School 2	Negative	Slight Positive	Negative
School 3	Negative	Slight Positive	Negative
School 4	Negative	Slight Positive	Negative
School 5	Negative	Slight Positive	Negative
Range 1	Negative	Negative	Negative
Range 2	Negative	Slight Positive	Negative
Range 3	Negative	Slight Positive	Negative
Range 4	Negative	Negative	Negative
Range 5	Negative	Slight Positive	Negative

Table 2. Experimental results from the microcrystal and microchemical examinations of the match head exemplars and the soil control samples.

hexagons were identified in the platinum chloride tests for 60% of the Range samples, but the predominant crystal forms observed were burrs of needles and curved needles, suggesting the presence of ammonium.

Results from the instrumental analyses of the match reference standards and the soil background samples are presented in Table 3. In 66% of the match reference standards, infrared absorptions at 970 cm^{-1} (dominant) and 940 cm^{-1} were identified by FTIR spectroscopy, with the consumed wooden "safety" match and the consumed "strike anywhere" match standards displaying no absorption bands in that region (Figure 1). Absorptions at these two wavelengths are indicative of potassium chlorate. Ion chromatography of the unconsumed match samples detected the presence of chlorates, chlorides, sulfates, and phosphates; with the "strike anywhere" standard containing nitrates as well. In the unconsumed samples, chlorate was the most abundant anion detected (Figure 2a). Ion chromatography of the consumed match reference standards detected chlorates (except in the wooden "safety" standard), chlorides, sulfates, phosphates, nitrates, nitrites, and bromides (only in the paper "safety" standard). The concentrations of the chloride and sulfate anions were greater than in the unconsumed samples, with the chloride anion being the most abundant anion detected (Figure 2b). In the FTIR analyses none of the soil background samples, from the School set or the Range set displayed absorption bands at 970 cm^{-1} or 940 cm^{-1} (Figure 3a, 3b). Ion chromatography of the School sample set detected chlorides, nitrates, sulfates, nitrites, and phosphates (only in sample School 3), with sulfate being the most abundant peak in each sample (Figure 4). Chlorides, nitrites, nitrates, and sulfates were detected in the Range samples, with sulfate being the most abundant anion detected (Figure 5).

Sample Name	FTIR (KCLO3)	Chlorates (IC)	Chlorides (IC)	Sulfates (IC)	Phosphates (IC)
Wood Safety Whole	Positive	Positive (Dominant)	Positive (.75 mg/L)	Positive (1.49 mg/L)	Positive (Moderate)
Wood Safety Burnt	Negative	Negative	Positive (883.98 mg/L)	Positive (75.20 mg/L)	Positive (Moderate)
Paper Safety Whole	Positive	Positive (185.72 mg/L)	Positive (.37 mg/L)	Positive (Moderate)	Positive (Miniscule)
Paper Safety Burnt	Slight Positive	Positive (Miniscule)	Positive (745.30 mg/L)	Positive (115.29 mg/L)	Positive (Moderate)
Strike Anywhere Whole	Positive	Positive (Dominant)	Positive (1.05 mg/L)	Positive (28.43 mg/L)	Positive (Miniscule)
Strike Anywhere Burnt	Negative	Positive (.72 mg/L)	Positive (1196.54 mg/L)	Positive (72.35 mg/L)	Positive (Moderate)
School 1	Negative	Negative	Positive (.76 mg/L)	Positive (4.47 mg/L)	Negative
School 2	Negative	Negative	Positive (1.64 mg/L)	Positive (10.55 mg/L)	Negative
School 3	Negative	Negative	Positive (1.54 mg/L)	Positive (9.70 mg/L)	Positive (Miniscule)
School 4	Negative	Negative	Positive (1.27 mg/L)	Positive (6.87 mg/L)	Negative
School 5	Negative	Negative	Positive (.95 mg/L)	Positive (5.75 mg/L)	Negative
Range 1	Negative	Negative	Positive (7.19 mg/L)	Positive (2166.89 mg/L)	Negative
Range 2	Negative	Negative	Positive (6.39 mg/L)	Positive (2177.40 mg/L)	Negative
Range 3	Negative	Negative	Positive (5.30 mg/L)	Positive (2141.14 mg/L)	Negative
Range 4	Negative	Negative	Positive (12.70 mg/L)	Positive (2417.16 mg/L)	Negative
Range 5	Negative	Negative	Positive (4.49 mg/L)	Positive (2179.35 mg/L)	Negative

Table 3. Experimental results from the FTIR and IC examinations of the match head exemplars and the soil control samples.

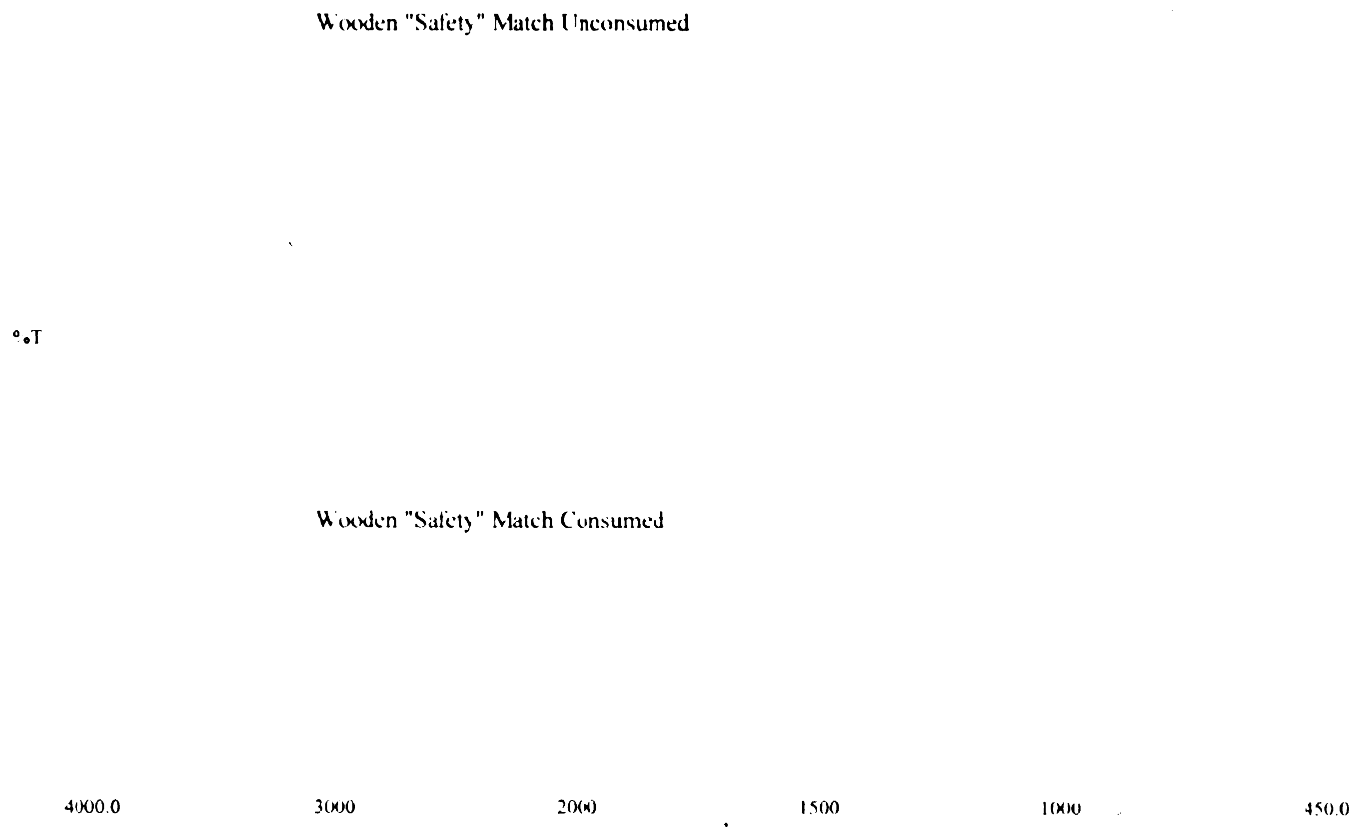

Figure 1. FTIR spectra of crystals recovered from the aqueous extracts of the wooden "safety" match exemplar. Both the consumed and unconsumed materials are represented. Note the presence of absorption bands at 970 cm^{-1} and 940 cm^{-1} characteristic of potassium chlorate.

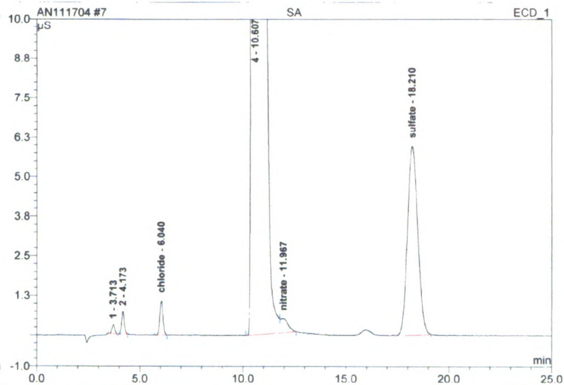

Figure 2. IC chromatograms of aqueous extracts from the "strike anywhere" match exemplar. **A.** Chromatogram of the extract from the unconsumed material.

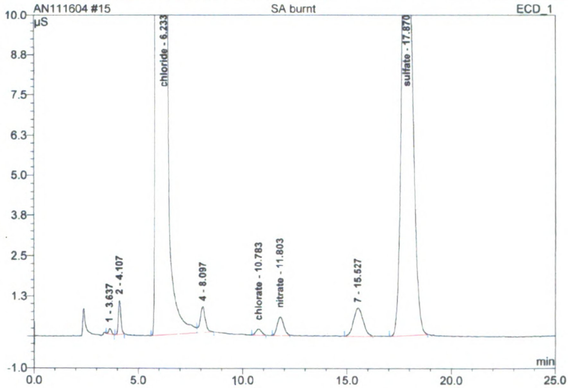

Figure 2B. Chromatogram of the extract from the consumed material

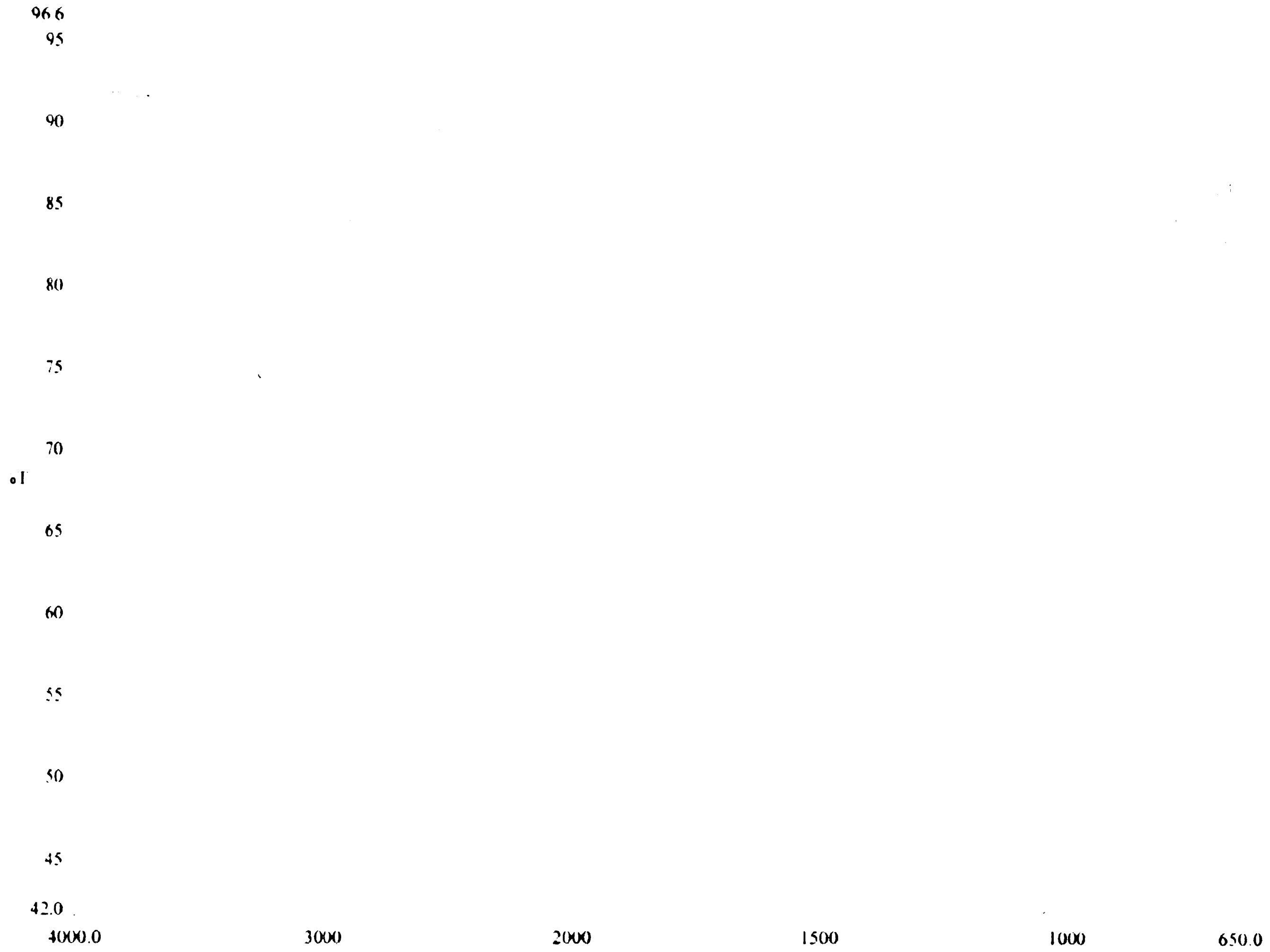

Figure 3. FTIR spectra of crystals recovered from the aqueous extracts of the soil control samples. Note the absences of the absorption bands at 970 cm^{-1} and 940 cm^{-1}. **A**. The FTIR spectra of the "School" soil sample.

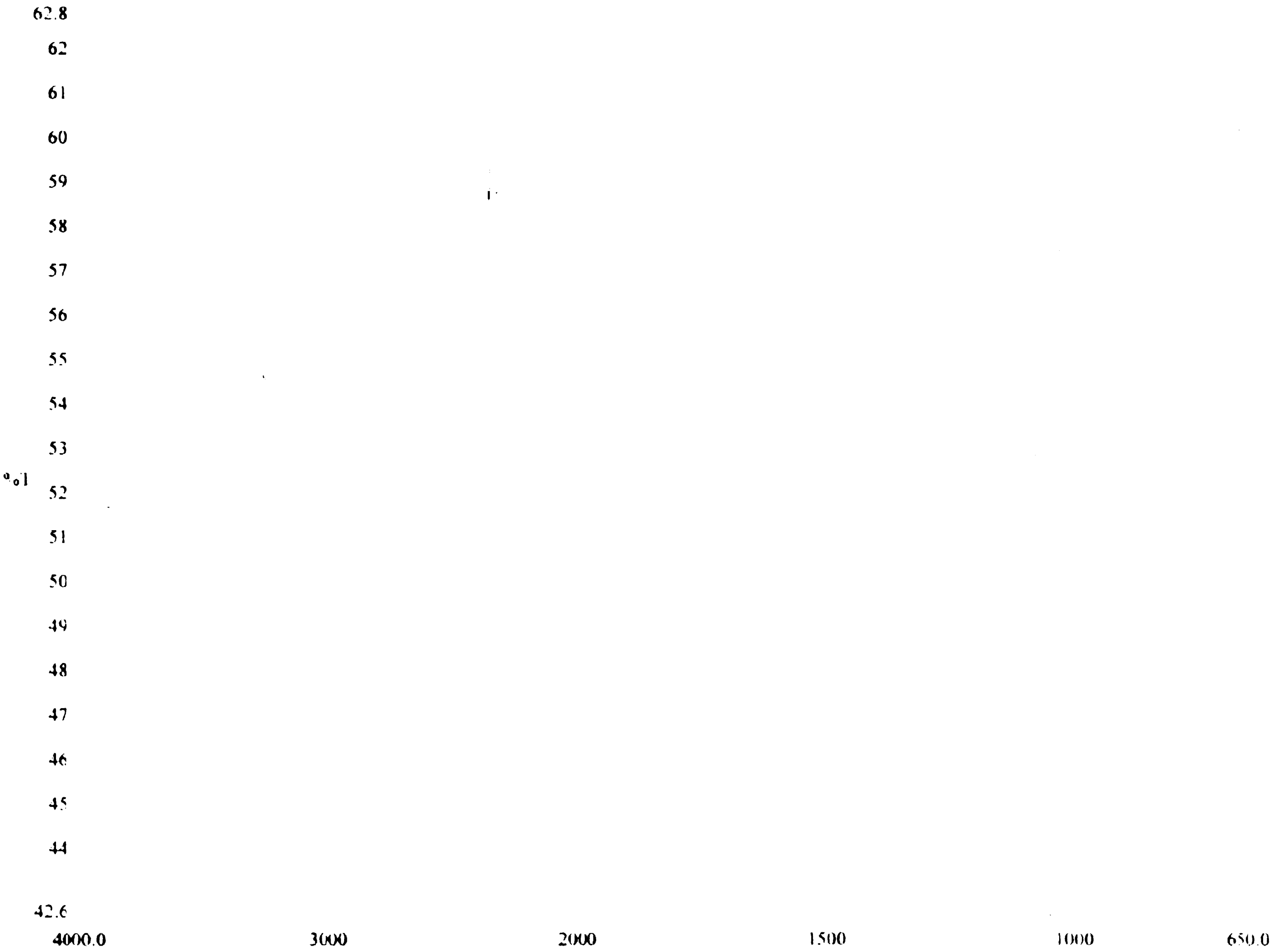

Figure 3B. The FTIR spectra of the "Range" soil sample.

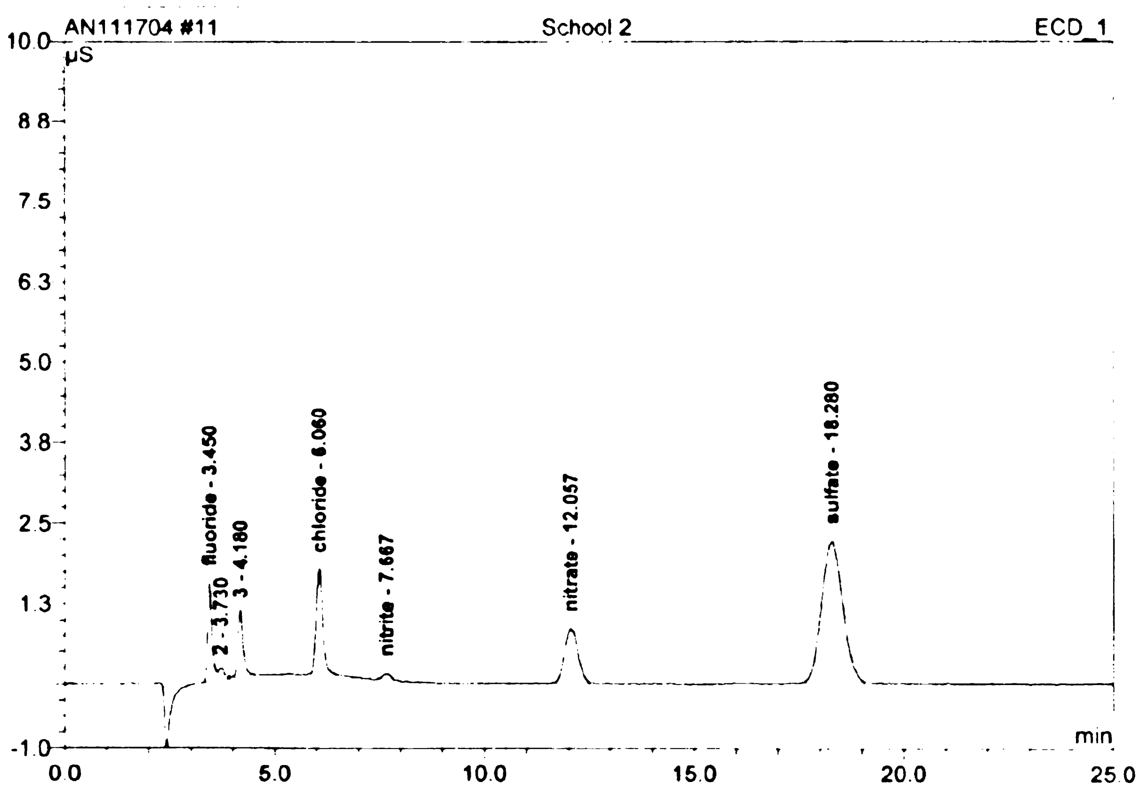

Figure 4. IC chromatogram of the aqueous extract from the "School" control soil sample. Note the absence of a chlorate peak.

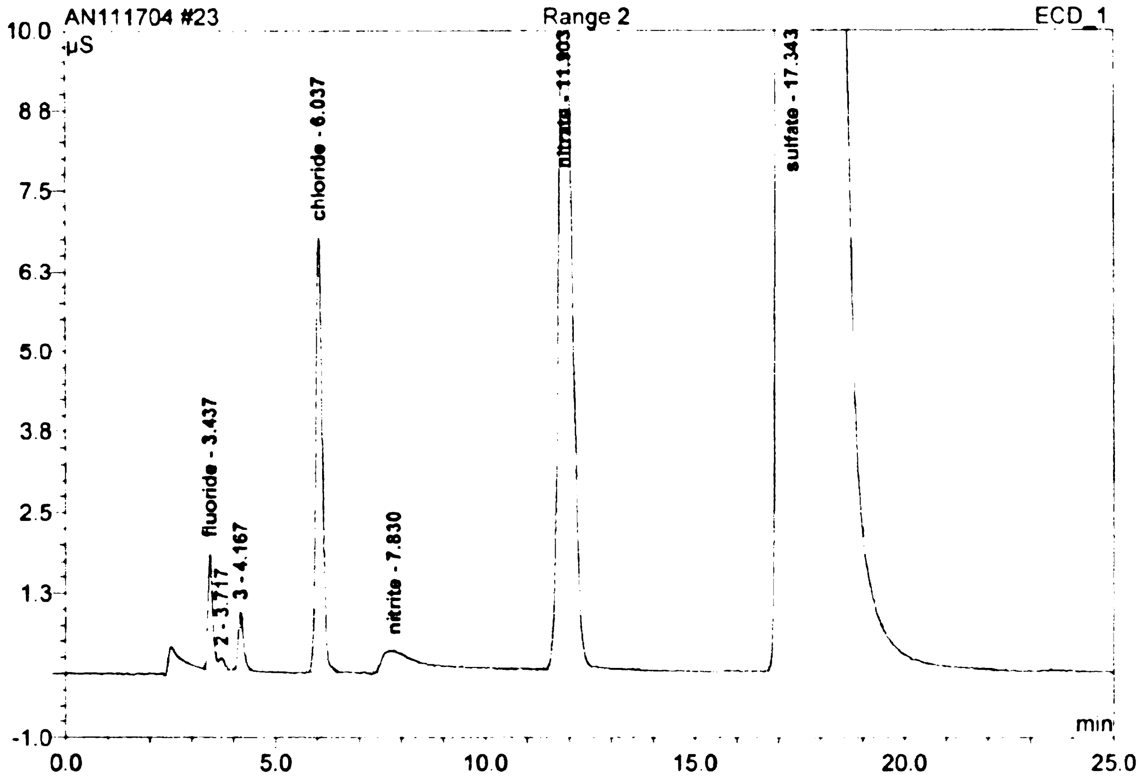

Figure 5. IC chromatogram of the aqueous extract from the "Range" control soil sample. Note the absence of a chlorate peak and the high concentration of sulfates.

Physical Examinations

Observations of all samples were recorded and compiled in Table 4. Physical examination of the steel IEDs consisted of observations of the relative amount of oxidation, or rust, present on the devices, as well as the color and nature of the interior residue. Of the entire device set, only devices S6 and S6B displayed any fragmentation. The residue material coating the interior of most of the devices consisted of a combination of gray and red particles; however a few of the devices (S3, S4B, and S6B) displayed only the gray particles. The residue material observed in the exemplars was a dark grayish black in the black powder device and a lighter gray in the smokeless powder device. Every sample in the set displayed some degree of oxidation, predominantly about the end caps and threading (Figure 6). Intact match heads were identified in all of the devices that employed whole heads as a filler. In 25% of the steel samples unconsumed material was recovered for chemical analysis; while in 75% of the steel samples consumed material was recovered.

Physical examination of the PVC devices consisted of observing the color and nature of the residue, and grouping the fragments into the size classes detailed in the methods section. The residue material observed on the IED interiors was a film of orange-brown color which varied in shade from light to dark. The residue observed in the exemplars was light brown for the black powder and gray for the smokeless powder. In 71% of the PVC samples unconsumed material was recovered; while in 100% of the PVC samples some consumed material was recovered for further analysis. In observing the PVC IED fragments, it was noted that some of the fragments from the sample set had

Sample Name	Unconsumed	Consumed	Residue Color	Other Info
P1	Present (Whole)	Present (Whole)	Orange/Brown	Pipe Intact
P1B	None	Present (Whole)	Orange/Brown	Pipe Intact
P2	None	Present (Whole)	Orange/Brown	Some Fractures
P2B	Present	Present (Whole)	Orange/Brown	Some Fractures
P3	Present	Present	Orange/Brown	Some Fractures
P3B	Present	Present	Orange/Brown	Some Fractures
P4	Present	Present	Orange/Brown	Some Fractures
P4B	Present	Present	Orange/Brown	Some Fractures
P5	Present	Present	Orange/Brown	Some Fractures
P5B	Present	Present	Orange/Brown	Some Fractures
P6	Present	Present	Orange/Brown	Some Fractures
P6B	None	Present	Orange/Brown	Some Fractures
P7	Present	Present (Whole)	Orange/Brown	Some Fractures
P7B	None	Present (Whole)	Orange/Brown	Some Fractures
P Black Powder	N/A	N/A	Light Brown	Multiple Fractures
P Smokeless	N/A	N/A	Gray	Multiple Fractures
S1	None	Present (Whole)	Red and Gray Flecks	Rust on Device
S1B	None	Present (Whole)	Red and Gray Flecks	Rust on Device
S2	None	Present (Whole)	Red and Gray Flecks	Rust on Device
S2B	Present	Present (Whole)	Red and Gray Flecks	Rust on Device
S3	None	Present	Gray Flecks	Rust on Device
S3B	Present	Present	Red and Gray Flecks	Rust on Device
S4	None	Present	Red and Gray Flecks	Rust on Device
S4B	None	None	Gray Flecks	Rust on Device

Table 4. List of all materials recovered from sample IEDs as well as important physical observations of sample IEDs, black powder, and smokeless powder exemplars.

S6	None	None	Red and Gray Flecks	Rust on Device, Fragmented
S6B	None	Present	Gray Flecks	Little Rust, Fragmented
S7	Present	None	Red and Gray Flecks	Rust on Device, Wooden Heads Present
S7B	None	Present (Whole)	Red and Gray Flecks	Rust on Device
S Black Powder	N/A	N/A	Grayish Black	Little Rust, Fragmented, Pipe Missing
S Smokeless	N/A	N/A	Light Gray	No Rust, Fragmented, Pipe Torn

Table 4 (cont'd)

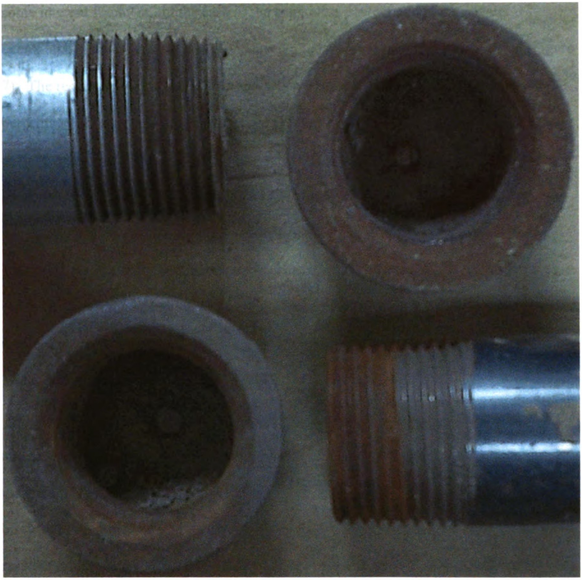

Figure 6. Examples of the oxidation observed on the threads and caps of the steel IEDs post-blast. The top pipe and cap are from sample S2B, the bottom pipe and cap are from sample S4.

some internal fractures. In the case of the smokeless powder and black powder exemplars, many of the fragments displayed a large amount of internal fracturing (Figure 7). The results of the fragmentation analysis are presented in Figure 8, which shows the percentage of fragments of each size type for each device in the PVC sample set as well as the two exemplar samples. All of the samples except for P1 and P1B fragmented to some degree, and only two samples (P6 and P6B) had fragments in the small size class exceeding 50% of the total number of fragments. The rest of the samples were fairly consistent in their fragmentation characteristics, displaying an even distribution of size elements. The only exception to this was sample P2, in which a low number of fragments were recovered for examination.

Microcrystal and Microchemical Examinations

All microchemical and microcrystal examinations were performed on the PLM, and all data reported in Table 5. In the analysis of the PVC samples, 100% of the unconsumed material samples displayed diamond shaped crystals of low order birefringence in the water recrystalization examination. The consumed material samples resulted in diamond shaped crystals of low order birefringence in 93% of the water recrystalizations, with sample P6 giving inconclusive results. In 86% of the residue samples low order diamond shaped crystals were formed during the water recrystalization, with P5B giving inconclusive results and P6 giving negative results. All material types from each sample in the PVC sample set formed hexagonal crystals in the platinum chloride microchemical test. In the chloroform recrystalization examination, 80% of the unconsumed samples formed saw shaped crystals, 29% of the consumed

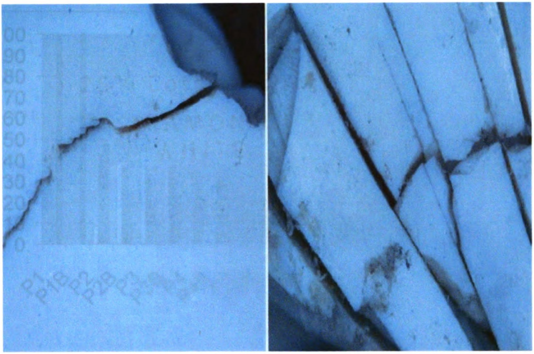

Figure 7. Examples of the fracturing observed in the fragments of the PVC IEDs The fragment on the left is from sample P3, the fragment on the right is from sam PSmokeless. Note that while some minor fractures are observed in the fragment the P3 sample IED ,extensive fracturing is observed in the fragment from the PSmokeless IED.

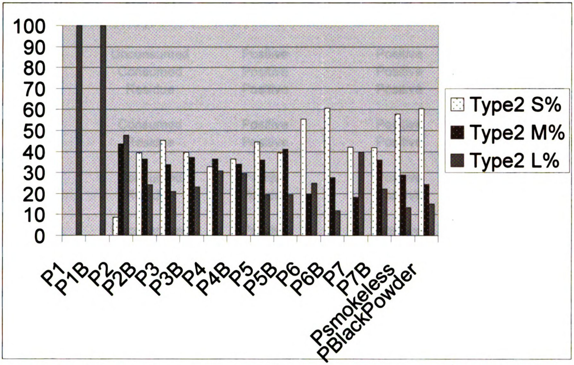

Figure 8. Plot of the percentages of total fragments in each size grouping for all unknown PVC samples, as well as the two PVC exemplars. Note that only samples P1, P1B, P6 and P6B, display significant differences in size distributions from the other unknown samples.

Sample Name	Material Type	H2O Recrystalization	PTCL Microchemical	CHCL3 Recrystalization
P1				
	Unconsumed	Positive	Positive	Positive
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
P1B				
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Positive (slight)
P2				
	Consumed	Positive	Positive	Positive (slight)
	Residue	Positive	Positive	Inconclusive
P2B				
	Unconsumed	Positive	Positive	Inconclusive
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
P3				
	Unconsumed	Positive	Positive	Positive
	Consumed	Positive	Positive	Positive (slight)
	Residue	Positive	Positive	Inconclusive
P3B				
	Unconsumed	Positive	Positive	Positive
	Consumed	Positive	Positive	Positive (slight)
	Residue	Positive	Positive	Inconclusive
P4				
	Unconsumed	Positive	Positive	Positive
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
P4B				
	Unconsumed	Positive	Positive	Positive
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
P5				
	Unconsumed	Positive	Positive	Positive
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
P5B				
	Unconsumed	Positive	Positive	Positive
	Consumed	Positive	Positive	Inconclusive
	Residue	Inconclusive	Positive	Inconclusive
P6				
	Unconsumed	Positive	Positive	Inconclusive
	Consumed	Inconclusive	Positive	Positive
	Residue	Negative	Positive	Inconclusive

Table 5. Experimental results from the microcrystal and microchemical examinations of the materials recovered from the sample IEDs.

Sample Name	**Material Type**	**H2O Recrystalization**	**PTCL Microchemical**	**CHCL3 Recrystalization**
P6B				
	Consumed	Positive	Positive	Positive
	Residue	Positive	Positive	Inconclusive
P7				
	Unconsumed	Positive	Positive	Positive
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
P7B				
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S1				
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S1B				
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S2				
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S2B				
	Unconsumed	Positive	Positive	Inconclusive
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S3				
	Consumed	Positive	Positive	Inconclusive
	Residue	Negative	Positive	Inconclusive
S3B				
	Unconsumed	Positive	Positive	Inconclusive
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S4				
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S4B				
	Residue	Positive	Positive	Inconclusive
S6				
	Residue	Positive	Positive	Inconclusive
S6B				
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S7				
	Unconsumed	Inconclusive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive
S7B				
	Consumed	Positive	Positive	Inconclusive
	Residue	Positive	Positive	Inconclusive

Table 5 (cont'd)

samples gave saw shaped crystals, and 7% of the residues formed saw shaped crystals. All other results in the chloroform recrystalization were inconclusive, with the sample displaying some crystal formation with no clearly discernable structure.

In the water recrystalization from the steel samples; 66% of the unconsumed materials, 100% of the consumed materials, and 92% of the residues resulted in the formation of diamond shaped crystals with low order birefringence. All of the steel IEDs had at least one type of material extracted from the device which resulted in the formation of low order diamond shaped crystals by water recrystalization. All of the material types from each of the steel samples formed hexagonal crystals in the platinum chloride microchemical examination. None of the recovered materials for any of the samples gave identifiable crystals in the chloroform recrystalization examination; all results consisted of the formation of amorphous crystal groups.

Instrumental Examinations

All of the instrumental data were collected and compiled in Table 6. In the FTIR of the PVC samples, 70% of the unconsumed material, 21% of the consumed material, and 21% of the residues displayed absorbance peaks at 970 cm^{-1} and 940 cm^{-1} (Figure 9). A chromatogram of the IC Standard solution is presented in Figure 10. Ion chromatography of the unconsumed material detected chlorates, sulfates, and chlorides in 100% of the samples, and phosphates in 60% of the samples (Figure 11a). The chlorate peak was the strongest peak in all of the unconsumed material samples, with the exception of the material from sample P6 where the sulfate peak was the strongest. In the consumed materials recovered from the PVC IEDs, ion chromatography detected

Sample	Material	FTIR (KCLO3)	Chlorates (IC)	Chlorides (IC)	Sulfates (IC)	Posphates (IC)
P1						
	Unconsumed	Positive	Positive (1508.48 mg/L)	Positive (3.69 mg/L)	Positive (29.08 mg/L)	Positive (Significant)
	Consumed	Slight Positive	Positive (1.00 mg/L)	Positive (723.79 mg/L)	Positive (135.41 mg/L)	Negative
	Residue	Negative	Positive (.46 mg/L)	Positive (443.03 mg/L)	Positive (174.38 mg/L)	Negative
P1B						
	Consumed	Negative	Negative	Positive (654.41 mg/L)	Positive (29.16 mg/L)	Positive (Miniscule)
	Residue	Negative	Positive (.68 mg/L)	Positive (302.68 mg/L)	Positive (6.06 mg/L)	Negative
P2						
	Consumed	Positive	Positive (.906 mg/L)	Positive (222.37 mg/L)	Positive (59.21 mg/L)	Negative
	Residue	Slight Positive	Positive (18.31 mg/L)	Positive (114.56 mg/L)	Positive (158.38 mg/L)	Negative
P2B						
	Unconsumed	Negative	Positive (28.56 mg/L)	Positive (.36 mg/L)	Positive (1.87 mg/L)	Negative
	Consumed	Slight Positive	Positive (25.80 mg/L)	Positive (61.51 mg/L)	Positive (15.04 mg/L)	Negative
	Residue	Negative	Positive (4.25 mg/L)	Positive (53.94 mg/L)	Positive (6.17 mg/L)	Negative
P3						
	Unconsumed	Positive	Positive (125.20 mg/L)	Positive (7.03 mg/L)	Positive (3.21 mg/L)	Positive (Miniscule)
	Consumed	Negative	Positive (.91 mg/L)	Positive (287.96 mg/L)	Positive (29.41 mg/L)	Positive (Miniscule)
	Residue	Slight Positive	Positive (8.24 mg/L)	Positive (99.74 mg/L)	Positive (11.56 mg/L)	Negative
P3B						
	Unconsumed	Negative	Positive (4.09 mg/L)	Positive (.69 mg/L)	Positive (2.42 mg/L)	Negative
	Consumed	Negative	Positive (4.01mg/L)	Positive (22.11 mg/L)	Positive (5.76 mg/L)	Negative
	Residue	Negative	Positive (5.68 mg/L)	Positive (100.52 mg/L)	Positive (7.02 mg/L)	Negative

Table 6. Experimental results from the FTIR and IC examinations of the materials recovered from the sample IEDs.

P4						
	Unconsumed	Positive	Positive (Dominant)	Positive (2.70 mg/L)	Positive (Weakest)	Positive (9.46 mg/L)
	Consumed	Negative	Negative	Positive (52.56 mg/L)	Positive (3.19 mg/L)	Negative
	Residue	Negative	Positive (.24 mg/L)	Positive (4.45 mg/L)	Positive (3.08 mg/L)	Positive (Miniscule)
P4B						
	Unconsumed	Positive	Positive (71.66 mg/L)	Positive (3.87 mg/L)	Positive (2.05 mg/L)	Positive (Miniscule)
	Consumed	Negative	Positive (Weakest)	Positive (909.24 mg/L)	Positive (39.78 mg/L)	Positive (Significant)
	Residue	Negative	Positive (1.10 mg/L)	Positive (62.60 mg/L)	Positive (5.86 mg/L)	Positive (Miniscule)
P5						
	Unconsumed	Positive	Positive (445.29 mg/L)	Positive (10.24 mg/L)	Positive (4.75 mg/L)	Negative
	Consumed	Negative	Positive (1.51 mg/L)	Positive (504.04 mg/L)	Positive (23.79 mg/L)	Positive (Miniscule)
	Residue	Negative	Positive (Weakest)	Positive (3.57 mg/L)	Positive (7.81 mg/L)	Negative
P5B						
	Unconsumed	Slight Positive	Positive (34.15 mg/L)	Positive (2.11 mg/L)	Positive (1.99 mg/L)	Positive (Miniscule)
	Consumed	Negative	Positive (3.22 mg/L)	Positive (116.32 mg/L)	Positive (14.79 mg/L)	Positive (Miniscule)
	Residue	Negative	Positive (2.45 mg/L)	Positive (17.31 mg/L)	Positive (3.74 mg/L)	Negative
P6						
	Unconsumed	Negative	Positive (2.32 mg/L)	Positive (.82 mg/L)	Positive (4.48 mg/L)	Positive (Miniscule)
	Consumed	Negative	Positive (.54 mg/L)	Positive (.84 mg/L)	Positive (5.34 mg/L)	Positive (Miniscule)
	Residue	Negative	Positive (.70 mg/L)	Positive (1.04 mg/L)	Positive (3.02 mg/L)	Negative
P6B						
	Consumed	Negative	Positive (Weakest)	Positive (31.11 mg/L)	Positive (Moderate)	Positive (Miniscule)
	Residue	Slight Positive	Positive (4.38 mg/L)	Positive (151.36 mg/L)	Positive (8.30 mg/L)	Positive (.51 mg/L)
P7						
	Unconsumed	Positive	Positive (12.93 mg/L)	Positive (1.10 mg/L)	Positive (Weakest)	Negative
	Consumed	Negative	Positive (.69 mg/L)	Positive (365.79 mg/L)	Positive (11.87 mg/L)	Positive (3.59 mg/L)
	Residue	Negative	Positive (.34 mg/L)	Positive (298.26 mg/L)	Positive (5.71 mg/L)	Positive (9.45 mg/L)

Table 6 (cont'd)

P7B						
	Consumed	Negative	Positive (1.72 mg/L)	Positive (249.66 mg/L)	Positive (19.08 mg/L)	Negative
	Residue	Negative	Positive (.23 mg/L)	Positive (99.89 mg/L)	Positive (2.65 mg/L)	Positive (Miniscule)
S1						
	Consumed	Slight Positive	Negative	Positive (Dominant)	Positive (27.13 mg/L)	Positive (Miniscule)
	Residue	Negative	Negative	Positive (48.58 mg/L)	Positive (28.82 mg/L)	Negative
S1B						
	Consumed	Negative	Negative	Positive (Dominant)	Positive (3.20 mg/L)	Positive (Miniscule)
	Residue	Negative	Negative	Positive (50.45 mg/L)	Positive (12.39 mg/L)	Negative
S2						
	Consumed	Negative	Negative	Positive (1048.07 mg/L)	Positive (4.02 mg/L)	Positive (1.60 mg/L)
	Residue	Negative	Negative	Positive (3762.23 mg/L)	Positive (74.51 mg/L)	Negative
S2B						
	Unconsumed	Negative	Positive (6.27 mg/L)	Positive (.34 mg/L)	Positive (Moderate)	Negative
	Consumed	Negative	Negative	Positive (417.82 mg/L)	Positive (8.13 mg/L)	Positive (Miniscule)
	Residue	Negative	Positive (Miniscule)	Positive (Dominant)	Positive (72.72 mg/L)	Negative
S3						
	Consumed	Negative	Negative	Positive (2308.99 mg/L)	Positive (2.68 mg/L)	Positive (Miniscule)
	Residue	Negative	Negative	Positive (19.71 mg/L)	Positive (45.60 mg/L)	Negative
S3B						
	Unconsumed	Positive	Positive (226.30 mg/L)	Positive (.49 mg/L)	Positive (1.80 mg/L)	Positive (Miniscule)
	Consumed	Negative	Positive (Miniscule)	Positive (4041.15 mg/L)	Positive (2.98 mg/L)	Negative
	Residue	Negative	Positive (Miniscule)	Positive (426.47 mg/L)	Positive (29.86 mg/L)	Negative
S4						
	Consumed	Negative	Negative	Positive (2275.37 mg/L)	Positive (70.59 mg/L)	Positive (32.39 mg/L)
	Residue	Negative	Positive (.21 mg/L)	Positive (262.24 mg/L)	Positive (16.63 mg/L)	Negative
S4B						
	Residue	Negative	Positive (Miniscule)	Positive (19.51 mg/L)	Positive (19.06 mg/L)	Negative

Table 6 (cont'd)

S6						
	Residue	Negative	Positive (Miniscule)	Positive (18.62 mg/L)	Positive (17.24 mg/L)	Negative
S6B						
	Consumed	Negative	Negative	Positive (84.17 mg/L)	Positive (1.17 mg/L)	Positive (Miniscule)
	Residue	Negative	Positive (.54 mg/L)	Positive (1839.41 mg/L)	Positive (23.92 mg/L)	Positive (.84 mg/L)
S7						
	Unconsumed	Slight Positive	Positive (Dominant)	Positive (.49 mg/L)	Positive (.70 mg/L)	Negative
	Residue	Negative	Positive (.10 mg/L)	Positive (95.72 mg/L)	Positive (11.06 mg/L)	Negative
S7B						
	Consumed	Negative	Positive (Miniscule)	Positive (511.46 mg/L)	Positive (2.34 mg/L)	Positive (5.85 mg/L)
	Residue	Negative	Negative	Positive (33.11 mg/L)	Positive (15.80 mg/L)	Negative

Table 6 (cont'd)

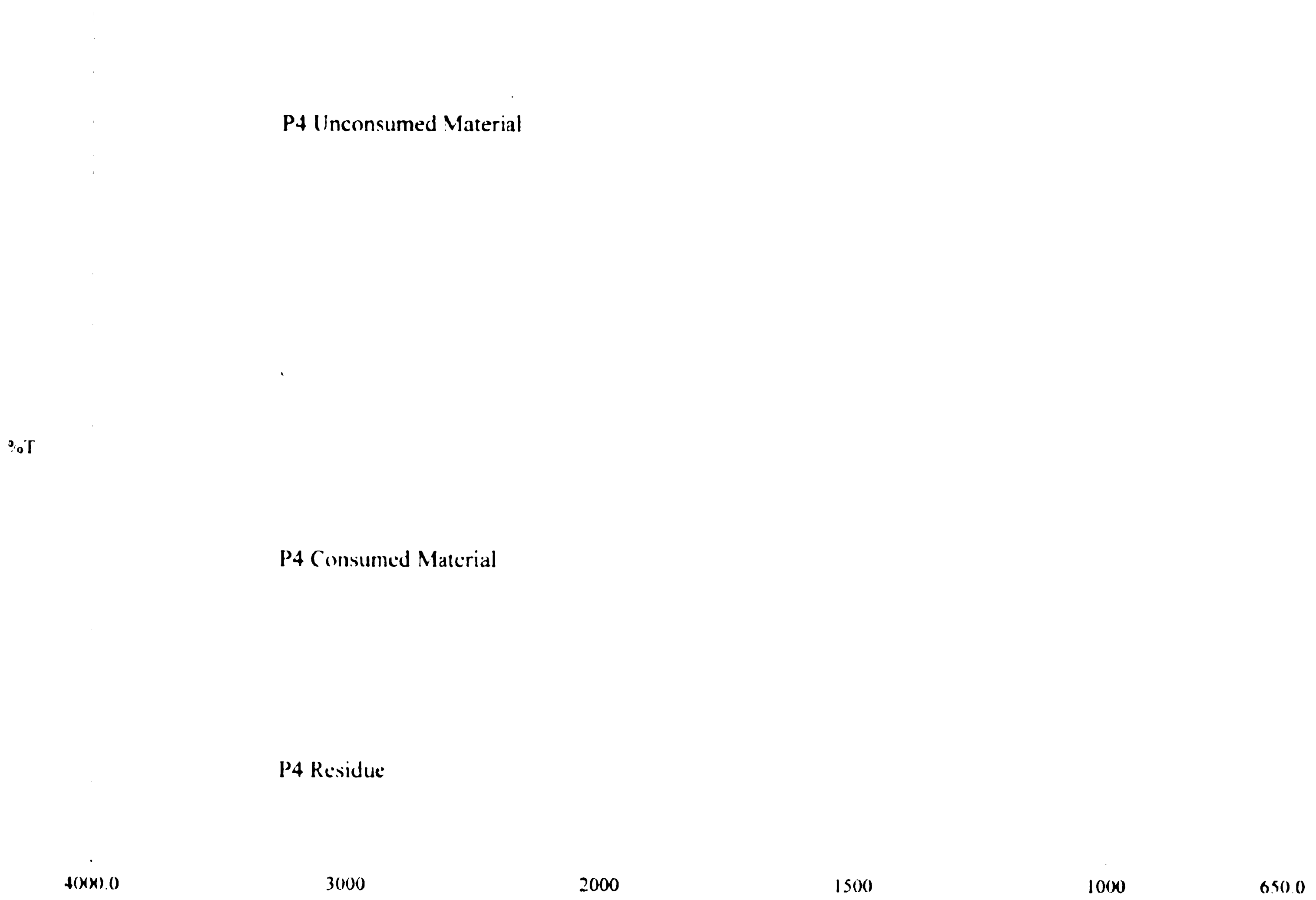

Figure 9. FTIR spectra of the aqueous extracts of the three material types recovered from sample P4. Note that only the unconsumed material extract displays the characteristic 970 cm^{-1} and 940 cm^{-1} peaks associated with potassium chlorate.

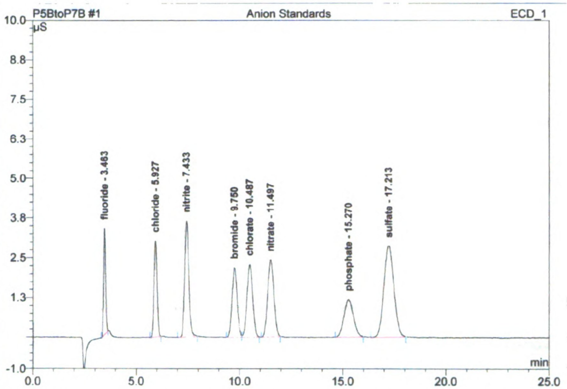

Figure 10. IC chromatogram of the standard sample run in the Anion method. Retention times of the 8 standard anions are displayed in minutes. Fluoride (3.4), chloride (5.9), nitrite (7.4), bromide (9.7), chlorate (10.4), nitrate (11.4), phosphate (15.2), and sulfate (17.2).

chlorates in 86% of the samples, sulfates and chlorides in 100% of the samples, and phosphates in 66% of the samples (Figure 11b). The chloride peak was the most dominant peak in the consumed material samples, with the exception of sample P6 where the sulfate peak was the strongest. In the PVC sample residues, chlorates, sulfates, and chlorides were detected in 100% of the samples, and phosphates were detected in 36% of the samples (figure 11c). The chloride peak was the strongest peak in the residue samples, with the exception of samples P2, P5, and P6 where the sulfate peak was the strongest.

In the FTIR analysis of the steel samples, 66% of the unconsumed material, 11% of the consumed material, and none of the residue material displayed absorbance peaks at 970 cm^{-1} and 940 cm^{-1} (Figure 12). Ion chromatography of the unconsumed materials from the steel IEDs detected chlorates, chlorides, and sulfates in 100% of the samples, and phosphates in 33% of the samples (Figure 13A). In the recovered consumed materials from the steel IEDs chlorides and sulfates were detected in 100% of the samples, chlorates were detected in 22% of the samples, and phosphates were detected in 89% of the samples (Figure 13B). In the steel IED residue samples chlorides and sulfates were detected in 100% of the samples, chlorates were detected in 58% of the samples, and phosphates were detected in 8% of the samples (Figure 13C). Bromides were detected in either the consumed materials or the residues recovered from 50% of the samples, but were not detected in any of the unconsumed materials recovered. The samples which indicated the presence of bromides were samples S1, S2, S2B, S3B, S4B, and S6. In addition, an unidentified peak with a retention time of approximately 23

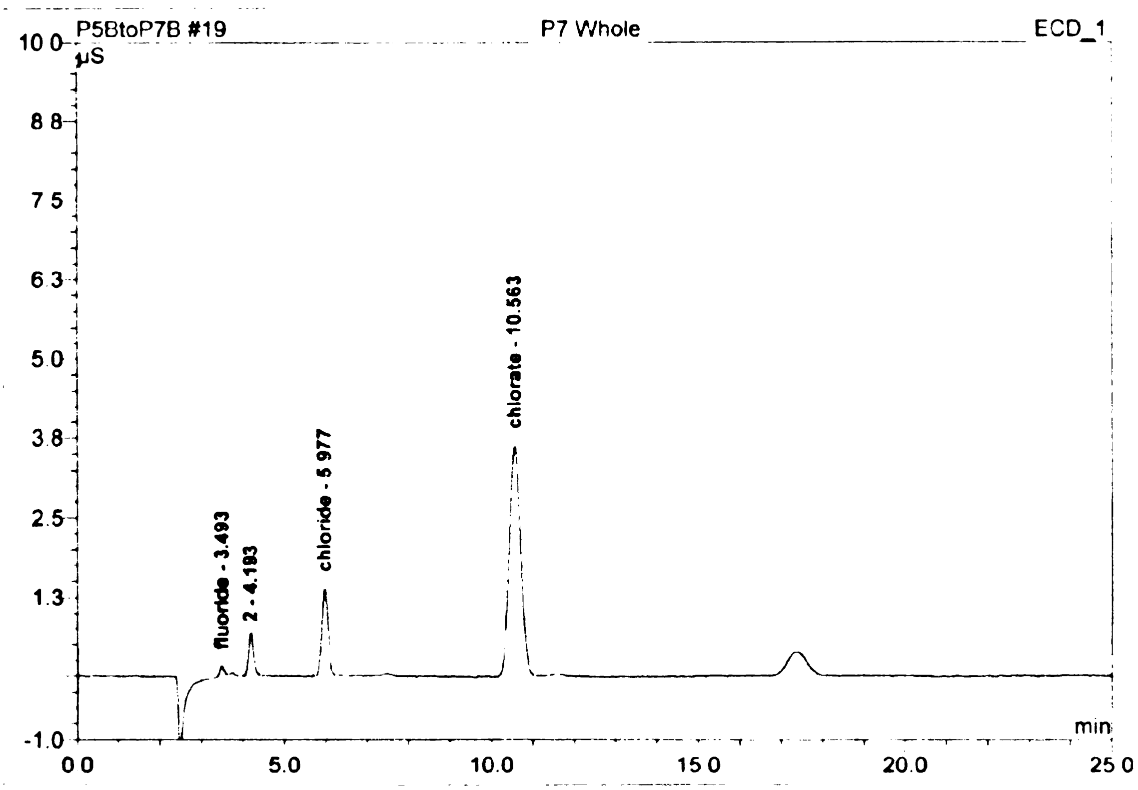

Figure 11. IC chromatograms of the aqueous extracts from the three material types recovered from sample P7. The sulfate peak is unlabeled in Figure 11a, but comparison with standard chromatograms indicates that the peak near 17 minutes is sulfate. A. Chromatogram of the extract from the unconsumed material.

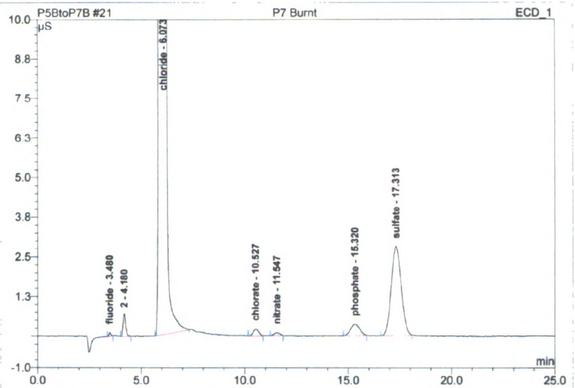

Figure 11B. Chromatogram of the extract from the consumed material.

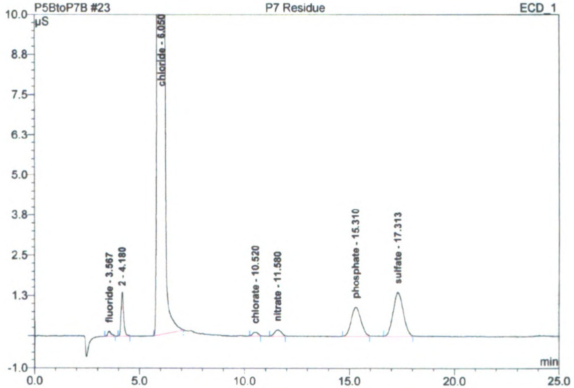

Figure 11C. Chromatogram of the extract from the residue material.

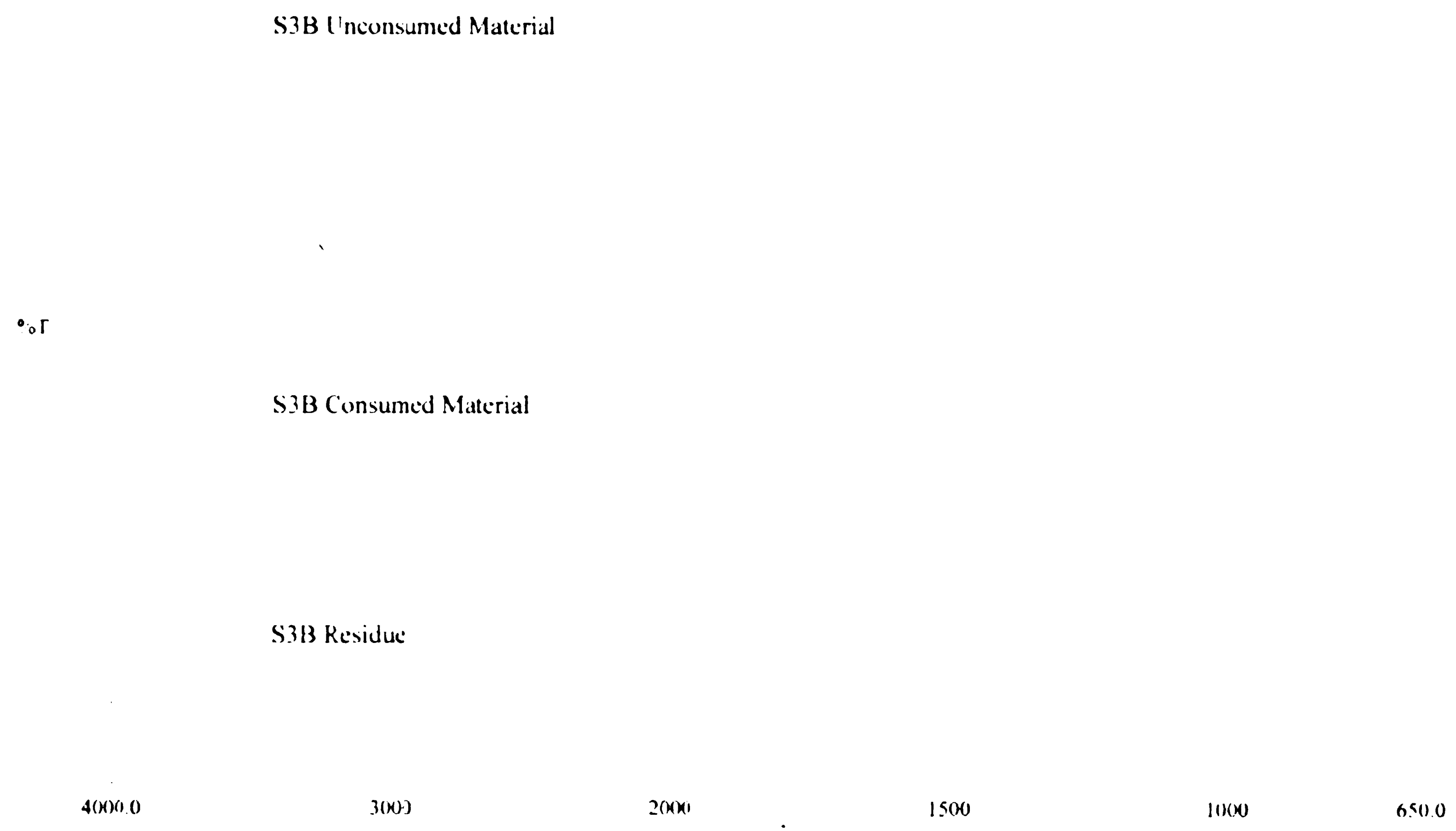

Figure 12. FTIR spectra of the aqueous extracts of the three material types recovered from sample S3B. Note that only the unconsumed material extract displays the characteristic 970 cm^{-1} and 940 cm^{-1} peaks associated with potassium chlorate.

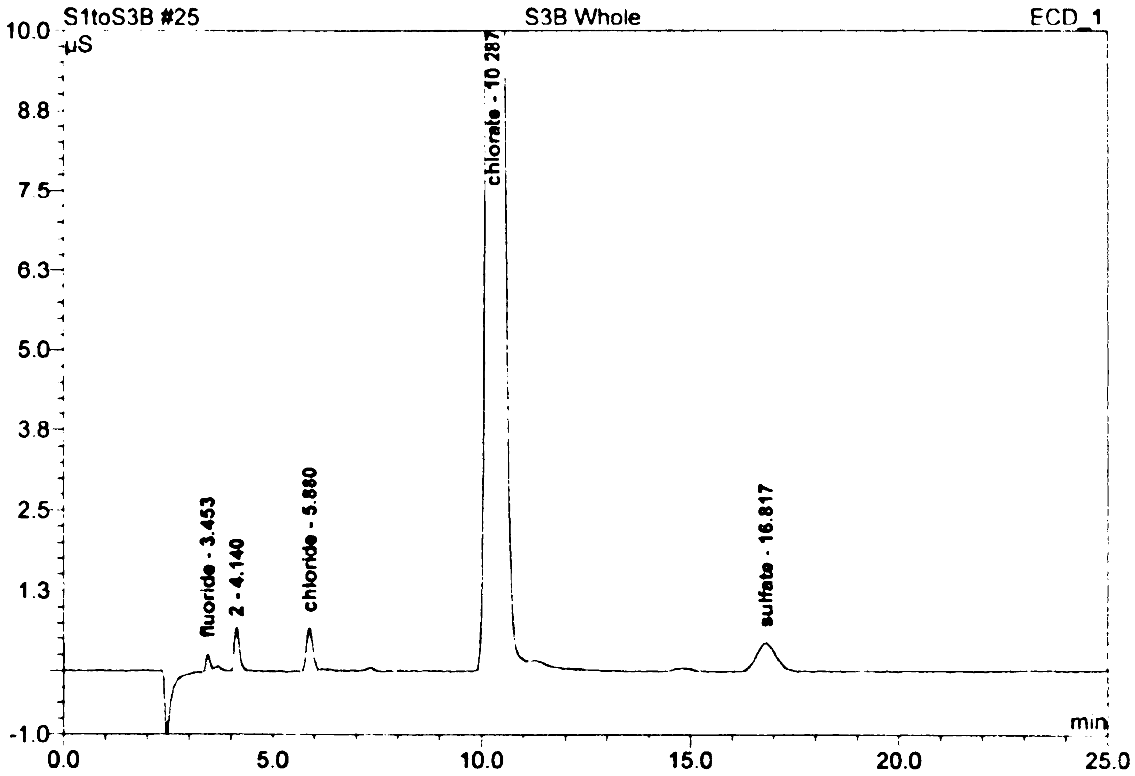

Figure 13. IC chromatograms of the aqueous extracts from the three material types recovered from sample S3B. Note the small unlabeled peak near 15 minutes in the unconsumed extract, indicating detection of phosphates. **A.** Chromatogram of the extract from the unconsumed material.

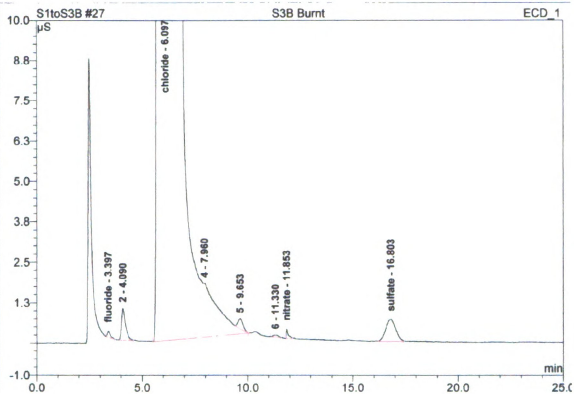

Figure 13B. Chromatogram of the extract from the consumed material.

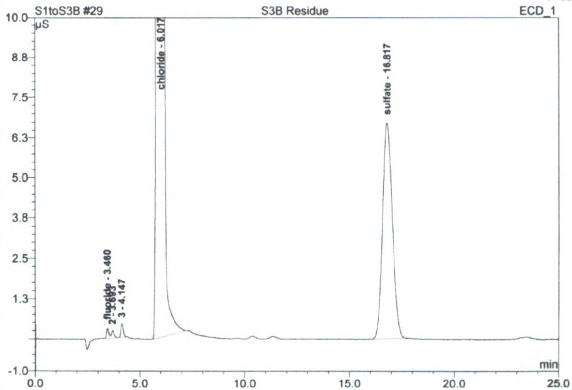

Figure 13C. Chromatogram of the extract from the residue.

minutes was observed in either the consumed material or residue recovered from 58% of the steel devices, this peak was not observed in any of the recovered unconsumed material (Figure 14). The samples with IC spectra displaying this peak were samples S1, S1B, S2, S2B, S3, S3B, and S7.

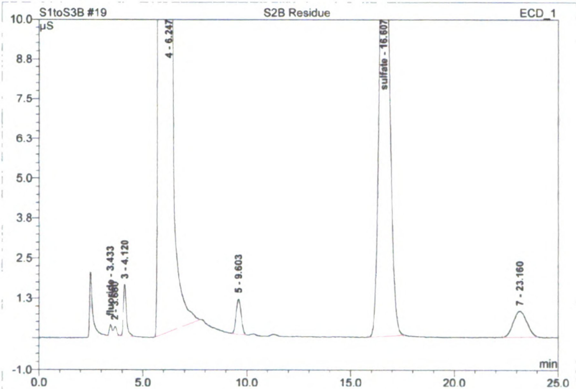

Figure 14. IC chromatogram of the aqueous extract from the residue of sample P2B. The peak at 9.6 minutes was identified as bromide by comparison with a standard spectrum. The peak near 23 minutes was unidentifiable by comparison with a standard spectrum.

DISCUSSION

Physical Examinations

The presence of rust on the steel pipes is most likely due to chemical interactions between the surface of the pipes and the potassium chlorate. As potassium chlorate is a strong oxidizer, it is likely that the residual potassium chlorate from the explosive material facilitated oxidation of the pipes during the time period between the detonation of the devices and their subsequent analyses four months later. This is of significance, as the oxidation process can consume the potassium chlorate, presenting difficulties in the analysis of devices.

In examining the residue on the devices, it was noted that 75% of the steel and 100% of the PVC devices displayed residues with a reddish or orange coloring. This is most likely due to the red coloring of the material from the match heads, as it was not observed in the black powder or smokeless powder exemplars (Figure 15). The brown coloring was present only in the PVC devices and may be due to one or both of the following possibilities. Due to the fact that most of the PVC IEDs fragmented to some extent, the interior surfaces of the devices were exposed to the soil that the samples were transported in, and because of this soil material may have become ingrained on the interior surfaces with the residue material. The other possibility is that the darkening observed in the PVC samples is due to scorching of the plastics during the deflagration of the devices. This seems likely as PVC melts at 175 degrees C, while the initial ignition temperature of a match is 260 degrees C and temperatures of match flames may reach up to 2500 degrees C. It is most likely that the brown coloring is a result of both of these factors. In addition to the coloring differences between the steel and PVC samples, the

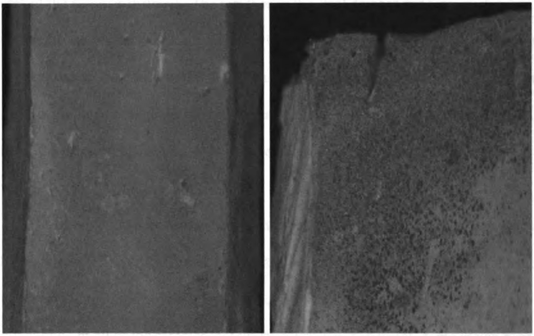

Figure 15. Photograph displaying the residue color from samples P4 (left) and PBlackPowder (right). Note the red-orange color of the P4 sample and the gray color of the PBlackPowder sample.

natures of the residues differed. The residues in the steel devices appeared particulate in nature, while the residues from the PVC devices had a more film-like consistency with some particulates embedded in the film. The film-like residue of the PVC IEDs can most likely be attributed to melting of the PVC surface during deflagration, allowing the particles from the explosive load to become embedded in the surface before the PVC resolidified.

The presence of unconsumed and consumed material is of particular importance in the analysis of these IEDs. In addition to the benefit of having load material to analyze for chemical content, the physical appearance of the material is useful in directing examinations. In all of the samples that utilized whole match heads, some match heads were found either within the device or scattered in the blast radius with the fragments of the IED during the collection phase. Most of the heads had unconsumed material on them, with only device P1 yielding a whole head with unconsumed material on it (Figure 16). Device S7 did not contain any whole heads with material on them, however whole heads without material were identifiable in the device. This is significant, as investigators can expect to find whole heads if a "cut" method was used in the construction of IEDs, which will in turn be helpful in ultimately characterizing the explosive load used in the device.

The coloring of the match head material makes it easily differentiable from most low explosive mixtures. Some smokeless powders contain red coloring, including the Red Dot powder used in the smokeless powder exemplars. In comparing the morphologies of the Red Dot smokeless powder to that of the unconsumed material, differences were observed (Figure 17). The Red Dot smokeless powder had crushed ball

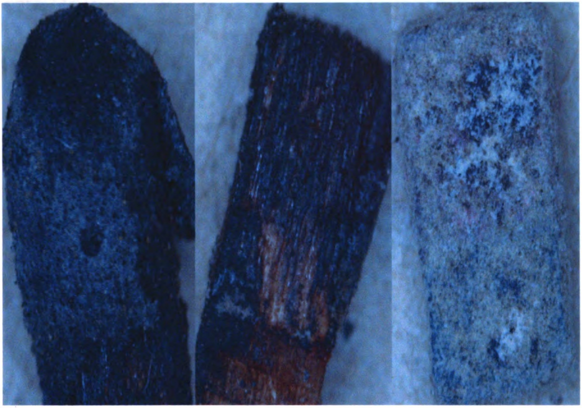

Figure 16. Photomicrograph of recovered intact match heads from various devices.

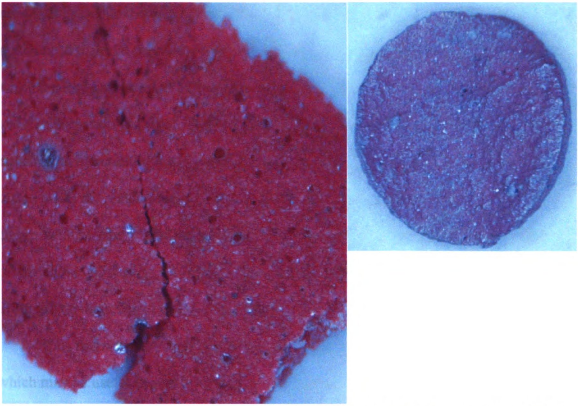

Figure 17. Photomicrograph of chipped match head material (left) and a flake of Red Dot commercial smokeless powder (right). Note the differences in shape, color, and luster in the two samples.

morphology, while the match head chips had more irregular shapes and edges. The match head material was much more granular in appearance and had a bright red color, while the Red Dot powder was smoother in surface texture with a more lustrous coating and a dull maroon-red color. Based on morphology, it was possible to differentiate the match head material from the Red Dot smokeless powder, and it may be possible to differentiate the match head material from other colored pyrotechnic mixtures and commercial powders. A point to note however is that many commercial matches are produced with different colorings, which may either make it easier or more difficult to differentiate them from other commercial low explosives.

The fragmentation analysis of the PVC IEDs gave some suggestive information which may be useful in characterizing the method used to prepare the explosive load in a match head based device. The P1 and P1B devices employed the cut wooden "safety" matches, and were the only devices of the set to display no fragmentation. This seemed consistent with the hypothesis that the wood from the match body inside the heads would decrease the overall brisance of the load. Conversely, the only steel IEDs that fragmented (S6 and S6B) employed ground "strike anywhere" loads, which seems to indicate that this preparation yields the explosive load with the greatest brisance. The fragmentation analysis seems to support this conclusion, as the two PVC devices employing ground "strike anywhere" match material (P6 and P6B) were the only PVC samples that had more than 50% of the fragments collected in the small size class. The fragmentation grouping of the P6 and P6B devices were similar to that of the exemplars (smokeless powder and black powder), being predominantly small fragments, suggesting again that the ground "strike anywhere" material is the explosive load with the greatest brisance. With the exception of the weakest devices (P1 and P1B) and the strongest devices (P6 and P6B), no clear differentiation between preparations can be made based on fragment size groupings. All of the remaining samples had fairly consistent size groupings with no discernable pattern. The only device, aside from the aforementioned exceptions, that was differentiable was sample P2, which had only 4% of the fragments in the small size class. This may not be due to the observed brisance of the device and is most likely due to the fact that only 23 fragments were recovered from sample P2. It is probable that many of the fragments were lost in the collection phase due to an incomplete collection of fragments, and it is more likely that the fragments that were not recovered would have fallen into the small size class, which would account for the low percentage of fragments in that size grouping. While there seems to be no clear differentiation between the intermediate explosive preparations by fragment sizes alone, the total sample population is not large enough to be considered representative. With such a restricted sample size, it may not be possible to see smaller differences between the various preparation types.

Microcrystal and Microchemical Examinations

The water recrystalization examination was an effective method for the characterization of potassium chlorate in the samples. No significant crystal formations were observed in the School soil control samples, and while the Range soil control samples displayed a multitude of various crystal structures, no low order diamond shapes indicative of potassium chlorate were observed. Based on these findings, the soil could be excluded as a possible source of the crystals observed in the analyses of the sample materials. In both the PVC and steel IED samples every device displayed a positive response to the water recrystalization with at least one form of recovered material; the unconsumed material, the consumed material, or the extracted residue. Of the PVC samples, only P5B and P6 gave non-positive responses in the water recrystalization. The consumed material recovered from sample P6 gave inconclusive results, while the residue from sample P6 yielded no appreciable crystals. Sample P6 was found to have a large amount of soil covering the device surfaces during the physical examination, which may explain the non-positive results observed with the materials recovered from that device. It is possible that some substances in the soil itself interfered with the recrystalization, or that the presence of the soil merely decreased the amount of potassium chlorate that was extracted in these two material types which then led to the non-positive results. The extracted residue from P5B resulted in an inconclusive response to the water recrystalization, and this is most likely due to a low concentration of potassium chlorate in the residue that was extracted. The unconsumed material from sample S7 gave inconclusive results and the residue extracted from sample S3 gave negative results, both of which may be due to low amounts of potassium chlorate in the samples. As was discussed in the physical examination section, potassium chlorate is a strong oxidizer and it is possible that the trace amounts originally in these samples diminished over time as the potassium chlorate reacted with the pipe surface. In addition, the presence of iron oxide could have caused interference in the recrystalization of these samples, resulting in the non-positive responses.

Slight positive responses were observed in some of the soil control samples during the platinum chloride microchemical examinations. This is most likely due to the presence of some small amounts of potassium or potassium salts in the soil samples, as the hexagonal shapes are presumptive of potassium. In a negative control of the deionized (NERL), water no crystal formations were observed. Based on this, it is unlikely that the deionized (NERL) water used in the extractions or a contaminant in the platinum chloride reagent were contributing factors to the results observed in the soil control examinations. In the School soil samples, the predominant structures observed were diamond shapes, with a few small scattered hexagons present, while in the Range samples the predominant structures observed were burrs of needles and curved needles, with only a few hexagons observed in 60% of the samples. In comparison, all material types from both the PVC and steel IEDs gave strong positive responses to the platinum chloride examinations. The predominant crystal structures observed were hexagons and large amorphous angular crystals. The hexagons were observed in greater size and abundance for the IED samples than in the soil control samples, which suggests that the positive response is a result of interaction between the platinum chloride and the potassium present in the samples and not due merely to contamination from the soil. The larger amorphous crystals are most likely the result of multiple hexagon complexes combining to form larger structures, which may occur in very concentrated samples.

The background soil controls resulted in no appreciable crystal formation in the chloroform recrystalization examinations, indicating an absence of elemental sulfur in the background of the samples. The PVC IED samples displayed the formation of saw shaped crystals in 86% of the devices, with only samples P2B and P7B yielding no conclusive results in any material examined. In 80% of the unconsumed material samples a positive response was observed, while in the consumed materials only 29% of the samples gave a positive response and in the residues only one sample gave a positive response. This is most likely due to the consumption of the elemental sulfur during the deflagration process. This is supported by the results from the IC examination of samples, in which the unconsumed materials contain low concentrations of sulfates, while the consumed materials and the residues display increased concentrations of sulfates. In addition, in the reference standards of consumed match heads only the paper "safety" matches resulted in a positive response to the chloroform recrystalization, while the wooden "safety" match and the "strike anywhere" match samples both gave inconclusive results. While it is possible to obtain positive results from the chloroform recrystalization of consumed and residue materials, it should not be expected, and also should not be grounds for the exclusion of match head material as a possible explosive load in a recovered IED. In the steel samples, all of the materials examined resulted in the formation of amorphous crystal groups in the chloroform recrystalization, leading to inconclusive results. It is uncertain why the unconsumed materials from the steel devices did not yield a positive result. In all of the materials examined by chloroform recrystalization, some crystal formation was observed even though structures were not identifiable in each sample. It is possible that due to the consumption of sulfur during deflagration the concentrations of sulfur in the extracts were too low to allow for complete crystal formation in most of the consumed materials and residues. It is also possible that secondary reaction products, such as the iron oxide, created interference which prevented the formation of characteristic crystals. In addition, the amounts of unconsumed materials that were recovered from the steel IEDs were much smaller than those of the recovered unconsumed materials from the PVC IEDs. Because of this, it is possible that the concentrations of elemental sulfur in the small amounts of unconsumed materials from the steel IEDs was too low to give appreciable crystal formation.

Instrumental Examinations

The absence of absorption peaks at 970 cm^{-1} and 940 cm^{-1} in the soil control samples confirms the absence of potassium chlorate to any appreciable amount in the background of the IED samples. The FTIR analyses of the extractions from the PVC sample materials proved effective, giving confirmation of the presence of potassium chlorate in 71% of the samples. Identification of potassium chlorate was much more effective in the unconsumed materials (70%) than in the consumed materials (21%) or the residues (21%). This was to be expected as most of the potassium chlorate would have been consumed in the deflagration process, making it more difficult to detect in the consumed and residue materials. The steel sample extractions had a significantly lower success rate, with only 25% of the samples confirming the presence of potassium chlorate by FTIR. The unconsumed materials had a much greater success rate (66%) than the consumed materials (11%) or the residues (0%), which is consistent with the observations made in the PVC sample material extractions. While the unconsumed materials from the PVC and steel devices had comparable success rates, the success rates for the consumed materials and the residues in the steel devices were significantly lower than those observed from the PVC devices. This may be due to interactions between the potassium chlorate and the surface of the pipes. If significant amounts of the potassium chlorate were consumed while oxidizing the steel pipe surfaces, there may not have been enough remaining in the extractions to give conclusive results by FTIR. Because this could be a significant factor in the characterization of the potassium chlorate, it should be noted that in the case of steel IEDs rapid analysis may be necessary to avoid the loss of valuable probative information.

Ion chromatography of the soil control samples failed to detect the presence of any chlorates, indicating that the chlorate results obtained in the samples were in fact due to the presence of chlorates in the match head material. Chlorates were detected in 100% of the PVC IED samples, and were detected in 67% of the steel IED samples. The high success rates observed with this technique may be attributed to that fact that IC is a separatory technique, which would allow for the isolation of the chlorates from other substances in the sample which may have led to interference in other examinations. The absence of chlorates in the consumed material samples from some of the devices is not unexpected, as IC analysis of the consumed wooden "safety" match head exemplar did not detect chlorates. It is possible that in the samples recovered from these devices, the consumption of the potassium chlorate went to completion to such a degree as to leave concentrations of chlorates that were below the limits of detection for the system. In the steel samples, it is possible that success rates lower than those observed in the PVC samples was due to the consumption of potassium chlorate via the oxidation of the steel pipes, as discussed in the FTIR results. For both the PVC and steel sample sets, the chlorate peak was the most abundant peak in the unconsumed material samples; which can be expected as potassium chlorate is the predominant water soluble ionic species in the unconsumed match material, as observed in the match head exemplars. The only exception to this was the unconsumed material from sample P6, in which the sulfate peak was dominant. It is probable that this was due to the unusually large amount of soil present on the surfaces of the device, which could have imparted greater amounts of sulfates to the sample during the aqueous extraction.

In the consumed material and residue samples the chloride peak was the dominant peak, with the exception of samples S3, P6, P5 and P2. In both the consumed material and the residue material from sample P6, sulfates were detected as the most abundant anion which is most likely due to contributions from the soil on the sample. The residue from sample P2 had sulfates as the most abundant peak; this is most likely due to contamination from the range soil, which displayed extremely high concentrations of sulfates. It is unclear as to why in the IC analyses of the residue materials from samples P5 and S3 sulfates were the most abundant ion. It is possible that the increased sulfate concentrations could be due to soil contamination, or it is possible that there was a lower than average amount of chlorides in the residues from these samples. While chlorides were detected in the background soil samples, the amounts detected in the IED samples was of a much greater concentration than that of the soil samples, which seems to indicate that at least some of the contribution to the chloride anion peaks was from the match head material in the IEDs. The chlorides detected were most likely formed as a reaction product from the deflagration of the explosive loads, which consumed the potassium chlorate converting them to more stable chlorides. Sulfates were also detected in significant amounts in the consumed material and residue samples from the IEDs. However, the amounts of sulfates detected were comparable to the amounts detected in the soil control samples. It is possible that sulfates were formed as reaction products from the deflagration of the match head material, but due to the comparable amounts of sulfates in the soil control samples it is not clear if any of the sulfates detected were definitely from the sample materials analyzed. Also, due to the design of the experiment, true quantitative comparison between samples and controls is not possible. Because of these factors, it does not appear that the detection of trace amounts of sulfates can be used as a means to include or exclude match heads as a possible source for the explosive load in an IED, especially in instances where the IED is recovered from soil material.

Phosphates were detected to some degree in all of the match head exemplars, both unconsumed and consumed. Phosphates were detected in at least one of the materials recovered from the IEDs in 79% of the PVC devices and in 75% of the steel devices. There seems to be no correlation between the type of match head used in the construction of the device ("strike anywhere" vs. "safety") and the detection of phosphates by IC. It was originally believed that the phosphorous sesquisulfide present in the "strike anywhere" matches would lead to detectable differences in the concentrations of phosphates between "safety" devices and "strike anywhere" devices, particularly in the consumed materials and the residues, but this does not appear to be the case. It is notable however that phosphates were detected in a miniscule amount in only one of the School soil samples, and thus the detection of phosphates in the IED samples seems resultant of contributions from the match head material used as the explosive load. Based on this, it seems that the detection of phosphates by IC may be suggestive of a match head based IED, though the absence of phosphates does not necessarily exclude match heads as a possible explosive load.

Aside from differing success rates, there were some observable differences between the ion chromatographs of the steel and PVC IEDs. The detection of bromides as well as the detection of an unidentified peak at 23 minutes was observed only in steel

IED samples. In addition, the detection of these two peaks occurred only in the consumed material samples and the residues recovered from some of the steel devices. While the peak near 10 minutes in the unconsumed material sample from S7 is labeled as bromide, comparison with the anion standard suggests that the peak is actually chlorate, and the mislabeling occurred due to misinterpretation by the software. As neither the bromide peak nor the peak at 23 minutes appear in any of the soil control samples, it seems most likely that the anions being detected are from the IEDs and are not due to contaminants from the environment. Because these peaks only occur in the consumed material and residues, and not the unconsumed material, they are most likely due to reaction products from the deflagration of the match head material. The IC analysis of the paper "safety" consumed material detected bromide, so it is possible that some component of the match head material forms bromides on deflagration. It is unclear as to whether these bromides are formed in small amounts and only occasionally form to such a degree that they are detectable by IC, or if they are due to some contaminant imparted to the match head material during manufacturing. It is possible that the peak near 23 minutes was only observed in the steel samples because the anions formed are the results of interactions between a component of the sample material and the steel devices during deflagration. The presence of oxidation on the steel devices indicates that some interaction is occurring, and it is unclear as to the contribution that this secondary reaction has on the formation of water soluble organic species.

CONCLUSIONS

The analytical scheme presented is a rapid and effective method of characterizing match heads as the explosive load used in IEDs. The combination of analytical techniques creates a class of physical and chemical characteristics that can exclude most common low explosives as possible materials used in the construction of such devices. By employing this system of analysis, 100% of the PVC IEDs and 75% of the steel IEDs were characterized sufficiently to identify the explosive load according to TWGFEX guidelines. Physical examination of the recovered device is effective in narrowing the range of focus for the chemical analysis of IED components. In the case of devices constructed using cut match heads it is expected that some heads would be recovered from a blast site, giving a clear indication of the type of material and preparation used. Particularly in the case of PVC devices the recovery of unconsumed materials can be expected, and a physical examination can be used to exclude most commercial low explosives and pyrotechnic mixtures as possible sources based on morphology alone. The coloration and appearance of residue material on the fragments may also suggest to the analyst that the device in question was a match head based IED. The high success rate observed in the water recrystalization analyses for all of the recovered material types allows for a rapid and inexpensive chemical analysis that can detect the presence of potassium chlorate; which is an uncommon low explosive oxidizer particularly in commercial preparations produced within the United States. The greatest drawback to the microchemical and microcrystal examinations is that they rely on the subjective interpretation of the analyst. Analysts uncomfortable or unfamiliar with PLM methods may not receive confident results with the microscopic examinations that were performed in this study. Specialized training course offered through various agencies such as the McCrone Institute of professional organizations may provide analysts with more in depth instruction in these methods. The detection of elemental sulfur by chloroform recrystalization was fairly effective in the PVC samples, and successful identification of sulfur can be a simple means for the exclusion of commercial smokeless powders as possible explosive loads, as they do not contain elemental sulfur in their formulations.

FTIR is an effective means of identifying potassium chlorate particularly in unconsumed material, and could be an effective method of analysis for analysts inexperienced with PLM examinations. However, due to the use of pressed KBr pellets in the scheme for the FTIR analyses some amount of sample was lost, and in cases with limited sample material this loss may not be acceptable. It should be noted that every sample material which detected potassium chlorate by FTIR also detected potassium chlorate in the PLM examinations, potassium in the microchemical examination, and chlorates in IC. In light of these observations, FTIR appears to be the least effective method of characterizing potassium chlorate from match head based IEDs, and may not be necessary if the other three methods are successful. IC has shown to be the most effective instrumental method used in this study for the analysis of match head based IEDs. The separatory nature of the system can accommodate for complex sample mixtures, allowing the isolation and detection of desired anions. In addition, the low limits of detection for the system allowed for the detection of chlorates in samples where FTIR was unable to clearly identify potassium chlorate, which was particularly useful in the examination of the consumed materials and residues. The success rate of the detection of chlorates by IC is exceptional, and on par with the success rates of the microchemical and microcrystal examinations. In addition, the high concentrations of chlorides detected in the consumed materials and residues seems suggestive of the consumption of chlorates during the deflagration process, which may be significant in the cases where the concentration of chlorates is at an undetectable level (such as is in samples P1B and P4). While it is possible that the detection of sulfates could be suggestive of a match head based IED, the extent of contamination from the soil at the detonation sites cannot adequately be accounted for in this study and as such no conclusions based on sulfate detection by IC can be made regarding the nature of the device. The detection of phosphates, while less successful than the detection of chlorates or chlorides, could be suggestive of a match head based device. However, their absence cannot be used as a means for the exclusion of match heads as having been used in an IED as they were not detected in all of the reference match head samples. The major drawback to analysis by IC is that only the anions of a sample are detected. While chlorates were detected in all of the samples, the IC methods employed were incapable of detecting the cations in the samples. Thus, while it is possible to identify the presence of chlorates, IC is incapable of characterizing potassium chlorate specifically. Thus, it is important to employ complementary techniques such as PLM examinations or SEM-EDS.

Despite the inability to differentiate "strike anywhere" match based IEDs from "safety" match based IEDs, this study has demonstrated that the analytical scheme employed is effective in characterizing commercial match heads as the explosive load used in IEDs. The combination of morphological examinations, instrumental techniques and microscopic analyses has been shown to be effective in identifying characteristics that can be used to include commercial match heads as the possible explosive load, while simultaneously eliminating other common low explosives. Most of the techniques that were used in this study are commonly employed by forensic analysts, so the implementation of this scheme presents no additional costs to crime laboratories. Most importantly, the analytical scheme presented in this study is a rapid and effective method for analyzing match head based IEDs.

FUTURE WORK

This study serves as a preliminary evaluation of methods that may be employed for the analysis of match head based IEDs. More in depth examinations of various aspects of this study would serve to strengthen the conclusions drawn herein. A larger sample population for each of the device construction types would allow for a more statistically significant population, which would in turn allow for more meaningful data to be obtained from fragmentation pattern analyses. Also, a more effective sample collection method should be employed to ensure that all fragments would be collected for size grouping, as some of the smaller fragments may have been lost during recovery in this study. Another area of this study that warrants further investigation is the detection of sulfates by IC. A method of sample collection would have to be employed that would eliminate possible background contamination, as deflagration in soil has been shown to be a likely source of contamination. A comparative study with other chlorate based explosive mixtures should also be performed. Such a study would strengthen the class characteristics identified in this study as being indicative of match head based IEDs. Finally, other methods of detection should be researched, as no method employed in this study was able to chemically differentiate samples constructed using "strike anywhere" matches from samples employing "safety matches". Such a differentiation would create smaller class sizes for match head based IEDs and thus help to eliminate Type II errors in casework.

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