



<b>ATF-LS-TE14</b> <b>Examination of General Unknowns and Uncommon Evidence</b>	Published Online: <b>March 2018</b>
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## I. SCOPE

Any substance or item that may be taken away from a crime scene or left at a crime scene by the suspect or victim may become important evidence. For this reason, a veritable plethora of different types of substances may become evidence in a case. These types of items may include but are in no way limited to items such as wood, paper, leather, feathers, matches, soils, tobacco, and household goods such as cleaning products or food items. As a part of the investigation, the trace evidence examiner may be asked on occasion to identify or compare these types of items.

It is impossible to design a single analytical scheme which is capable of analyzing or identifying all substances. Due to this fact, the examiner must utilize common methods, laboratory equipment, and known reference materials or standards to affect identifications and/or conduct a comparison as needed on a case by case basis.

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Note: Refer to Periodic Table of Elements for Chemical abbreviations.

### **Validation**

The techniques described below for examination of general unknowns and uncommon evidence are all well known and scientifically accepted in the forensic science community and in the relevant private industry of each material. Relevant examples of related literature can be found in Section II (References).

### **III. SAFETY PRECAUTIONS**

1. Use appropriate safety garments and apparatus (glasses, gloves, lab coat)
2. Care should be taken when handling chemicals and/or any physical evidence.

### **IV. APPARATUS / REAGENTS**

Due to the wide variety of substances that may be encountered, the following is a list showing some of the equipment and/or materials which may commonly be used:

1. Stereomicroscope
2. Microscope with high magnification such as polarized light microscope or comparison microscope
3. SEM/EDS
4. XRF
5. XRD
6. GC-MS or Py-GC-MS
7. FTIR

8. Microspectrophotometer
9. Hot Stage Microscopy
10. Miscellaneous solvents and/or chemicals
11. Glass microscope slides, cover slips, mounting media
12. Litmus paper

#### **Calibration / Performance Checks and Adjustments**

Microscopes, micrometers / measuring devices, and all scientific equipment should be properly calibrated or performance checked according to the protocols for each instrument.

#### **V. PROCEDURES**

When attempting to identify general unknown substances, contact with the investigating officer prior to any analyses may provide useful information about items related to the victim, suspect, or crime scene. This could prove useful in narrowing down potential sources or possible identity of the general unknown in question.

When a particular substance is suspected or known to the examiner as a possible source/identity of the unknown item of evidence, it may prove useful to call the manufacturer of the consumer product for information about product processing, ingredients, and packaging. Internet searches are also a good source of information.

Controls or standards are often not submitted with evidence. A similar store bought item may prove useful as a reference.

#### **Visual Examination**

Visual examination of the submitted item is often the first step in identification or comparison of general unknowns or uncommon evidence items. Low power magnification may be used when applicable. This may be the only step necessary to affect an identification of some evidence items. Any significant physical characteristics such as size, color, texture, shape, or odor should be noted.

If the specimen is a liquid, check for sediments, suspensions and any liquid interface. Foaming upon shaking may indicate soap or detergent. Items such as soap, detergents and cleaning powders are frequently encountered in criminal complaints.

#### **Analytical Methods**

Due to the wide range of samples encountered in this type of case work, the type of analyses conducted on the specimen will be determined on a case by case basis. Using the case history, the type of sample submitted as a guide if available, and the observations made during the visual examination; the examiner should decide which analytical methods are appropriate. The following are just a few of the common laboratory methods that may be utilized:

1. Microscopical examinations may lead to identification of the unknown substance and may be the only method necessary for comparison of some uncommon evidence items. General morphology as well as observation of the substance under controlled lighting conditions will aid in the identification and comparison. Starch, fungus, soil, feathers, leather, wood, paper, and plant material are just a few of the substances which can be identified and compared using stereomicroscopy and polarized light microscopy (See TE02 Set-up and Use of the Microscope).

2. Test pH of a liquid sample and if possible compare to pH of control. If pH is unusual, the examiner may test for acids or bases—typical are hydrochloric acid and sodium hydroxide (See Appendix I).
3. If unusual odors are present, a sample of the headspace injected on the GC/MS or via carbon strip (see Fire Debris protocols) may identify volatile substances. Some halogenated compounds can be detected by spot tests (See Appendix I).
4. Toxic metals can be detected by using the Reinsch test. This test can be applied directly to body fluids, tissue slurries, food and drink. Mercury, arsenic, silver, bismuth and antimony can be detected with this test (See Appendix I).
5. Water extractions are sometimes needed to test for inorganic substances. Silver nitrate and barium chloride are good reagents for general testing of samples for cyanide, arsenic and numerous anions. Silver nitrate, barium chloride and other reagents are described in Appendix I.
6. Acidic/Basic organic extractions can be tested for the presence of drugs, pesticides and other organic substances on the GC/MS. The extraction may include clean up steps to eliminate unwanted compounds, e.g., fats.
7. Some solid samples may be analyzed and compared on a variety of laboratory instruments such as the FTIR, SEM/EDX, XRF, XRD, MSP, or Py-GC-MS (See individual instrument protocols).

For specific tests and analytical schemes consult Appendices I through V or appropriate references listed in Section II.

### **Sampling / Sample Selection**

After close visual examination, any item (solid, liquid or powder) appearing homogenous will be assumed to be homogeneous unless further evidence is developed to believe otherwise. In these cases, a small portion of the item may be analyzed further and yet the results in reports may represent the item/substance as a whole (sampling). For substances that appear non-homogenous, sample selection should be utilized for both testing and for reporting of results.

### **VI. QUALITY ASSURANCE / QUALITY CONTROLS**

Appropriate controls, blanks and reference materials should be used for each test. Appropriate blanks, controls and calibrations / performance checks and adjustments will be employed per individual instrument protocols.

#### **APPENDIX I--Frequently Used Micro Chemical Tests**

It should be noted that slight variations in the formulations of each of these reagents may be acceptable. Regardless, all chemical reagents should be tested on a known sample prior to each use in order to test the reliability of the reagent. When a reagent is made, the bottle should be labeled with the name of the reagent and the date it was made or lot number at a minimum. Records should be kept as to who made the reagent and that it was tested for reliability. The list below is not all inclusive, but

any reagents or tests used in the laboratory must be well documented in literature and generally accepted in the scientific community.

### General Tests

**10% HCl**--acidify test sample with drops of dilute HCl. Gas evolution indicates bicarbonates, carbonates, cyanides, hypochlorites (bleach), nitrates or nitrites. Use caution as cyanide gas is very poisonous.

**5% AgNO<sub>3</sub>**--precipitates many ions. Most precipitates are white.

**5% BaCl<sub>2</sub>**--precipitates many ions. Most precipitates are white.

#### **--Precipitated by AgNO<sub>3</sub> and insoluble in HNO<sub>3</sub>:**

iodide, I <sup>-</sup>	sulfide, S <sup>-2</sup>
bromide, Br <sup>-</sup>	cyanide, CN <sup>-</sup>
chloride, Cl <sup>-</sup>	thiocyanate, SCN <sup>-</sup>
hypochlorite, ClO <sup>-</sup>	

#### **--Precipitated by AgNO<sub>3</sub> and soluble in HNO<sub>3</sub>:**

cyanates, CNO <sup>-</sup>	boric acid, H <sub>3</sub> BO <sub>3</sub>
carbonic acid, H <sub>3</sub> CO <sub>3</sub>	iodic acid
oxalic acid, C <sub>2</sub> H <sub>2</sub> O <sub>4</sub>	

#### **--Precipitated by AgNO<sub>3</sub> and BaCl<sub>2</sub>; soluble in HNO<sub>3</sub>:**

sulfites, SO <sub>3</sub> <sup>-2</sup>	thiosulfates, S <sub>2</sub> O <sub>3</sub> <sup>-2</sup>
arsenite, As <sup>+3</sup> , As <sub>2</sub> O <sub>3</sub>	arsenate, As <sup>+5</sup> , AsO <sub>4</sub> <sup>-3</sup>
*phosphate, PO <sub>4</sub> <sup>-3</sup> yellow w/ AgNO <sub>3</sub>	chromic acid
carbonate, CO <sub>3</sub> <sup>-2</sup>	bicarbonate, HCO <sub>3</sub> <sup>-</sup> cream w/ AgNO <sub>3</sub>

\*silver nitrate does not precipitate phosphoric acid due to acidic medium

#### **--Precipitated by BaCl<sub>2</sub> and insoluble in HNO<sub>3</sub>:**

sulfate, SO<sub>4</sub><sup>-2</sup> (high concentrations of sulfate can cause crystal formation with silver nitrate)  
fluoride, F<sup>-</sup>

**1% Diphenylamine/Concentrated Sulfuric Acid (fresh)** -- blue color develops with the presence of the following oxidizers: chloride, bromide, iodide, chlorates, nitrates, nitrites, hypochlorite, bromate, iodate, permanganate, Fe<sup>+3</sup>, Sb<sup>+5</sup>, and peroxides. An immediate and permanent blue/purple indicates NO<sub>3</sub><sup>-</sup>. A similar color is obtained with relatively concentrated solutions of FeCl<sub>3</sub>. Immediate blue colors are produced by ClO<sub>3</sub><sup>-</sup> and NO<sub>2</sub><sup>-</sup> but color from the latter fades rapidly and in about 1 minute is yellow green. At low levels, color development may occur after standing a short time. Similar reactions may also be

observed with chloride, bromide, iodide, hypochlorite, bromate, iodate, permanganate,  $\text{Fe}^{+3}$ ,  $\text{Sb}^{+5}$ , and peroxides.

**Fujiwara Test**--indicates presence of chloral hydrate, trichloroacetic acid, chloroform, bromoform, iodoform, and other compounds with at least two halogen atoms attached to one carbon. *Procedure:* to 1 ml of sample, add 1 ml 5N NaOH and 1 ml pyridine. Heat for two minutes in boiling water. Red or pink color in pyridine layer is positive.

**Reinsch Test**--indications for mercury, silver, arsenic, antimony and bismuth. *Procedure:* Add 3 mls conc. HCl to 15 mls sample. Immerse a copper wire that has been cleaned with concentrated  $\text{HNO}_3$  in sample and heat gently ( $80\text{-}90^\circ$ ) for 1 hour. Examine copper for discoloration every fifteen minutes. A silvery deposit is given by mercury and silver. A black deposit is given by bismuth and arsenic. A purple deposit is given by antimony.

**5% Brucine Sulfate in  $\text{H}_2\text{SO}_4$** --orange to red color indicates nitrates, nitrites or chlorates.

**Sugar test**--to a drop of sample or solid sample add 1 drop of 15% 1-naphthol in EtOH and then 3-4 drops of conc. sulfuric acid. If sucrose or fructose is present a blue to purple color will appear; if glucose or maltose is present a pink-red color will develop.

**Metals by Ammonium sulfide**--to a drop of liquid sample acidified with 5% HCl, add a drop of aqueous  $(\text{NH}_4)_2\text{S}$ . Perform tests in hood. Many **metal ions** give colored precipitates:

Black precipitate: indicates Hg, Pb, Ag, Bi, Cu, Co, Ni, or Fe. With addition of concentrated HCl: Bi dissolves; Pb turns grey; Fe turns rust colored or dissolves to orange solution.

Yellow precipitate and solution indicates Cd.

Dark brown precipitate indicates Sn.

Reddish-brown precipitate indicates Pt.

Peach precipitate and solution indicates Mn.

Orange precipitate indicates Sb.

Milky white precipitate indicates Zn.  $\text{ZnS}$  is soluble in excess  $(\text{NH}_4)_2\text{S}$ .

### Specific Tests

**Ethchlorvynol**--add crystals of diphenylamine to an alcoholic solution of the sample; slowly trickle in concentrated  $\text{H}_2\text{SO}_4$ . Red color positive.

**Thiocyanate** (nitroprusside)--add drop of 5% ferric chloride. Red color is positive.

**Cyanide**--add two drops of concentrated  $\text{H}_2\text{SO}_4$  to 2-3 drops sample in test tube. Cover top of tube with a cover slip with a hanging drop of  $\text{AgNO}_3$ ; warm at  $80^\circ\text{C}$  for 4-5 min. Search hanging drop for crystals

of AgCN--tiny, highly refractive, short rods or sheaves of slender needles. Rod's RI's  $n_{\perp} = 1.685$  and  $n_{\parallel} >> 1.685$ .

**Arsenates**--red precipitate with AgNO<sub>3</sub>. View crystals with microscope.

**Arsenites**--yellow precipitate with AgNO<sub>3</sub>. Best if ammoniacal AgNO<sub>3</sub> is used. Add concentrated ammonium hydroxide to 5% AgNO<sub>3</sub> until precipitate dissolves upon mixing. Add drop of this reagent to drop of sample. View crystals with microscope.

**Oxalic acid, oxalate salts**--to the acid or acid solution of the salt add drop of 10% ferrous sulfate. Yellow precipitate positive.

**Lithium ion**--add sample drop to glass slide and heat to dryness to remove any possible ammonium salts. Add drop of 15% hexamethylenetetramine (hexamine) to dried residue. Transfer this drop to another glass slide in two separate drops. To one drop add a crystal of K<sub>3</sub>Fe(CN)<sub>6</sub> (potassium ferricyanide); to the other a crystal of K<sub>4</sub>Fe(CN)<sub>6</sub> (potassium ferrocyanide). The ferricyanide yields yellow octahedra that appear birefringent due to high strain within the crystal; ferrocyanide yields short rods and radial clusters of rods. To help form the ferrocyanide crystals, push crust at edge of drop back into the middle and scratch slide with a glass rod. *Negative samples* of the ferricyanide also yield stars and yellow octahedra; however, these crystals are of *very low* birefringence.

**Bleach containing Hypochlorite** -- pH should be basic. Test with hanging drop of 5% silver nitrate by acidification with 5% HNO<sub>3</sub>. Wash and dry precipitate in reagent drop with distilled water and dissolve precipitate with drop of 50% ammonium hydroxide. Add coverslip and using PLM look for formation of highly refractive cubic crystals of silver chloride along edge of coverslip. This indicates the presence of chloride ion from evolution of Cl<sub>2</sub> from the test drop. Crystals are then confirmed as AgCl via X-ray analysis.

**Iodine Solution** - Place a small amount of material on a microscope slide and cover with a cover slip. Add I<sub>2</sub> reagent and allow it to flow under the coverslip. Examine utilizing PLM. Starch grains and gelatinized starch particles stain purple/blue to red/brown. Color produced depends on the amylase content.

**10% Povidone-Iodine (Betadine) Solution** - Examine utilizing PLM. Starch grains and gelatinized starch particles stains purple/blue to red/brown. Color produced depends on the amylase content. Advantage of this test over the Iodine Solution is that that "Maltese" cross can be observed after the starch grains pick up the stain.

**Fehling's Test for Reducing and Non-Reducing Sugars** -- A material to be tested is gently heated to a boil in a drop or two of Fehling's solution. If a reducing sugar (e.g. lactose, maltose, etc) is present, the solution will turn yellow/orange. For a non-reducing sugar, the solution will stay blue. To test for a non-reducing sugar (e.g. sucrose), warm the material to be tested in dilute HCl and then add the Fehling's solution. The solution will turn yellow/orange if a non-reducing sugar was originally present.

**Selleger's Stain and Graff "C" for cellulose fibers** -- Add stain to paper fibers which have been disintegrated and dispersed on a microscope slide. Cellulose fibers will stain different colors depending on pulp make-up and previous chemical treatment.

**Ammonia or Ammonium Ion** -- precipitate using hanging drop of 10% platinum chloride by volatilizing ammonium ion to ammonia by adding 10% sodium hydroxide to test sample. To test for presence of ammonia gas (anhydrous ammonia) place drop of reagent on glass slide and place slide in air tight container with specimen. Allow to sit an appropriate amount of time (overnight if necessary) to allow for the formation of octahedral crystals indicative of the ammonium ion reaction product. Crystals thus formed can be rinsed with distilled water, dried and analyzed via IR spectroscopy.

**Ethylene Glycol**—See “Analytical Methods, Section 3 above or follow protocols for Fire Debris.

**Hydrogen Peroxide** -- Use two tests.

1) Reduction test: Place one drop of 1.0% potassium ferricyanide/0.5% ferric chloride in spot well. Add test drop(s). Prussian blue coloration indicates hydrogen peroxide. Very dilute solutions may give a green coloration.

2) Oxidation test: Soak filter paper with 0.5% lead acetate. Hold over open bottle of 24% ammonium sulfide. Paper will become brown due to formation of PbS (Lead Sulfide). Allow paper to dry. Spot paper with drop of sample. A white coloration indicates hydrogen peroxide. If only one of the tests is positive something other than hydrogen peroxide is indicated.

\*\*Several other spot tests and micro chemical tests can be found in reference articles such as “Characterization and Identification of Water Soluble Explosives” by Hopen and Kilborn and “Extended use of Squaric Acid as a Reagent in Chemical Microscopy” by Whitman and Wills.

## **APPENDIX II -- Wood Examinations**

Because wood examinations may require special preparation, additional procedures follow. A low power microscopical examination (10-30X) of prepared wood samples can be used to classify wood as soft or hard or, if enough sample is present, to genus or, ultimately, species. The later classifications will sometimes require thin sectioning of the wood sample for examination via high power microscope (100-400X). The botanical features observed to classify a wood fiber or piece can be found in the literature listed in the bibliography (Appendix IV).

### **Analytical Equipment and Materials**

- 1) Stereomicroscope
- 2) Microscope with high magnification such as a PLM or comparison microscope
- 3) Razor blades, glass slides, cover slips, mounting media
- 4) 0.25% Safranin in 20% ethyl alcohol

### **Procedure**

1. Determine if the piece of wood is large enough for stereomicroscopic examination and thin sectioning. If not, only a microscopical examination of wood fibers can be performed.
2. If only wood fibers are to be examined a stain such as Safranin may be used and the sample can be mounted in an appropriate mounting medium. Examine using a high powered microscope. Look for microscopical characteristics, if present, that will allow classification of fibers as hard or soft wood; and, if appropriate, mechanically or chemically pulped. Some characteristic features may be present to determine a more specific classification.

3. For larger wood fragments, razor cuts are made on the whetted wood to obtain either a clean cross-sectional surface for stereoscopic examinations, or thin sections for high power microscopic examinations. Thin sections from the cross-sectional, radial and tangential are made, if possible.

4. Cross-sectional surfaces are examined via low power microscopy and keyed according to Hoadley. Comparison to standard wood blocks can be helpful.

5. Thin sections may be treated with a stain such as Safranin and mounted in an appropriate mounting medium between slide and cover slip. The preparation may be heated to remove air bubbles. Examine sections via high power microscope. Samples are classified according to Hoadley and/or Trimpe (MAFS) key. Comparison to the thin section standards can be helpful.

Report results to the appropriate level of classification.

### **APPENDIX III -- Soil Examinations**

Because soil examinations may require special preparation or techniques, additional procedures follow. Soil is comprised of a number of different components in a variety of combinations (e.g. minerals, vegetation).

When possible, known soil samples should be collected and submitted for comparison purposes.

#### **A. Initial Color and Gross Composition**

A visual examination is first conducted to see if the soil samples are similar in color and gross composition. If the soil samples are not dry they can be dried in an oven for several hours or overnight. Visually compare color of dry soils.

##### **1. Initial Color:**

Place similar amounts of the dried soil samples on a watch glass or other suitable glassware and evaluate the samples for color. Also, the soil samples may be moistened and the color of the damp soil samples compared. If differences in color are observed, the examination may be complete. In addition, the soil may be compared and classified using Munsell color charts if desired.

##### **2. Gross Composition:**

Examine each sample under the stereoscope to determine if the gross composition is similar or different between the control and questioned soils. Note presence of man-made materials such as glass, brick, paint or fibers that may also be useful for comparison purposes.

#### **B. Sieving**

If sample size allows for calculation of fraction sizes, the examiner may weigh the amount of soil to be sieved. Large clumps may be broken up by mortar, rubber mallet, gloved hands, glass rod or sonication. Place soil samples in a beaker and add distilled water. Turbidity and the amount of floating organic debris may be noted. Check pH if desired. Wet sieve using a set of mesh sieves and collect the silt fraction in the pan or filter paper. Collect fractions and dry. Compare colors of the soil samples of like sized mesh sizes. The examiner may calculate the weight percent of each fraction to the total original weight and record. Soil samples with a common origin should have similar weight fractions;

however, questioned samples may have lost all or some of the larger sized fractions, i.e., soil from pants may be primarily small particle sized or clay.

### C. Polarized Light Microscopy

Using a representative portion of one of the soil fractions, the light minerals may be separated from the heavy minerals using bromoform ( $d = 2.89$ ). The density fractions may be mounted and examined by PLM using Cargille liquids (light fractions are generally examined using 1.550 HD or 1.545 HD; heavy minerals are examined in 1.660). The examiner may record the number and types of minerals present. If a large amount of organic matter is present, it can be removed by adding 30% hydrogen peroxide. The number and type of minerals should be similar for soils with a common origin.

### D. Instrumental Methods

A variety of instruments such as SEM/EDS, XRF and XRD may be utilized in order to further identify or compare portions of the soil samples.

## **APPENDIX IV – Paper Match Examinations**

### **The Forensic Examination and Analysis of Paper Matches**

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#### **Abstract**

A brief discussion on the history and production of paper matches will be presented plus an overview of 10 key physical characteristics the analyst can determine when comparing a paper match to a book of matches. Also, a summary will be presented on the information that can be obtained by the examination and comparison of match heads and/or match stems by PLM, SEM-EDS, XRF, TLC, and MSP. In addition to these traditional techniques listed above, this presentation will discuss the value of characterizing the paper content of the match stems as well as the use of Adobe Photoshop in the comparison of the match stem color.

#### **Introduction**

Paperbook matches are sometimes encountered at crime scenes and submitted as evidence to a forensic laboratory. Arson and bombing incidents are the most common types of cases where matches are utilized in the commission of a crime. A match collected at a crime scene may be intact or partially burned and sometimes both types may be present. Match evidence becomes extremely important when a matchbook from a suspect is obtained and submitted to be compared with a match or matches recovered from a scene.

This paper will present a brief overview of the history of matches, how paper matches are manufactured, and the physical characteristics that one can quickly determine to provide a wealth of comparative information. Also, this paper will discuss the use of several different analytical procedures that can be employed to provide additional discriminatory information. In addition, the use of Adobe Photoshop to compare match stems from different books with similar colors will be addressed.

#### **HISTORY**

The discovery of elemental phosphorus by German alchemist Hannig Brandt in 1669 and the invention of the first friction match by Englishman John Walker in 1827 made fire generally accessible to man.

Walker's matches were simply wood splints, tipped with sulfur, potassium chlorate, and other ingredients (1). The more convenient paper "flexible" book matches were patented by Joshua Pusey, a Pennsylvania patent attorney, in 1892 who then sold the patent to the Diamond Match Company in 1894 (2). This basic matchbook consisting of a cover folded over the cardboard matches and stapled, with one end of the cover tucked into the other remains basically unchanged today (Fig.1). Often the matchbook includes advertising on the cover, an idea sparked in 1896 by Diamond Match Company salesman Henry Traute.

### **PRODUCTION**

Matchbooks are produced from paperboard which is finished and treated with an anti-afterglow solution. The paperboard rolls are cut into long strips called combs. These combs are then dipped into a wax, dried, and then dipped into the match-head solution and dried again. The head is mainly composed of potassium chlorate (oxidizer), sulfur (fuel) and glue with some inert ingredients. The standard match book will contain two combs of 10 stems, a total of 20 matches. The advertising printing on the covers is applied prior to the friction plate (strickers). With sales targeted to the cigarette smoker, the match market reached its peak in the 1940s and 1950s but the increase in lighters in the 90's and, lately, the enforcement of smoking bans have resulted in an estimated 90% market loss (3). Thus the original "big five" manufacturers have been reduced to three major companies in North America:

- Diamond Match Co. (wooden matches)
- Bradley Industries (owns Atlas Match Co. and produces special production matches for small businesses, hotels, and restaurants)
- D. D. Bean and Sons produces matches for resale/vending market, such as grocery stores, large retail chains, military sales and convenient stores (4). D.D. Bean and Sons currently produces approximately 80% (8 to 10 million match books a day on a four-day work week) of all matchbooks in the United States (3).

### **Physical Characteristics**

Initially, the examination and comparison of matches is made by visual inspection including utilization of a stereobinocular microscope. Early work by H. J. Funk (5) and K. C. Dixon (6) described a number of key physical features one can determine. Some features may only provide class characteristics, whereas others may be unique and provide individual characteristics. These features are as follows:

#### **Match Head**

The match head color, porosity, shape and size should be noted. Even burned heads may reveal this information.

#### **Stem Color**

Match stems are made from cardboard and may have several observable layers when viewed on edge using a stereomicroscope. A holder is described by Funk (5), embedding clips, or a small strip of doubled-sided sticky tape on a microscope slide that can be used to aid in maintaining the match on edge. The front facing surface layer of the match stem frequently has a distinctly different color as compared to the underlying match stem body due to pigmentation and/or dying. Even the front surface of brown/tan stem matches can have a slightly different appearance than the interior of the match body. The use of a simple longwave UV lamp or alternate light source (7) may also be employed during the examination of match stems which may provide additional comparative information.

### Wax Line

The wax on the match stem can normally be seen as a slight darker discoloration on the upper portion of the match stem. The depth of the wax line on the match stems can vary between books and within a book of matches.

### Stem Width

The width of matches usually fall into two groups; ones that have a width of approximately 3.3 mm and ones that have a width of approximately 2.7 mm. The approximately 2.7 mm (specification is 0.0108 inches) width is a patented dimension and matches exhibiting this width are only manufactured by D. D. Bean & Sons (3). However, it must be noted that this does not mean that the matchbook will have "D. D. Bean & Sons" markings on the match cover since D. D. Bean & Sons produces matches with this dimension for other companies and other companies produce matches other than 2.7 mm for D.D. Bean & Sons.

### Stem Length and Thickness

The match stem length, when placed at the cardboard base of the matchbook should correspond to the length of the known unburned matches in the matchbook. If the match is burned, a portion of the head must still be present to conduct an accurate comparison. The match thickness does not vary much and cannot be related to a particular manufacturer.

### Base Stem Cut/Indent

As far as these authors know, this feature has not been previously addressed in previous literature. Some matchbooks may be cut or have an indentation at the base of the match stem to aid in removal of the match from the matchbook. The cut/indent may be consistent on every match or vary within a book.

### Cut Edge Abnormalities

Cut edge abnormalities appear along the vertical edge of the match body as small irregular cuts or tears. These imperfections are due to a cutting blade becoming dull over time and are another potential point of comparison to an adjacent match in a book.

### Cross Cut and Torn Fibers

Cross cut and torn fibers may provide unique comparable features that can associate a match to a particular matchbook. Cross cut (horizontal) and torn (vertical) fibers are noted as darker colored fibers contrasted against the more lightly colored fibers. Cross cut (horizontal) and torn (vertical) fibers are recognized under low magnification utilizing a stereomicroscope. The horizontal fibers are fibers which cross individual match stems and have been cut during the manufacturing process. Vertical fibers are the contrasting fibers which run from the base into the match stem and are torn in two when the match is removed from the book. Torn fibers are less useful when attempting to make a positive association since the tearing action of the match from the cardboard base may distort any comparison. Many times the vertical fibers may not be torn in two but are completely pulled from the base or stem when the match is removed.

One can increase the contrast between the fibers in the match stems by use of stains but it should be noted that the use of stains may permanently alter the color of the match stems. One simple way to increase the contrast between fibers is to place a droplet of an 80:20 deionized water:ethanol on the match stems, allow it to set for a moment, and then wick off any excess liquid.

Dixon (6) proposed that if two fibers on the front and two fibers on the back of two matches or four fibers on the front surfaces of two matches match, that it would be sufficient criteria to assert a positive association providing, of course, the other class characteristics are the same.

#### Inclusions

Foreign matter inclusions are common artifacts in match stems and many times are cut in two when adjacent stems are cut by the blade. The strength of association is dependent upon the uniqueness or number of corresponding inclusions. To help reveal the inclusions Gerhart, et al, (8) proposed a submersion method for the comparison of match stems. A positive association can be asserted if one observes unique features for one corresponding inclusion or there are several inclusions corresponding between two matches.

#### Torn End

Assuming the general class characteristics are the same, an examination to determine if there is a physical association between the questioned match and matchbook should be made. Unfortunately, a physical association is not common due to the small match stem area available for comparison.

#### **Analytical Techniques**

Analytical techniques common to most laboratories can be used to characterize and compare matches. For this study, polarized light microscopy (PLM), paper fiber analysis, scanning electron-energy dispersive X-ray spectroscopy (SEM-EDS), X-ray fluorescence (XRF), thin layer chromatography (TLC), microspectrophotometry (MSP), and Adobe Photoshop were employed. The information that each technique might provide is summarized as follows:

#### PLM

Polarized light microscopy (PLM) is a well established analytical technique used to characterize and identify particulate material (9). Some of the common inert ingredients that may be present in the match head that can be quickly identified by PLM include quartz (irregular grains,  $\omega = 1.544$  and  $\epsilon = 1.553$ ), glass fragments (irregular chips,  $n \sim 1.52$ ), diatoms ( $n \sim 1.44$  with very fine structure), and wollastonite (fibrous,  $\alpha \sim 1.62$ ,  $\beta \sim 1.63$ ,  $\gamma \sim 1.64$ ). Pigments and starch grains may also be noted during a PLM examination. The presence or absence of any constituent may provide quick differentiation. Also, it helps if one removes the water soluble components with warm water using micro extraction techniques. Pigments and inclusions in stems can also be characterized by PLM.

#### Fiber Analysis of Paper Stems

The cardboard from which paper matches are manufactured is sometimes referred to as sulfate board, which relates to the alkaline chemical process for separating the fibers from wood. This pulping method is also known as the kraft process. Many of the paper matches examined appear to be from old corrugated container (OCC), which is mainly composed of used unbleached kraft paperboard. Bleached kraft fiber, hardwood semichemical pulp, and grass fibers, i.e. cereal straws, reeds, and sugar cane bagasse, can also be present in OCC.

Dixon (6) suggested the potential of analyzing the fibers in the match paperboard by differential staining. In the paper industry, this type of fiber analysis on papers is a common practice (10). Herzberg and Selleger's Stains have been used for this type of testing. However, Graff "C" Stain is more commonly utilized. These stains give color reactions which serve to differentiate chemical wood pulps, such as sulfate (kraft), soda, and sulfite (acid process) along with mechanical pulps, such as groundwood and thermomechanical pulp. These colors also vary depending on whether the wood fiber is hardwood

(broad leafed trees) or softwood (conifers). There are also described color reactions for non-woody fibers, such as bast, leaf stem, and grass fibers.

To differentiate between certain pulp types that are similar, other stains can be utilized such as the Green and Yorston Stain. This stain detects only unbleached sulfite fiber by displaying a pink color. In general, a fiber analysis method is a destructive test, which reduces the paperboard to a fibrous slurry in water. The slurry is deposited on a glass microscope slide and dried down with an even distribution of fibers across the slide. The stain is then applied to the dried fibers on the slide and examined under the transmitted light microscope. The percentages can be determined by counting the fiber types in traverses across the slide. The identification of the species present is determined by the morphology of the cell types and the anatomical features on the softwood fibers or the hardwood vessel elements.

The ability to identify the species comes from experience, familiarity with TAPPI Test Method T263, wood anatomy keys, and fiber atlases. The precise species can not always be determined due to common features within a given genus. For example, one can determine that a vessel of Yellow Birch is at least a type of birch, but not that it is particularly that species.

When comparisons were performed using matches within the same matchbook, the variance of pulp type percentage was within the tolerance ranges of 2% to 5% depending on proportion.

When comparing the paperboard of match stems from different books, including those produced by the same company, enough variance was found to state that they were significantly different. This was true for every comparison tested in our study. This probably reflects the nature of the product, since the board has been made from recycled fibers. This study suggests there is considerable variability within match book paperboard from different batches. It should be noted that there is a possibility that the same mix could be found in different batches, but that likelihood is certainly low.

#### SEM-EDS

SEM-EDS can provide bulk elemental information (11) and can also be employed to characterize and identify particulate material and can confirm the constituents identified by PLM. Quartz grains have an irregular shape containing silicon and oxygen; glass fragments are irregular chips containing mainly silicon, oxygen and calcium with minor/trace amounts of sodium, aluminum, magnesium, and iron; diatoms have very fine structure and are composed mainly of silicon and oxygen; and wollastonite is fibrous containing mainly calcium, silicon and oxygen. Also, as with PLM, the pigments and inclusions in stems can be characterized by SEM-EDS.

#### XRF

The use of x-ray fluorescence (XRF) for the elemental analysis of forensic samples has been utilized for over 20 years and found popularity partially due to its easy sample prep and non-destructive testing. Several authors have studied the elemental analysis of match heads and stems using both SEM/EDS and XRF (11) (12).

In order to determine the discriminating ability of XRF for match heads and stems, different groups of matches were evaluated with each group sharing common visual gross characteristics such as red heads with white stems. Samples were analyzed using a 40 KeV excitation energy to allow heavier elements such as strontium (Sr) and zirconium (Zr) to be detected.

Spectra of the match heads were obtained by placing the beam near the center of the head and testing several random areas to detect homogeneity of the sample. Most heads were very homogenous in nature with few minor variations.

Spectra for stem samples were obtained by analyzing the finished side of the stem and below the wax line to ensure the elemental profile reflected only that of the paper stock. Several areas were analyzed to determine homogeneity of the stems. As was the case with the heads, some variability existed within a single stem but most stems were homogeneous.

The head and stem elemental profiles of matches from the same book were consistent with one another while matches from different books varied considerably. Although several samples shared either similar head or similar stem profiles, the combined head and stem profile discriminated all matches in this group. It is also important to note that five of the books have printing on the book indicating they were manufactured at the same Universal Match plant location.

Burned and unburned heads from the same match book for several match samples were analyzed to determine if a close elemental profile exists as noted from previous work. The overall elemental profile of the heads and stems did not significantly differ between burned versus unburned matches from the same book. However, unlike earlier reports (11) (12) where sulfur levels only were reported as varying in burned heads from the same book, spectra in this work showed variations in the sulfur (S), chlorine (Cl), and potassium (K) levels.

No absolute elemental profile was noted that would distinguish between heads of differing colors. It is interesting to note that significant Titanium (Ti) levels were present in all white head matches tested other than the D.D. Bean samples. Contact with the D.D. Bean Company supports this finding with the confirmation that D.D. Bean does not use titanium oxide as a pigment in any of its match formulations. A significant Ti level was considered to be a Ti Ka peak intensity larger than the Fe Ka peak. Two of the three green head matches and one of the three blue head matches had Ti Ka intensities larger than the Fe Ka.

XRF analysis has shown to be highly effective at discriminating matches, especially when both the head and stem profiles can be obtained.

### TLC

Thin-layer chromatography (TLC) has been used for decades as a separation technique and the possibility of discriminating colored match heads based on the TLC of their dye content has not been investigated. Since dyed match heads are common, the use of TLC to discriminate between visually similar red-colored match heads was investigated. A match head contains approximately 20 mg. of material, 0.05 to 0.3% of which is dye. Therefore, approximately one-half of an intact match head should be sufficient to perform a TLC analysis. In order to determine the applicability of TLC to discriminate colored match heads, 16 red match heads with similar shading from different match books with red heads were selected for analysis.

Water is the most effective solvent for the extraction of dyes from heads but using water also extracts potassium chlorate which interferes with the TLC analysis. A double extraction procedure was employed as follows: Acetone extraction (x2 in warm water bath) followed by a single dye extraction using methanol (warm water bath). The TLC development systems used were n-butanol/ethanol/water (4:1:1) and n-butanol/pyridine/water (3:1:3). The TLC of match heads did not provide a high degree of individualization but did reveal a similar dye pattern for matches of similar manufacture/brand origin.

### MSP

Transmission microspectrophotometry (MSP), an indispensable method for the comparison of color, was utilized to compare colored match stems. To evaluate this method several visually similar black stem matches from hundreds of match books were selected. Although most spectra from the different

stems were distinguishable, a few spectra from some of the black pigmented stems produced little or no spectral curves or slope for comparative purposes.

Reflectance MSP was also attempted for the comparison of red match heads. Reflectance MSP examination of visually similar red head matches disclosed that differences could be detected between three manufacturers that were tested.

Adobe® Photoshop®

Although not normally considered an analytical technique, utilizing Adobe® Photoshop shows promise for the comparison of paper matches. The matches are scanned together and the comparison of the image of the matches can be conducted using two techniques within Photoshop; Hue/Saturation and LAB color mode. The adjustment of the hue within the Hue/Saturation window was able to distinguish between black match stems that visually looked the same but were from different matchbooks. No differences were noted when comparing matches from the same matchbook. The image viewed within LAB color mode channels that can be observed within the channels palette: L= lightness (luminance) shows how bright or dark the image is; A= the A chromatic component/channel identifies colors in the image between green/red; and B= the B chromatic component/channel identifies colors in the image between blue/yellow. Normal image adjustments using "levels" can be performed in each channel to improve image quality. These grayscale stem images within a channel are compared to determine if the stems are consistent or inconsistent with each other. The B channel appears to reveal the most information in this preliminary study of black match stems. Using both techniques worked extremely well in differentiating black pigmented stems which were visually similar and indistinguishable when examined with transmission MSP.

### **Conclusions**

The observation of the physical characteristics as described in this paper will produce a wealth of information in paper match comparisons. The torn end of a match may be unique enough to make a positive association to a matchbook. Also, the observance of corresponding features between two matches such as cross-cut fibers or inclusions may provide a basis for a positive association. Information obtained by non-physical feature analytical techniques in a match comparison may affirm an association or eliminate the samples under comparison. Although this paper does not address the analysis/comparison of wood matches, many of the examinations and techniques described in this paper can be used.

Note

The opinions or assertions contained herein are the private views of the authors and are not to be construed as official or as reflecting the views of the Department of the Army of the Department of Defense, the Georgia Bureau of Investigation, Bureau of Alcohol, Tobacco, Firearms, and Explosives.

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## APPENDIX V – Examination of Tobacco, Cigarettes, Cigarette Butts and Tax Stamps

Cigarettes and tax stamps may be submitted to determine product authenticity. In addition, tobacco materials such as cigarettes, cigarette butts, packs or cartons may be submitted for determination of brand and style identification or for comparison. Examinations typically include physical comparison with exemplars obtained from manufacturers or purchased from reputable retailers and testing of security features. Further examination by chemical and instrumental techniques may be utilized particularly when comparing products that are not commonly seen in the laboratory or for products with few unique identifying features. If known exemplars and/or product information is not available, such as with foreign produced cigarettes and tax stamps, evidence may have to be sent to the manufacturer for determination of authenticity.

### Cigarettes, cigarette butts and other tobacco products

Analysis of cigarette evidence typically includes physical comparison of the following components: carton board, pack board, pack wrap, foil wraps, tipping paper, plug wrap, filters, cigarette paper and the shredded and blended tobacco. Major domestic manufacturers such as Philip Morris, Lorillard and RJ Reynolds produce very consistent products and will often provide proprietary information about packaging features that are not easily counterfeited. Additionally, these manufacturers have routinely provided ATF with exemplars for purposes of comparison.

Counterfeiters will copy anything that is readily observable. Poor quality counterfeits are often easy to spot due to off odors and off colors. However, better quality counterfeits are not readily distinguishable

by visual inspection. To determine cigarette authenticity there are multiple items that should be examined and compared with an exemplar(s). These items vary by brand and are often proprietary. The analyst should obtain information about particular products from examination of exemplars and/or contact with the manufacturer. Some important features to consider are the type of printing on packs and cartons, tear strip characteristics, pack and carton codes, presence of optical brightening agents (observed under UV), ventilation holes, fire safe paper characteristics (banding), filter design, and manufacturing marks. Proprietary features, such as taggants and specialty inks, may require testing by equipment provided by the manufacturers. Pack and carton codes are often counterfeited repeatedly. Lists of known counterfeit codes are provided by manufacturers such as Lorillard and Philip Morris.

Examination of tobaccos should include comparison of the blends and examination for foreign materials. Counterfeits often smell moldy and may include bits of plastic, foil, soil, bug parts and other materials. Exemplars examined in the ATF laboratory have not had these items, however the presence of soil or bug parts (tobacco beetle) in genuine products is possible as tobacco is an agricultural commodity. Tobaccos in typical American blended cigarettes (ABC) contain the following: Burley (20-25%), Flue-cured (25-35%), Oriental (5-15%), Reconstituted Tobacco, RECON (5-25%), Expanded cut tobacco, ECT, (5-15%), Cut-rolled expanded stems, CRES, (0-15%) and Flavorings, 0.5-1.0%. Counterfeit products will often have high levels of CRES and very little RECON. The cut size of the tobaccos and characteristics of the RECON may help identify a product.

Additional analytical tests may be utilized to determine authenticity or for comparative purposes. These include, but are not limited to, lengths and weights of cigarettes, tipping papers and filters, phloroglucinol testing for the presence of ground wood, FTIR testing of adhesives, color analysis of cigarette packs and cartons using microspectrophotometry, and X-ray analysis for determining bulk elemental information of packaging materials or tobaccos.

### Tax Stamps

States, cities, counties, municipalities and reservations may have cigarette tax stamps. There are several manufacturers of tax stamps, however Meyercord Revenue (a SICPA company) manufactures the majority of the stamps in the United States. These stamps are heat applied and have a variety of security features that may be present depending on the stamp. Security features may include water marks, color shifting inks, short and long wave fluorescent inks, taggants that are authenticated with hand-held scanners, holograms and microprint. California and Massachusetts have sticker type stamps produced by SICPA that are encrypted with information that is only available to the state authorities. Meyers, Sekuworks and De La Rue are other tax stamp manufacturers. Tax stamp design and security features can change frequently.

The examiner should discuss the security features and obtain exemplars and the tools to analyze the security features from the manufacturer prior to analysis. If a more than one stamp is submitted, such as those in a carton of cigarettes, examine all the stamps for the presence of security features under normal light, UV light and with a stereomicroscope. If the stamps all exhibit the same characteristics (serial number, watermark, color, size, print process, etc) a sample may be analyzed as long as the report clearly indicates that further testing was only conducted on the sample (see below). Non-destructive tests such as taggant tests and destructive tests, such as acid-base reactions may be conducted. The stamp may further be examined using SEM, SEM-EDS, microspectrophotometry or

other techniques as determined by the examiner. The examiner will determine if further verification by the manufacturer is required.

### Sample Selection and Report Wording Guidelines

Due to the manufacturing and packaging processes for cigarettes, cartons within a master case, packs within a carton, and cigarettes within a pack, will likely have come off the same manufacturing line within a very short period of time. Similarly, due to the manufacturing and application processes of tax stamps, tax stamps within a carton will likely have been manufactured on the same line within a very short time frame. However, it is responsibility of the analyst to determine what variability may exist and determine which analyses are required. If an exhibit contains multiple cartons, packs, cigarettes, or tax stamps a visual/microscopic examination should be performed to assess any differences between the items. Important features to consider include, carton and pack codes, serial numbers, features under UV light and packaging characteristics. Based on the results of the initial analyses and the products being analyzed, the examiner may then choose certain items for further analyses. A random number generator may be used for sample selection.

When sample selection is utilized it must be clearly stated in the report. The following is an example of appropriate wording:

*Exhibit 1 consists of a carton board and (#) packs which were examined for the presence of characteristics found in authentic products produced by Altria (the manufacturer of Marlboro products in the United States). The carton board and packs were found to be visually dissimilar with genuine products. The carton board was also found to be inconsistent with authentic Altria products based on chemical testing. All of the packs in exhibit 1 were visually consistent with one another; therefore two of the packs were randomly selected for further analysis. Within the selected packs, three cigarettes were randomly selected for further analysis. The analyzed packs and cigarettes had physical and chemical characteristics that are inconsistent with authentic products produced by Altria. The results of the aforementioned testing indicate that Exhibit 1 is counterfeit.*

*All of the packs in Exhibit 1 contained Virginia tax stamps with the serial number XXXX. All of the stamps in Exhibit 1 were visually consistent with one another; therefore two of the stamps were randomly selected for further analysis. The analyzed stamps had physical and chemical characteristics that are inconsistent with authentic products produced by SICPA. The results of the aforementioned testing indicate that the stamps in Exhibit 1 are counterfeit.*

When exhibits are sent to manufacturers for examination it must be stated in the report along with the manufacturer's findings. A copy of the manufacturer's report, if provided, will be attached to the laboratory report.

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