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Law Enforcement and Corrections Standards and Testing Program

SMOKELESS POWDER RESIDUE ANALYSIS BY CAPILLARY ELECTROPHORESIS

NIJ Report 600-91

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Smokeless Powder Residue Analysis by Capillary Electrophoresis

NIJ Report 600-91

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FOREWORD

The Office of Law Enforcement Standards (OLES) of the National Institute of Standards and Technology (formerly the National Bureau of Standards) furnishes technical support to the National Institute of Justice program to strengthen law enforcement and criminal justice in the United States. OLES's function is to conduct research that will assist law enforcement and criminal justice agencies in the selection and procurement of quality equipment.

OLES is: (1) Subjecting existing equipment to laboratory testing and evaluation, and (2) conducting research leading to the development of several series of documents, including national standards, user guides, and technical reports.

This document covers research conducted by OLES under the sponsorship of the National Institute of Justice. Additional reports as well as other documents are being issued under the OLES program in the areas of protective clothing and equipment, communications systems, emergency equipment, investigative aids, security systems, vehicles, weapons, and analytical techniques and standard reference materials used by the forensic community.

Technical comments and suggestions concerning this report are invited from all interested parties. They may be addressed to the Office of Law Enforcement Standards, National Institute of Standards and Technology, Gaithersburg, MD 20899.

David G. Boyd, Director
Office of Science and Technology
National Institute of Justice

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LIST OF ACRONYMS USED IN THIS REPORT

CE	capillary electrophoresis
DBP	dibutylphthalate
DEP	diethylphthalate
DNT	dinitrotoluene
DPA	diphenylamine
EC	ethyl centralite
MC	methyl centralite
MECE	micellar electrokinetic capillary electrophoresis
NC	nitrocellulose
nDPA	nitrodiphenylamine
NG	nitroglycerin
NGU	nitroguanindine
N-nDPA	N-nitrosodiphenylamine
SDS	sodium dodecylsulfate
SEM	scanning electron microscopy
SEM/EDX	scanning electron microscopy/energy dispersive X-ray analysis
OLES	Office of Law Enforcement Standards
NIST	National Institute of Standards and Technology

COMMONLY USED SYMBOLS AND ABBREVIATIONS

A	ampere	H	henry	nm	nanometer
ac	alternating current	h	hour	No.	number
AM	amplitude modulation	hf	high frequency	o.d.	outside diameter
cd	candela	Hz	hertz (c/s)	Ω	ohm
cm	centimeter	i.d.	inside diameter	p.	page
CP	chemically pure	in	inch	Pa	pascal
c/s	cycle per second	ir	infrared	pe	probable error
d	day	J	joule	pp.	pages
dB	decibel	L	lambert	ppm	part per million
dc	direct current	L	liter	qt	quart
°C	degree Celsius	lb	pound	rad	radian
°F	degree Fahrenheit	lbf	pound-force	rf	radio frequency
diam	diameter	lbf·in	pound-force inch	rh	relative humidity
emf	electromotive force	lm	lumen	s	second
eq	equation	ln	logarithm (natural)	SD	standard deviation
F	farad	log	logarithm (common)	sec.	section
fc	footcandle	*M*	molar	SWR	standing wave radio
fig.	figure	m	meter	uhf	ultrahigh frequency
FM	frequency modulation	min	minute	uv	ultraviolet
ft	foot	mm	millimeter	V	volt
ft/s	foot per second	mph	mile per hour	vhf	very high frequency
g	acceleration	m/s	meter per second	W	watt
g	gram	N	newton	λ	wavelength
gr	grain	N·m	newton meter	wt	weight

area=unit^2 (e.g., ft^2, in^2, etc.); volume=unit^3 (e.g., ft^3, m^3, etc.)

PREFIXES

d	deci (10^{-1})	da	deka (10)
c	centi (10^{-2})	h	hecto (10^2)
m	milli (10^{-3})	k	kilo (10^3)
μ	micro (10^{-6})	M	mega (10^6)
n	nano (10^{-9})	G	giga (10^9)
p	pico (10^{-12})	T	tera (10^{12})

COMMON CONVERSIONS
(See ASTM E380)

ft/s×0.3048000=m/s
ft×0.3048=m
ft·lbf×1.355818=J
gr×0.0647989 1=g
in×2.54=cm
kWh×3 600 000=J
lb×0.4535924=kg
lbf×4.448222=N
lbf/ft×14.59390=N/m
lbf·in×0.1129848=N·m
lbf/in^2×6894.757=Pa
mph×1.609344=km/h
qt×0.9463529=L

Temperature: $(T_{°F}-32)\times 5/9=T_{°C}$

Temperature: $(T_{°C}\times 9/5)+32=T_{°F}$

SMOKELESS POWDER RESIDUE ANALYSIS BY CAPILLARY ELECTROPHORESIS

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A procedure is presented that can be used for the analysis of residues generated by the combustion of ammunition and explosives prepared with smokeless gunpowder. The bases of the test are the qualitative and quantitative identification of characteristic organic components present in the post-combustion residues. The residues are collected by adhesive film lift and/or alcohol swabbing of hands, clothing, spent shell casings, and explosive debris, and analyzed using micellar electrokinetic capillary electrophoresis (MECE). The MECE technique provides identification of organic additives in the smokeless powder. The procedure described here may provide positive identification of the use of materials containing smokeless powder.

Key words: explosive residue; gunshot residue; micellar electrokinetic capillary electrophoresis; organic constituents; plasticizers; primers; propellants; stabilizers.

1. PURPOSE

The purpose is to provide evidence in crimes involving the use of firearms and improvised explosive devices (such as pipe bombs), based on the analysis of residues from fired smokeless powder collected from the hands of shooters, clothing, spent shell casings, or explosive remains.

2. SCOPE

The test is based on the compositional analysis of gunshot and explosive residues left on surfaces that (1) provide limited interferences from complex matrices such as soil, blood, sweat, etc., and (2) are easily sampled using adhesive film lifts or by swabbing with solvent. Quantitative compositional data on more than 100 commercially available smokeless powders provides the framework for a data base for matching purposes. The possible occurrence of characteristic smokeless powder components that could result in false positive tests has been extensively evaluated. Samples were collected from 100 people in the general population to identify interfering components for the MECE analysis. Results obtained by the MECE method are compared to the results obtained by scanning electron microscopy/ energy Dispersive X-ray analysis (SEM/EDX). Finally, this procedure is used to evaluate forensic casework samples for the presence and composition of gunpowder residues. MECE analysis of residues left on the hands of a suspect may provide a link to a shooting. The composition of these residues may also provide a link to a weapon.

3. DEFINITIONS

3.1 Adhesive Film Lift

An adhesive film lift is a method used for the collection of gunshot residues from surfaces, such as skin, and clothing. This method uses an adhesive tape to collect the residue. Residues are recovered from the tape by physical removal of visible particles with tweezers, or solvent extraction for microscopic residues.

3.2 Capacity Factor

An indexing term that allows normalization for random variations in migration times for a given component in multiple MECE analyses. The capacity factor, k, is defined by the following equation:

$$k = \frac{t_r - t_0}{t_0(1 - t_r/t_m)}$$

where t_r is the migration time of a solute, t_0 is the migration time for ethanol, which migrates with the electroosmotic flow, and t_m is the migration time for dibutylphthalate (DBP—a compound that moves with the micellar agent).

3.3 Capillary Electrophoresis

Capillary electrophoresis (CE) is an analytical technique in which chemical compounds are separated based on their relative movement through a conductive liquid (buffer) in a small diameter capillary tube under the influence of a high voltage electric field. Compounds separate as a result of differences in size and positive or negative charge.

3.4 Characteristic Residue Components

These are smokeless powder additives that are good markers for the MECE identification of gunshot residues. These compounds are typically present in amounts greater than 0.1 percent in the unfired powder, can be detected by optical absorbance, and occur in many types of ammunition powders. These may include, but are not limited to, nitroglycerin, dinitrotoluene isomers, diphenylamine, and ethyl centralite. Although alkyl phthalate esters are present in most smokeless powders, they are also present in many plastic and adhesive materials. Therefore the phthlate esters are not suitable for consideration as characteristic components of gunshot residue.

3.5 Electroosmotic Flow

Electroosmotic flow is the bulk flow of buffer towards the detector end of the capillary, caused by electrostatic interactions of the buffer with the charged walls of the capillary under the influence of the high voltage field.

3.6 Explosive Residue

Explosive residue is defined as the traces of material that remain on fragments of an explosive device and/or are deposited on other objects as a result of the detonation of such a device. The sources of these materials are the propellants, stabilizers, and plasticizers used in the explosive device as well as the compounds formed from the decomposition of these materials as a result of detonation.

3.7 False Positive Results

A false positive result is the erroneous identification of characteristic residue components in a sample that by definition should not contain any of these components.

3.8 Gunshot Residue

Gunshot residue is defined as the traces of material that remain after the use of a firearm using smokeless gunpowder ammunition. The major sources of these materials are the primer, propellant and associated stabilizers, and plasticizers that are used in ammunition, as well as any decomposition products of these materials formed during detonation.

3.9 Micellar Electrokinetic Capillary Electrophoresis

Micellar electrokinetic capillary electrophoresis (MECE) is a CE method in which electrically neutral compounds, not possessing a positive or negative charge, may be separated based on differences in their interaction with a charged micellar agent (such as sodium dodecylsulfate—SDS) added to the buffer. See figure 1.

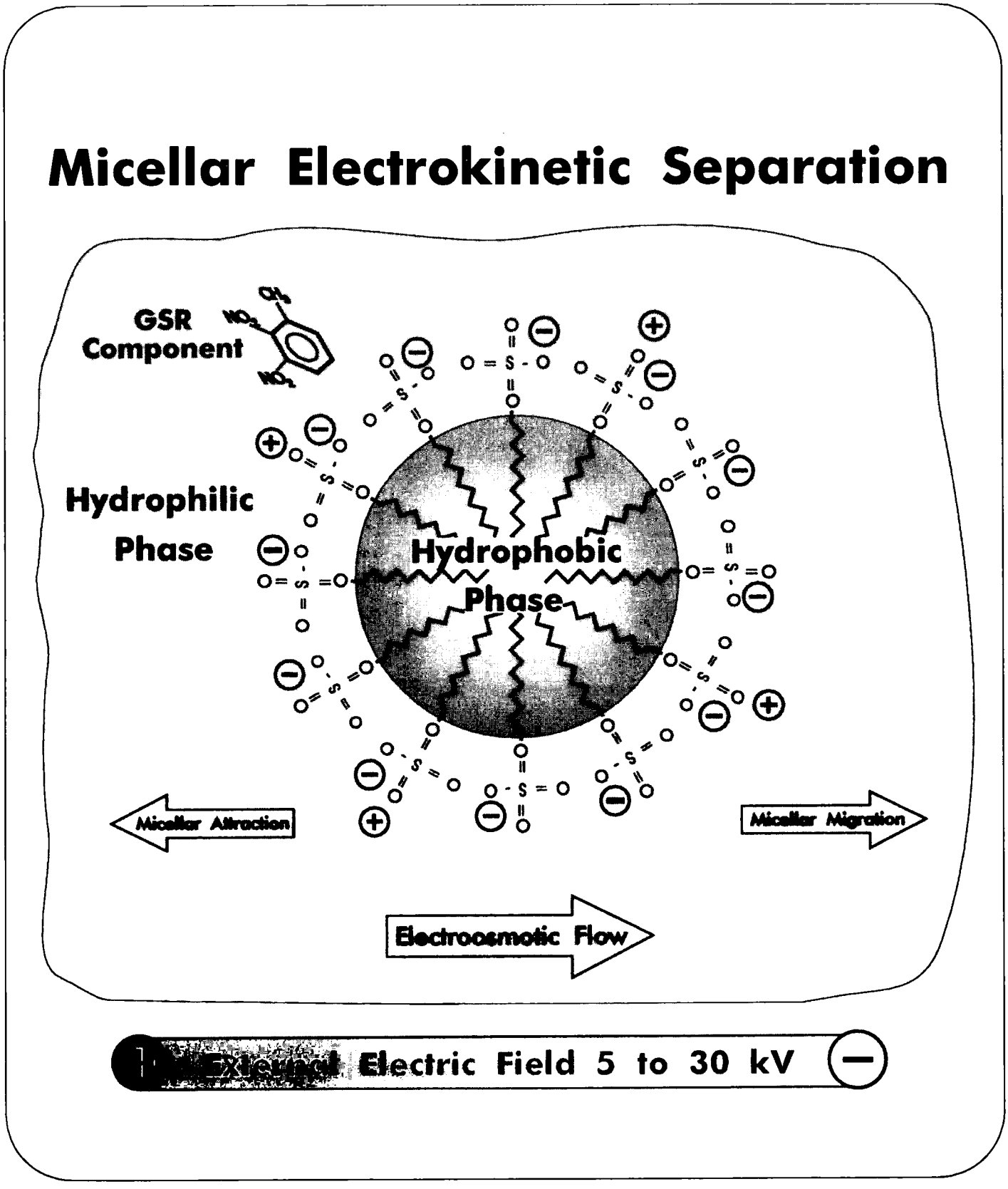

Figure 1. Schematic of MECE Separation.

3.10 Migration Time

Migration time (t_m) is the unique time required for the electrophoretic movement of a single organic component to travel from the beginning of the capillary to the detector. Using current MECE instruments, small variations in the migration time for a given component occur from test to test as a result of system instability. Computing capacity factors and normalizing to an internal standard substantially eliminates these variations. See figure 2.

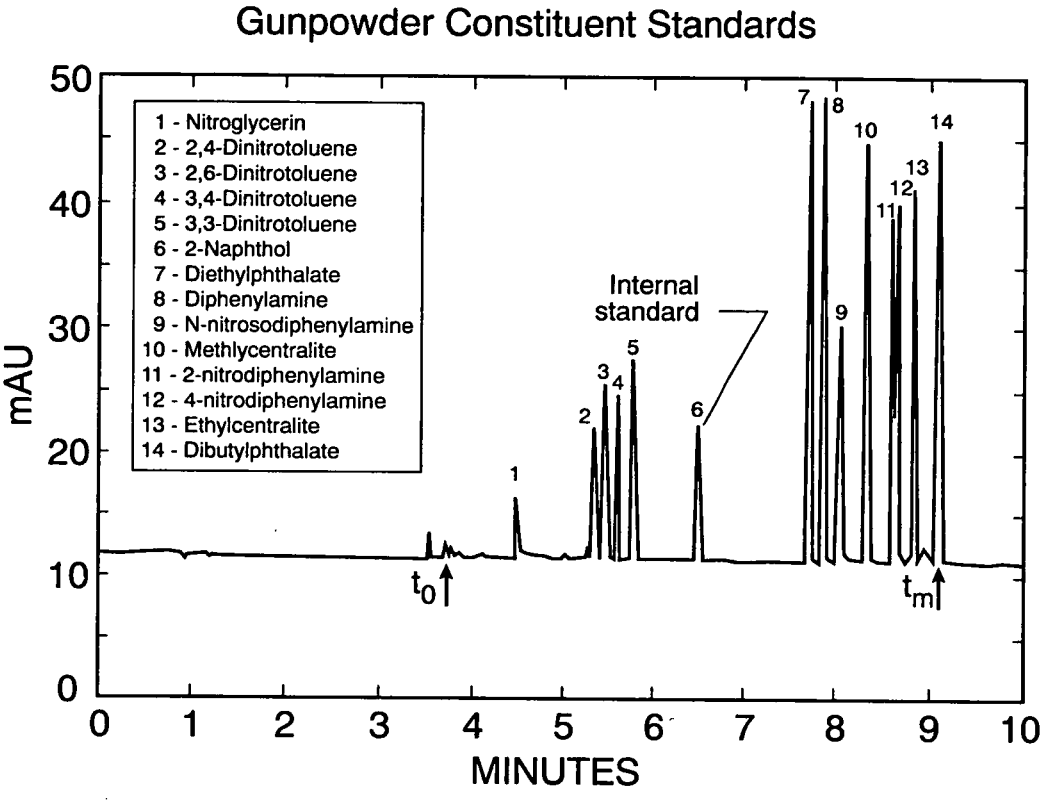

Figure 2. MECE Analysis of Gunpowder Constituent Standards.

3.11 Plasticizers in Smokeless Powders

Plasticizers are organic materials added during manufacture of propellant and explosive mixtures to aid in their fabrication. These materials may include, but are not limited to, short chain aliphatic phthalic acid esters, of which dibutylphthalate is the most common.

3.12 Primers

Primers are devices used to initiate the propellant in ammunition, and may consist of a single component or a mixture of various inorganic and organic materials. Primer ingredients may include, but are not limited to, lead azide, lead styphnate, tetracene, diazodinitrophenol, barium nitrate, strontium nitrate, and antimony sulfide.

3.13 Propellant Components of Smokeless Powder

Smokeless powder propellants are organic materials that undergo rapid combustion when initiated with a primer. In smokeless gunpowder, the bulk material is nitrocellulose (NC). Propellant materials found in double-and triple-base smokeless gunpowders may also include, but are not limited to, nitroglycerin (NG), nitroguanidine (NGU) (rare in small arms propellants), and the isomers of dinitrotoluene (DNT).

3.14 Residue Extract

The residue extract is prepared by taking either an adhesive film lift or collection swab and extracting with an organic solvent to remove the residue.

3.15 Scanning Electron Microscopy/Energy Dispersive X-Ray (SEM/EDX) Analysis

SEM is an analytical technique that uses an electron beam to examine microscopic particles. In the SEM/EDX technique, an x-ray detector provides additional information of the chemical composition of selected particles. This technique has been used to identify gunshot residues based on the selective detection of primer compounds containing lead, and/or barium, and/or antimony.

3.16 Stabilizers

Stabilizers are organic materials that are added to propellants and explosives to retard their decomposition during storage. These materials may include, but are not limited to, diphenylamine (DPA) and ethyl centralite (EC). Both decomposition and combustion of the propellants results in the formation of nitrated stabilizer derivatives such as N-nitrosodiphenylamine (N-nDPA), 2-nitrodiphenylamine (2-nDPA), and 4-nitrodiphenylamine (4-nDPA).

4. TEST EQUIPMENT AND ANALYTICAL PROCEDURES

4.1 Residue Collection and Preparation

The MECE test relies on preparing a liquid extract of the fine particles that remain following the use of ammunition and explosives. Both adhesive film lift and swabbing methods can be used for residue collection. Gloves and tweezers rinsed with alcohol are used to handle all materials.

4.1.1 Adhesive Lift Method

The adhesive material used for sample collection should be a masking-type tape with an adhesive that does not dissolve in alcohol. The tape should be precleaned using methanol in an ultrasonic bath to eliminate any alcohol-soluble material on the tape. Samples are collected by using a 2.5 cm × 2.5 cm section of masking tape held by tweezers and pressed onto the surface to be examined. This method can be adapted for analysis by both SEM/EDX and MECE by using a double-sided masking type adhesive tape placed on an aluminum SEM/EDX sample stub. Samples are then collected by holding the aluminum stub while pressing the adhesive onto the surface to be examined.

Samples from the hands of a suspected shooter should be collected using a separate adhesive lift for both back and palm of each hand. Samples from clothing should be collected using a separate adhesive film lift pressed onto the clothing near any bullet hole or area suspected of being exposed to gunshot residue. All samples are placed in capped vials and refrigerated until analyzed.

A blank sample also should be collected as a part of the crime scene protocol. A blank sample tests both the sample collection and preparation systems for interferences or contamination. To collect a blank sample, an adhesive film lift should be collected from the skin of a suspected shooter in an area that could not be exposed to gunshot residue, e.g., a foot, leg, back, etc. From clothing samples, the blank should be collected from an area of the clothing that could not have been exposed to any gunshot residue. The blank sample should then be tested in the same manner as evidence samples. The following protocols are used for MECE analysis:

Sample Preparation for Residue Samples

Place a 2 mm × 2 mm section of adhesive lift in 50 μL of methanol,
Agitate in an ultrasonic bath for 15 min,
Add 5 μL of ethylene glycol to prevent complete evaporation,
Evaporate methanol under a stream of dry nitrogen,
Reconstitute residue in 50 μL of MECE buffer.

Sample Preparation for Unfired Powder

Place 0.050 g of powder in 5.00 mL of methanol,
Agitate in an ultrasonic bath for 15 min,
Add 5 μL of the methanol extract to 50 μL of MECE buffer.

4.1.2 Swab Residue Collection Method

Cotton used for swabbing is precleaned in an ultrasonic bath with high purity ethyl alcohol. Alcohol-cleaned cotton, moistened with ethanol and held with tweezers, is used for swab collection of residues from the surface to be sampled.

Samples are collected from shell casings or debris from improvised explosive devices by swabbing each surface of interest with a separate swab. The swabs are placed in capped glass vials and refrigerated prior to analysis.

As with the adhesive lift method, a blank sample also should be collected for the swab method. A swab should be made of a surface that could not have been contaminated with gunshot residue.

Samples for MECE analysis are prepared as follows:

Place swab in 500 μL of ethanol,
Agitate in an ultrasonic bath for 15 min,
Add 5 μL of ethylene glycol to prevent complete evaporation,
Centrifuge sample through a 1 μm fluoropolymer filter,
Evaporate ethanol under a stream of dry nitrogen,
Reconstitute residue in 50 μL of MECE buffer.

4.2 Residue Generation

Controlled firing range studies were used to generate test residue samples for method validation. Eight handguns, varying from 22 to 45 caliber, with both revolver and semiautomatic mechanisms, were used to generate residues. The hands of the individual firing the weapons are thoroughly washed with soap and water and dried with paper towels before firing. A blank sample is then collected to verify that the hands are free of any residues prior to firing the weapon. This procedure is repeated before each test. Gunshot residues for this test are generated by firing three rounds of commercial ammunition from the test weapon. Examination of gunpowder composition before and after firing is achieved by analysis of unfired powder and the residues generated by firing a single round of commercial ammunition with the same powder through a 15 cm × 15 cm section of sterile 100 percent nylon cloth placed over a white paper target from a distance of about 20 cm (muzzle-to-target distance). These tests are repeated with each weapon and ammunition studied.

4.3 Residue Persistence and Component Aging

In casework, it is important to establish how long residues from a particular weapon will persist on the hands or clothing of a shooter after deposition. This persistence may be studied by firing ammunition from the weapon and collecting residues at hourly intervals. In this study, adhesive film lift samples were collected at hourly intervals from 0 h to 6 h from hands used to fire three rounds from a semiautomatic handgun. Individuals were involved in normal activities during the time period of this study.

In order to simulate the effect of environmental exposure on gunpowder components, bulk gunpowder was placed in glass test tubes, positioned outside in direct sunlight, and sampled every hour for 28 h.

4.4 Capillary Electrophoresis Equipment

Commercially available apparatus for capillary electrophoresis, providing controlled voltage to 30 kV and using on-column ultraviolet absorbance detection, is required. A schematic of a typical capillary electrophoresis system is shown in figure 3. The capability to select the exact detection wavelength is required. Wavelength programming or simultaneous multiwavelength detection is advantageous, permitting measurement of component spectra to enhance the certainty of component identification. Table 1 lists information on the wavelength maxima for the gunpowder component standards studied. An example of the diode array display for four gunpowder components is shown in figure 4.

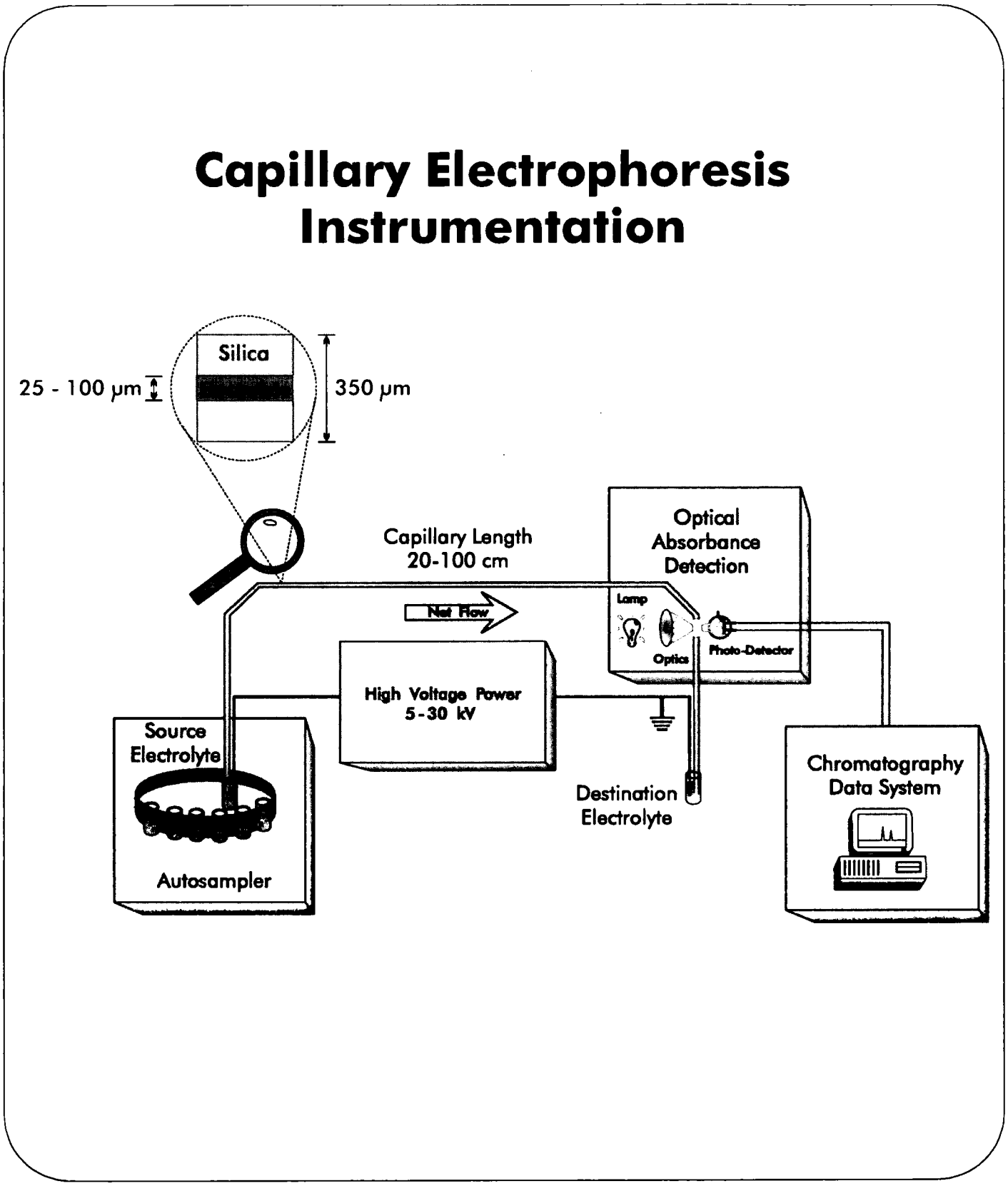

Figure 3. Schematic of Capillary Electrophoresis System.

TABLE 1. *Information values of wavelength maxima of residue components*

Sample	*Wavelength maxima in nm*[1]	*Sample*	*Wavelength maxima in nm*[1]
DBP	200, 235	DEP	200, 235
DPA	200, 285, 235	2-nDPA	< 190, 275, > 450
4-nDPA	< 190, 410, 260	2,3-DNT	210, 265
2,4-DNT	260, 200	2,6-DNT	<200, 240
3,4-DNT	<190, 220, 270	EC	< 190, 250
MC	< 190, 250	N-nDPA	< 190, 230, 300
NG	< 190	NGU	270, 220

[1] Maxima estimated to the nearest 5 nm on an uncalibrated spectrometer; first value is largest maximum, with succeeding values in descending order of intensity.

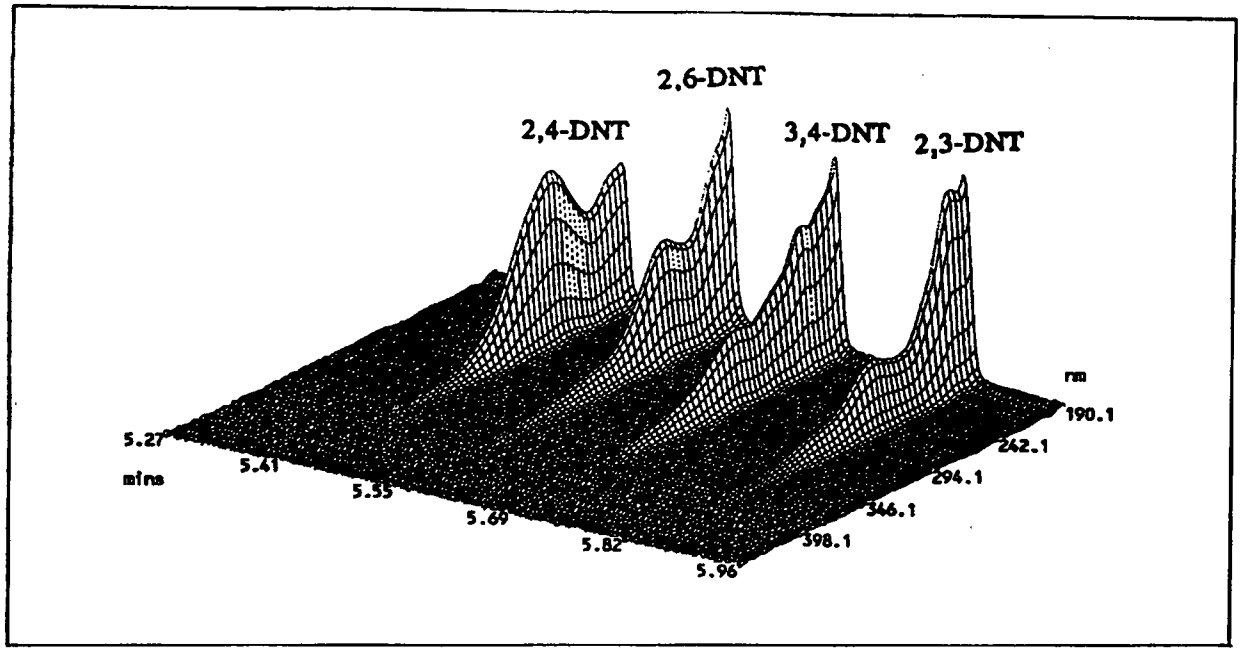

FIGURE 4. *Diode array UV spectra of dinitrotoluene isomers.*

4.5 Chromatography Data Station

A chromatography data system, capable of precise measurement of peak retention time, peak height and peak area is required. The capability to edit graphically the peak measurements and to overlay multiple sample runs is desirable.

4.6 MECE Operating Conditions

The operating conditions used for the MECE analysis are as follows:

SDS buffer: 10 mmol/L sodium tetraborate decahydrate (adjusted to pH 8.50 with boric acid), 25 mmol/L sodium dodecylsulfate (SDS) ,

Sample preparation buffer: SDS buffer with 1×10^{-4} mol/L 2-naphthol as an internal standard,

Sample injection: samples are injected using pressure injection at 300 Pa (30 mbar) for 1.5 s,

Separation column: 75 μm diameter capillary of 82 cm length with an extended path length optical cell,

Separation voltage: analyses are conducted at 30 kV,

Detection mode: diode array ultraviolet absorbance monitored at 200 nm.

Typical results for a mixture of standards at 10^{-4} mol/L are shown in figure 2.

5. DISCUSSION

In this work, the MECE method for use in forensic gunshot residue and explosive cases has been validated by collecting the following information: 1) a reliable and reproducible residue collection and sample preparation protocol has been developed; 2) the minimum detection limits of the characteristic smokeless powder components using the MECE apparatus have been determined; 3) the occurrence of false positive results in the general population was studied and was determined to be unlikely; 4) a database of the composition of more than 100 commercially available smokeless powders has been generated; 5) the persistence of characteristic residue components on sample surfaces and the loss of residues from the shooter's hands over time has been investigated; 6) changes in characteristic residue component composition resulting from environmental exposure have been determined; 7) multiple firings of two weapons were performed to determine if residues would be deposited each time a given weapon was fired; 8) MECE analysis of firing range samples was compared to those obtained using SEM; and, 9) casework samples were examined using both the MECE method and SEM/EDX.

5.1 Residue Collection and Sample Preparation

Adhesive film lifts, as evidenced by this study, provide a reliable method for sample collection of gunshot residues from the hands of individuals who have fired a weapon and from clothing. The adhesive used for the film lift must be evaluated for method interferences by extraction using the residue protocol and analysis by MECE before being used for collection. Adhesives that do not dissolve in methanol must be used. Various commercially available solvent resistant masking tapes (both single-and double-sided tapes) are most likely to meet these requirements. The double-sided tape may be used on a SEM/EDX sample holding aluminum stub so that gunshot residue samples can be collected for both MECE and SEM/EDX. This is the collection method of choice.

Alcohol swab collection can be used to recover residues from spent cartridges and bomb fragments. However, it was found that recovery of characteristic residue components from cotton swabs is less than 50 percent using solvent extraction. Swabbing is not suitable for residue collection from the hands since the alcohol recovers large quantities of fats and oils from the skin that can interfere with quantitative residue recovery and analysis.

Ultrasonic agitation of residues in methanol for 15 min was found to be sufficient to completely extract the characteristic residue components from gunpowder. This was determined by performing a second extract on all commercial gunpowders examined. Negligible quantities of the components were detected in the second extract. An evaluation of solvents found that methanol was more efficient than ethanol as a residue extraction agent. The addition of ethylene glycol, when used at a volume fraction of 5 percent, was found to prevent losses of the residue constituents during the evaporative concentration of the extract, and did not alter the quantitative analysis by MECE.

5.2 Minimum Detection Limits

The minimum detection limits for gunshot residue constituents using four commercially available CE instruments were found to be in the picogram mass range using the MECE method. Detection limits were approximately 1 pg to 2 pg for the components with aromatic functionalities and 4 pg for nitroglycerin. Future improvements in detection technology may improve the sensitivity of the test.

5.3 Interferences and False Positives

Using the outlined protocols and MECE analysis, no false positive tests for the characteristic residue components were found on the adhesive film lifts from the hands of 100 volunteers representing a wide variety of occupational backgrounds. The individuals sampled included law enforcement officers, mechanics, teachers, construction workers, farmers, chemists, secretaries, and many other professions. The sample group included men, women, right-handed, left-handed, employed, and unemployed individuals, as well as people with visibly clean hands and dirty hands. No peaks were identified with capacity factors that match any of the characteristic residue components.

All blank samples collected during this study were also found to be negative for the characteristic residue components. Hand washing with soap and water was determined to effectively remove all MECE-detectable residue components.

These results indicate that the identification of the characteristic residue components by MECE can only occur if an individual has been exposed to a recently fired weapon. False positive results because of occupational duties or environmental exposure were not found to occur.

5.4 Gunpowder Identification

In order to provide evidence to identify a gunpowder or its residues, including the type of propellant, manufacturer, and lot, it is necessary to identify and quantitate the characteristic components of both the residues and the unfired powder. In determining a match between a residue sample and unfired powder, it is also necessary to take into account any chemical compositional changes that may result from the firing of the gunpowder.

Information values on the approximate composition of 106 commercially available cartridges and reloading powders as determined by MECE are provided in Table 2. These results are provided as a first step in identifying the source of an unknown residue or powder sample so that subsequent tests can provide final identification.

5.4.1 Factors to Be Considered in Matching of Smokeless Powders

There are several factors that need to be considered in the identification of a smokeless powder or residue sample based on matching known and unknown materials. In a matching study of the residue composition of 17 smokeless powders before and after firing, clear matches were not always obtained. The matching criteria consisted of a qualitative test and a quantitative test. Both tests had to be satisfied for a match to be obtained. The qualitative test identified the characteristic residue components in the residue and compared them to the components in the unfired gunpowder. The quantitative test compared the measured value of each component in the residue to the measured value of those components in the unfired powder. For this exercise, a match was considered to be achieved if the relative amount of a component in the residue was within one standard deviation of the measured value of that component in the unfired powder. In this test, residues obtained from 5 of the 17 powders met this match criteria, while 12 of the powders did not. In addition, multiple sub-samples from the same residue gave significantly different quantitative results. Therefore, there may be a number of factors that need to be considered in interpreting the results.

Some of the factors that should be considered in comparing pre- and post-firing composition are: 1) nonuniformity in the manufactured composition of the unfired powder; 2) nonuniformity in the individual residue particles collected; 3) nonuniformity resulting from changes in composition from burning or partial burning of the gunpowder; 4) nonuniformity from changes in composition that may result from environmental exposure; and, 5) nonuniformity resulting from the use of different ammunition in the same weapon.

The relative contribution of each of these 5 factors to the interpretation of matches between known and unknown materials has yet to be determined.

5.5 Persistence of Residues After Firing

The persistence of post-firing residues on shooters' hands was studied to evaluate the effect of normal activity on the MECE test results. The residues from a weapon discharged at a firing range were studied to examine residue persistence. Samples were collected immediately after the weapon was fired (time zero), and test samples were taken to correspond to hourly collections up to 6 h. One hour after firing, no residues were found on any of the subjects. Subsequent samples at 2 h to 6 h also were found to be negative for gunshot residues. Initial residues (at time zero) were at low concentrations, thus it is possible that weapons that deposit higher concentrations of residues may result in longer residue persistence. As discussed previously, all residues can be deliberately removed from the hands by washing with soap and water.

Table 2. Information Values of Gunpowder Composition

AMMUNITION																			
9mm	LOT #	NG	dev*	2,4-DNT	dev	DPA	dev	N-nDPA	dev	MC	dev	2-nDPA	dev	4-nDPA	dev	EC	dev	DBP	dev
3-D Inv. Inc.	80317941	12.5%	0.3%	0.063%	0.007%	0.44%	0.01%	0.40%	0.01%			0.041%	0.002%	0.024%	0.002%	0.98%	0.03%	0.18%	0.01%
CCI Blazer	3509	41.8%	0.7%													0.72%	0.02%		
Eldorado Starfire	ELD95FA-008	24.4%	1.4%			0.34%	0.04%	0.43%	0.05%			0.040%	0.003%	0.019%	0.003%	0.041%	0.006%	0.19%	0.02%
Federal	431572H067	45.2%	0.6%													0.86%	0.06%	0.15%	0.02%
Federal Am. Eagle	2310812592	17.3%	0.2%			0.05%	0.01%	0.08%	0.01%										
Federal JHP	43A-5106	47.6%	1.5%													0.78%	0.04%		
Federal Parabellum	9AP																	0.77%	0.04%
Makarov (9X18)	NA					0.72%	0.06%	0.11%	0.02%										
Norinco	NA	42.2%	2.0%							1.6%	0.03%							0.020%	0.01%
Norma No. 19026	11308Y					0.42%	0.01%	0.26%	0.01%			0.034%	0.004%	0.096%	0.010%	0.41%	0.01%		
Remington	Y07YC8501	42.9%	2.7%													0.80%	0.08%	0.25%	0.02%
Remington Kleanbore	T110	40.9%	0.9%													0.95%	0.08%		
S & W	41041	44.1%	1.8%			0.05%	0.002%	0.08%	0.01%							0.61%	0.03%	0.022%	0.004%
S & W Nyclad	AE0441	48.3%	1.2%													0.82%	0.01%	0.059%	0.01%
Sellier & Bellot	891					0.84%	0.05%	0.12%	0.01%			0.038%	0.003%					0.039%	0.01%
Sellier & Bellot	891					0.86%	0.01%	0.10%	0.0001%										
Speer "Lawman"	4100T4	36.1%	2.5%													0.60%	0.05%		
Speer Blount	D05Z23	13.7%	2.4%	0.064%	0.010%	0.52%	0.13%	0.23%	0.06%			0.033%	0.008%	0.019%	0.006%	0.021%	0.005%	0.20%	0.05%
SuperVel	ME173	44.8%	2.1%	0.28%	0.003%			0.80%	0.02%									0.25%	0.03%
Winchester	28MC473	50.9%	2.1%													0.87%	0.01%		
Winchester Luger Cartridge #10	56HN61/85	19.2%	0.6%			0.71%	0.02%	0.34%	0.01%										
Winchester Luger Cartridge #30	56HN61/85	19.0%	0.3%			0.71%	0.004%	0.33%	0.01%										
Winchester Luger Cartridge #10	36HE10/64	19.5%	0.1%			0.76%	0.01%	0.35%	0.01%										
Winchester Luger Cartridge #30	36HE10/64	19.0%	1.5%			0.74%	0.05%	0.32%	0.03%										
Winchester Luger Cartridge #10	56HN61/85	20.3%	0.9%			0.75%	0.03%	0.36%	0.02%										
Winchester Luger Cartridge #30	56HN61/85	20.2%	0.5%			0.75%	0.02%	0.36%	0.01%										
Winchester Luger Cartridge #10	46HN61/98	18.7%	0.1%			0.71%	0.01%	0.34%	0.01%										
Winchester Luger Cartridge #30	46HN61/98	18.5%	0.3%			0.72%	0.01%	0.33%	0.01%										
Winchester Luger Cartridge #10	36HE10/64	20.6%	0.9%			0.81%	0.04%	0.38%	0.01%										
Winchester Luger Cartridge #30	36HE10/64	19.3%	0.5%			0.76%	0.01%	0.34%	0.01%										
Winchester Subsonic	73FN11	14.6%	1.6%	0.061%	0.009%	0.42%	0.05%	0.24%	0.03%			0.038%	0.004%	0.021%	0.003%	0.025%	0.004%	0.18%	0.02%
Winchester Super-X	66HA91	15.8%	0.4%	0.079%	0.004%	0.64%	0.02%	0.30%	0.01%			0.043%	0.003%	0.025%	0.001%	0.024%	0.002%	0.25%	0.01%
Winchester Supreme	54HF30	17.6%	0.6%			0.66%	0.02%	0.31%	0.02%									0.25%	0.001%

* Represents the uncertainty of deviation of 9 replicates and does not reflect the uncertainty in the measured value.

Table 2. Information Values of Gunpowder Composition Continued

38 caliber	LOT #	NG	dev	2,4-DNT	dev	DPA	dev	N-nDPA	dev	MC	dev	2-nDPA	dev	4-nDPA	dev	EC	dev	DBP	dev
3-D Inv. Inc.	150929931	24.4%	1.2%	0.050%	0.009%	0.45%	0.03%	0.54%	0.03%			0.037%	0.003%	0.021%	0.003%	0.075%	0.01%	0.20%	0.01%
CCI - Blazer	L04M2	41.8%	2.2%													0.53%	0.05%		
CCI - Speer	210014	25.6%	2.3%			0.35%	0.04%	0.071%	0.01%			0.055%	0.005%	0.023%	0.003%				
CCI-Blazer	G20N4	50.2%	1.0%													0.58%	0.002%		
Eldorado PMC	38G-582	27.9%	0.4%			0.61%	0.01%	0.18%	0.004%			0.029%	0.002%	0.015%	0.002%			0.064%	0.05%
Eldorado Starfire	38SFA-014	24.2%	0.7%	0.075%	0.009%	0.37%	0.01%	0.44%	0.03%			0.043%	0.005%	0.021%	0.002%	0.045%	0.004%	0.19%	0.01%
Federal Lot	12A-4670	55.9%	2.9%													0.91%	0.06%		
Federal Lot	12B-4634	55.0%	0.2%													1.0%	0.02%		
Fiocchi	5330223610	59.0%	4.8%													1.5%	0.4%		
G&S	3	38.0%	0.5%													0.49%	0.01%	0.12%	0.004%
Hornady's Frontier	5937915	31.9%	0.8%	0.43%	0.007%	0.072%	0.01%	1.1%	0.01%									0.38%	0.01%
Lapua	6011FJSU950					0.44%	0.01%	0.45%	0.01%					0.17%	0.011%				
Norma	NA					0.27%	0.02%	0.34%	0.03%	0.51%	0.06%								
Remington	LE03H	33.3%	1.5%			0.21%	0.01%	0.13%	0.01%			0.058%	0.005%	0.024%	0.003%	0.40%	0.04%	0.068%	0.01%
Remington Kleanbore	RA5169	18.6%	0.3%			0.082%	0.001%	0.72%	0.02%			0.026%	0.001%	0.043%	0.004%				
Remington	LA08R	39.6%	7.2%													2.9%	0.5%		
Remington	LA08R	31.8%	6.7%													2.2%	0.5%		
S&W	7230321	44.7%	0.7%													0.69%	0.02%		
Scorpion Hydra-Shok	X13456	29.7%	0.3%	0.41%	0.01%	0.17%	0.01%	0.97%	0.02%			0.094%	0.004%	0.031%	0.006%				
SuperVel	GC09228	16.3%	0.5%			0.18%	0.01%	0.18%	0.01%										
USAC	2030593	13.3%	0.2%			0.67%	0.02%	0.53%	0.02%			0.097%	0.004%	0.071%	0.001%			2.1%	0.07%
WAHIB	MAR 5, 1982	22.8%	0.2%			0.35%	0.01%	0.11%	0.01%									0.35%	0.04%
Winchester	60VM62/2	26.3%	1.1%	0.20%	0.02%	0.44%	0.02%	0.24%	0.01%			0.077%	0.002%	0.031%	0.002%	0.23%	0.02%	0.21%	0.09%
Winchester	36UM92/95	48.4%	1.3%													1.0%	0.04%		
Winchester	60VM62/2	30.9%	3.0%	0.19%	0.006%	0.40%	0.02%	0.61%	0.05%							0.34%	0.03%	0.20%	0.004%
380 auto	**LOT #**	**NG**	**dev**	**2,4-DNT**	**dev**	**DPA**	**dev**	**N-nDPA**	**dev**	**MC**	**dev**	**2-nDPA**	**dev**	**4-nDPA**	**dev**	**EC**	**dev**	**DBP**	**dev**
3-D Inv. Inc.	100314941	24.4%	0.60%			0.46%	0.006%	0.52%	0.01%			0.037%	0.002%	0.019%	0.002%	0.026%	0.002%	0.16%	0.01%
CCI - Blazer	NA	48.9%	1.8%					0.089%	0.002%							0.71%	0.03%		
Federal	090534H116	36.4%	0.60%			0.17%	0.006%	0.034%	0.004%							0.59%	0.02%		
Federal Am. Eagle	949663600	52.7%	1.4%													0.93%	0.04%		
Fiocchi	333008N137	63.9%	1.3%													1.5%	0.04%		
Remington	J17T5114	47.7%	0.50%													0.59%	0.01%		
Remington Kleanbore	R18U	50.2%	1.5%													0.72%	0.02%		
S&W	7210682	44.6%	1.0%			0.071%	0.003%	0.070%	0.004%							0.61%	0.02%		
S&W	8050981	45.7%	0.20%			0.031%	0.001%	0.13%	0.004%							0.64%	0.002%		
Winchester	042HB42/99	43.3%	0.99%			0.47%	0.005%	0.37%	0.02%									0.15%	0.005%
Winchester	05SL62/49	51.3%	0.60%													0.63%	0.02%		
Winchester	46KD1103	30.7%	0.40%	0.38%		0.15%	0.006%	0.98%	0.007%			0.12%	0.002%	0.081%	0.002%				
Winchester	73GK70/56	49.2%	2.7%			0.54%	0.029%	0.55%	0.02%			0.045%	0.003%	0.021%	0.002%	0.026%	0.002%		

Table 2. Information Values of Gunpowder Composition Continued

25 auto	LOT #	NG	dev	2,4-DNT	dev	DPA	dev	N-nDPA	dev	MC	dev	2-nDPA	dev	4-nDPA	dev	EC	dev	DBP	dev
CCI - Blazer	K15R6	48.3%	1.2%													0.72%	0.029%		
Daisy & Heddon	RF-U/L-101					0.26%	0.002%	0.79%	0.006%			0.069%	0.005%	0.18%	0.002%				
Dynamit GECO	NA					0.30%	0.006%	0.13%	0.003%										
Dynamit Sinoxid	43 MA					0.27%	0.02%	0.12%	0.004%										
Eldorado PMC	25A-089	28.9%	0.80%			0.51%	0.02%	0.32%	0.02%			0.027%	0.002%	0.013%	0.002%			0.073%	0.004%
Federal	17A-3250	48.6%	2.0%													0.66%	0.026%		
Fiocchi	704001-075	26.3%	1.4%	0.15%	0.004%											1.82%	0.16%		
Remington	LB20D	41.4%	0.30%					0.19%	0.004%							0.54%	0.018%		
Winchester	65SH62/97	48.7%	1.4%													0.62%	0.012%		
Winchester ACP	03GA41/37	37.4%	0.60%			0.30%	0.006%	0.56%	0.01%									0.13%	0.01%
Winchester	11GF03/1190	39.5%	0.70%			0.14%	0.008%	0.072%	0.003%							0.64%	0.023%	0.18%	0.02%
RELOADING POWDERS	**LOT #**	**NG**	**dev**	**2,4-DNT**	**dev**	**2,6-DNT**	**dev**	**DPA**	**dev**	**N-nDPA**	**dev**	**2-nDPA**	**dev**	**4-nDPA**	**dev**	**EC**	**dev**	**DBP**	**dev**
Dupont IMR SR4756	P73JY22B			2.3%	0.1%	0.028%	0.002%	0.44%	0.01%	0.17%	0.01%	0.021%	0.002%	0.068%	0.001%				
Dupont IMR SR7625	P71MY03C			2.3%	0.1%	0.035%	0.001%	0.47%	0.02%	0.17%	0.02%	0.021%	0.001%	0.083%	0.009%				
Dupont IMR 4198	OE11/3087			4.3%	0.2%	0.055%	0.001%	0.34%	0.02%	0.16%	0.01%	0.057%	0.004%	0.069%	0.005%				
Dupont IMR 4350	P75MA10B			4.6%	0.1%	0.076%	0.002%	0.11%	0.003%	0.22%	0.003%	0.070%	0.070%	0.071%	0.002%				
Dupont IMR 3031	P73A1323			6.1%	0.3%	0.069%	0.003%	0.10%	0.004%	0.32%	0.03%	0.072%	0.006%	0.10%	0.01%				
Dupont IMR 4064	P75JA25C			6.3%	0.4%	0.10%	0.02%	0.10%	0.01%	0.30%	0.02%	0.061%	0.002%	0.088%	0.007%				
Hercules Rx7	11	8.28%	0.6%					0.33%	0.01%	0.17%	0.004%	0.030%	0.002%	0.021%	0.001%	3.0%	0.1%		
Hercules Unique	UN294	28.0%	0.9%					0.44%	0.004%	0.056%	0.002%								
Hodgdon H414	NA	10.3%	0.4%	0.24%	0.01%			0.54%	0.02%	0.30%	0.004%	0.092%	0.004%	0.041%	0.002%			4.2%	0.1%
Hodgdon H100	50685	13.7%	0.3%	0.26%	0.01%			0.65%	0.01%	0.44%	0.01%					3.0%	0.03%		
Hodgdon H322	50484							0.16%	0.01%	0.11%	0.00%	0.081%	0.007%	0.16%	0.02%				
Norma 1010	NA	34.8%	1.6%													1.5%	0.2%		
Norma 2120	NA	38.9%	1.8%													1.2%	0.1%		
Winchester 680	680081H5	12.2%	0.2%	0.61%	0.01%			0.63%	0.01%	0.34%	0.01%							3.2%	0.1%
Winchester 748	748018NE91	12.9%	0.5%	0.38%	0.004%			0.40%	0.01%	0.59%	0.01%	0.18%	0.004%	0.13%	0.001%			4.6%	0.1%
Winchester 760	760010HG2	12.9%	0.5%	0.57%	0.01%			0.20%	0.01%	0.51%	0.02%	0.14%	0.014%	0.061%	0.005%			4.1%	0.1%
Winchester 748	748018NE91	13.3%	0.5%	0.39%	0.01%			0.50%	0.02%	0.56%	0.02%	0.18%	0.004%	0.12%	0.006%			4.5%	0.1%
Winchester 296	296003KH72	13.8%	0.4%	0.56%	0.01%			0.45%	0.01%	0.34%	0.01%	0.13%	0.005%	0.64%	0.005%			2.4%	0.1%
Winchester 452AA	452KB81	17.7%	0.6%	0.52%	0.02%			0.18%	0.004%	0.79%	0.02%	0.070%	0.005%	0.050%	0.002%	2.2%	0.03%		
Winchester 473AA	473009KB42	18.5%	0.5%	0.51%	0.01%			0.27%	0.01%	0.73%	0.01%	0.087%	0.002%	0.066%	0.003%	1.8%	0.03%		
Winchester 231	27092NB11	28.9%	1.1%	0.25%	0.01%			0.41%	0.02%	0.83%	0.03%	0.082%	0.004%	0.036%	0.003%				
Winchester 540	540080822	30.1%	0.4%	0.14%	0.002%			0.51%	0.003%	0.74%	0.01%	0.12%	0.002%	0.054%	0.003%				
Winchester 571	500094 E61	30.2%	2.2%	0.49%	0.03%			0.051%	0.001%	0.64%	0.02%					0.35%	0.02%		
Winchester 630	63000XE21	53.3%	0.3%	0.32%	0.01%					0.85%	0.01%								

Based on this experiment, residues found on the hands of a suspect are a clear indication that a weapon has been fired or handled within a short period of time prior to the collection of the sample. These results also indicate that sampling for gunshot residues from the hands must occur as soon as possible. A suspect that is not captured until several hours after a shooting is not likely to be found to have residues present on his/her hands at that time, as determined by the MECE test.

Clothing was found to retain residues for a long period after firing. Residues may persist for days, weeks or months if the clothing is not washed or involved in significant frictional contact with other objects.

5.6 Environmental Exposure and Residue Decomposition

Changes in the chemical composition of a given gunpowder might be expected to occur over time, particularly when exposed to heat and/or light. However, when an unfired powder was exposed to direct summer conditions over a 28 h period, no significant change was found in the measured value of the following characteristic gunpowder components: NG, 2,4-DNT, DPA, N-nDPA, 2-nDPA, and 4-nDPA. Although unfired powder composition appeared to be unchanged in this simple experiment, the decomposition of the characteristic components when deposited as residues might be expected to be more rapid. We evaluated whether this compositional change could be used to provide an estimate of the time of firing.

Samples of post-firing residues on cloth targets obtained by point blank firing of a handgun were stored in the laboratory and examined over a 2-month period of time. Residue composition immediately after firing, in some cases, was different from the unburned powder and varied from sample to sample as determined by replicate analyses, as noted previously. Very slow loss of nitroglycerin was seen over the 2 months of the study. However, the measured variation in composition is similar in magnitude to the particle-to-particle variability both of unfired gunpowder particles and collected residue particles. Thus, a determination of when a weapon was discharged based on the change in residue composition does not appear to be possible. The decomposition rate is too slow relative to the measurement uncertainty.

5.7 Frequency of Residue Deposition

In cases where a positive MECE test for gunshot residues is obtained, it is a strong implication that the individual has been exposed recently to a discharged weapon. We evaluated the frequency of positive MECE test results by firing two weapons. Multiple firings were made using two different 9 mm semiautomatic weapons. Each weapon was fired multiple times with each hand. The first weapon, a Mac 9, has an ejection port on the right side of the weapon. The second weapon, a Walther P38, has an ejection port on the top of the weapon. Samples collected from the Mac 9 were positive for gunshot residues 53 percent of the time when the weapon was fired with the right hand and 20 percent of the time when fired by the left hand. Samples collected from the Walther P38 were positive for gunshot residues 93 percent of the time when the weapon was fired with the right hand and 71 percent of the time when fired by the left hand.

These results indicate that, even in controlled conditions, detectable gunshot residues may not be deposited on the hands of the shooter every time the weapon is fired. Deposition may be dependent on the caliber of weapon, type of weapon (e.g., semiautomatic versus revolver, etc.), configuration of ejection port, mechanical condition of the weapon, how clean the weapon is, the ammunition composition, completeness of the ammunition combustion, wind conditions, perhaps weapon temperature, random trajectories of residue particles, and which hand the shooter used to fire the weapon. Thus, a negative residue test does not prove that a weapon was not fired.

5.8 MECE Analysis Versus SEM/EDX Analysis

If MECE analysis is to augment the current technology for gunshot residue detection, it is necessary to compare the results obtained by MECE to those obtained using SEM/EDX on the same sample. Firing range samples examined using both MECE and SEM/EDX were found to be positive for gunshot residues by both methods. Some samples found to be positive by SEM/EDX were found to be negative by MECE. However, all samples that were positive by MECE were positive by SEM/EDX. Some false positive SEM/EDX results were obtained on blank samples. Since multiple firings were conducted by the same volunteer, it was assumed that hand washing would remove the inorganic residues as efficiently as the organic residues. This was not always the case with all blank samples. The false negative MECE results obtained on these identical samples could be caused by a difference in the quantities of inorganic versus organic residues. However, the most important result is that no false positive results were obtained by MECE analysis, and SEM/EDX analysis confirmed the positive results.

5.9 Casework Results

MECE and SEM/EDX has been conducted simultaneously on samples collected from the hands of individuals suspected of having fired a weapon in seven criminal investigations. No gunshot residues were conclusively identified by MECE in any of these cases. However, SEM/EDX analysis has conclusively identified gunshot residues in two of these cases. MECE analysis in one of those cases suggested the presence of nitroglycerin, but at quantities too small to confirm. The negative MECE results for the five other cases were confirmed by SEM/EDX. This would indicate that the organic gunshot residues were either not deposited, or were not present at high enough concentrations to be detected.

MECE analysis has been performed on a total of 16 samples from two cases involving the examination of clothing for the presence of gunshot residues. Gunshot residues were conclusively identified in seven of the samples from these cases using MECE analysis. SEM/EDX analysis of the samples confirmed the presence of gunshot residues on five of these samples and three additional samples that were negative by the MECE method. The results on five of the samples were the same using both MECE and SEM/EDX analysis. However, three samples were positive by SEM/EDX and negative by MECE, and two samples were positive by MECE and negative by SEM/EDX.

6. CONCLUSION

The results of this research suggest that MECE analysis is a valid analytical method for gunshot residue analysis. MECE analysis has been achieved on adhesive film lifts from hands and clothing, and these same lifts are compatible for SEM/EDX of the inorganic residues. Risk of false positive tests from occupational exposure to characteristic residue components does not appear to be a concern. Positive MECE results are not always obtained when a weapon is fired. This could be due to a number of factors, including low efficiency of residue deposition, low residue persistence, and lack of sensitivity of the MECE test. Recoverable residues do not appear to persist on skin for more than an hour. Thus, residues must be collected immediately from skin. However, residues on clothing are stable for a long period of time. Quantitative analysis for the purpose of generating a "chemical fingerprint" to match residues to known gunpowders must be interpreted with great care. Compositional variations of unfired and fired gunpowder particles has been noted. However, the presence or absence of components may provide valuable information for the inclusion or exclusion of an ammunition type or manufacturer. Residue decomposition over time resulting from environmental exposure is slow compared to the time that residues persist on samples. Thus, time of firing information cannot be obtained from quantitative analysis of the residues.

Sample preparation and analysis can be achieved in about 2 h per case. Qualitative and quantitative information is generated concerning the characteristic organic constituents in gunshot residues. The cost of instrumentation is four to five times less than the cost of an SEM/EDX system. MECE analysis does provide a rapid and complimentary analytical tool for gunshot residue analysis.

7. REFERENCES

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