

Gunshot Residue Analysis by Micellar Electrokinetic Capillary Electrophoresis: Assessment for Application to Casework. Part I*

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ABSTRACT: Usually gunshot residue (GSR) analysis examines samples for the presence of inorganic primer compounds, in particular lead (Pb), barium (Ba), and antimony (Sb). Alternative methods are needed because of the advent of primers that do not contain these metals. Micellar electrokinetic capillary electrophoresis (MECE) has been used to examine characteristic organic gunpowder components (COGC), including nitroglycerin (NG), diphenylamine (DPA), ethylcentralite (EC), and others. The purpose of this project was to develop MECE for implementation in GSR casework. In order to do this, it was necessary to: (a) establish a sample collection and preparation method for use with both MECE and scanning electron microscopy (SEM); (b) determine that the minimum detection limits for COGC were in the picogram range; (c) show that no residues were identified in samples from the general population at or above these levels; and (d) quantitatively identify the common chemicals used in more than 100 commercially available ammunitions and reloading powders. Results from the study covering organic GSR persistence, environmental exposure effects, comparison to SEM results, and casework using both CE and SEM are described in a subsequent paper.

KEYWORDS: forensic science, criminalistics, gunshot residue, capillary electrophoresis, gunpowder

Most commonly, gunshot residue (GSR) analysis involves examinations for the inorganic compounds used in the manufacture of ammunition primers. Concerns about environmental contamination from lead and other toxic primer elements have resulted in the development and introduction of lead-free ammunition (1,2). Scanning electron microscopy (SEM) and atomic absorption spectroscopy (AAS) are the most commonly used methods for conducting GSR analysis of primer residues (3). These methods will become less useful for GSR analysis if the complete elimination of the inorganic compounds eventually is achieved. New analytical methods need to be developed to provide an alternative approach. One approach is to examine GSR for the presence of organic residues from the gunpowder used in smokeless ammunition. Table 1 lists some of the characteristic organic gunpowder components (COGC) that may be found in

manufactured ammunition. It should be noted that phthalate esters (such as DEP and DBP) are not unique to gunpowder and thus have limited utility by themselves as COGC. Micellar electrokinetic capillary electrophoresis (MECE) has been used for the analysis of organic gunpowder residues (4,5) and thus provides a possible alternative technique.

The purpose of the work described in this paper and the following paper (6) was to evaluate implementation of MECE in GSR casework. Validation of new methods in forensic laboratories must be completed before they can be used independently in casework. For GSR analysis, issues to be addressed include: (1) sample collection and preparation procedures, (2) detection limits, (3) interferences and occurrences of GSR chemical residues in the general population, (4) composition of common ammunitions, (5) frequency and persistence of residue deposition, (6) comparison to existing methods, and (7) potential significance in casework. The results of work on the first four issues will be described in this paper. Results of work conducted on the last three issues are the subject of the second paper (6).

Sample collection and preparation for GSR analysis has been extensively researched for use with the SEM and AAS techniques (7–11). An adhesive film lift collection and preparation methods for organic gunshot residues (O-GSR) by MECE has been previously described (5). The goal of this project was to adapt these methods so that a single method could be used to collect GSR samples for both MECE and SEM analysis.

Inorganic compounds common to GSR have been identified at significant levels by AAS analysis of samples collected from the general population, thus indicating that occupational or environmental exposure must be considered in GSR analysis (12). If COGC are unique to gunpowder or are not found in the environment at MECE detectable levels, identification of O-GSR containing these materials provides strong evidence of handgun use or exposure. An examination of commercially available gunpowder was conducted to identify components that may be used for this purpose. The minimum detectable quantity for each of these COGC was determined. Based on these detection limits, samples collected from the general population were examined to determine the potential for environmental exposure to any of the COGC used in the manufacture of gunpowder.

Materials and Methods

Capillary Electrophoresis

Analyses were conducted using a Hewlett-Packard (Waldbronn, Germany)^{3D}CE equipped with a diode array UV detector operating

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TABLE 1—Characteristic organic gunpowder components.*

Compound	Abbreviation	Usage
2,3-Dinitrotoluene	2,3-DNT	Flash inhibitor
2,4-Dinitrotoluene	2,4-DNT	Flash inhibitor
2,6-Dinitrotoluene	2,6-DNT	Flash inhibitor
3,4-Dinitrotoluene	3,4-DNT	Flash inhibitor
2-Nitrodiphenylamine	2-nDPA	Stabilizer reaction product
4-Nitrodiphenylamine	4-nDPA	Stabilizer reaction product
Dibutylphthalate	DBP	Plasticizer
Diethylphthalate	DEP	Plasticizer
Diphenylamine	DPA	Stabilizer
Ethylcentralite	EC	Stabilizer
Methylcentralite	MC	Stabilizer
Nitroglycerin	NG	Propellant
N-Nitrosodiphenylamine	N-nDPA	Stabilizer reaction product

* These compounds do not represent a comprehensive list of chemicals that may be found in gunpowder formulations. These are some of the most commonly occurring components, with the exception of the 4 DNT isomers. These were included to demonstrate the resolving power of the MECE technique for closely related compounds. The list also does not include nitrocellulose, which is a major gunpowder component. The analytical protocols described herein do not provide for the examination of nitrocellulose.

from 190 to 600 nm with monitoring conducted at 200 nm. The analytical column (from Hewlett-Packard) was a fused silica capillary, 80 cm in length, with an internal diameter of 75 μm . The column's optical cell was an extended path length cell of about 225 μm . The column was thermostated at 35°C using the instruments onboard thermostating system, and the sample tray was thermostated at 25°C using a Haake FJ (Paumus, NJ) external water circulating bath. Samples were placed in glass-lined 300 μL polypropylene sample vials (Hewlett-Packard). The buffer used for both the analytical analysis and sample preparation was 25 mmol/L sodium dodecylsulfate (Ultrapure Bioreagent grade—J. T. Baker, Phillipsburg, NJ), 10 mmol/L sodium tetraborate (Baker Analyzed grade—J. T. Baker), 10 mmol/L Boric Acid (Ultrapure Bioreagent grade—J. T. Baker) in water (Omnisolve HPLC grade—EM Science, Gibbstown, NJ) pH 8.5. The analysis method used a 3 min pressure flush of the capillary with run buffer, sample injection at 30 mbar for 1.5 s, separation using an applied voltage of 30 kV for 10 min.

Analytical Standards

Standards for the COGC listed in Table 1 were obtained from the following sources: DBP, DEP, 2,3-DNT, 2,4-DNT, 2,6-DNT, 3,4-DNT, DPA, 2-nDPA, 4-nDPA, MC, and EC were obtained from Aldrich (Milwaukee, WI), N-nDPA was obtained from Sigma (St. Louis, MO), and NG was obtained from Accustandard (New Haven, CT). These standards were dissolved in ethyl alcohol (U.S. Industrial Chemical Co.—Anaheim, CA) to provide a 10 mmol/L stock solution. 2-naphthol was obtained from Aldrich for use as the internal standard. A 10 mmol/L stock solution of 2-naphthol in ethyl alcohol was diluted in run buffer to provide a buffer solution that contained 100 $\mu\text{mol/L}$ internal standard. The GSR standards stock solution was diluted with the internal standard buffer solution to generate standard solutions at 100, 10, and 1 $\mu\text{mol/L}$. These solutions were used to generate four-point calibration curves (zero forced) for the quantitation of each compo-

nent. The internal standard buffer solution was used for all sample preparations.

Sample Collection

Two adhesive film lift sample collection procedures were used in this study. The first method, used in the initial phase of this study, has been previously described (5) and uses 1 in.² sections of masking tape (Tesa Tuck Inc.—New Rochelle, NY) held with tweezers to recover samples from hands and clothing. The tweezers were cleaned with methanol (Baker Analyzed grade—J. T. Baker) before and after each sampling. All adhesive tape was cleaned by ultrasonic agitation for 15 min in methanol. This was to ensure that any alcohol-soluble components of the adhesive were removed prior to sample collection. After samples were collected, the adhesive film lift was placed in a clean glass vial, capped, and refrigerated until analyzed. Samples were collected by pressing the adhesive film onto the surface in question. Four samples were collected from each of 100 individuals from the general population to test for the presence of any interfering compounds. Individual samples were collected from the palm and the back of both the left and right hand of each person (especially including the web between the thumb and forefinger). Each sample was collected by pressing a single piece of tape onto the skin in multiple locations on each surface. In addition to the four samples collected for each participant, a control sample of unused adhesive tape was saved for analysis along with the other samples.

The second method was an adaptation of the first method using a sampling device that could be used for both SEM and MECE analysis. The sampling device consisted of double-sided adhesive tape (Shurtape—Hickory, NC) attached to an aluminum SEM sample mount (Ted Pella, Inc.—Redding, CA) held with a single mount holder (Ted Pella, Inc.). This particular holder has a cover that allows it to serve as both a storage and mailing container. Samples collected with this device could be refrigerated after collection. The double-sided adhesive tape was cleaned with methanol prior to use in the same manner described above. Surfaces were sampled for GSR by pressing the sampling device onto the surface. Multiple pressings were made over the entire region of interest using a single sampling device.

Sample Preparation

Residue extracts for CE analysis were recovered from the adhesive film lifts by placing a 2 mm² section of the adhesive film lift in 50 μL of methanol and agitating in an ultrasonic bath for 15 min. Following extraction, 5 μL of ethylene glycol (J. T. Baker) was added to the extract to prevent dryness upon evaporation. The methanol was then removed by evaporation under a stream of dry nitrogen. The concentrate was then reconstituted in 50 μL of internal standard buffer solution.

Unfired gunpowder was collected from 9 mm, .38 caliber, 380 automatic, and 25 automatic ammunition purchased at a local gun shop. Canister reloading powders were obtained from the Washington State Patrol Crime Laboratory's firearm's reference collection. The gunpowder was prepared for CE analysis by placing 0.050 g of gunpowder in 5.00 mL of methanol and agitating in an ultrasonic bath for 15 min. A 5.0 μL aliquot of the methanol extract was diluted in 50 μL of the internal standard buffer solution. To verify completeness of extraction, undissolved material was extracted a second time and analyzed. No

significant quantity of the COGC was found in any of the powders that were re-extracted. This suggests that the initial extraction was sufficient to quantitatively extract the components of interest.

Results and Discussion

The efficacy of adhesive film lifts for the collection of O-GSR was previously demonstrated (5). The current study found that a double-sided masking type adhesive tape placed on an aluminum SEM sample stub could be used to collect GSR. The value of this design is that a sample can be analyzed first by SEM for inorganic GSR and then by MECE for O-GSR, thus providing collaborative evidence. MECE evaluation must be done of the chemical composition of any solvents, reagents, and adhesive tapes to identify and/or eliminate any possible interfering components. A blank sample using the extraction protocol outlined above should be analyzed by MECE, since the sample preparation method involves methanol extraction of the adhesive tape used for the film lift. Adhesives that do not dissolve in methanol must be selected. The adhesive tapes used in this study were selected because they met these requirements. Various other commercially available organic solvent resistant masking tapes (both single- and double-sided tapes) may meet these requirements, but should be evaluated on a case-to-case basis. If SEM analysis is to be conducted on samples the double-sided tape must also be able to meet the requirements of the SEM system used. The tape selected for this study was found to be suitable for SEM analysis (operational details and results are given in the following paper (6)). Chromatography grade (or higher) solvents and reagents should be used. The current study also determined that quantitative extraction of gunpowder can be achieved using ultrasonic extraction in methanol as described in the methods section above.

MECE analysis is a high-resolution separation technique. Complex mixtures can be examined in short periods of time as was previously demonstrated for COGC (4). That report utilized a CE system with a variable wavelength UV detector. Migration time information could be supplemented with spectral information only by conducting multiple analyses at several different detector wavelengths. Since that study, diode-array UV detectors have been made available as CE detectors. It is now possible to generate both high-resolution migration time separations and complete UV spectral information on each component during a single analysis using the diode-array UV detector on a CE system. Figure 1 shows the diode-array UV spectra of the common gunpowder additives and the 2-naphthol internal standard as generated during MECE analysis. Notable spectral differences can be seen between most of the components, including the 2-nDPA and 4-nDPA that are only partially resolved chromatographically. This spectral identification coupled with the migration time data provided by the high resolution CE separation provides a powerful 2-dimensional analysis technique for COGC.

Minimum detection limits for most of the compounds in Table 1 were determined in the previous study (4) using the variable wavelength UV detector. Since this study utilized a CE with a diode-array UV detector, it was necessary to determine the minimum detection limits again. The mass detection limits for the COGC are listed in Table 2. It should be noted that the injection volume for the

TABLE 2—GSR standard's detection limits.*

Compound	Detection Limit
2,4-Dinitrotoluene	1.1 picograms
2,6-Dinitrotoluene	1.2 picograms
3,4-Dinitrotoluene	1.0 picograms
2,3-Dinitrotoluene	1.3 picograms
2-Nitrodiphenylamine	1.9 picograms
4-Nitrodiphenylamine	2.1 picograms
Dibutylphthalate	2.6 picograms
Diethylphthalate	2.2 picograms
Diphenylamine	0.9 picograms
Ethylcentralite	1.8 picograms
Methylcentralite	1.1 picograms
Nitroglycerin	3.8 picograms
N-Nitrosodiphenylamine	1.0 picograms

* Calculations made based on a signal-to-noise ratio of about 3 to 1 at 200 nm and using an injected volume of 5.8 nL. All values represent the average of 10 replicate runs. Standard deviations from these values did not exceed 3%.

MECE analysis is on the order of nanoliters, thus the mass detection limits listed in Table 2 would translate into concentration detection limits that are not as good as those obtained in other chromatographic analyses. However, since MECE utilizes very small quantities of sample, the mass detection limits are good enough to allow for the determination of the COGC in partial fragments from a single grain of gunpowder.

In order for the detection of O-GSR to be considered a direct indication of the use of a handgun or contact with someone who had recently discharged a weapon, the uniqueness of detecting the COGC must be established. No reference has been found in the literature to the examination of the general population for COGC. One investigation, of workers in the explosives industry, found traces of NG on the hands following the handling of explosives containing NG (13). To establish whether any of the COGC can be found on the hands of people who have not recently fired a handgun, samples were collected from 100 volunteers representing a variety of occupations. A questionnaire/data sheet was developed to generate information concerning occupation, activity prior to sample collection, when hands had been previously cleaned, etc. This questionnaire is shown in Fig. 2. Most of the samples were collected at a state patrol operated vehicle inspection station located adjacent to the laboratory. Since vehicle inspection is mandatory in this state, the individuals participating in this study represented a good cross-section of occupations, economic status, age, and supposed hand cleanliness. The results of this survey are listed in Table 3. It is significant that based on the sampling and analysis protocols used in this study, none of the COGC were identified (based on the minimum detection limits) on the hands of any of the volunteers, regardless of occupation, etc. Seven of the participants indicated that they had some contact with a weapon within 24 h of sample collection. All of these indicated that they had washed their hands prior to the samples being collected. If O-GSR had been deposited on the hands of these individuals as a result of their activity with the weapon, the hand washing was sufficient to remove any residue to concentration levels below the minimum detection limits. It is also significant that no interferences were observed

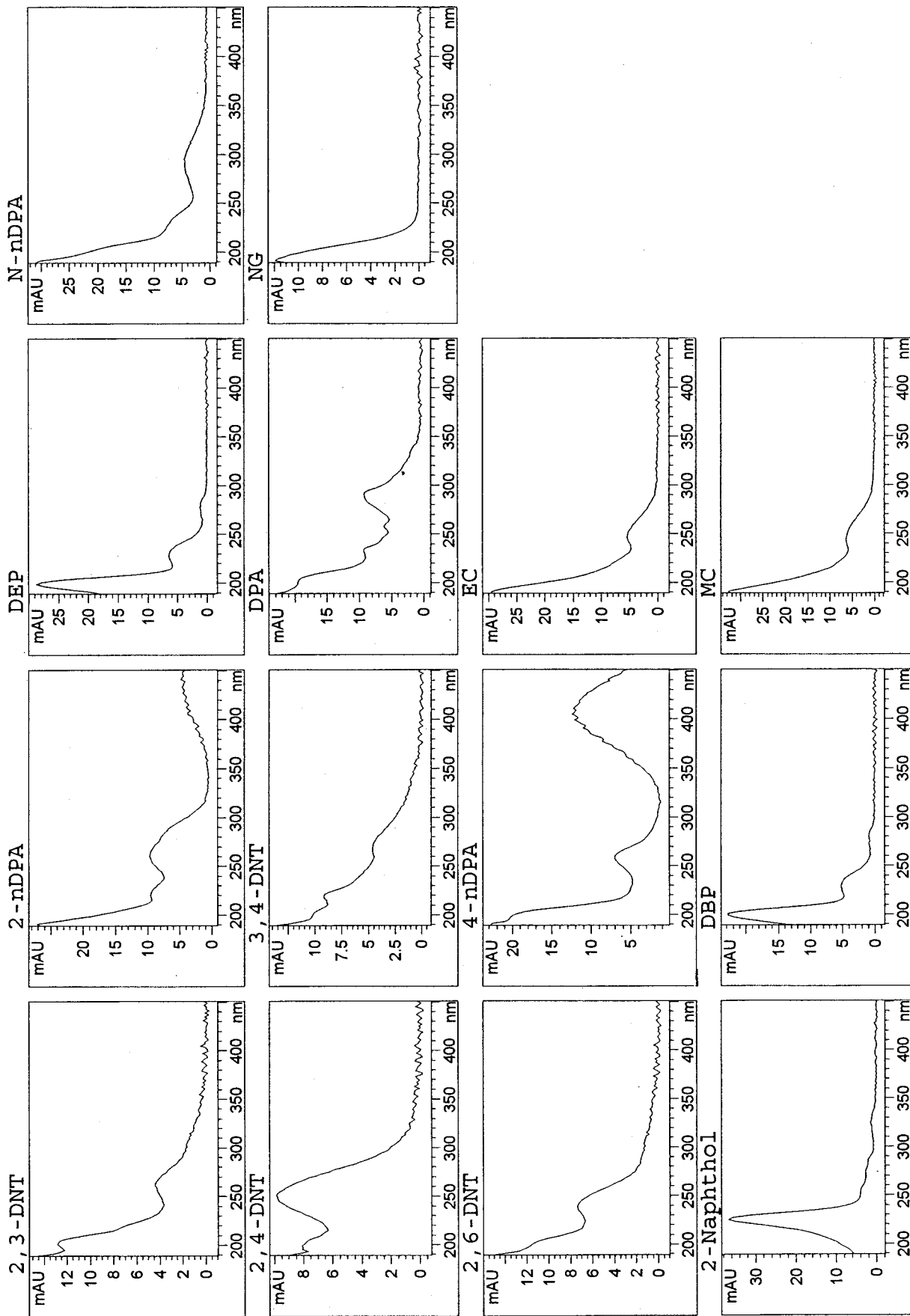


FIG. 1—Diode-Array UV spectra of smokeless gunpowder chemicals (from Table 1) and MECE internal standard 2-naphthol.

TABLE 3—Gunshot residues found in the general population.

Kit Numb	Occupation	Sex	R/L handed	Hand Size	At Work	Weapon	Collected	Time	Analyzed	SamCon	C	RB	RP	LB	LP
94-0001	Forensic Scientist	m	r	medium	yes	no	4/1/1994	10:50	4/4/1994	dry clean	0	0	0	0	0
94-0002	Teacher	f	r	small	no	no	4/4/1994	13:30	4/5/1994	dry clean	0	0	0	0	0
94-0003	Admin. Assist.	f	l	small	yes	no	4/4/1994	15:40	4/5/1994	dry clean	0	0	0	0	0
94-0004	Data Entry Oper.	f	r	medium	no	yes washed	4/5/1994	15:15	4/6/1994	dry clean	0	0	0	0	0
94-0005	State Trooper	m	r	medium	yes	yes washed	4/5/1994	15:20	4/12/1994	dry clean	0	0	0	0	0
94-0006	Janitor	f	r	large	yes	no	4/5/1994	16:30	4/14/1994	dry clean	0	0	0	0	0
94-0007	Vin Inspector	m	r	large	yes	no	4/14/1994	15:45	4/20/1994	dry clean	0	0	0	0	0
94-0008	UPS driver	m	r	medium	yes	no	4/20/1994	12:00	4/20/1994	dry dirty	0	0	0	0	0
94-0009	Mail Carrier	m	r	large	yes	no	4/20/1994	10:55	4/21/1994	dry clean	0	0	0	0	0
94-0010	Laundry Service	m	r	large	yes	no	4/20/1994	14:35	4/21/1994	dry clean	0	0	0	0	0
94-0011	Writer	m	r	medium	yes	no	4/20/1994	15:20	4/21/1994	dry clean	0	0	0	0	0
94-0012	Retiree	m	r	medium	yes	no	5/3/1994	13:05	5/11/1994	dry clean	0	0	0	0	0
94-0013	Unemployed	m	r	medium	yes	no	5/3/1994	13:15	5/11/1994	dry crack	0	0	0	0	0
94-0014	Undersheriff	m	r	medium	yes	no	5/3/1994	13:20	5/16/1994	dry clean	0	0	0	0	0
94-0015	Physical Therapist	f	r	small	no	no	5/3/1994	13:25	5/16/1994	dry clean	0	0	0	0	0
94-0016	Homemaker	f	r	small	yes	no	5/3/1994	13:35	5/18/1994	dry clean	0	0	0	0	0
94-0017	TV News Anchor	f	r	small	yes	no	5/3/1994	13:43	5/18/1994	dry clean	0	0	0	0	0
94-0018	Laborer	m	r	medium	yes	no	5/3/1994	13:50	5/19/1994	dry clean	0	0	0	0	0
94-0019	Retiree	m	r	medium	no	no	5/3/1994	13:55	5/19/1994	dry dirty	0	0	0	0	0
94-0020	Welder	m	r	large	no	no	5/3/1994	14:00	5/20/1994	dry dirty	0	0	0	0	0
94-0021	Homemaker	m	r	medium	yes	no	5/3/1994	14:05	5/23/1994	dry clean	0	0	0	0	0
94-0022	Security Guard	m	r	medium	yes	no	5/3/1994	14:10	5/23/1994	dry clean	0	0	0	0	0
94-0023	Laborer	m	r	medium	no	no	5/3/1994	14:20	5/23/1994	moist clean	0	0	0	0	0
94-0024	Nurse	f	r	small	no	no	5/3/1994	14:25	5/23/1994	dry clean	0	0	0	0	0
94-0025	Laborer	m	r	medium	no	no	5/3/1994	14:30	5/23/1994	dry dirty	0	0	0	0	0
94-0026	Homemaker	f	r	small	yes	no	5/3/1994	14:35	5/23/1994	dry clean	0	0	0	0	0
94-0027	Laborer	m	r	small	no	no	5/31/1994	10:20	6/10/1994	dry clean	0	0	0	0	0
94-0028	Maintenance	m	r	medium	yes	no	5/31/1994	10:30	6/10/1994	dry clean	0	0	0	0	0
94-0029	Driver	m	r	medium	yes	no	5/31/1994	10:35	6/10/1994	dry clean	0	0	0	0	0
94-0030	Driver	m	r	medium	no	no	5/31/1994	10:40	6/14/1994	dry clean	0	0	0	0	0
94-0031	Public Relations	m	r	medium	yes	no	5/31/1994	10:45	6/14/1994	dry clean	0	0	0	0	0
94-0032	Homemaker	f	r	small	yes	no	5/31/1994	10:50	6/14/1994	dry clean	0	0	0	0	0
94-0033	Tire Sales	m	r	medium	yes	no	5/31/1994	10:55	6/14/1994	dry dirty	0	0	0	0	0
94-0034	Nurse	f	r	small	no	no	5/31/1994	10:58	6/14/1994	dry clean	0	0	0	0	0
94-0035	Laborer	m	r	large	yes	no	5/31/1994	11:05	6/15/1994	dry grease	0	0	0	0	0
94-0036	Homemaker	f	r	small	yes	no	5/31/1994	13:05	6/15/1994	dry clean	0	0	0	0	0
94-0037	Retired	m	r/l	medium	yes	no	5/31/1994	13:10	6/15/1994	dry dirty	0	0	0	0	0
94-0038	Apart. Manager	f	r	small	yes	no	5/31/1994	13:15	6/15/1994	dry clean	0	0	0	0	0

(continued)

94-0039	Scientist	m	r	medium	yes	no	5/31/1994	13:20	6/15/1994	dry clean	0	0	0	0
94-0040	Fabricator	f	r	small	no	no	5/31/1994	13:25	6/16/1994	dry clean	0	0	0	0
94-0041	Firefighter	m	r	large	no	no	5/31/1994	13:30	6/16/1994	dry clean	0	0	0	0
94-0042	Quality Engineer	m	r	medium	no	yes washed	5/31/1994	13:35	6/16/1994	dry clean	0	0	0	0
94-0043	Retired	m	r	medium	yes	no	5/31/1994	13:40	6/17/1994	dry clean	0	0	0	0
94-0044	Realtor	m	r	large	yes	no	5/31/1994	13:45	6/17/1994	dry clean	0	0	0	0
94-0045	TV Journalist	m	r	medium	no	no	5/31/1994	13:46	6/17/1994	dry clean	0	0	0	0
94-0046	Truck driver	m	r	medium	yes	no	5/31/1994	13:50	6/17/1994	dry clean	0	0	0	0
94-0047	Forensic Scientist	m	r	medium	yes	no	5/31/1994	14:20	6/20/1994	moist dirty	0	0	0	0
94-0048	Forensic Scientist	m	r	medium	yes	no	5/31/1994	14:30	6/20/1994	dry dirty	0	0	0	0
94-0049	College Prof.	m	r	large	yes	no	5/31/1994	14:40	6/20/1994	dry clean	0	0	0	0
94-0050	Cook	f	r	medium	no	no	5/31/1994	14:55	6/20/1995	moist clean	0	0	0	0
94-0051	Car Sales	m	r	medium	yes	no	5/31/1994	15:00	6/20/1994	moist dirty	0	0	0	0
94-0052	Health Phys. Tech	m	r	medium	no	no	5/31/1994	15:10	6/21/1994	dry dirty	0	0	0	0
94-0053	Construction	m	r	medium	yes	no	5/31/1994	15:25	6/21/1994	dry dirty	0	0	0	0
94-0054	Secretary	f	r	medium	no	no	5/31/1994	15:30	6/21/1994	dry clean	0	0	0	0
94-0055	Retail	f	r	medium	yes	no	5/31/1994	15:35	6/21/1994	dry clean	0	0	0	0
94-0056	Retired	m	r	medium	yes	no	5/31/1994	15:37	6/22/1994	moist clean	0	0	0	0
94-0057	Firefighter	m	r	medium	no	no	5/31/1994	15:45	6/22/1994	dry dirty	0	0	0	0
94-0058	Construction	m	r	large	yes	yes washed	5/31/1994	15:50	6/22/1994	dry dirty	0	0	0	0
94-0059	Construction	m	r	medium	yes	yes washed	5/31/1994	16:00	6/22/1994	moist dirty	0	0	0	0
94-0060	Carpenter	m	r	medium	yes	no	5/31/1994	16:10	6/24/1994	moist dirty	0	0	0	0
94-0061	Mechanic	m	r	large	no	no	5/31/1994	16:15	6/24/1994	dry clean	0	0	0	0
94-0062	Restaurateur	f	r	small	yes	no	5/31/1994	16:20	6/24/1994	dry clean	0	0	0	0
94-0063	Geologist	m	r	medium	yes	no	5/31/1994	16:30	6/24/1994	moist dirty	0	0	0	0
94-0064	Laborer	m	r	medium	yes	yes washed	5/31/1994	16:35	6/27/1994	dry dirty	0	0	0	0
94-0065	Baker	m	r	small	yes	no	5/31/1994	16:40	6/27/1994	moist clean	0	0	0	0
94-0066	TV Reporter	f	r	small	yes	no	6/7/1994	12:00	6/27/1994	dry clean	0	0	0	0
94-0067	Fisherman	m	r/l	medium	yes	no	6/28/1994	10:25	6/30/1994	dry clean	0	0	0	0
94-0068	Physicist	m	r	medium	yes	no	6/28/1994	10:35	7/18/1994	dry clean	0	0	0	0
94-0069	Restaurateur	m	r	medium	no	no	6/28/1994	10:42	7/18/1994	dry clean	0	0	0	0
94-0070	Beverage Supervisor	f	r	small	no	no	6/28/1994	10:45	7/18/1994	dry clean	0	0	0	0
94-0071	Gardener	m	r	medium	yes	no	6/28/1994	10:50	7/18/1994	dry dirty	0	0	0	0
94-0072	Pipe fitter	m	r	medium	no	no	6/28/1994	11:00	7/19/1994	dry clean	0	0	0	0
94-0073	Teacher	m	r	large	no	no	6/28/1994	13:00	7/19/1994	dry clean	0	0	0	0
94-0074	Accountant	m	r	large	yes	no	6/28/1994	13:10	7/19/1994	dry clean	0	0	0	0
94-0075	Correctional Officer	m	r	medium	no	no	6/28/1994	13:15	7/19/1994	moist clean	0	0	0	0
94-0076	Retired	m	r	large	yes	no	6/28/1994	13:20	7/19/1994	dry dirty	0	0	0	0
94-0077	Research Scientist	m	r	medium	no	no	6/28/1994	13:35	7/20/1994	dry clean	0	0	0	0

(continued)

TABLE 3—Continued.

Kit Numb	Occupation	Sex	R/L handed	Hand Size	At Work	Weapon	Collected	Time	Analyzed	SamCon	C	RB	RP	LB	LP
94-0078	Teacher	f	r	small	no	no	6/28/1994	13:40	7/20/1994	dry clean	0	0	0	0	0
94-0079	Youth Ranch Counselor	m	r	medium	yes	no	6/28/1994	13:55	7/20/1994	dry clean	0	0	0	0	0
94-0080	Student	m	r	medium	yes	no	6/28/1994	14:00	7/20/1994	dry clean	0	0	0	0	0
94-0081	Student	m	r	medium	yes	no	6/28/1994	14:05	7/20/1994	dry clean	0	0	0	0	0
94-0082	Homemaker	f	l	small	yes	no	6/28/1994	14:10	7/21/1994	dry clean	0	0	0	0	0
94-0083	Truck Driver	m	r	medium	no	no	6/28/1994	14:30	7/21/1994	dry clean	0	0	0	0	0
94-0084	Lease Broker/Sales	m	r	large	yes	no	6/28/1994	14:40	7/21/1994	dry clean	0	0	0	0	0
94-0085	Teacher	f	r	one arm	no	no	6/28/1994	15:15	7/21/1994	dry clean	0	0	0	0	0
94-0086	Teacher	f	r	small	no	no	6/28/1994	15:15	7/21/1994	moist clean	0	0	0	0	0
94-0087	Electrician	m	r	medium	yes	yes washed	6/29/1994	9:55	7/22/1994	moist dirty	0	0	0	0	0
94-0088	Farmer	m	r	large	yes	no	6/29/1994	10:00	7/22/1994	dry dirty	0	0	0	0	0
94-0089	Audiologist	f	r	small	no	no	6/29/1994	10:10	7/22/1994	moist clean	0	0	0	0	0
94-0090	Engraver	m	l	medium	no	no	6/29/1994	10:15	7/22/1994	dry clean	0	0	0	0	0
94-0091	Auto Broker	f	r	medium	yes	no	6/29/1994	13:00	7/22/1994	dry clean	0	0	0	0	0
94-0092	Auto Broker	m	r	medium	yes	no	6/29/1994	13:05	7/25/1994	dry clean	0	0	0	0	0
94-0093	Car Dealer	m	r	medium	yes	no	6/29/1994	13:25	7/25/1994	dry dirty	0	0	0	0	0
94-0094	Unemployed	f	r	small	yes	no	6/29/1994	13:35	7/25/1994	dry clean	0	0	0	0	0
94-0095	Computer Technician	m	r	medium	no	no	6/29/1994	13:40	7/25/1994	dry dirty	0	0	0	0	0
94-0096	Social Worker	f	r	small	no	no	6/29/1994	13:45	7/25/1994	moist clean	0	0	0	0	0
94-0097	Warehouse Worker	m	r	large	yes	no	6/29/1994	13:50	7/26/1994	moist dirty	0	0	0	0	0
94-0098	Truck Driver	m	r	large	yes	no	6/29/1994	14:00	7/26/1994	moist dirty	0	0	0	0	0
94-0099	Daycare Worker	f	r	small	yes	no	6/29/1994	14:10	7/26/1994	moist clean	0	0	0	0	0
94-0100	Student	m	r	large	no	no	6/29/1994	14:15	7/26/1994	dry dirty	0	0	0	0	0

NOTE: SamCon = sample condition; C = control; 0 = no GSRs found; r = right; l = left; r/l = ambidextrous; RB = back of right hand; RP = palm of right hand; LB = back of left hand; LP = palm of left hand; Weapon = was a weapon handled or fired within 24 h, if so hands have been washed since then.

TABLE 4—Information values of gunpowder composition.

AMMUNITION	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%						0.78%	0.04%		0.04%	
Federal Parahellum	43A-5T06	47.6%	1.5%																
Makarov (9X18)	9AP					0.72%	0.06%	0.11%	0.02%	1.6%	0.03%								
Norinco	NA					0.42%	0.01%	0.26%	0.01%							0.41%	0.01%	0.02%	
Norma No. 19026	11308Y	42.2%	2.0%																
Remington	Y07YC8501	42.9%	2.7%													0.80%	0.08%	0.25%	
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%	
S. & W. Nyclud	AE0441	48.3%	1.2%													0.82%	0.01%	0.053%	
Sellier & Bellot	891					0.84%	0.05%	0.12%	0.01%		0.038%	0.003%						0.039%	
Sellier & Bellot	891					0.86%	0.01%	0.10%	0.0001%										
Speer "Lawrman"	4100T4	36.1%	2.5%													0.60%	0.05%		
Speer Blount	D05223	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%	
Super-Vel	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	56HN61785	19.2%	0.6%			0.71%	0.02%	0.34%	0.01%										
Winchester Luger Cartridge #30	56HN61785	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	56HN61785	20.3%	0.9%			0.75%	0.03%	0.36%	0.02%										
Winchester Luger Cartridge #30	56HN61785	20.2%	0.5%			0.75%	0.02%	0.36%	0.01%										
Winchester Luger Cartridge #10	46HN61/98	18.7%	0.1%			0.72%	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%	0.051%	0.009%	0.42%	0.05%	0.24%	0.03%						0.038%	0.004%	0.18%	0.02%	
Winchester Super-X	68HA91	15.8%	0.4%	0.079%	0.004%	0.64%	0.02%	0.30%	0.01%						0.043%	0.003%	0.25%	0.01%	
Winchester Supreme	54HF30	17.6%	0.6%			0.66%	0.02%	0.31%	0.02%								0.25%	0.001%	
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%			
Dynamit GECCO	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%	
Federal	17A-3250	48.6%	2.0%																
Fioschi	2704001-075	26.3%	1.4%	0.15%	0.004%														
Remington	LB20D	41.4%	0.30%																
Winchester	65SH62/97	48.7%	1.4%													0.66%	0.026%		
Winchester ACP	03GA41/37	37.4%	0.60%			0.30%	0.006%	0.56%	0.01%							1.82%	0.16%		
Winchester	11GF03/1190	39.5%	0.70%			0.14%	0.008%	0.072%	0.003%							0.54%	0.018%		
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%	0.008%				
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	P76MA10B			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%	0.44%	0.04%	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%	0.18%	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	680031H5	12.2%	0.2%	0.61%	0.01%			0.63%	0.01%	0.34%	0.01%						3.2%	0.1%	

(continued)

TABLE 4—Continued.

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
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%	0.13%	0.001%	0.001%	0.001%	4.6%	0.1%	
Winchester 760	760010HQ2	12.9%	0.5%	0.57%	0.01%	0.20%	0.01%	0.51%	0.02%	0.14%	0.014%	0.061%	0.005%	0.14%	0.014%	0.005%	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%	0.18%	0.004%	0.006%	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%	0.13%	0.005%	0.005%	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%	0.070%	0.005%	0.002%	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%	0.087%	0.002%	0.003%	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%	0.082%	0.004%	0.003%	0.003%			
Winchester 540	540080R22	30.1%	0.4%	0.14%	0.002%	0.51%	0.003%	0.74%	0.01%	0.12%	0.002%	0.054%	0.003%	0.12%	0.002%	0.003%	0.003%			
Winchester 571	500094 E61	30.2%	2.2%	0.49%	0.03%	0.051%	0.001%	0.64%	0.02%											
Winchester 630	63000XE21	53.3%	0.3%	0.32%	0.01%	0.051%	0.001%	0.85%	0.01%											
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.	160929931	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%			0.35%	0.04%	0.071%	0.01%			0.055%	0.005%	0.023%	0.003%	0.53%	0.05%			
CCI - Speer	210014	25.6%	2.3%																	
CCI-Blazer	G20N4	50.2%	1.0%																	
Eldorado PMC	38G-582	27.9%	0.4%			0.61%	0.01%	0.18%	0.004%			0.029%	0.002%	0.015%	0.002%	0.045%	0.004%	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%							0.49%	0.01%	0.38%	0.01%	
Lapua	6011FJUSU950					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	48.1%	0.5%													3.5%	0.2%			
Remington	LA08R	39.2%	1.2%													2.8%	0.1%			
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%					
SuperViel	GC0922B	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%	0.089%	0.002%							0.71%	0.03%			
Federal	090534H116	36.4%	0.60%			0.17%	0.006%	0.034%	0.004%											
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	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	042H42/99	43.3%	0.99%			0.47%	0.005%	0.37%	0.02%									0.15%	0.005%	
Winchester	05SL2/49	51.3%	0.60%																	
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	79GK70/56	49.2%	2.7%			0.54%	0.029%	0.55%	0.02%			0.045%	0.003%	0.021%	0.002%	0.026%	0.002%			

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

from any of the soaps, lotions, dirt, oil, and other unknown materials that were on the hands of those that participated in this study. These results suggest that COGC identified by MECE do not occur as a result of occupational duties or environmental exposure in a manner similar to the inorganic compounds previously studied (12). This eliminates the possibility of a false positive result as an artifact of these activities and thus the necessity of establishing a threshold concentration level to determine residue significance as is done for the metallic GSR residues. It should be noted that a meaningful blank sample should be collected for casework so that the lack of COGC can be verified for each individual. The following report (6) will discuss the persistence of residues after deposition and how this relates to attaining positive results in instances where a weapon has been utilized.

In order to provide evidence to identify a gunpowder or its residues, including the type of propellant, manufacturer, and lot, it is necessary to quantitatively identify the characteristic components of both the residues and the unfired gunpowder. The use of a unique compound as a tagging material for gunpowder and explosives has been discussed a number of times over the years. However, a recent report has indicated that tagging is not a currently feasible or advisable way to identify the source of these materials (14). In the absence of any unique gunpowder identifier, a catalogue of gunpowder composition based on MECE analysis would be a valuable tool in attempting to generate information regarding the source of a GSR or gunpowder. Table 4 is a compilation of qualitative and quantitative data on more than 100 gunpowders from both commercially prepared ammunition and canister reloading powders. Table 4 represents the type of data that can be generated to develop a database of gunpowder composition. It is important to note that N-nDPA, 2-nDPA, and 4-nDPA are the reaction products of the propellants with the stabilizer DPA. When these components are found in a sample their individual molar quantities should be added to that of the DPA to determine the original DPA concentration in the gunpowder at manufacture. The presence of the decomposition products is a result of storage time and conditions (15) and thus may provide additional useful information. Detection of these products must be considered when evaluating the composition of O-GSR.

It is also important to realize that the data provided in Table 4 represents the average composition of the bulk gunpowder. Gunpowders are formulated to produce specific ballistic properties, not chemical properties. Thus, the gunpowder in a single ammunition cartridge may not be homogeneous chemically because sometimes more than one powder are blended to achieve desired ballistic characteristics (personal communication during tour of the Olin Ordnance facility, St. Marks, FL, 1990). The results for a 38 caliber ammunition from Remington, Lot No. LA08R, demonstrate this fact. There are two entries in Table 4 for this gunpowder because there were two visually different gunpowder flakes contained in this ammunition. The results of the separate examination of each flake type shows a significant difference in composition between them. The implications of these results are that individual O-GSR particles could have compositions that differ from the average bulk composition of the original gunpowder. This consideration along with a number of other conditions that are generated when a weapon fires the ammunition will affect the degree of identification that can be obtained with quantitative MECE analysis. These factors will be evaluated in the following paper (6).

Conclusion

This study has determined that COGC generated in GSR can be readily examined by MECE analysis. A database of COGC in selected commercial gunpowder has been generated as a reference. Detection of the COGC is a strong indication of O-GSR with little or no likelihood that they resulted from environmental exposure. Thus, the identification of these compounds in a sample would suggest exposure to gunpowder as opposed to some other source. Sample collection methods that can be used for both MECE and SEM analysis were readily developed for the purpose of identifying both organic and inorganic GSR at the same time.

As a result of these studies, further research was conducted to examine other factors that contribute to the value of O-GSR information as developed by MECE analysis. These studies are detailed in the following paper (6).

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