

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

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ABSTRACT: Micellar electrokinetic capillary electrophoresis has been used to examine characteristic organic gunpowder compounds, including nitroglycerin, diphenylamine, ethylcentralite, and others. The purpose of this project was to develop MECE for implementation in gunshot residue casework. Studies were designed to provide data for evaluating casework samples. The first study compared the results of MECE analysis of organic gunshot residues (O-GSR) from firing range samples, to SEM results for inorganic GSR (I-GSR). MECE analysis found detectable O-GSR for each caliber of weapon examined except for the .22 caliber weapon. SEM analysis confirmed the presence of I-GSR in selected samples where O-GSR was identified by MECE, as well as vaporious lead for the .22 caliber sample. Repetitive firing experiments demonstrated that detectable O-GSR were not deposited each time the same weapon was fired under the same conditions. This leads to the conclusion that residue deposition mechanisms and collection efficiency significantly effect the outcome of O-GSR analyses. Another study found that detectable O-GSR do not persist for more than an hour after firing (under the conditions examined). Decomposition of O-GSR from environmental exposure occurs at a rate that is slow compared to residue persistence, thus, the value of O-GSR composition is not compromised by short term exposure. However, since time dependent changes do not occur, time of firing determinations cannot be made from this data. Finally, casework samples from the hands of suspected shooters and from victims' clothing were examined for O-GSR and I-GSR. O-GSR and I-GSR were identified on some of the samples. MECE has been found to be a potentially valuable tool in the examination of GSR evidence for characteristic organic gunpowder compounds.

KEYWORDS: forensic science, criminalistics, gunshot residue, capillary electrophoresis, gunpowder, scanning electron microscopy

Micellar electrokinetic capillary electrophoresis (MECE) is a comparatively new technique for the analysis of organic gunpowder residues (O-GSR) (1–3). The purpose of this paper and the preceding paper (4) was to provide data and reference information to assess MECE for use in forensic GSR casework. The previous study (4) addressed sample collection and preparation procedures,

detection limits, interferences, and occurrences of characteristic organic gunpowder compounds (COGC) in the general population resulting from environmental or occupational exposure, and the characteristic composition of some common ammunitions. The purpose of this paper is to address additional issues that relate to the significance of identifying O-GSR.

The first issue was to determine if detectable O-GSR could be identified after weapons of different caliber were fired. If O-GSR can be detected, it is then important to know if those residues can be detected every time the same weapon is fired. So, the second issue examined was to identify the frequency with which detectable O-GSR were deposited on a given surface (such as the hands of a shooter) when a weapon is fired. Lack of detectable O-GSR after shooting may be a result of either inefficient deposition or shooter activity afterwards, or a combination of both. A review of GSR analysis (5) described work by Wessel and colleagues at the Aerospace Corporation. They found a number of factors that can significantly impact the efficiency of O-GSR deposition and thus recovery, including: the type and condition of the weapon; number of shots fired; the amount of oil, moisture, or perspiration material on the deposited surface; and the direction and force of air currents.

If detectable O-GSR could be identified, it was necessary to find out how long detectable O-GSR persisted on a surface (in particular the hands of a suspected shooter) after deposition. Persistence of residues has a bearing on the value of the data collected. If residues persist for relatively long periods of time, it may not be possible to connect the residue to the time of a specific event. If O-GSR persist for too short a time, they may not be found upon collection. Previous studies (6) of I-GSR have found they persisted for approximately 1 to 2 h on the hands of the shooter. Reports indicate that the persistence of nitroglycerin in O-GSR (7,8) may vary anywhere from less than 30 min to 7 h.

The value of O-GSR data may be useful beyond just a presence or absence determination. Because of the potentially unique nature of COGC, compositional analysis of O-GSR may provide additional information such as type of ammunition, time of firing, etc. Various factors must be considered in evaluating O-GSR data for this purpose. One issue to examine was whether environmental exposure results in significant alteration of the original O-GSR composition after a residue has been deposited. It is well known that long-term storage of ammunition results in its degradation (9). Diphenylamine (DPA) is one of the stabilizers that is added to many gunpowder formulations. Over time, the decomposition of nitrocellulose results in the formation of several nitrated products of DPA. If O-GSR are to be matched to a specific ammunition or manufacturer, the compositional analysis of the residue must be

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similar to the unfired powder, or changes that occur from the firing process or environmental exposure need to be rendered sensible.

Another related issue was the comparability of O-GSR composition (post fired) to the original gunpowder composition (pre-fired). Work on O-GSR using fluorescence detection for ethylcentralite (EC) has suggested that O-GSR compositions may not match those of the original ammunition (10). This issue was examined by MECE analysis.

O-GSR data generated using MECE also needed to be compared to I-GSR data generated using SEM on the same samples, to show any correlation between I-GSR analysis and O-GSR analysis. This investigation involved the examination for both I-GSR and O-GSR on the same samples. It has been suggested that I-GSR and O-GSR results may be independent of each other since I-GSR and O-GSR come from different sources (11).

Finally, GSR analysis using both MECE and SEM on case samples was conducted. Laboratory generated GSR samples had a higher success rate in obtaining positive I-GSR results when compared to I-GSR results from actual case samples (12). It was suggested that this may be partially because actual case related GSR samples are generated and collected under nonideal, noncontrolled situations. This leads to a low success rate for the identification of GSR.

Materials and Methods

Capillary Electrophoresis, SEM, Analytical Standards, and Sample Preparation

The MECE instrumentation, methods, analytical standards, and sample preparation methods were described previously (4). The sample collection method used in this work was the adhesive lift method previously described (4) using double-sided masking type adhesive tape (Shurtape—Hickory, NC) on an aluminum SEM stub (Ted Pella, Inc., Redding, CA) or tweezer-held single-sided masking type adhesive tape (Tesa Tuck Inc., New Rochelle, NY). In this paper, the terms test samples and blank samples were defined as follows: Test samples were adhesive film lift samples collected after a weapon had been fired. These include a separate film lift from the back of each hand and the palm of each hand for a total of four samples. Blank samples were adhesive film lift samples collected after a shooter had washed his or her hands but prior to handling or discharging a weapon. The blank sample was used to demonstrate that no GSR was left from any previous exposure. Again, four samples were collected, one each from the back and palm of both hands. Hand washing was done using soap and warm water, and drying with a clean towel. Hand washing was done prior to each experiment and in between each series of firings if new samples were to be collected. All samples were stored in individual sealed containers at 0°C until analyzed. Weapons were cleaned prior to each experiment by a trained firearms examiner using clean swabs and gun cleaning oil. Both interior and exterior surfaces were cleaned.

SEM analysis was conducted using a CamScan Series 2 SEM (Cambridge, England) with an EDAX PV9800 detector using a tungsten filament at 20 keV and an acquisition energy of 1-20 keV, specimen current of $0.5 - 1 \times 10^{-9}$ amps, and a count rate of approximately 11 000. The samples were examined using an automated stage controlled with CamScan automated GSR software version 2.02. Samples were mounted at a tilt of 10°, magnified 2000× at a working distance of 30 mm and resolution of spot size 3. The criterion for a positive I-GSR result was the finding of one or more particles containing the three elements lead, barium, and antimony.

Ammunition Quantitation

A representative sample of each ammunition, used in all the studies described below, was saved for quantitative MECE analysis of the unfired gunpowder. The quantitative analysis method, and results for each ammunition, are included in the quantitative compositional study reported previously (4).

Experiments, Results, and Discussion

Effect of Weapon Caliber and Ammunition

The first experiment conducted was to determine if detectable O-GSR could be identified when weapons of different caliber and various brands of ammunition were used. Samples were generated under controlled firing range conditions using one of the laboratory's ventilated indoor firing ranges. The shooter did not handle any door or surface within this area during the experiment. For this study, weapons of different caliber were tested using a number of different ammunitions. Both blank and test samples were collected. Samples for MECE analysis were collected using the single-sided adhesive film lift method. Recovery of O-GSR from the adhesive was done by taking a single 2 mm² section of the adhesive tape, extracting with methanol, and preparing for MECE analysis as previously described (4). Samples for SEM analysis were collected using the double-sided adhesive tape method. Comprehensive SEM analysis of all samples was not conducted due to time and instrumentation constraints. However, representative samples from each caliber of weapon used, were examined by SEM. The shooter was right-handed and fired using only that hand. Weapons used were, a Jennings J-22, .22 caliber semiautomatic handgun, a Colt .25 caliber semiautomatic handgun, a Mauser .380 semiautomatic handgun, a Speed 6 Ruger .38 special revolver, and a Browning High Power 9 mm semiautomatic handgun. These caliber of weapons were selected because they represented the more commonly encountered types of weapons examined in this laboratory system. Ammunition used in this study is shown in Table 1.

Results from these experiments (Table 2) are listed in the order that the samples were generated and collected. A number of observations can be made from this data. Detectable O-GSR were obtained on various types and sizes of weapons and ammunition, confirming previously published work (2).

I-GSR was identified on the same samples on which O-GSR was identified for the 9 mm and .38 caliber tests. Identification of I-GSR for the .380, .25, and .22 caliber weapons cannot be conclusively connected to the finding of O-GSR because I-GSR was identified on the blank samples as well. This indicates that the hand washing did not completely remove the I-GSR from previous firings. The finding of no O-GSR on any of the blank samples confirms that hand washing successfully removes detectable O-GSR, as noted previously (2).

Detectable O-GSR was sometimes found on palm samples, back of firing hand samples, or both. This O-GSR location-specific inconsistency suggests the need to continue to collect samples from both locations, and a need to examine O-GSR deposition mechanisms to determine how, where, and with what frequency O-GSR is deposited. These issues will be examined later.

SEM analysis identified a large amount of vaporous lead on samples from the .22 caliber weapon. These results are consistent with GSR from a .22 caliber weapon. No detectable O-GSR were identified for the .22 caliber weapon used in this experiment. O-GSR analysis on samples from other .22 caliber weapons needs to be done to determine if the lack of detectable O-GSR is due

TABLE 1—Firing range study #1 ammunition.

Caliber	Manufacturer	Name	Lot Number
9 mm	Federal Cartridge Co.	Federal Premium Hydra-Shok	431572H067
9 mm	Eldorado Cartridge Co.	Starfire Classic 9 mm Luger	ELD95FA-008
9 mm	3-D Inv., Inc.	3-D Remanufactured	80317941
9 mm	Winchester	Subsonic Deep Penetrator	73FN11/82
9 mm	Winchester	Super-X Parabellum	66HA91/87
9 mm	Blount Inc.	Speer Gold Dot	D05Z23
9 mm	Remington	Golden Saber HPJ	Y07YC8501
9 mm	Sellier and Bellot	9 mm Luger	891
.38 Special	3-D Inv., Inc.	3-D Remanufactured	150929931
.38 Special	Eldorado Cartridge Corp.	Starfire Classic +P	385FA-014
.38 Special	Winchester	Super-X	60VM62/2
.38 Special	Eldorado Cartridge Corp.	PMC 38G	38G-582
.38 Special	Fiocchi	38 S&W Special	5330223610
.38 Special	Remington	38 Special	LE03H
.380	Federal	Premium Hydra-Shok	090534H116
.380	Winchester	Super-X	042HB42/99
.380	3-D Inv., Inc.	3-D Remanufactured	100314941
.25	Winchester	Full Metal Case	03GA41/37
.25	Eldorado Cartridge Corp.	PMC FMJ	25A-089
.25	Winchester	Super-X	11GF03/1190
.22	Winchester	Wildcat 22	2CB30L
.22	Winchester	Super-X	2HC40L
.22	Federal	American Eagle	38H133
.22	Federal	Hi-Power Long Rifle	3A4525
.22	CCI	Stinger 22LR	M08Y06
.22	CCI	Mini Mag	A17Z23

to the size of the weapon and ammunition, or if these results are connected to the specific weapon configuration, i.e., location of the ejection port and other factors that influence the amount and direction of O-GSR discharge. The importance of weapon configuration is demonstrated later in the discussion of O-GSR deposition experiments.

One result was obtained that should urge caution in the interpretation of data. Zeichner et al. (13) noted that detectable I-GSR could be recovered that had compositional characteristics from different ammunitions being fired from the same weapon. This same result was seen in one instance in this firing range study. The O-GSR composition found for the 9 mm Sellier and Bellot ammunition consisted of the COGC nitroglycerin (NG), diphenylamine (DPA), and N-nitrosodiphenylamine (N-nDPA), as well as dibutylphthalate (DBP). However, the original gunpowder composition of that ammunition (4) did not contain any NG (assuming that the gunpowder in the unfired cartridge was the same as the one that was fired). The most likely source for the NG in the O-GSR was from the weapon (even though the weapon was cleaned in between the use of each new ammunition), because the blank adhesive lifts indicated that the hands had been thoroughly cleaned prior to the test. A more thorough examination of the composition

of unfired gunpowder versus O-GSR composition will be discussed later.

Detectable O-GSR was not identified for every ammunition fired from the same weapon. There are at least two explanations for these results. One explanation is that there may be an ammunition-dependent component to O-GSR deposition. Ammunition is manufactured to meet velocity and pressure requirements, not chemical composition standards. The chemical composition, gunpowder quantity, shape, burn rate, and age of the gunpowder can vary from ammunition to ammunition. Each of these factors may effect the amount and location of O-GSR deposition. Thus it is possible that detectable O-GSR may not be deposited for all ammunitions. The second explanation is that a combination of these various ammunition characteristics, and different weapon characteristics, prevent detectable O-GSR from being deposited every time a weapon is fired.

O-GSR Deposition Frequency

A controlled firing range experiment was conducted to determine the frequency of detectable O-GSR deposition. Variables that were examined included differences in the location of the ammunition ejection port and which hand was used to fire the weapon. The two weapons examined were a Mac 9, 9 mm semi-automatic handgun with a right side ejection port, and a Walther P38, 9 mm semi-automatic handgun with a top ejection port. The shooter's hands were washed prior to firing and in between each series of experiments following the collection of postfiring samples. The double-sided adhesive film lift collection method was used to collect both blank and test samples. Three rounds of ammunition were fired for test samples. The shooter fired a series of three rounds from each weapon a total of 15 times, both with the left hand and with the right hand, for a total of 60 experiments using 180 rounds of ammunition. The ammunition used was Winchester 9 mm Luger 115 grain FMJ obtained from a local sporting goods store. Five boxes of 50 cartridges each were used. Every tenth cartridge in each box was saved as a reference for quantitative compositional analysis representing that box of ammunition. Experiments were conducted at an outdoor firing range. After the first two series of firings with the Mac 9 and first series of firings with the Walther P38, the firing was moved to an inside room open to the outdoor target, to shield the shooter from strong winds. Although extensive tests were not conducted, detectable O-GSR were not identified in any firing range experiment done out-of-doors in conditions other than calm air. This includes several other experiments that were conducted, but not reported, in this study.

Results of O-GSR deposition frequency experiments are shown in Figs. 1 and 2. The results for the Walther P38 (having a top ejection port) show that O-GSR were identified 100% of the time on the back of the shooting hand when the weapon was fired with the right hand, but at best only 71% of the time when fired with the left hand using O-GSR found on either the back or palm of the hand as criteria for a positive result. It should also be noted that residues were found variously on the back and palm of the hand, but most consistently on the back of the hand.

The results for the Mac 9 (having a right side ejection port) show a poor detectable O-GSR deposition frequency. The highest frequency, 62%, was achieved when using O-GSR found on either the back of the hand or the palm of the hand as the criteria for a positive result. If only the back or only the palm of the hand is considered, positive results were obtained less than 40% of the time. Detectable O-GSR results were even worse for the Mac 9 when fired

TABLE 2—Weapon caliber and ammunition experiment.

Weapon	Ammunition	Capillary Electrophoresis Results					SEM Results	
		Control	RB Blank	RP Blank	RB	RP	RB Blank	RB
9 mm Browning	Federal	no	no	no	A & F	no	no	>3
	Eldorado	no	no	no	A & F	no		
	3-D Inv. Inc.	no	no	no	A, B, C, D & E	no		
	Winchester Subsonic	no	no	no	A	no		
	Winchester Super-X	no	no	no	no	A		
	Sellier and Bellot	no	no	no	no	A		
	Speer Gold Dot	no	no	no	no	no		
	Remington 3-D Inv. Inc.	no	no	no	no	no	no	2
.38 Special Ruger	Eldorado Starfire	no	no	no	no	A		
	Winchester Super-X	no	no	no	no	no		
	Eldorado PMC	no	no	no	no	no		
	Fiocchi	no	no	no	no	no		
	Remington	no	no	no	A, B, & C	no		
	Federal	no	no	no	A	no	2	2
.380 Mauser	3-D Inv. Inc.	no	no	no	A, B & C	A, B & C		
	Winchester Super-X	no	no	no	A, B & C	A, B, C, D & E		
	Winchester FMC	no	no	no	A, B & C	no	>3	>3
.25 Colt	Eldorado PMC	no	no	no	A, B & C	no		
	Winchester Super-X	no	no	no	no	no		
	Winchester Wildcat	no	no	no	no	no	vaprous lead	vaprous lead
.22 Jennings	Winchester Super-X	no	no	no	no	no		
	Federal Am. Eagle	no	no	no	no	no		
	Federal Hi-Power	no	no	no	no	no		
	CCI Stinger	no	no	no	no	no		
	CCI Mini Mag	no	no	no	no	no		

NOTE: RB = Back of Right Hand; RP = Palm of Right Hand; no = no GSRs; A = nitroglycerin; B = diphenylamine; C = N-nitrosodiphenylamine; D = 2-nitrodiphenylamine; E = 4-nitrodiphenylamine; F = Ethylcentralite; SEM Results = the number of 3 component particles found.

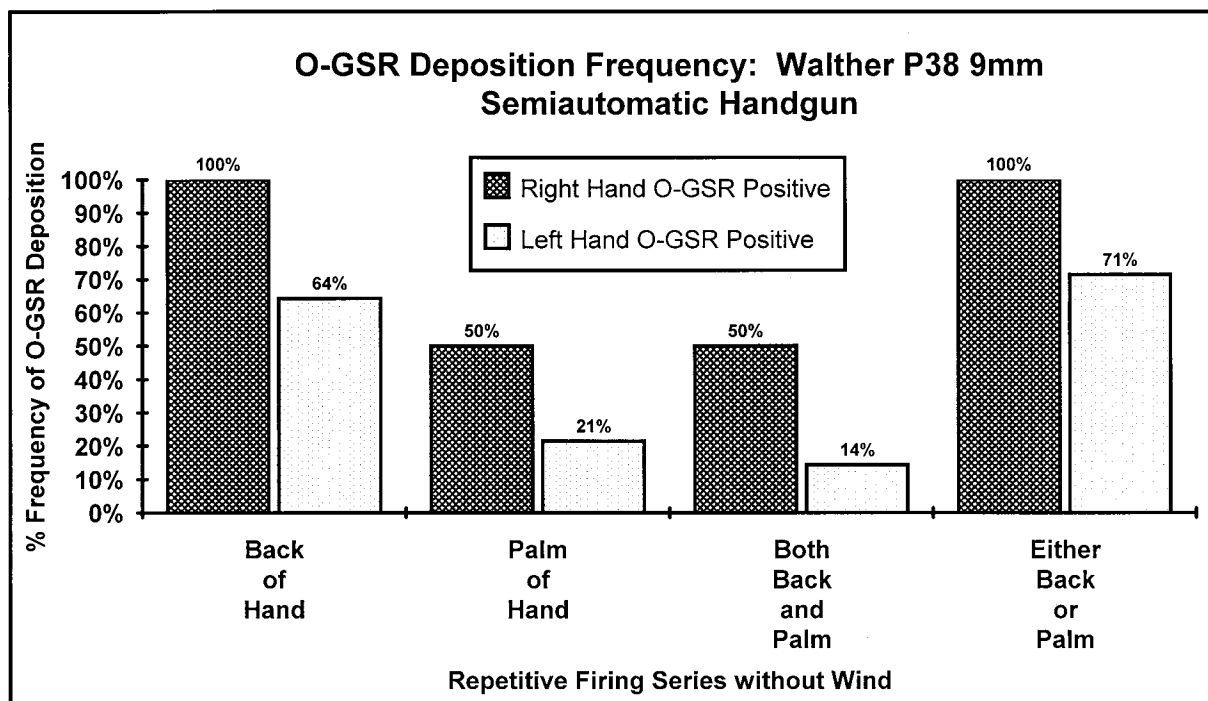


FIG. 1—O-GSR deposition frequency: Walther P38 9 mm semiautomatic handgun.

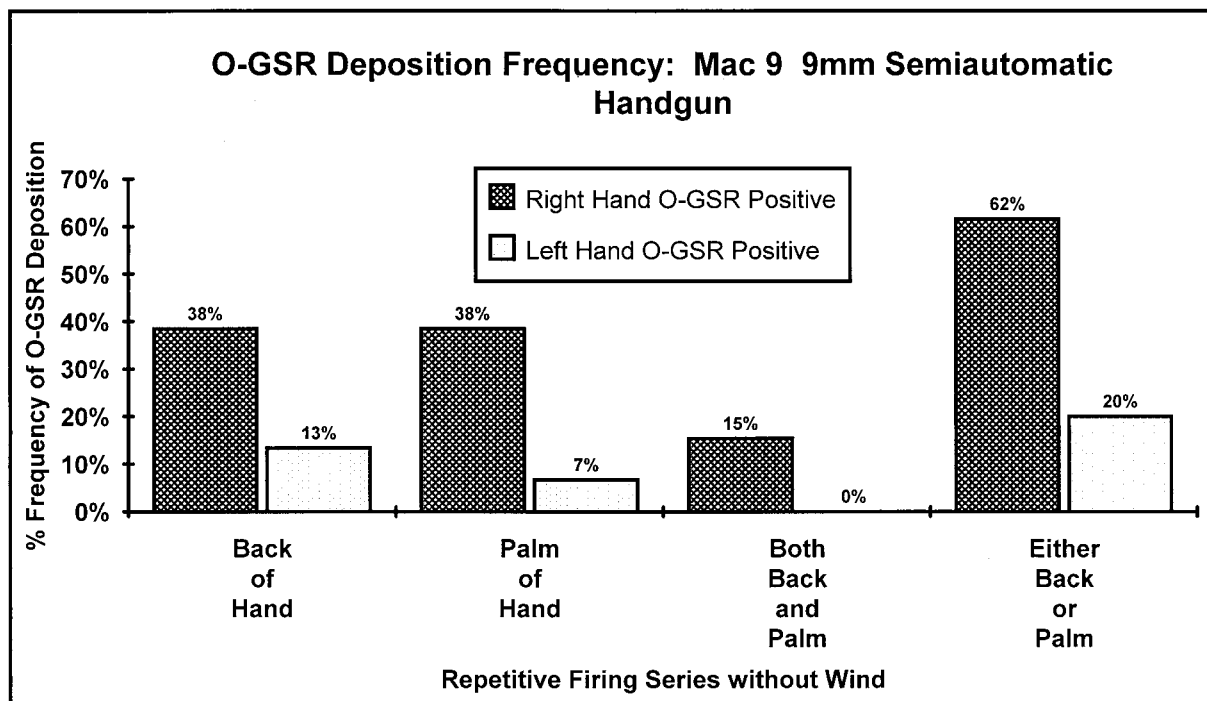


FIG. 2—O-GSR deposition frequency: Mac 9 9 mm semiautomatic handgun.

with the left hand. Using the selection criteria of detectable O-GSR on either the back or palm of the shooter's hand, resulted in only a 20% positive rate. If a positive GSR result was called only when residues were present on both the back and palm of the shooter's hand, none of the firings were positive.

These results clearly support the idea that the type of weapon can play a significant role in whether detectable O-GSR will be identified. This study also demonstrates that how the weapon is handled when fired will effect the results. Samples need to be collected from the back and palm of both hands to ensure that all possible methods of shooting are covered. Even in the best of circumstances, detectable O-GSR may not be found.

Once the presence of O-GSR on a surface (such as the shooter's hands) has been identified, it is important to know how long that residue may have been on that surface. The following experiment was conducted to determine the persistence of O-GSR after the firing of a weapon has occurred.

O-GSR Persistence

When GSR is introduced as evidence of handgun use, it is important to be able to evaluate both the possible origin and time of residue deposition. The suggestion that a suspect had been to a firing range the previous day or been involved in some other prior activity is commonly used to explain the presence of GSR. To address these types of arguments, it is necessary to show how long GSR are likely to persist on a given surface (such as the shooter's hands). A controlled firing range study was conducted in order to identify how long residues would persist on a shooter's hands given normal activity. Normal activity was defined as any activity that the shooters were engaged in during their work. However, that activity was to exclude any additional contact with firearms or ammunition, and hand washing. The weapon used was a Colt .25 caliber semiautomatic handgun (one of the weapons as used in the

first experiment) with Winchester ACP 50 grain .25 auto ammunition. Initially, the weapon was cleaned and then six shots of ACP ammunition were fired from the weapon (to simulate a used, uncleaned weapon). Blank samples were collected from each volunteer. Each shooter then fired three shots from the weapon and a sample was collected to represent Time Zero. This was followed by three additional shots fired by each shooter to redeposit a residue for collection after a specified time interval. Samples were collected at hourly intervals for 6 h. Four different shooters were used so that each hourly sample would be a fresh sample that had remained uncollected for that length of time. For example, if the sampling was done properly at Hour 1, no residues would remain for collection at subsequent times, thus in order to collect samples at subsequent time intervals, a new residue needed to be deposited prior to waiting the specified time interval. Shooter #1 was used for samples at 1 h, then the shooter's hands were cleaned, more shots fired, a new time zero sample collected, hands cleaned, more shots fired and sampling was done at 4 h. Shooter #2 was used in a similar manner to generate samples for collection at 2 h and 3 h. Shooter #3 was used for the 5 h sample and Shooter #4 was used for the 6 h sample. Samples were collected from the pockets of the shooters at the conclusion of the test period to determine if O-GSR had been transferred to that location. Each shooter was right-handed and fired using only that hand. Sample preparation and analysis were done as previously described. The firing range for this set of experiments was the same one used in the first experiment. As before, none of the shooters were allowed to touch any surfaces in the firearms section of the laboratory to eliminate any possibility of inadvertent contamination from a source other than the firing of the weapon.

All samples collected from the shooters immediately after firing the .25 caliber weapon (time zero) had detectable O-GSR. However, no detectable O-GSR was identified on the hands of the shooter after one hour. Subsequent samples at 2 to 6 h also were

found to have no detectable O-GSR. Time zero residues were at low concentrations, thus it is possible that weapons depositing greater quantities of O-GSR may result in longer residue persistence. The implication of these results is that normal activity is likely to cause the loss of O-GSR in a very short period of time following the discharge of the weapon. No detectable O-GSR was found on the samples collected from the pockets of the shooters, suggesting that the loss of O-GSR in this instance was not caused by putting hands in pockets.

One factor that was not studied, but may be important, is the effect of hand washing. It has been demonstrated that hand washing was effective in removing O-GSR, however, it may also contribute to the prevention of O-GSR retention. Hand washing tends to remove the natural fats and oils from the hand. Thus, cleaning hands prevents carry-over contamination, but it may also make the hands less adhesive for O-GSR subsequently deposited. A detailed examination of this factor has not yet been done, but previous reports suggest this may be an issue (14).

Since, under the conditions studied, detectable O-GSR do not persist on a shooter's hand for more than 1 h, any O-GSR identified cannot have resulted from an event that occurred on another day, such as shooting at a firing range. Thus, MECE identification of O-GSR could be considered highly indicative of recent firearms activity. However, not finding O-GSR does not indicate lack of firearms activity. Additional experiments need to be conducted to determine if these results hold true for weapons of different caliber, etc.

The experiments discussed so far have demonstrated some of the reasons that O-GSR is not identified even when firearms activity had occurred. Table 3 lists a number of factors that must be considered when evaluating O-GSR data. For example, given these considerations, it would be premature to suggest that the finding of no detectable O-GSR in the experiment using the Jennings .22 caliber handgun was related solely to the caliber of the weapon and ammunition. Another example would be the futility of collecting samples from hands of individuals suspected of being involved in a shooting where conditions allowed for significant airflow over the weapon and hand during discharge, as in a drive-by shooting.

Additional O-GSR Informational Value

The experiments to this point were centered around the question of the presence or absence of O-GSR. If O-GSR is identified, can additional information be generated, such as, time of shooting, or brand of ammunition used? For the purpose of using MECE analysis in identifying the source of a O-GSR, it is necessary to determine if the qualitative and compositional results are consistent from unfired gunpowder to O-GSR to environmentally exposed

O-GSR. Changes in the chemical composition of a given gunpowder might be expected to occur over time, particularly when exposed to heat and/or sunlight. It would also be expected that the conditions in a weapon when it is fired could cause chemical compositional changes. The following experiments were conducted to examine some factors related to these issues.

O-GSR Composition

A controlled firing range study was conducted to determine if O-GSR composition is similar to the composition of unfired gunpowder. This experiment was conducted in the laboratory's indoor firing range. To generate sufficient residue for repetitive quantitative analysis, a single shot was fired through a sterile nylon cloth (100% nylon, 9 in. × 9 in. Miracle Wipe 4000, Texwipe. Upper Saddle River, NJ) placed over a clean piece of paper placed in front of the firing range. Each shot was fired from a range of approximately 6 in. and fresh targets were used for each ammunition. The weapons used were a Smith & Wesson 9 mm semiautomatic handgun, a Smith & Wesson .38 caliber revolver, a Llama .380 semiautomatic handgun, and a Raven .25 caliber semiautomatic handgun. Each weapon was cleaned prior to the firing of each ammunition. The ammunition used for this study is listed in Table 1, and is the same as that used in the first firing range study with the exclusion of all the .22 caliber ammunition and three other ammunitions (Sellier and Bellot 9 mm, Remington .38 Special and Winchester .38 Special) that were no longer available. The cloth targets were individually placed in clean zip-loc plastic bags and refrigerated at 0°C until analyzed. At analysis time, samples were recovered from each cloth using the double-sided adhesive film lift method. For quantitative analysis, five replicate samples were examined from each adhesive film lift. These results were compared to the quantitative results on the unfired gunpowder from each ammunition. The unfired gunpowder was examined using the quantitative MECE methods previously described (4). In most double-based gunpowder, NG has the largest concentration of any of the COGC. Since it was not practical to accurately weigh the O-GSR collected from the cloth targets, comparison of the O-GSR composition was made to the original gunpowder by examining the concentration ratio of each of the other COGC to NG.

The results of this study are shown in Table 4. A review of these results shows they are more complicated than anticipated. Several examples will be discussed to illustrate this complexity. The results shown in Fig. 3 are from a gunpowder found to contain NG, diphenylamine (DPA), and n-nitrosodiphenylamine (N-nDPA) as COGC. The before-firing gunpowder composition and the composition of the O-GSR after firing are very similar. The differences in composition are well within the standard deviation

TABLE 3—*Factors effecting O-GSR identification.*

O-GSR Formation	O-GSR Deposition	O-GSR Persistence	O-GSR Recovery	O-GSR Analysis
gunpowder shape, size completeness of gunpowder burn gunpowder composition burn rate burn thermodynamics weapon condition—clean, worn etc	caliber of weapon type of weapon e.g., semiauto vs. revolver ejection port location weapon condition wind conditions random particle trajectories which hand was used to fire the weapon	time since deposition cleanliness of the hands or other surface sample surface postfiring activity quantity of samples	sampling method sample location, e.g., hands, face, etc. quantity of sample time since deposition cleanliness of hands	sample preparation type of gunpowder, e.g., single base etc. original COGC detection limits analysis method environmental exposure

TABLE 4—Gunpowder quantitation before and after firing.

9 mm 3-D Inv., Inc.					9 mm Federal				9 mm Speer			
Ratio to N	%Before	dev	%After	dev	%Before	dev	%After	dev	%Before	dev	%After	dev
2,4-DNT	0.500	0.056	0.320	0.092								
DPA	3.480	0.072	3.280	0.400					3.790	0.950	3.180	0.240
N-nDPA	3.170	0.096	2.560	0.200					1.690	0.450	1.580	0.150
EC	7.800	0.220	0.290	0.031	1.900	0.130	2.440	0.280				
9 mm Remington					9 mm Eldorado				9 mm Winchester Subsonic			
Ratio to N	%Before	dev	%After	dev	%Before	dev	%After	dev	%Before	dev	%After	dev
2,4-DNT												
DPA					1.390	0.160	1.160	0.200	2.880	0.340	2.220	0.190
N-nDPA					1.760	0.220	1.380	0.130	1.640	0.210	1.200	0.087
EC	1.860	0.180	2.160	0.300								
9 mm Winchester Supersonic												
Ratio to N	%Before	dev	%After	dev								
2,4-DNT												
DPA	4.050	0.130	2.890	0.360								
N-nDPA	1.900	0.076	1.110	0.340								
EC												
.38 cal Eldorado					.38 cal 3-D Inv., Inc.				.38 cal PMC			
Ratio to N	%Before	dev	%After	dev	%Before	dev	%After	dev	%Before	dev	%After	dev
DPA	1.530	0.058	1.200	0.550	1.880	0.120	1.990	0.270	2.190	0.032	1.200	0.350
N-nDPA	1.820	0.130	1.750	0.700	2.140	0.140	2.110	0.140	0.650	0.014	0.630	0.200
EC												
.38 cal Fiocchi												
Ratio to N	%Before	dev	%After	dev								
DPA												
N-nDPA												
EC	2.620	0.750	2.940	0.200								
.380 auto Federal					.380 auto Winchester				.380 auto 3-D Inv., Inc.			
Ratio to N	%Before	dev	%After	dev	%Before	dev	%After	dev	%Before	dev	%After	dev
DPA	0.470	0.016	0.200	0.043	1.070	0.012	0.480	0.048	1.880	0.025	1.580	0.120
N-nDPA	0.093	0.011	0.160	0.035	0.860	0.035	0.730	0.055	2.140	0.045	2.000	0.047
EC	1.620	0.055	2.440	0.800								
.25 auto ACP					.25 auto PMC				.25 auto Winchester			
Ratio to N	%Before	dev	%After	dev	%Before	dev	%After	dev	%Before	dev	%After	dev
DPA	0.800	0.016	0.610	0.073	1.760	0.069	1.300	0.190	0.350	0.020	0.230	0.042
N-nDPA	1.470	0.032	1.450	0.090	1.110	0.066	1.300	0.150	0.180	0.007	0.310	0.036
EC									1.620	0.058	2.130	0.690

NOTE: dev = standard deviation; NG = Nitroglycerin; 2,4-DNT = 2,4-dinitrotoluene; DPA = Diphenylamine; N-nDPA = N-nitrosodiphenylamine; EC = Ethylcentralite.

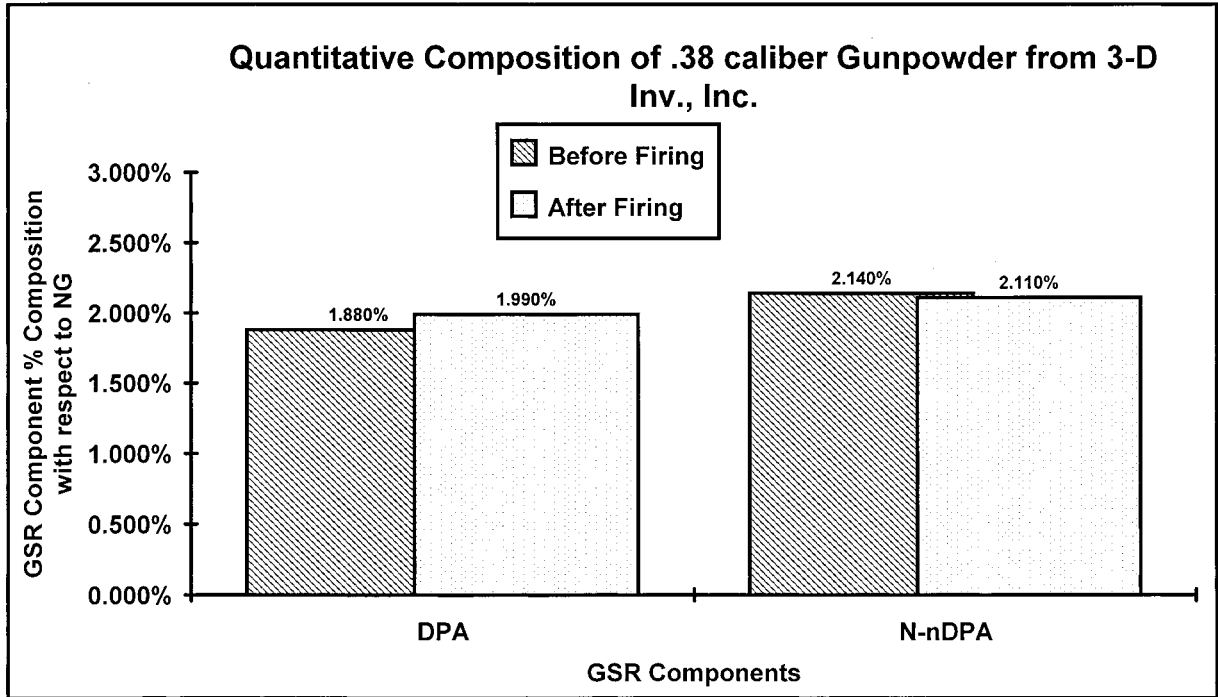


FIG. 3—Quantitative composition of .38 caliber gunpowder from 3-D Inv., Inc. comparison before and after firing.

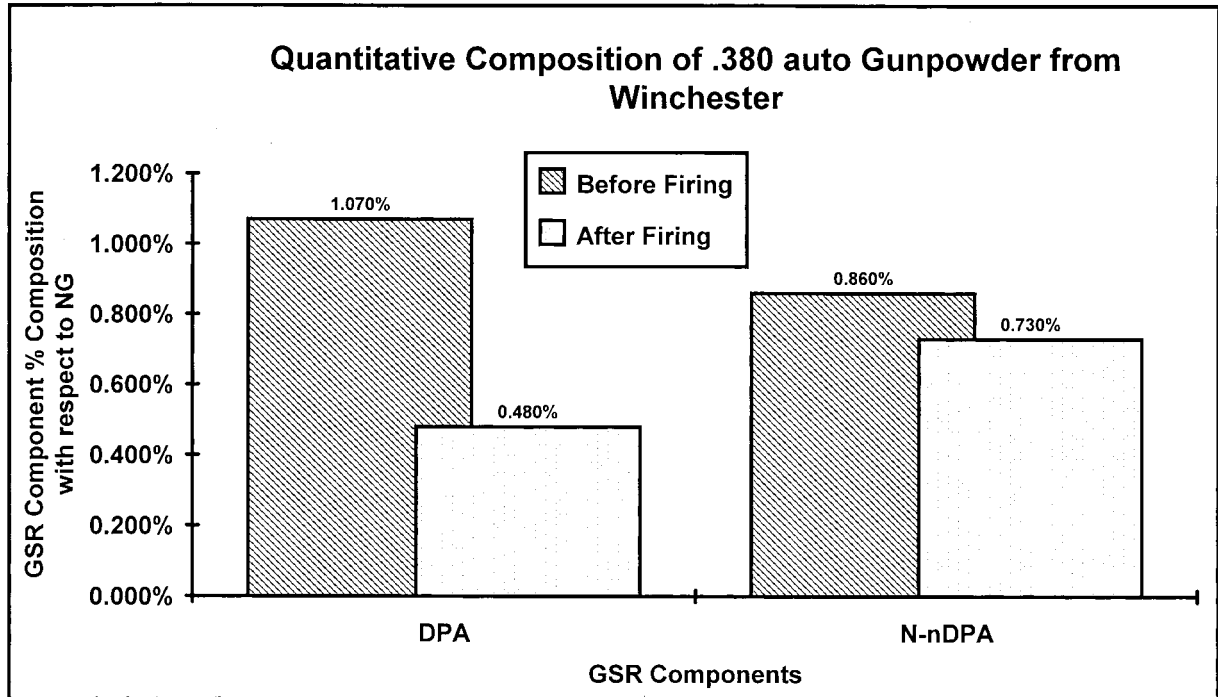


FIG. 4—Quantitative composition of .380 auto gunpowder from Winchester comparison before and after firing.

of the experimental data. These results would suggest that residues do not change significantly from the original gunpowder composition. However, the results shown in Fig. 4 seem to contradict this observation. These results are from a gunpowder that has an original COGC similar to the gunpowder in Fig. 3, and yet the O-GSR quantitative composition is significantly different

from the original gunpowder composition. What is not known is if the difference in composition is a sampling effect resulting from gunpowder and O-GSR inhomogeneity, or an actual chemical change resulting from the firing process. Because a ratio of components is being used, the change in ratio could be from a change in either the NG or other COGC concentration. Identify-

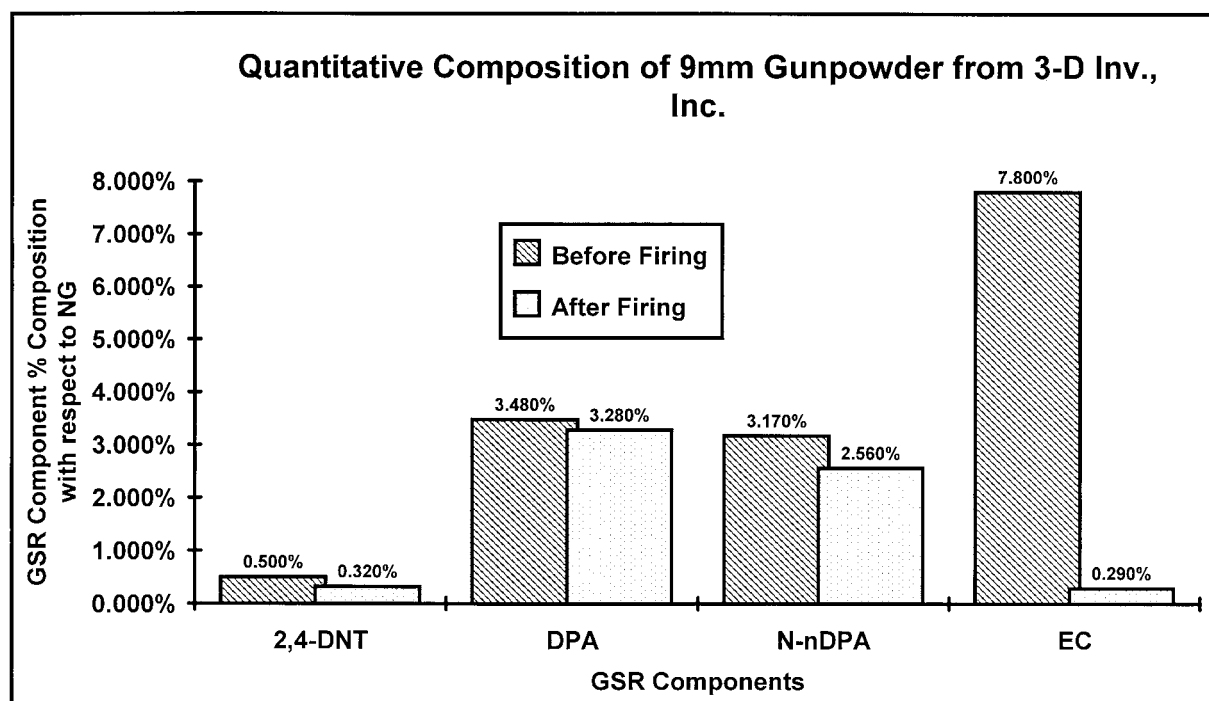


FIG. 5—Quantitative composition of 9 mm gunpowder from 3-D Inv., Inc. comparison before and after firing.

ing which component has changed in the O-GSR is not possible without weighing the sample first.

The results shown in Fig. 5 demonstrate what is likely to be a sampling effect difference and is similar to results previously reported (10). The gunpowder in this figure contains NG, 2,4-dinitrotoluene (2,4-DNT), DPA, N-nDPA, and ethylcentralite (EC). Some gunpowder is actually a mixture of two or more different batches of gunpowder as discussed previously (4). The significant difference in the EC concentration is almost certainly an indicator that the original gunpowder was an inhomogenous mixture. The O-GSR collected consisted of material that had almost no EC in its composition, whereas the bulk gunpowder contained a larger quantity of EC. Typically there are as many as several hundred grains of gunpowder in a single cartridge. The O-GSR that is collected may consist of only a few grains or less of the original material. As a result, it is very easy for the O-GSR collected to be a nonrepresentative sample of the original material if that original material was not initially uniform in composition. Another factor to remember is the potential for carryover from other ammunitions previously fired from the same weapon. In most instances, a carefully cleaned weapon resulted in no carryover, however, as discussed earlier, carryover occasionally can occur.

O-GSR composition data should be assessed with caution if it is to be used to identify a specific gunpowder as a source. O-GSR composition has been seen, in some cases, to be dramatically different from the original gunpowder, even under the controlled conditions of these experiments. Assessment of the results therefore is a complex issue. Added to that complexity is the possibility that exterior environmental exposure could alter the composition of O-GSR once it has been deposited on a surface. The next series of experiments were designed to evaluate some of the aspects of this problem.

Environmental Exposure

If environmental exposure results in significant degradation of gunpowder and O-GSR, it is expected that differences in the ratios of the various COGC would be observed. Typically, long-term (months/years) storage of ammunition shows this change, particularly in the stabilizer composition (9). For example, as an ammunition decomposes, DPA concentrations decrease, while the nitrated derivatives of DPA increase. Characterizing these types of changes was the goal of these studies. Additionally, this information could be used to estimate the time-of-firing.

The first environmental experiment was to evaluate the stability of O-GSR stored for days at ambient temperature ($22^{\circ}\text{C} \pm 1^{\circ}\text{C}$) in the laboratory. When O-GSR samples were generated for the quantitative analysis experiment, an extra sample of the Winchester .25 semiautomatic ammunition was made. This cloth sample, in its protective zip-loc plastic bag, was placed on a counter in the laboratory that was directly exposed to sunlight throughout the day. Samples were collected from the cloth after 38 days and 65 days on the bench. The quantitative analysis results were compared to those obtained on an O-GSR sample of this ammunition that was refrigerated and not exposed to the environment, and comparisons were made to the original gunpowder.

The O-GSR left on the bench for more than two months appeared to slowly lose NG, as evidenced by a small increase in the ratio of stabilizer to NG. However, the ratios of stabilizer to decomposition products did not change, and the quantity of the loss of NG was less than sample to sample variations arising from sample inhomogeneity. Thus, even over a long time (days) period, sunlight and room temperature exposure of O-GSR do not produce compositional changes significant enough to generate any time course information. This also means that under most normal circumstances, the composition of O-GSR is not likely to be effected

by indoor conditions during the time between deposition, collection, and analysis.

The second environmental exposure experiment was to evaluate the stability of gunpowder placed outside the laboratory. This was to simulate more extreme heat and light exposure. Gunpowder was chosen because of the difficulty in weighing sufficient O-GSR to do quantitative measurements. Environmental alteration of exposed gunpowder could then be connected to O-GSR composition by examining the COGC composition ratios of both.

A 50 mg sample of Winchester 748 canister reloading powder was placed in each of ten glass test tubes. The tubes were capped and placed outside the laboratory in direct sunlight. Once an hour for the first 8 h a sample was brought inside for quantitative MECE analysis. The last two samples were collected at 24 h and 28 h. Ambient air temperatures recorded at the time of each sample collection ranged from 62 to 84°F (17 to 29°C), with the knowledge that the temperature inside the glass tube was likely to be higher. A sample of unexposed gunpowder was also examined as a time zero sample. Each sample was split into three aliquots and each aliquot was run three times for a total of nine runs on each sample. The shorter time duration of the experiment was chosen given the relevance of the persistence experiments that showed retention of O-GSR to be less than 1 h. Containment of the samples in test tubes was done to prevent mechanical sample loss, however, this could cause a possible change in the ultraviolet radiation wavelength resulting from attenuation from the glass. Other factors, such as the presence or absence of the graphite coating, may also impact the results and were not studied at this time.

The gunpowder samples appeared to lose a small amount of NG over time, similar to the indoor O-GSR samples. However, as before, the change was less than one standard deviation for the measurements, and represented a deviation smaller than compositional variations from sample to sample. It does appear that sample decomposition may occur differently for gunpowder and O-GSR when in unconfined spaces. Confined spaces, such as the interior of the ammunition cartridge, prevent the loss of volatile components, such as NG. In an unconfined space, the generation of significant decomposition products may occur at a slower rate and preferential loss of volatile components may be increased. Extensive study of these aspects of compositional changes has not been done. The initial conclusions from the environmental study are that during the short period of time over which O-GSR may be successfully collected, environmental factors will not contribute to measurable changes in composition. Additional exposure experiments should be conducted to confirm these findings. Estimating the time-of-firing by looking at exposure-caused compositional change in O-GSR is thus not likely to be relevant. However, as mentioned above, the presence of O-GSR on a suspect's hands suggests recent activity.

Since short term environmental exposure does not appear to contribute to compositional changes in O-GSR, the examination of COGC ratios can provide valuable information about long term (months/years) storage effects. For example, if the O-GSR is found to have very small quantities of stabilizer decomposition products, it is possible to suggest that the ammunition was either relatively new, or had been stored in a cool location that does not encourage the decomposition process. Also, even in samples that show significant degradation, the sum of all the stabilizer components can suggest an approximate original concentration of stabilizer. Finally, to avoid any possible damage to O-GSR samples prior to analysis, they should be stored under refrigeration and away from sunlight.

Casework

The final phase of this project was to successfully examine casework samples using both O-GSR and I-GSR analysis, and to compare the relative value of both types of data. In addition, clothing samples in casework were tested using the Griess nitrate visualization test and sodium rhodizonate lead visualization test (15). Those results were also compared to the O-GSR and I-GSR data.

Fifty GSR sample collection kits were provided to two law enforcement agencies for use in cases involving shootings. The kits consisted of four adhesive film lift collection devices, an instruction sheet (Fig. 6), and a sample *Request for Analysis* form (Fig. 7). Additionally, sample kits were provided to firearms examiners in this laboratory system to be used on clothing examinations.

Only six sample kits of the original 50 sent out for hand GSR collection were returned, thus the significance of the findings is very limited. However, the results are discussed so as to highlight some of the difficulties encountered with case samples. More extensive studies need to be done before more definitive conclusions as to the utility of O-GSR analysis can be made. The results of the analyses of the six kits that were returned are listed in Table 5. I-GSR was detected on two of those six cases. O-GSR analysis on

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GSR SAMPLING INSTRUCTIONS

1. **Make absolutely certain that the person collecting the samples, thoroughly cleans his/her hands before collecting the samples. He/She also must not touch any potentially contaminated surfaces after cleaning hands and before collecting the samples.**
2. Four (4) samples are to be collected as follows:
 - 1) Back of right hand - including thumb, forefinger and web in between
 - 2) Back of left hand - including thumb, forefinger and web in between
 - 3) Face - especially around any self inflicted wound - avoid blood
 - 4) Control - any area of body that could not be contaminated, e.g. foot
3. To collect sample: remove cover from the sample stub holder; discard the paper that covers the adhesive; dab the stub on the selected area very thoroughly; replace the cover on the sample stub. Use only one stub per sample.
4. If the samples are not already labeled, place one of the labels provided on each sample for identification purposes.
5. **Keep samples refrigerated and in the dark until delivered to the laboratory.**
6. Fill out a GSR sample data request sheet provided with the sample kit. The questions concerning the activity of the samplee are designed to determine if the individual had contact with a weapon or occupational material prior to the actual shooting event for which the sampling is being conducted. The question about the condition of the sample refers to issues such as sweaty, dirty or bloody sampling surfaces, or any other condition that may contribute to the integrity of the sample. As indicated above, collection of blood should be avoided to prevent the spread of blood borne pathogens. If this is unavoidable, then a biohazard sticker should be applied to the outside of the sample kit, and indicated on the request sheet.

GSR SAMPLE KIT

Return Samples to the Kennewick Crime Laboratory

FIG. 6—Instruction sheet for GSR collection.

KENNEWICK CRIME LABORATORY
CASE GSR DATA

Sample Kit No. _____	Date Collected _____
Agency Case No. _____	Time Collected _____
Suspect _____	Offense Date _____
Victim _____	Date Analyzed _____

Person's Normal Occupation: _____

<input type="checkbox"/> Male	<input type="checkbox"/> Right-Handed	<input type="checkbox"/> Small Hands
<input type="checkbox"/> Female	<input type="checkbox"/> Left-Handed	<input type="checkbox"/> Medium Hands
		<input type="checkbox"/> Large Hands

Was person engaged in normal occupation within the last two hours? Yes, No
If [No], what activity was person engaged in?

Had person handled , or fired a weapon in the last 24 hours? Yes, No
If yes, had person washed hands since that activity? Yes, No

Misc. Notes: _____

Samples Collected and Condition of Sample

Right Back _____
Left Back _____
Face _____
Control _____

Submitted By: _____ Submitted Via: _____ Rec'd By: _____
Signature _____ Date/Time: _____

Disposition of Evidence _____ Date/Time: _____
Returned By: _____ Returned Via: _____

LABORATORY USE ONLY

<u>Results of Analysis</u>	Compounds Found
Right Back _____	
Left Back _____	
Face _____	
Control _____	

FIG. 7—GSR request for analysis form.

one of the six cases indicated NG at a concentration near the detection limit. No detectable O-GSR was found in any of the other cases returned by detectives. In all but one case, sample collection occurred hours after the event. In the homicide case, the samples were collected from the victim who had fired multiple shots at the assailant. Despite the time delay in sampling, the victim was deceased and therefore no activity caused transfer of O-GSR from the hands could have occurred. In the suicide case, finding no detectable O-GSR would be expected since the victim was cleaned by emergency personnel at a hospital prior to sample collection. Reasons for not detecting O-GSR on the other cases are not known, but could be due to any of the factors listed in Table 3. It is of interest to note that on four of the six cases no O-GSR or I-GSR was detected. Thus, the lack of detectable GSR is not likely to be an analysis issue, but rather a deposition, retention mechanism or collection issue. A finding of no detectable GSR must therefore be reported as inconclusive of handgun activity.

Sample kits provided to firearms examiners for clothing examinations were used in two homicide cases. Sixteen adhesive film lift samples were collected in these two cases and subjected to

both O-GSR and I-GSR analysis. Table 6 shows the results of the examinations conducted on those samples. O-GSR analysis on the first case collaborated the other examinations with the exception of the SEM results on the back of the cap. In the second case the results demonstrate the value of testing for both the I-GSR and the O-GSR. Detectable GSR were identified on 7 of the 13 samples. However, two of the samples that had no detectable O-GSR, had detectable I-GSR (two 3-component particles each). Conversely, there were also two samples that had no detectable I-GSR, but detectable O-GSR. These results support the idea that I-GSR and O-GSR may not be deposited on the same surface by the same mechanism or at equally detectable levels, thus supporting analysis for both. SEM and MECE analyses should be seen as generating independent data. Conducting both examinations may provide a higher degree of certainty as to the presence or absence of GSR. The results from these cases demonstrated the feasibility of using the same sample collection kit for both O-GSR and I-GSR analysis. Also, GSR may be found on clothing for longer periods of time and at apparently higher concentrations than on the hands of the shooter.

TABLE 5—Case analysis by MECE and SEM—hand samples.

Case Number	Type of Case	Weapon	Time After Firing	Sample	CE Results	SEM Results
6950773	Homicide	Browning 9 mm	7 h	Back R-hand Back L-hand Face Control	Trace of A No O-GSR No O-GSR No O-GSR	>3 1 1 No I-GSR
6951495	Assault	Unknown 9 mm	Unknown	Back R-hand Back L-hand Face Control	No O-GSR No O-GSR No O-GSR No O-GSR	No I-GSR No I-GSR No I-GSR No I-GSR
6951500	Armed Robbery	Unknown	Approx. 6 hours	Back R-hand Back L-hand Face Control	No O-GSR No O-GSR No O-GSR No O-GSR	No I-GSR No I-GSR No I-GSR No I-GSR
		Unknown	Approx. 5 hours	Back R-hand Back L-hand Face Control	No O-GSR No O-GSR No O-GSR No O-GSR	No I-GSR No I-GSR No I-GSR No I-GSR
95CF11268	Suicide	Jennings .22	Approx. 2 hours	Back R-hand Back L-hand Face Control	No O-GSR No O-GSR No O-GSR No O-GSR	No I-GSR No I-GSR No I-GSR No I-GSR
6960036	Assault	Semiauto	less than 1 hour	Back R-hand Back L-hand Face Control	No O-GSR No O-GSR No O-GSR No O-GSR	1 >3 >3 No I-GSR
6960037	Assault	Unknown	Approx. 3 hours	Back R-hand Back L-hand Face Control	No O-GSR No O-GSR No O-GSR No O-GSR	No I-GSR No I-GSR No I-GSR No I-GSR

NOTE: CE Results: A = nitroglycerin, B = diphenylamine, C = N-nitrosodiphenylamine, SEM Results = number of 3 component particles.

TABLE 6—Case analysis by MECE and SEM—clothing samples.

Case Number	Type of Case	Weapon	Sample Location	CE Results	SEM Results	Greiss	Na Rhodizonate
3951433A	Homicide	Charter Arms .38 Special	Cap - near hole	A & C	>3	Positive	Positive
			Cap - near hole & blood	A	>3	Positive	Positive
			Cap - back	No O-GSR	>3	Negative	Negative
3950568	Homicide	Taurus PT92c	Shirt R-sleeve - near hole	A	2	Negative	Positive
			Shirt front - near hole	A, B & C	>3	Inconcl.	Positive
			Shirt front - near collar hole	A	1	Negative	Positive
			Shirt shoulder - near collar	No O-GSR	No I-GSR	Negative	Positive
			Shirt L-sleeve - near hole	No O-GSR	2	Negative	Positive
			Sweater - near hole #1	No O-GSR	No I-GSR	Negative	Negative
			Sweater - near hole #2	No O-GSR	2	Negative	Positive
			Sweater - near hole #3	No O-GSR	No I-GSR	Negative	Negative
			Sweater - near hole #4	A	No I-GSR	Negative	Positive
			Sweater - near hole #5	No O-GSR	No I-GSR	Negative	Negative
			Cardigan - around collar	No O-GSR	No I-GSR	Negative	Negative
			Black jacket - near hole #1	No O-GSR	No I-GSR	Negative	Negative
Black jacket - near hole #2	A, B & C	No I-GSR	Negative	Negative			

NOTE: CE Results: A = nitroglycerin, B = diphenylamine, C = N-nitrosodiphenylamine SEM Results = the number of 3 component particles found.

Conclusion

The results of this research demonstrate the value of MECE analysis for O-GSR. Because MECE analysis can identify COGC, it is possible to generate more information than is possible with I-GSR analysis. The significance of detecting O-GSR on a sample is magnified by the finding of no false positive results in any of the experiments conducted. MECE detection limits for COGC provide for the ability to obtain quantitative analysis on samples smaller than an individual grain of gunpowder. It was found that O-GSR deposition and retention mechanisms significantly effect the ability to identify detectable O-GSR. A number of factors that contribute to these mechanisms were identified as reasons for not detecting O-GSR. Recoverable O-GSR may not persist on skin for more than 1 h, thus samples must be collected immediately. This short recovery time may pose a problem for effectively obtaining meaningful samples. Quantitative analysis for the purpose of generating a "chemical fingerprint" to match O-GSR to known gunpowder must be interpreted with care. Compositional variations of unfired and fired gunpowder particles occur and can have a significant impact on this data. Elimination of possible carry-over or cross-contamination from different ammunitions must be evaluated prior to making any ammunition source determinations. Time of firing information cannot be generated from compositional O-GSR analysis because the time needed for significant compositional change to occur appears to be longer than the O-GSR persistence time.

Since the deposition and retention mechanisms for O-GSR and I-GSR are not completely dependent on each other, evaluating evidence for both can provide valuable complementary information. O-GSR analysis may eventually be the only option if the trend towards "lead free" ammunition continues. MECE analysis also provides for higher sample through-put than SEM analysis methods. Sample preparation and analysis can be achieved in about 2 h per case using MECE analysis. Qualitative and quantitative information can be generated for the O-GSR with an instrument that costs four to five times less than for SEM equipment. The most significant limitation to MECE analysis for O-GSR is specifically an issue of residue recovery. This issue is also a problem in traditional I-GSR analysis. More effective recovery of residues would greatly enhance the information that could be generated in GSR analysis by MECE. Additional work needs to be conducted on the issues raised in this study in order to more clearly understand O-GSR analysis. However, the results of this study continue to demonstrate the flexibility of CE as a general analytical tool in the forensic laboratory and in particular its use in rapidly generating valuable information in GSR casework.

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