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## Investigating the Effect of Changing Ammunition on the Composition of Organic Additives in Gunshot Residue (OGSR)

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**ABSTRACT:** The measurement of the organic additives in smokeless gunpowder is an attractive approach for the detection of handgun use because it provides compositional information that can help associate residues and unfired gunpowder. We investigate several factors that will be required to advance the characterization of organic gunshot residue (OGSR) as a useful forensic tool, including evaluating residue contamination from previously fired ammunition, particle-to-particle compositional variability, and compositional features resulting from the type of firing primer used. Using ammunition loaded with known smokeless powders containing different stabilizers, a sequence of shots was fired from a .357 magnum revolver, and the muzzle exit residues were collected. Compositional analysis of the residues, both in bulk and as single particles, showed only a trace of the previously fired powder in the first shot and none in subsequent shots. In an additional experiment testing conventional leaded and the new lead-free firing primers, the OGSR composition was found not to depend on the primer type.

**KEYWORDS:** forensic science, capillary electrophoresis, compositional analysis, diphenylamine, ethyl centralite, gunpowder, gunshot residue, handgun, nitroglycerin

Characterization of organic compositions in gunshot residue (OGSR) by measuring the amount of propellant and stabilizer additives is a relatively new approach for the detection of the use of handguns (1–4). Qualitative identification of the various gunpowder additives provides information that will help associate residues with unfired gunpowder, as well as to determine whether or not a suspect fired a weapon (3). Recently, quantitative compositional measurements of nitroglycerin (NG), diphenylamine (DPA), N-nitrosodiphenylamine (NnDPA), and N,N'-diethyl-N,N'-diphenyl urea (ethyl centralite, EC) in OGSR have been used to determine if residue and unfired powder compositions can be reliably associ-

ated under ideal conditions (5). In order for the analysis of OGSR to become a useful forensic tool, a number of factors that may affect OGSR composition need to be evaluated, including: a. the possibility of residue contamination from previously fired ammunition, b. uncertainties resulting from particle-to-particle compositional variability, and c. compositional features that are influenced by the type of firing primer used.

The problem of residue contamination has been previously evaluated for the inorganic primer GSR. Zeichner et al. (6) performed a test firing experiment in which gunpowders containing different metallic primer compositions were fired from the same weapon. Analysis of the residues by scanning electron microscopy combined with energy dispersive X-ray analysis (SEM/EDX) showed the presence of mixed residues with primer composition change. Similarly, Wolten and Nesbitt (7) reported that when firing a used weapon, a small percentage of particles containing copper or antimony were observed fairly often in GSR from cartridges that did not contain either element. Harris (8) studied the effect that conventional leaded ammunition had on the subsequent firing of CCI Blazer lead-free ammunition. In addition to observing lead, barium, and antimony particles in the lead-free ammunition GSR, these same elements were also found in shavings taken from the inside of the weapon chamber. Gunaratnam (9) found lead and barium in GSR recovered from firing lead-free Sintox ammunition from a thoroughly cleaned weapon. Although the precise mechanism by which the metallic accumulations are deposited and released is not known, it has been proposed to be a function of the high temperature and pressure that is created inside the chamber when the primer and gunpowder rapidly vaporize and condense (9). Evidently, the volume of material that constitutes metallic GSR is small compared to the volume retained in the barrel of the weapon. Significant residue contamination is observed as the metallic barrel accumulations are released (8).

The problem of OGSR contamination from previous weapon use has been considered in two previous studies (10,11). After evaluating individual residue particles and qualitatively identifying the presence of specific additive components, Mach et al. (10) found some evidence for contamination of the most recently fired OGSR from past weapon use. Andrasko (11), employing an additive compositional pattern correlation, found no contamination from previous firings. In this paper, we rigorously evaluate the OGSR contamination issue using a quantitative additive determination of residues obtained from known gunpowders fired in sequence.

In a previous study of gunpowder composition, the particle-to-particle variability of seven commercial reloading powders was

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\* Certain commercial equipment, instruments, or materials are identified in this paper to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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studied. While some powders demonstrated consistent single particle compositions, others showed a wide range of additive concentrations (5). Calculating the ratio of propellant NG to stabilizer content provided additional information to help associate residues to unfired powders, even for powders with particle-to-particle heterogeneity. Because OGSR particles appear to be largely a result of incomplete combustion of individual grains, the variability in the unfired powder composition will be reflected in the resulting fired residues. This variability will be important to consider when only a few particles of OGSR can be recovered. We made a preliminary investigation of OGSR heterogeneity in this study.

OGSR composition may also be influenced by the primer material that is used in the ammunition (12). Study of this effect is particularly timely due to the recent introduction of new, heavy-metal-free primer compositions. Responding to increasing concern over health and environmental risks associated with the use of conventional leaded ammunition, manufacturers and the United States military have developed lead-free primers and bullets. Therefore, attention should be directed toward studying the characteristic components that are associated with the primer as well as the gunpowder. In the new primers, more common metals such as zinc and strontium or metal-free organic formulations are used rather than the conventional combinations containing lead, barium, and antimony. Since the detection of zinc and strontium is not unique to GSR, other means of identifying gunshot residue must be explored. Analyses were performed to determine whether any significant differences existed between the concentrations of the recovered components produced by lead-free primers versus conventional primers. We also investigated whether lead-free primer ammunition had characteristic organic compounds for OGSR detection using capillary electrophoresis.

## Materials and Methods

### *Reagents and Standards*

HPLC grade methanol (J.T. Baker, Phillipsburg, NJ) was used to extract the OGSR samples using the ultrasonic solvent extraction (USE) method (5). The buffer solution for the capillary electrophoresis (CE) consisted of 25 mmol/L sodium dodecyl sulfate (SDS) (sequanal grade, Pierce, Rockford, IL) and 10 mmol/L sodium borate buffer, pH 9.1 (granular boric acid, Mallinckrodt, Paris, KY and crystalline sodium borate, J.T. Baker) in HPLC grade water. A standard solution containing nominally 2000 mg/L NG, 1000 mg/L DPA, and 1000 mg/L EC (AccuStandard Inc., New Haven, CT) in methanol was diluted to 200 mg/L NG (100 mg/L other components) with the buffer solution for calibration. NnDPA (Chem Service, West Chester, PA) was also added to the standard solution for a final concentration of 100 mg/L. The internal standard, 2-methyl-6-nitroaniline (MNA) from Fluka (Milwaukee, WI), was prepared in methanol (5).

### *Ammunition Testing*

The two commercially available gunpowders used were Hi-Skor 700X, distributed by IMR Powder Company (Washington, PA), and Universal Clays, distributed by Hodgdon (Shawnee Mission, KS). Samples of the gunpowders were retained for analysis, while the remaining portions were reloaded into brass cartridges with either conventional or CCI lead-free primers (Small Pistol O. E. M. Primer, Blount Inc., Lewiston, ID) at the National Institute of Standards and Technology firing range, Gaithersburg, MD. For the powder change experiments, each type of gunpowder was reloaded

into 100 Remington brass cartridges (.357 magnum), primed with conventional leaded primer. For the primer change experiments, 92 rounds with each powder and both primer types were prepared. The weapon was a used Smith and Wesson, Model 19-4, .357 magnum revolver. The projectiles were 125 grain jacketed hollow points. Each cartridge was loaded with 4.5 grains (about 290 mg) of powder.

### *Residue Collection*

Gunshot residues were collected in twelve plastic target boxes (41 cm × 38 cm × 17 cm). Each had a removable lid and a 10 cm diameter hole drilled through the bottom. The holes were covered with 15 cm × 15 cm Teflon squares (Bytac, chemical resistant Teflon® FEP film, PGC Scientifics) through which the bullets were fired. A photo of the collection box is presented elsewhere (5). During the test firings, the box lid was removed and the weapon was fired at a distance of 2 m, perpendicular to the target, at a downward angle of approximately 30°. The test firings were conducted free hand and indoors.

The first step in the experimental protocol involved cleaning the weapon with a commercial firearms cleaning fluid (Klean-Bore Formula 3 gun conditioner), spraying it with Generation 2000 Contact Clean, freon flush, then allowing it to air dry. To “condition” the weapon, five rounds of Hi-Skor loaded ammunition were fired from the cleaned weapon. Muzzle exit residues from the conditioned weapon were obtained by firing five shots of Hi-Skor ammunition into collection box #1. Next, a single shot using ammunition loaded with different gunpowder, Universal Clays, was fired into collection box #2. A second single shot of Universal Clays was fired into box #3. Rounds three through five of Universal Clays were fired into box #4, and rounds six through ten of Universal Clays were fired into box #5. Each box was covered, when not in use, to prevent contamination. This entire sequence was repeated four times to insure that a sufficient amount of residues had been collected for analysis. Then the order in which the Hi-Skor and Universal Clays ammunitions were fired was reversed, collecting residues in five additional boxes.

In order to determine the effect of lead-free primer on the composition of OGSR, 92 rounds each of ammunition reloaded with either Hi-Skor or Universal Clays gunpowders were fired into two separate collection boxes.

### *Extraction and Analysis*

The bulk composition of the collected residue was determined by removing five 2 mg samples from each box and analyzing by USE/CE. The procedure used had been previously developed in our laboratory (2,5). Each of the five independent sample extracts was analyzed in triplicate on the CE to give a total of fifteen measurements. Five 2 mg samples of the unfired powders were extracted and analyzed in the same manner.

The “individual particle” composition was determined by removing three particles from each collection box and performing a single analysis on each.

Concentrations of the additives in the bulk residues, unfired powders, and single residue particles were calculated based on the internal standard method, using peak area counts of a gunpowder additive standard, internal standard (MNA), and the samples to determine the final concentration in mg/L. The mass of each sample and any dilutions were included in the final calculation to produce concentrations in mg/g. Average mg/g concentrations (for five measurements of residues or powders and three measurements of

single particles) were determined, as well as standard deviations of the mean.

Analyses were performed on a capillary electrophoresis system (Beckman P/ACE 5510, Brea, CA) with fixed wavelength absorbance detection at 214 nm and computer-based chromatography data collection. Extended path length (200  $\mu\text{m}$  window diameter) capillaries were used (Agilent Technologies, Palo Alto, CA) with a 75  $\mu\text{m}$  inner diameter and an overall length of 77 cm. A 1 s pressure injection was used with a separation voltage of 22 kV, a column temperature of 30°C, and a 20 min analysis time. A controlled temperature bath kept the samples at a constant 10°C during analysis on the CE.

## Results and Discussion

### Comparing Powder and Residue Composition

Initially, we evaluated the relationship between the composition of unfired powder and residues from a clean, conditioned weapon that fired ammunition loaded with Hi-Skor and Universal Clays powders. The quantitatively-determined values of the additive composition of the two unfired powders and of the bulk (2 mg sample size) residues from five firings of a conditioned weapon are presented in Table 1. Since identical canisters of the two gunpowders were used in a similar earlier study, those previously measured concentrations are also tabulated. The data for DPA and its primary decomposition product, NnDPA, are included on a single line since the total stabilizer content is the sum of DPA and NnDPA. The concentrations of the NG and EC in the Hi-Skor are similar in unfired powder and fired residues for both studies, a result that is consistent with a study of the handgun-fired residues of seven gunpowders (5). This powder showed very consistent particle-to-particle composition. However, when the concentrations of NG and DPA + NnDPA in the unfired powder are compared to the residue concentrations for Universal Clays, considerable differences are found. This result is in agreement with data from a previous study (5).

We can partly attribute the observed differences to the high compositional heterogeneity of our canister of Universal Clays. When ten individual particles of this powder were evaluated, the compositional variations, expressed as percent relative standard deviation of the mean, was 35% (5). Measurement agreement between the composition as determined by the average value of ten single particles and on bulk 2 mg samples was also poor for this canister of powder (5). Perhaps, as is often the practice in the smokeless powder industry (13), this canister contained two or more batches of manufactured powder that were blended to achieve specific burn

rate criteria. For the evidentiary evaluation of a limited number of residue particles, powders with such dramatic heterogeneity will impede the association of a residue to an unfired powder based solely on quantitatively determined additive composition. In such cases, the evaluation of other characteristic factors, such as the qualitative identification of other additives (2) or powder/residue morphology (14,15), would be needed to confirm associations.

### Effect of Changing Ammunition on Residue Composition

To evaluate the effect of changing ammunition on residue composition, two double-base reloading powders were used. Universal Clays contained DPA as the primary stabilizer and Hi-Skor contained EC. In addition, for the ammunition powder loads used for this experiment, the amount of residue collected per shot was quite different, where the relatively efficient-burning Hi-Skor produced few collected residues ( $\approx 1.0$  mg/shot) and the relatively inefficient-burning Universal Clays produced a much larger amount ( $\approx 7.6$  mg/shot). With this experimental sequence, a completely different stabilizer could be detected from the previous shots, and any effect of different burning efficiencies could also be noted. In most cases, sufficient residue was present in each collection box to make five independent compositional determinations, measured in triplicate. However, for the single first and second shots of the efficiently burning Hi-Skor ammunition, only enough residue was present to comprise three and two samples, respectively. In order to maintain uniformity in the number of measurements made, replicates were taken from the extracted samples to give a total of fifteen measurements per box.

Test firings were made with a used .357 magnum revolver that was cleaned and conditioned. Hi-Skor residues were collected by firing downward five times into the residue collection box, which was then covered. Then, without cleaning the weapon, single shots were fired with ammunition loaded with Universal Clays into separate collection boxes representing the first, second, third through fifth, and sixth through tenth shots. To assure the collection of sufficient residues, the sequence was repeated four times. In a second set of experiments, firing of ammunition loaded with Universal Clays was followed by Hi-Skor ammunition.

The results of bulk measurements made on 2 mg powder and residue samples are presented in Table 2. Examining the composition of the first shot after the ammunition change reveals only a trace of contamination from the residues of previous firings. In the first experiment, when changing from the relatively efficient burning Hi-Skor (stabilized with EC) to the Universal Clays (stabilized with DPA), only an unquantifiable trace (detected in one sub-sam-

TABLE 1—Concentration of gunpowder additives based on bulk measurements of unfired gunpowder and from the first five shots from a clean weapon. Concentrations represent average values and one standard deviation of the mean from five independent samples.

Powder	Composition	Avg Conc (mg/g) This Study	Avg Conc (mg/g) (5)	
Hi-Skor powder	NG	300 $\pm$ 6	251 $\pm$ 9	
	EC	10.9 $\pm$ 0.2	7.9 $\pm$ 0.3	
	.38 residues	NG	259 $\pm$ 7	279 $\pm$ 12
		EC	7.4 $\pm$ 0.2	8.3 $\pm$ 0.4
Universal Clays powder	NG	113 $\pm$ 6	70 $\pm$ 4	
	DPA (NnDPA)	12.8 $\pm$ 0.2 (0.7 $\pm$ 0.1)	7.1 $\pm$ 0.3 (ND)	
	.38 residues	NG	38.6 $\pm$ 1.6	40 $\pm$ 2
		DPA (NnDPA)	6.4 $\pm$ 0.9 (1.9 $\pm$ 0.1)	7.2 $\pm$ 0.2 (2.6 $\pm$ 0.1)

TABLE 2—Bulk residue compositions and concentrations recovered from changing ammunition. Concentrations represent average values and one standard deviation of the mean from five independent samples.

Ammunition Load	Composition	Avg Conc in mg/g
initial load: Hi-Skor five shots	NG	259 ± 7
	EC	7.4 ± 0.2
second load: Universal Clays first shot	NG	36 ± 3.5
	DPA (NnDPA)	4.6 ± 0.1 (1.4)
	EC	ND*
second shot	NG	42.4 ± 6.5
	DPA (NnDPA)	5.8 ± 0.1 (1.6 ± 0.1)
third through fifth shot	NG	33 ± 1.9
	DPA (NnDPA)	6.4 ± 0.2 (2.1 ± 0.1)
sixth through tenth shot	NG	40.8 ± 3
	DPA (NnDPA)	7.1 ± 0.2 (2.6 ± 0.5)
initial load: Universal Clays five shots	NG	38.6 ± 1.6
	DPA (NnDPA)	6.4 ± 0.9 (1.9 ± 0.1)
second load: Hi-Skor first shot	NG	237 ± 7
	DPA	0.4 ± 0.1
	EC	6.5 ± 0.1
second shot	NG	255 ± 7
	EC	7.3 ± 0.4
third through fifth shot	NG	272 ± 4
	EC	9.3 ± 0.2
sixth through tenth shot	NG	258 ± 8
	EC	8.9 ± 0.3

\* A low concentration of EC (0.01 mg/g) was detected in one replicate of one residue sub-sample.

ple) of EC from the Hi-Skor was found in the first shot with the new ammunition. No trace of EC was found in the second or subsequent shots. In the second experiment, changing from the relatively inefficient burning Universal Clays to the Hi-Skor, a quantifiable trace of DPA was found in the first shot. No trace of DPA was found in the second or subsequent shots. Even in the first shot, the amount of contamination from previous firings is very small, representing less than 6% of the total stabilizer detected. These experimental conditions probably represent a “worst case” sequence where an inefficiently burning ammunition was followed by a very efficiently burning load.

We have found it useful to calculate the ratio of NG propellant (P) to the combined stabilizer (S) content as a characteristic powder identifier (5). The P/S ratio is defined as  $[NG] / [EC]$  for EC-stabilized powders and  $[NG] / \sum [DPA] + [NnDPA]$  for DPA-stabilized powders. The accumulated results presenting the P/S ratios are displayed in Fig. 1 and will be discussed under specific sub-headings.

#### Bulk Powder/Residue Measurements

The P/S ratio bulk compositional results from the first shot after changing from Hi-Skor to Universal Clays loaded ammunition is shown in the upper left-hand quadrant of Fig. 1 (white diamond symbols). The horizontal line width represents the 95% confidence intervals of the measurements. Since no stabilizer of the previous ammunition was detected in the second through tenth shots, the data for these firings were combined. For Universal Clays ammunition, residues from all experiments had a somewhat lower P/S than the unfired powder (black diamond), although the uncertainties overlap. The first shot had a slightly larger P/S than the subse-

quent firings, but the uncertainties overlap significantly. In addition, the first shot average closely agrees with the results from a weapon that was conditioned with the Universal Clays ammunition (lead primer data, gray symbols). Note that these residue measurements show a high uncertainty value that reflects the heterogeneity of the unfired powder.

Results for bulk measurements, switching the order of firing from Universal Clays to Hi-Skor loaded ammunition, are presented in the right upper quadrant of Fig. 1 (white circles). The first shot appears to have a slightly higher P/S value than the combined second through tenth shot, albeit with an overlap of the confidence intervals. However, as noted with Universal Clays, the first shot results are in good agreement with data from a conditioned weapon (lead primer, gray circles). Thus, we believe the P/S ratio differences noted for the first and subsequent shots of both ammunitions are well within the uncertainty associated with repeated firing and analysis. Contrary to the results for Universal Clays, all residue P/S ratios are somewhat higher than the unfired powder (black circle). This result is in agreement with that found by Reardon et al. where no consistent trend could be discerned for the comparison of P/S values for unfired powder and residues (5).

#### Single Particle Residue Analysis

Although evaluation of the bulk characteristics of residues collected from changing ammunition composition was a good approach for learning about the average behavior of fired gunpowder, it is not closely related to the type of information that might be ob-

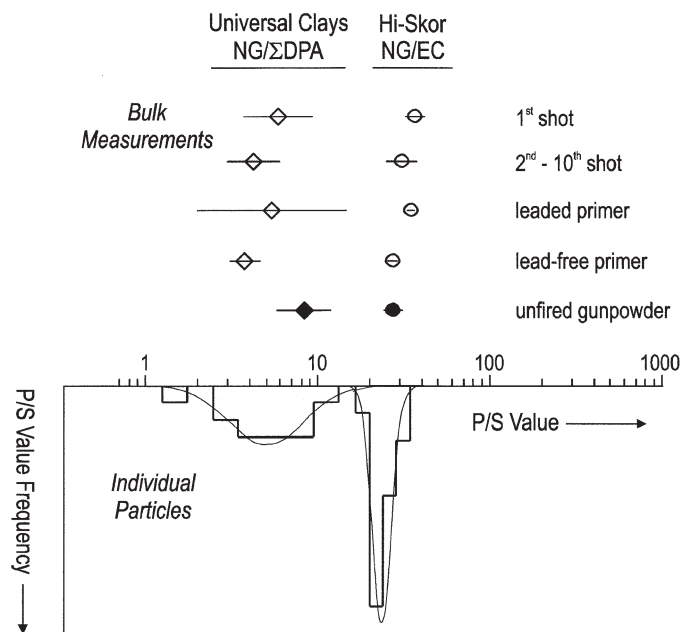


FIG. 1—Comparison of the P/S values of unfired gunpowder to bulk and single particle handgun residues. Left most data: residues collected after changing from Hi-Skor to Universal Clays loaded ammunition. Rightmost data: residues collected after changing from Universal Clays to Hi-Skor ammunition. Upper points: average value (symbols) and 95% confidence intervals (lines) for the bulk residue measurements obtained in the ammunition change experiment (white symbols), primer change experiment (gray symbols) and unfired gunpowder (black symbols). Lower traces: area normalized distributions of the 15 individual particles analyzed as part of the ammunition change experiment (boxes). Smooth curves are Gaussian distributions with the mean and standard deviation of the experimental data.

TABLE 3—Comparison of .38 caliber residues primed with lead-free and conventional leaded primers. Concentrations represent average values and one standard deviation of the mean from five independent samples.

Powder	Component	Lead-free Primer	Leaded Primer	
			This Study	Ref 5
Hi-Skor	NG	261 ± 8	259 ± 7	280 ± 12
	EC	9.6 ± 0.4	7.4 ± 0.2	8.3 ± 0.4
Universal Clays	NG	41 ± 3	39 ± 2	40 ± 2
	DPA (NnDPA)	7.9 ± 0.3 (2.8 ± 0.1)	6.4 ± 0.9 (1.9 ± 0.1)	7.2 ± 0.2 (2.6 ± 0.1)

tained in forensic case investigations. Tape lifts taken from handgun shooters collect only a very limited number of particles for evaluation (4). In order to simulate more closely the type of OGSR information that might be obtained in forensic casework, we evaluated the composition and P/S ratio of individual residue particles. Three particles were removed from each of the collection boxes representing the sequence of individual shots after changing the ammunition composition, pictured graphically in the lower section of Fig. 1. We present the results from all of the residue particle measurements that followed the ammunition change and from the test firing of the conditioned weapon (leaded primer), amounting to evaluation of 15 particles for each ammunition. Data from these experiments were combined for two reasons: 1) The bulk measurements on the sequential firings gave no indication that residues from the first shot were statistically different from subsequent shots or from those obtained from a conditioned weapon, and 2) in a casework investigation, the forensic scientist would not have the luxury of accounting for the previous use of a different ammunition in the weapon.

For Universal Clays (lower left curve), the equal-area plot of the frequency of residue particles having a P/S value shows a broad distribution of values, reflecting the compositional heterogeneity found for this canister of unfired powder (5). The smooth Gaussian curve representing these data is well centered over the average bulk P/S values and shows a confidence interval similar to the conditioned weapon residues (leaded primer data). However, it should be noted that only data for 13 of the 15 particles fit onto the plot for Universal Clays. Two particles had no NG detected, only the DPA stabilizer was detected. If only these two residue particles were recovered and analyzed, the ammunition powder might be incorrectly classified as single-base.

For Hi-Skor loaded ammunition (lower right curve), the range of P/S values for the 15 residue particles was much narrower, which is consistent with previous results of single particle analysis of the unfired gunpowder from this canister (5). The Gaussian fit to the frequency histogram is centered at a P/S value that is somewhat lower than for the bulk residue measurements with a wider confidence interval. No particles containing the DPA stabilizer were found from this EC-stabilized ammunition.

Comparing both the bulk and single particle residue measurements to those made on the unfired gunpowders, residues from Universal Clays ammunition had consistently lower P/S values, whereas for Hi-Skor ammunition, the residues had a consistently higher value. This is the same trend for residue and powder P/S values as was found in a previous study (5) for bulk residues with Universal Clays ammunition, but is the opposite trend for Hi-Skor.

#### Effect of Lead-Free Primer on Residue Composition

CE analysis of the bulk residues produced by ammunition using CCI lead-free primer revealed that the concentrations of the de-

tected additives were consistent with those recovered from the conventional leaded ammunition. The measured values of propellant and stabilizers for the two primers from this and a previous study of these same canisters of powder are shown in Table 3. The measured values of residue NG agreed within the uncertainty of the measurements for both powders tested. However, the measured stabilizer values appear to be somewhat higher in the residues from the lead-free primers.

When evaluated as the P/S value (Fig. 1, center, gray symbols), comparing the residues obtained with conventional leaded and lead-free primer, the lead-free had slightly lower P/S values with overlapping confidence intervals. Since only two powders were evaluated, it is not clear if this is a trend. Ammunition loaded with Hi-Skor gave very similar amounts of collected residue,  $\approx 1.0$  mg/shot for leaded and lead-free primers. However, for the Universal Clays loaded ammunition, combustion was much less efficient with the lead-free primers, providing  $\approx 12$  mg/shot versus  $\approx 7.6$  mg/shot for leaded primers. A number of misfirings were also observed for the 92 shots of lead-free primer Universal Clays ammunition.

The CE analyses of the lead-free primer ammunition did not reveal the presence of any additives that were not previously detected in residue obtained with leaded primers.

#### Conclusions

Sequential firing of two compositionally different double-base gunpowders from the same weapon revealed only the slightest trace of residual OGSR from previous firings, limited to the first shot after the ammunition change. Even for this first shot, the compositional change resulting from previous-shot residues was not sufficient to result in qualitative misidentification of the primary stabilizer, nor was it sufficient to perturb significantly the compositional pattern of the most recent shot. Analyses of residues collected after firing two to ten rounds of new ammunition did not show any traces of the organic components characteristic of the initial gunpowder. This is in contrast to the significant residue contamination from previous firings noted for metallic residues when the primer composition is changed. This may be explained by the differences in the mechanisms of formation of the organic and inorganic GSR. The metallic residue spherules are formed by condensation of volatilized metal from both the primer components and ablated residues from the weapon barrel (6–9,12) and constitute a small fraction of the total residue volume. In contrast, the OGSR are largely incompletely combusted powder, retaining much of the characteristic shape of the original grains (14) and form the bulk of the total residue volume. Evidently, the contribution to OGSR from the organic material released from the weapon barrel is small when compared to the unburned powder.

Evaluation of the composition of a limited number of individual OGSR particles, as might be encountered in trace evidence cases, gave reasonably good agreement with measurements of the unfired gunpowder and of bulk measurements of residues. Determining the propellant/stabilizer ratio, which does not require the OGSR particles to be weighed, appears to be a useful factor to characterize powders and residues.

Testing the new lead-free primers did not reveal any new CE-detectable organic additives nor did their use cause a dramatic change in the composition of the residues when compared to conventional leaded primers.

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