

Associating Gunpowder and Residues from Commercial Ammunition Using Compositional Analysis

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ABSTRACT: Qualitatively identifying and quantitatively determining the additives in smokeless gunpowder to calculate a numerical propellant to stabilizer (P/S) ratio is a new approach to associate handgun-fired organic gunshot residues (OGSR) with unfired powder. In past work, the P/S values of handgun OGSR and cartridges loaded with known gunpowders were evaluated. In this study, gunpowder and residue samples were obtained from seven boxes of commercial 38 caliber ammunition with the goals of associating cartridges within a box and matching residues to unfired powders, based on the P/S value and the qualitative identity of the additives. Gunpowder samples from four of the seven boxes of ammunition could be easily differentiated. When visual comparisons of the cartridge powders were considered in addition to composition, powder samples from all seven boxes of ammunition could be reliably differentiated. Handgun OGSR was also collected and evaluated in bulk as well as for individual particles. In some cases, residues could be reliably differentiated based on P/S and additive identity. It was instructive to evaluate the composition of individual unfired gunpowder and OGSR particles. We determined that both the numerical centroid and dispersity of the P/S measurements provide information for associations and exclusions. Associating measurements from residue particles with those of residue samples collected from a test firing of the same weapon and ammunition appears to be a useful approach to account for any changes in composition that occur during the firing process.

KEYWORDS: forensic science, compositional analysis, diphenylamine, ethyl centralite, gunpowder, gunshot residue, handgun, nitroglycerin, propellant, stabilizer, smokeless powder

Introduction

Determining the identity and concentration of the organic additives in smokeless gunpowder and handgun residues is a promising approach to source identification. In addition to the primary propellant, nitrocellulose (NC), smokeless gunpowder contains additives that include other propellants, usually nitroglycerin (NG), and stabilizers, commonly either diphenylamine (DPA) or ethyl centralite (EC). Handgun residues have been associated with unfired double-base gunpowders by measuring these additives using ultrasonic solvent extraction/capillary electrophoresis and then calculating the propellant nitroglycerin (P) to stabilizer (S) ratio (1,2) as

a numerical identifier. By firing ammunition with known gunpowder composition, we determined that the organic gunshot residues (OGSR) retain much of the compositional characteristics of the unfired powders, providing the possibility of associating residues with a gunpowder using the P/S value. In this paper, we further evaluate composition and P/S value to associate ammunition cartridges and handgun residues.

One issue that directly impacts the certainty of residue/ammunition associations is the variability in the gunpowder composition found within a box of cartridges. Since gunpowders are often blended to achieve ballistic goals (3,4), the compositional heterogeneity within and between cartridges must be considered. A few studies have considered this issue by examining qualitative (5) and quantitative compositional variations (6–10) of cartridges within and between manufacturing lots and for individual particles of reloading powder (1,7). Systematic evaluation must be made of the compositional variation of powder and handgun-fired residue particles from commercial ammunition based on organic compositional analysis.

To associate handgun OGSR reliably to powder in unfired cartridges, the changes in composition during the firing process must also be considered (1,7,10). As a general rule, the propellant and stabilizer additives in the unfired powder are retained in the handgun residues in similar concentrations. Andrasko (7) evaluated the similarity of residues and powders by determining a complex numerical correlation factor, developed by Keto (11), of major additive chromatographic peaks. Although the correlation factor allowed ammunition from different manufacturers to be distinguished reliably, its complexity and interpretation would be difficult for a lay audience to understand. The P/S ratio developed by Reardon et al. (1) provides a much simpler numerical identifier that focuses on the evaluation of the primary additives in double-base gunpowder in a manner that is simple to determine and interpret. Neither numerical identifier has been evaluated for association of unknown residues and source ammunition.

It may also be possible to associate residues with ammunition more accurately by comparing unknown residues to known residues recovered from a test firing of the source ammunition. Other studies (1,2,7) have not determined whether such an approach might provide more associative information than by examining the characteristics of the unfired powder alone.

This paper evaluates the powder and residues from commercial ammunition with cartridges that were given a coded identity. Residues were collected in duplicate using a .357 magnum revolver and coded. Cartridge gunpowder and residues were analyzed, and the additive P/S ratio was determined. Associations of the coded powder and residue samples were then made both in

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bulk and as individual particles using the P/S value, and in the case of unfired powders, with an additional evaluation of particle morphology.

Experimental Materials and Methods*

Ammunition and Gunpowder

Seven brands with 50 rounds each of .38 Special ammunition were obtained from a local retailer. The cartridges in each Box were assigned numbers of 1 through 50. Cartridges 1, 10, 20, 30, 40, and 50 from each of the seven Boxes were given random codes. Each bullet was pulled, and the powder was transferred to an amber vial labeled only with the code. The remaining cartridges from each Box were divided into two groups of 22 by removing alternate cartridges from the box and coded.

Collection of Organic Gunshot Residues

A photograph of the OGSR collection boxes can be seen elsewhere (1). OGSR collection was conducted at the National Institute of Standards and Technology firing range, Gaithersburg, MD. A .357 Magnum Smith and Wesson Model 19-4 was used to prepare the OGSR. The weapon was cleaned initially and between each set of cartridges with Outers Nitro Solvent (Blount, Inc., Onalaska, WI) and Generation 2000 Contact Clean (Rite Off, Seaford, DE). Each group of 22 cartridges were pulled from each ammunition cartridge box and fired into separate coded collection boxes. A detailed description of the OGSR collection protocol can be found in previous publications (1,2).

OGSR was removed from each box at the laboratory and examined under a microscope for any extraneous debris. OGSR was then weighed in labeled amber glass vials.

Ultrasonic Solvent Extraction/Capillary Electrophoresis

A Branson ultrasonic bath (Model B220, 125 watts, Shelton, CT) filled with water was used for the ultrasonic solvent extractions (USE). Unfired gunpowder (10 mg), OGSR (2 mg), and individual OGSR particles (≈ 0.10 mg) were extracted with 1.0 mL, 200 μ L, and 60 μ L, respectively, of 2-butanol:methanol (1:3) for 15 min. Analyses of the extracts were performed on a capillary electrophoresis (CE) system (Beckman, P/ACE 5510, Brea, CA). Specific information on the USE and CE procedures can be found elsewhere (12).

One 10 mg sample and one individual particle of unfired gunpowder were analyzed from each cartridge. To assess the source of compositional variability in residues collected, three particles from each unfired cartridge were evaluated for Ammunition box 3. From every residue Box, measurements were made on a single 2 mg OGSR sample. Three OGSR particles for analysis were collected visually from each of the 14 collection boxes using tweezers.

Additive concentrations were calculated using the internal standard method and a recovery factor specific to the analyte (12). Average concentrations in mg/g and mean standard deviations were also determined.

* 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.

Results and Discussion

The overall design of this study was to evaluate the ability of the P/S value to differentiate unfired cartridge powders, to associate residues with the source powders, and to pair residues with residues.

Associating Unfired Cartridges

Bulk samples (10 mg) of the coded powder samples from the seven boxes of ammunition were analyzed by USE/CE and the P/S ratios were determined. For grouping, seven groups of six cartridges were assumed by the investigator. Care was taken to exclude any morphological observations in the initial group assignments. The results of these compositional measurements are presented in Table 1 in order of increasing P/S value.

Of the 42 cartridge powders from the seven boxes of ammunition, six were found to contain only DPA and N-nitrosodiphenylamine (NnDPA - a decomposition product of DPA), with no detectable NG present. These single-base powder samples were not assigned a P/S ratio. The results for the total stabilizer content ($[DPA] + [NnDPA]$) were consistent for this box with values of 14.2 mg/g, 13.3 mg/g, 13.9 mg/g, 11.7 mg/g, 14.2 mg/g, and 14.3 mg/g. Thus, the six cartridge powder measurements assigned to Ammunition box 1 are clearly distinguished from the other groups based on their unique single-base composition and were correctly grouped when compared to the known box code.

There were twelve cartridges that contained NG, DPA, and NnDPA. Since the total stabilizer content includes DPA and its nitrated derivatives, the P/S ratio for these powders was calculated as $[NG] / \sum ([DPA] + [NnDPA])$. It is reasonable to divide these measurements into two groups given that the P/S values of the groups differ by more than two standard deviations (approximately $\pm 1.2\%$ for a NG/DPA powder (1)). Six of the cartridge powders had values of approximately 15 (Group 2), and the other six were calculated to be approximately 19 (Group 3). Thus, P/S differences for these two groups of cartridges should be statistically significant and were found to be correct when compared to the code.

The remaining 24 cartridges contained powder with NG and EC. Six of the 24 were easily associated with a P/S value of approximately 66 (Group 7), but the other 18 had P/S ratios ranging from 33 to 39 (designated as Groups 4–6). Previous work estimated the uncertainty of P/S measurements for an NG/EC powder to be approximately $\pm 5.3\%$ (1). Thus, these 18 measurements fell into a single statistically indistinguishable group. Given the expected measurement uncertainty in the P/S value (roughly ± 1.5 for this P/S value) and the similarity in P/S values of these 18 powders, differentiating these powders into three clearly defined groups of six would be impossible based on the P/S value alone. However, P/S values that are characteristic of Box 4 should be statistically different from Box 6, which was born out when the code was revealed. The four incorrect box assignments, made solely by considering the P/S numerical order for groups of six cartridges, are highlighted in bold in Table 1.

A brief morphological examination of the unfired gunpowder from each of the cartridges was performed, which included a visual evaluation of shape, particle dimension, and color.

Gunpowders from Boxes 1 through 3 were classified as flattened ball and Boxes 4 through 7 as disk (13). A representation of the dimensional measurements of the cartridge powders is presented in Fig. 1. Each shaded bar is the mean value and the error bars represent ± 1 standard deviation of the mean ($n = 10$). Both the average value and the variability of these values provides information for classification (13).

Of the three groups closely associated by P/S value (Groups 4 through 6), powders belonging to Group 5 had a somewhat larger thickness and a much larger particle diameter than Groups 4 and 6. In addition, gunpowders from Group 4 were a darker shade of gray than Group 6. Neither P/S composition or these simple morphological evaluations alone could make clear discriminations between these powder samples. However, using the combination P/S

values and morphology, all cartridges could be appropriately grouped as determined by comparison to the code (Table 1).

We also considered the variation among a limited number of individual particles of the unfired cartridge powders. In addition to one bulk measurement, an individual particle was taken from each cartridge and analyzed. A graphical representation, based on Youden (14), of these bulk and individual particle measurements is shown in Fig. 2. The line shown is $y = x$. Individual particle measurements are noted by the symbols. All of the measurements clearly group according to the cartridge box number. Each ellipse (15) represents the 95% confidence interval of these measurements. A rightward tilt of the ellipses indicates results where the bulk and individual particle measurements are positively correlated within that cartridge, i.e., it is likely that both a high individual particle and high bulk measurement would be found for a given cartridge powder sample. The height of the envelope gives a measure of the heterogeneity of the powder measurements within each cartridge. For Ammunition Boxes 2 through 5 and 7, individual cartridge results are well correlated between the bulk and individual particle measurement. As noted in Fig. 2, the individual particle measurements for Box 6 are highly variable, suggesting particle-to-particle heterogeneity. Thus, measurements on an individual or limited number of particles would not be likely to represent accurately the mean P/S value for ammunition Box 6.

Associating Unfired Cartridges and OGSR Particles

Given the "basis set" of measurements on the cartridge powders, we then determined if associations between handgun-fired residues and unfired gunpowders were possible. Two sets of samples were collected into randomly coded collection boxes by firing 22 rounds of 38 caliber ammunition from a revolver and collecting the muzzle-exit residues. A bulk (2 mg) and three individual particle samples were taken from each collection box for compositional evaluation.

As noted previously, Ammunition Box 1 was single-base with DPA + NnDPA values ranging from 11.7 mg/g to 14.3 mg/g for the six evaluated cartridges. Bulk handgun residue measurements found 10.9 mg/g and 11.1 mg/g for the total stabilizer content. In

TABLE 1—Unfired gunpowder from the cartridges grouped using the P/S ratio. Box 1 contained single-base gunpowder; thus no P/S value was assigned.

	P/S (DPA)	Ammunition Box		P/S (EC)	Ammunition Box
Group 1	DPA only	Box 1	Group 4	33.1	Box 4
	DPA only	Box 1		33.1	Box 4
	DPA only	Box 1		33.3	Box 4
	DPA only	Box 1		33.5	Box 4
	DPA only	Box 1		33.5	Box 5
Group 2	DPA only	Box 1	Group 5	33.8	Box 4
	14.8	Box 2		34.0	Box 4
	15.0	Box 2		34.3	Box 5
	15.1	Box 2		35.2	Box 5
	15.3	Box 2		35.4	Box 5
	15.3	Box 2		36.3	Box 6
Group 3	15.5	Box 2	Group 6	36.6	Box 5
	19.1	Box 3		37.8	Box 5
	19.3	Box 3		37.8	Box 6
	19.4	Box 3		38.2	Box 6
	19.4	Box 3		39.0	Box 6
	19.5	Box 3		39.2	Box 6
	19.9	Box 3		39.4	Box 6
		Group 7	63.5	Box 7	
			65.5	Box 7	
			66.6	Box 7	
			66.7	Box 7	
			66.8	Box 7	
			66.9	Box 7	

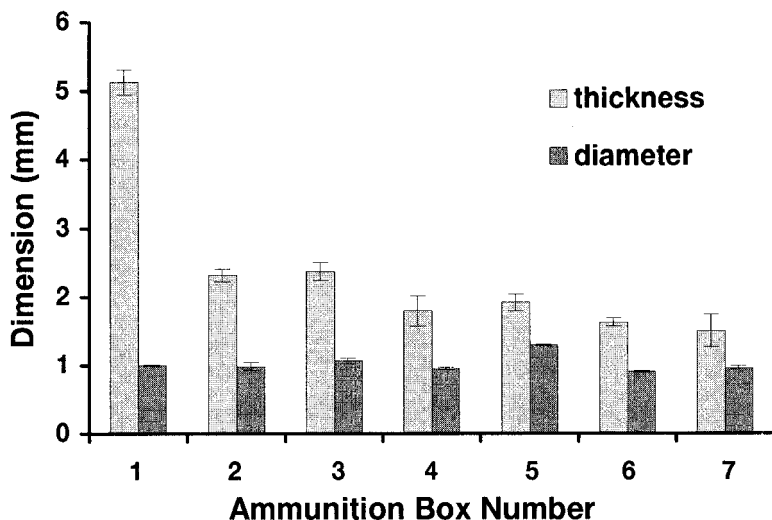


FIG. 1—Evaluation of dimensional morphology of unfired cartridge powders. Error bars represent ± 1 standard deviation of the mean for 10 measured particles.

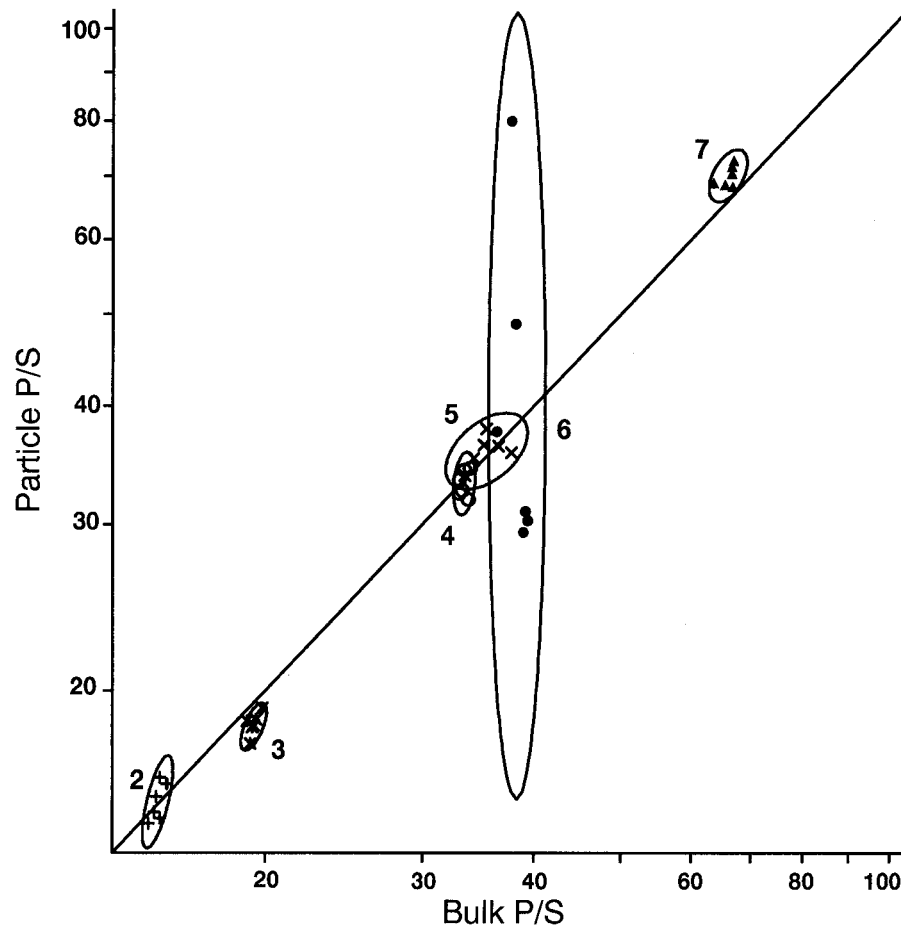


FIG. 2—Graphical comparison of unfired cartridge gunpowder individual particle versus bulk cartridge P/S compositional measurements. Line determined by the mean values of each set, points represent individual particle measurements.

addition, one bulk measurement found low levels of NG, 22.7 mg/g. Measurements on three individual residue particles from the two boxes found 15.6 mg/g, 15.5 mg/g, 16.9 mg/g, 14.0 mg/g, and 14.0 mg/g total DPA. Unfortunately, all of the sets of individual particle and bulk unfired gunpowder and individual particle and bulk residue measurements could not be made on the same day, so calibration errors may contribute to the differences observed in these measurements. In addition, one individual particle measurement for this ammunition found NG (235 mg/g) with EC (4.3 mg/g) stabilizer.

Other unexpected results were also obtained for individual residue particle measurements from Ammunition Boxes 3 and 6, see Table 2. Two of the samples of residues from Box 3 had only NG detected and two had no additives detected. One residue sample from Box 6 had DPA stabilizer, which was not consistent with all of the other powder/residues measurements for this cartridge Box.

Two factors are likely to have contributed to the observed anomalous measurements. Heterogeneity of powder composition within cartridge boxes is possible since commercial ammunition was used. Ammunition powder is manufactured and blended to achieve ballistic targets, not uniform additive composition (4). Although no inconsistent results were found in any of the unfired cartridge measurements, only six cartridges out of 50 were sampled. Secondly, although we used a cleaned weapon and residue collec-

tion box for each set of cartridges, the possibility of contamination by background firing range residues and plastic debris from our collection protocol at the test firing facility must be considered a possible limitation in these individual particle evaluations. Microscopic examination of the residues should have been used to assure collection of particles that only showed characteristic features of OGSR.

The result of all of these experiments for the six double-base powders is shown graphically in Fig. 3. The unfired powder measurements are represented by the smooth curves, where the apex represents the mean determined from the bulk and individual particle measurements, the height relates the frequency of occurrence, and the width represents the 95% confidence interval as determined by considering the combined uncertainties of bulk as well as the individual particle measurements. The position of the bell curve on the P/S axis provides a visual means of determining if the compositional measurements of powders can be distinguished. In addition, the width of the measurement curves is also a characteristic feature that must be considered when evaluating limited numbers of individual particles. This "peak" shaped representation of the measurements makes the strengths and limits of the P/S data to differentiate powders readily apparent. The two DPA-stabilized double-base powders (Boxes 2 and 3) are easily categorized qualitatively from the EC-stabilized powders (Boxes 4 through 7). With six bulk and six individual particle measurements from each box,

TABLE 2—OGSR samples paired according to code. P/S ratios determined on one bulk (2 mg) sample and three individual OGSR particles. Collection Boxes 1a and 1b contained single-base OGSR; therefore P/S ratios were not calculated, unless otherwise noted.

P/S Based on Individual Measurements					
Ammunition Box	Bulk	Particle 1	Particle 2	Particle 3	Additive
Box 1					
a	2.0 ^a	N/A	N/A	N/A	DPA
b	N/A	N/A	N/A	54.4 ^b	
Box 2					
a	17.2	15.8	17.5	19.2	DPA
b	16.4	19.5	17.5	18.4	
Box 3					
a	20.4	c	c	d	DPA
b	23.8	19.2	d	28.1	
Box 4					
a	35.6	29.1	31.4	34.8	EC
b	34.7	31.2	31.4	33.3	
Box 5					
a	35.8	33.7	35.4	35.6	EC
b	35.1	41.9	30.6	36.6	
Box 6					
a	40.7	33.8	e	39.1	EC
b	40.8	44.2	42.9	58.9	
Box 7					
a	67.5	66.9	76.3	69.0	EC
b	68.7	70.4	92.2	65.0	

^a Trace NG detected.

^b NG and EC detected, P/S value for NG/EC.

^c Only NG was detected.

^d Neither NG nor stabilizers were detected.

^e OGSR grain contained NG, DPA, and NnDPA, no EC.

cartridge powders from ammunition Boxes 2 and 3 can be differentiated from each other with some certainty based on P/S measurements. Ammunition from Box 7 could also be distinguished from the other EC powders. However, cartridges from Boxes 4, 5, and 6 would not be distinguishable based on this limited number of measurements. In particular, because of the high variability noted for cartridge Box 6, making a clear association for a limited number of exemplar cartridges or powder particles would be impossible based solely on P/S determination.

The graphical representation of the individual particle residue measurements in Fig. 3 are shown as numbered Boxes. Three particles were analyzed from each of the two collection Boxes. Box 3 has only two measured particles, as four of the other individual residue particles were not found to contain either propellant nitroglycerin or stabilizer or both.

In general, the individual residue particles exhibit a higher variability in P/S value than the unfired powders. This may be a result of the combustion process. The P/S values of the two DPA double-base powders, Boxes 2 and 3, are shifted to higher values than the unfired powder. Whereas unfired powder from Boxes 2 and 3 could be reliably distinguished, the residue particle P/S values overlap somewhat.

For the EC-stabilized powders, the residue particle P/S values are generally in good agreement with the unfired values, albeit with a higher variability. It would not be possible to distinguish reliably residues from ammunition Boxes 4, 5, and 6 from each other. However, residues from Box 7 could be distinguished from residues collected from the three other boxes of EC-stabilized powders.

Overall, among the 42 individual residue particle evaluations, six proved indeterminate with one or both of the expected additives not measurable above the detection limit of the method. This may have been the result of the poor choice of particles for evaluation via unaided visual selection. One particle of the 42 evaluated was found to contain a completely different stabilizer additive than was found in all other unfired and bulk residue measurements for that powder.

Associating Residues with Residues

The next step in this study was to determine if OGSR could be more reliably associated with other samples of OGSR fired from the same box of ammunition. Individual bulk measurements from each of the two collection boxes are represented by numbered circles in Fig. 3.

For five of the six ammunition boxes, the P/S measurements from the two bulk residue samples are generally much closer to the mean of the individual particle residue measurements than to the mean unfired powder values. It might be anticipated that individual particle and bulk residue P/S measurements might be in closer agreement than with unfired powder measurements. Only for Ammunition Box 4 do the bulk residue P/S values provide a poorer correlation of residue particle P/S mean value than do the unfired powder measurements.

In general, the individual residue particles exhibit a greater range of P/S values than is associated with the unfired powder and bulk OGSR measurements. In addition, as might be anticipated, powders that exhibit a greater variability in the unfired cartridge measurements also showed a greater range of P/S values in the residue particle measurements.

Conclusions

The P/S ratio has proven to be a useful trait for relating gunpowder and OGSR. Measuring the P/S ratio is an attractive technique for the practicing forensic scientist, since it is a quantitative measurement that can be calculated on gunpowder or OGSR samples that may be too small to weigh accurately. This study revealed some of the strengths and limitations of compositional evaluation of OGSR as a means of association with unfired gunpowder samples. For these seven ammunition samples, the identity of the gunpowder additives immediately provided a gross categorization of the powders/residues into three general categories: single-base, double-base, and stabilizer type. Evaluation of the P/S value of the double-base cartridges adds an additional level of discrimination. Cartridge powder from four of the seven boxes could be reliably distinguished. However, three of the ammunition boxes had enough compositional similarity that they could not be reliably distinguished based on the limited number of measurements used for this study.

Our simple evaluation of the morphology was not sufficient to provide complete discrimination between powders. However, when combined with the P/S determinations, all of the cartridge samples could be correctly associated into seven boxes of ammunition. In addition, for this very limited evaluation of six cartridges from each of seven boxes of ammunition, the identity and concentration of the additives were consistent within each box of cartridges. Further evaluation of a much larger sample of ammunition would be warranted before this could be considered as a general observation.

Careful consideration was needed in interpreting information available from the compositional evaluation of handgun-fired

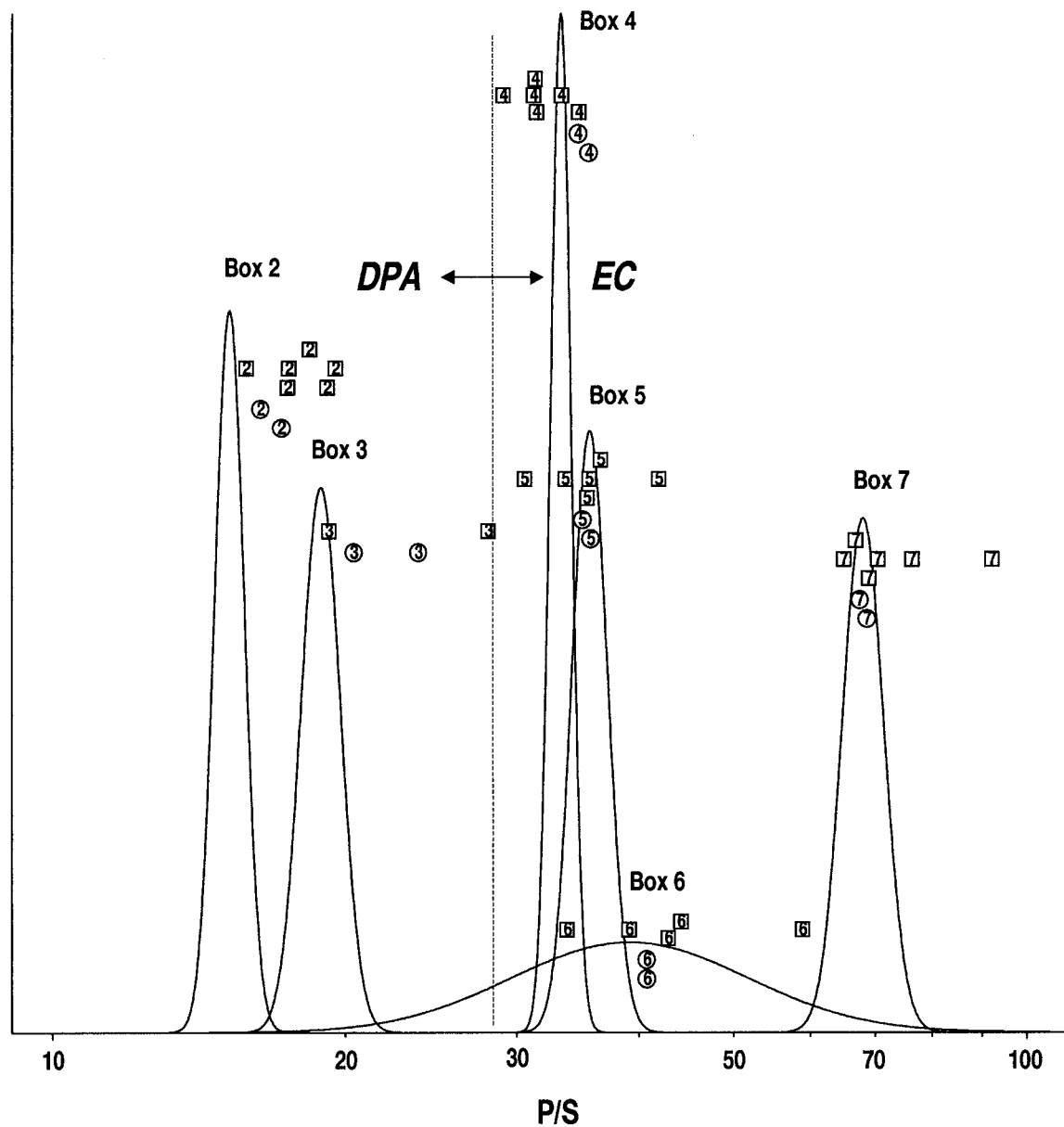


FIG. 3—Graphical evaluation of unfired cartridge powder, individual particle, and bulk residue P/S compositional measurements. Smooth curves represent the 95% confidence interval of the bulk and individual particle unfired cartridge powder measurements. Circled points represent the bulk residue measurements and the numbered squares are the individual residue particle measurements.

residues. Graphical comparison of individual residue particle measurements to a basis measurement set that consisted of unfired cartridge powder and bulk handgun-fired residue samples proved useful in making associations and exclusions. Variability in the individual particle measurements must be considered when a limited number of questioned samples or exemplars is available for evaluation.

After reviewing all of the test results, three aspects of OGSR and cartridge powder evaluation could have been done more effectively: (1) same day compositional measurement of the questioned residue samples and powder/residue basis set should be used to minimize calibration errors in the determination of P/S values; (2) the basis set of powder measurements should have a higher proportion of individual particles than bulk measurements to better understand the expected range of measurements for the cartridge

powders; and (3) protocols for the careful microscopic collection of viable OGSR particles should be developed.

Acknowledgments

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References

1. Reardon MR, MacCrehan WA, Rowe WF. Comparing the additive composition of smokeless gunpowder and handgun-fired residues. *J Forensic Sci* 2000;54(6):1196–1202.
2. MacCrehan WA, Patierno ER, Duewer DL, Reardon MR. Investigating the effect of changing ammunition on the composition of organic additives in gunshot residue (OGSR). *J Forensic Sci* 2001;46(1):54–9.

3. Wallace CL, Midkiff CR. Smokeless powder characterization: an investigative tool in pipe bombings. In: Yinon J editor. *Advances in the analysis and detection of explosives*. The Netherlands: Kluwer Academic Publishers, 1993;29–39.
4. Black and smokeless powders: technologies for finding bombs and the bomb makers. Washington, DC: National Research Council, 1998.
5. Mach MH, Pallos A, Jones PF. Feasibility of gunshot residue detection via its organic constituents. Part I: analysis of smokeless powders by combined gas chromatography-chemical ionization mass spectrometry. *J Forensic Sci* 1978;23(3):433–45.
6. Northrop DM, MacCrehan WA. Smokeless powder residue analysis by capillary electrophoresis. Washington DC: U.S. Department of Justice, National Institute of Justice, Report No.: 600–91, 1997.
7. Andrasko J. Characterization of smokeless powder flakes from fired cartridge cases and from discharge patterns on clothing. *J Forensic Sci* 1992;37(4):1030–47.
8. Northrop DM gunshot residue analysis by micellar electrokinetic capillary electrophoresis: validation and application to casework—Part 1. *J Forensic Sci*. 2001;46(3):549–59.
9. Meng H-h, Caddy B. Detection of N, N'-diphenyl-N, N'-diethylurea (ethyl centralite) in gunshot residues using high-performance liquid chromatography with fluorescence detection. *Analyst* 1995;120:1759–62.
10. Northrop DM. Gunshot residue analysis by micellar electrokinetic capillary electrophoresis: validation and application to casework—Part 2. *J Forensic Sci*. 2001;46(3):560–72.
11. Keto RO. Comparison of smokeless powder by pyrolysis capillary gas chromatography and pattern recognition. *J Forensic Sci* 1989;34(1): 74–82.
12. Reardon MR, MacCrehan WA. Developing a quantitative extraction technique for determining the organic additives in smokeless handgun powder; *J Forensic Sci*. 2001;46(4):802–7.
13. Selavka CM, Strobel RA, Tontarski RE. The systematic identification of smokeless powders: an update. *Proceedings of the Symposium on Analysis and Detection of Explosives*; 1989 Jul 10–13; Mannheim-Neustadt, Germany. Fraunhofer-Institut für Chemische Technologie, 3-1-3-27, 1989.
14. Youden, WJ. Graphical diagnosis of interlaboratory test results; *Industrial quality control*, 1959;15(11):1–5.
15. Hall IJ, Sheldon DD. Improved bivariate normal tolerance regions with some applications. *J Quality Tech* 1979;11:13–19.

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