# Comparing the Additive Composition of Smokeless Gunpowder and Its Handgun-fired Residues

**REFERENCE:** Reardon MR, MacCrehan WA, Rowe WF. Comparing the additive composition of smokeless gunpowder and its handgun-fired residues. J Forensic Sci 2000;45(6):1232–1238.

ABSTRACT: Detecting the use of handguns via the determination of the organic additives in smokeless gunpowder residues (OGSR) presents a promising alternative to primer metal residue analysis. Compositional analysis of the gunpowder additives nitroglycerin, diphenylamine, and ethyl centralite provides information that can associate residue samples with unfired gunpowder. We evaluated the composition of seven reloading smokeless gunpowders, both in bulk and as single particles, by ultrasonic solvent extraction/capillary electrophoresis. Handgun-fired residues obtained from three common weapon calibers loaded with the known reloading powders were compared with the unfired powders. In general, the composition of the residues was similar to that found in the unfired powders. For double-base powders, comparing the ratio of the propellant (P) to the total amount of stabilizer (S) for both residue and gunpowder samples proved to be a useful measurement for identification. This P/S ratio demonstrated that the additives in the residues did not greatly change relative to the unfired powder, providing a useful indicator to aid in forensic powder and residue evaluation.

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

Smokeless gunpowder residues contain useful information that can aid forensic investigators in connecting a suspect to the discharge of a firearm. With the advent of new primers containing less toxic (and more common) metals or nonmetallic compositions (1,2), the traditional methods of analysis involving measurements of the metals lead, barium, and antimony may become less useful in determining handgun use. The chemical composition of propellants has changed little over the years and can be an excellent indicator of the presence of organic gunshot residue (OGSR) (3–6). The primary detectable organic additives most commonly seen in double-base propellants are nitroglycerin (NG), diphenylamine (DPA), and N,N'-diethyl-N,N'-diphenyl urea (ethyl centralite, EC). Singlebase powders do not have the energetic additive, NG, and will usually only contain either DPA or EC. A number of additional addi-

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Received 28 Sept. 1999; and in revised form 30 Nov. 1999; accepted 6 Dec. 1999.

tives occur in some double- and single-base propellants including methyl centralite, dialkyl phthalates, triacetin, and dinitrotoluene (DNT) isomers. These may also be used for identification.

The possibility of "tagging" smokeless and black gunpowder to aid in forensic identifications was recently explored by the National Research Council Committee on Smokeless and Black Powder (7). The technical, logistical, and economical feasibilities of gunpowder taggants were evaluated and included such technologies as isotopic labeling, dyeing specific particles, and incorporating a chemical code into the propellant, as either a separate particle or as part of the gunpowder itself. None of the tagging techniques were deemed practical at the current time. One of the recommendations of the committee was to make better use of chemical compositional analysis for identifications, rather than to introduce taggants.

It may be possible to associate an evidentiary sample of recovered handgun residues with the unfired gunpowder, based on organic gunpowder compositional analysis (3-5,8,9). Previous studies were all based on a very limited number of test firings, and none have been made with direct control over the powder composition of the ammunition. Because the relationship of the composition of OGSR and unfired gunpowder has not been reliably established, forensic investigators prefer to analyze intact or minimally burnt smokeless powder grains that were found on an individual or at a crime scene (7,9). A single particle may be enough for a confirmation on the presence of OGSR, but partially consumed grains might also provide useful compositional information that could be linked to the unfired ammunition. In the investigation presented here, powders of known composition were loaded into ammunition for three common handgun calibers. Repetitive test firings were performed to prepare weighable residues for quantitative compositional comparison of fired and unfired gunpowder. In addition, single-particle analysis of the unfired powder was used to explore the variability of composition that might be expected when a single particle is used for evaluation.

# **Materials and Methods**

# Reagents and Standards

HPLC-grade methanol (J. T. Baker, Phillipsburg, NJ) was used in the extraction procedure for the gunpowder additives. The buffer solution for the capillary electrophoresis (CE) contained 25 mmol/L sodium dodecyl sulfate (SDS) (sequanal grade, Pierce, Rockford, IL) and 10 mmol/L sodium borate buffer, pH 9.1 in HPLC grade water (J. T. Baker). A standard solution of the three gunpowder additives with 2000 mg/L NG, 1000 mg/L DPA, and 1000 mg/L EC (AccuStandard Inc., New Haven, CT) in methanol was diluted with the CE buffer for calibration. N-nitrosodiphenylamine (NnDPA) (Chem Service, West Chester, PA) was also added to the calibration standards. As internal standard for the CE, we used 2-methyl-6-nitroaniline (MNA) from Fluka (Milwaukee, WI) dissolved in methanol.

## Gunpowder Samples

Seven commercially available gunpowders were purchased from United States and European manufacturers (see Table 1). Powder obtained in more than one canister was mixed thoroughly to ensure homogeneity before analysis and transport to the test firing facility. Samples of the powders were retained for analysis, while the remaining portions were shipped to National Technical Systems (NTS) in Camden, AR, for reloading into blank ammunition cartridges and for collection of handgun-fired residues.

#### Gunshot Residue Collection

Twenty-one plastic boxes (41 cm  $\times$  38 cm  $\times$  17 cm) with integral lids (from KMart) were used to collect the residues. A 10 cm

diameter hole was cut in the bottom of each box. An adhesive Teflon square (Bytac, chemical resistant Teflon<sup>®</sup> FEP film, PGC Scientifics), approximately 15 cm  $\times$  15 cm, was cut and placed over the hole from each side of box. The Teflon covered area served as the target for the firings (Fig. 1).

The powders were loaded into a total of 2100 new Remington brass cartridges for three different types of ammunition commonly used in criminal activities (9 mm, .38 Special, and .45 ACP). The cartridges were primed with either Winchester small or large pistol primers. The powder load for the 700 9 mm cartridges was 5.5 grains of gunpowder (where 1 grain was approximately 65 mg of powder); for the 700 .38 Special cartridges the load was 4.5 grains of gunpowder; and the 700 .45 ACP cartridges were loaded with 6.0 grains of gunpowder. The weapons used were a 9 mm Beretta (Model 92FS, S/N BER4064572, barrel length 12.5 cm), Smith and Wesson .357 Magnum (Model 586, S/N AUD6092, barrel length 14.0 cm), and Ruger .45 ACP (Model P90DC, S/N 661-25448, barrel length 11.4 cm). The projectiles were a 115 grain full metal jacket for the 9 mm, a 125 grain hollow point for the .38 Special, and a 230 grain full metal

TABLE 1—Smokeless reloading powders used in this research.

Powder	Powder Manufacturer		Morphology	Target Additives	
Bullseye	Alliant Powder* (Radford, VA)	Alliant	extruded	NG, EC	
Universal Clays	Alliant Powder	Hodgdon	extruded	NG, DPA, NnDPA	
HP-38	Primex Technologies (St. Marks, FL)	Hodgdon	ball	NG, DPA, NnDPA	
231	Primex Technologies	Winchester	ball	NG, DPA, NnDPA	
Hi-Skor 700×	IMR Powder Company† (Washington, PA)	IMR Powder	extruded	NG, EC	
No. 2	Synthesia (Czech Republic)	Accurate Arms	ball	NG, EC	
N320	Vihtavouri Oy (Lapua, Finland)	Kaltrone-Pettibone	extruded	DPA	

\* Formerly Hercules.

‡ Formerly Dupont.

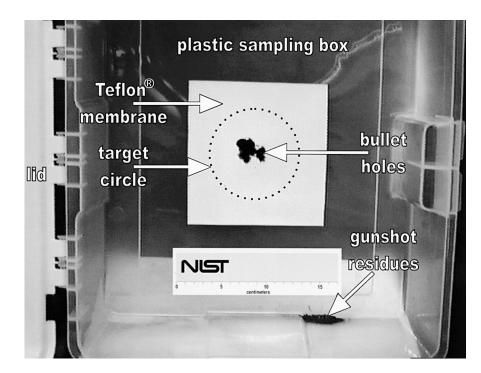


FIG. 1-Sampling box for gunshot residue collection.

jacket for the .45 ACP. Each powder was loaded into 100 9 mm cartridges, 100 .38 Special cartridges, and 100 .45 ACP cartridges. The plastic boxes were designed to hold the muzzle residues obtained by firing 100 rounds of ammunition.

For the test firings, the box lid was removed, and swab-cleaned handguns were fired at a distance of 1.4 m perpendicular to the open face of the box. The tests were conducted free hand, out of doors, and with the muzzle having a 30° downward angle. After 100 rounds of ammunition had been fired into the box, the residues were carefully swept away from the target area, and fresh Teflon squares were placed over the holes, reducing the chance of losing residues during shipment. The lid was replaced, secured to the box with duct tape, and shipped back to the laboratory.

Residues were collected from each box with a spatula and examined under a microscope. Any trace of extraneous debris was removed, and the residues were weighed into amber glass vials to protect the light-sensitive additives. Microscopic examination demonstrated that the quantity of inorganic particles present in the residues was negligible. No effort was made to remove comingled inorganic residue from the sample before weighing or extraction.

#### Ultrasonic Solvent Extraction for Gunpowder/Residue

Extractions of bulk powder, OGSR, and single grains were all performed by ultrasonic solvent extraction (USE), using a Branson ultrasonic cleaner bath (Shelton, CT). The procedure used was adapted from conditions previously developed in our laboratory (4). A 10 mg portion of gunpowder was weighed into a sealed 15 mL centrifuge tube using a balance capable of measuring to 0.01 mg. Methanol (1.0 mL) was added, and the sample was vortex-mixed for 10 s and agitated in the ultrasonic bath for 15 min. Some of the powders completely dissolved during the agitation in methanol, but the ball powders, HP-38, Winchester 231, and Accurate No. 2, only swelled in the solvent. The samples were then vortexed for another 10 s and finally centrifuged for 5 min. A 10 µL portion of the extract was added to 250 µL of the CE buffer and 5 µL of 2000 mg/L MNA (internal standard). This solution was also mixed for 10 s, and then 200 µL of the solution was transferred to a CE sample vial for analysis. Sample preparation and extraction was performed either in the absence of light or under incandescent light to minimize photodecomposition of the additives (10). Complete recovery by this extraction approach was tested by a second extraction of the gunpowder sample with fresh solvent which yielded no additional recovery of the additives (10).

Single gunpowder particles were weighed  $(\pm 1 \mu g)$  into the same type of sealed containers. The above procedure was followed, although 30  $\mu$ L of methanol were used for extracting the additives from each grain, instead of 1.0 mL. OGSR was extracted in a similar manner, except that 2 mg samples and 200  $\mu$ L of methanol were used.

When possible, powder and residue samples were extracted in replicates of ten. For several weapon calibers, insufficient OGSR was present for ten 2 mg samples. In these cases, fewer samples were extracted as noted in the table and multiple portions of the extracts were analyzed to yield a total of ten measurements. For the individual particle determinations, a single extraction and analysis was performed. Concentrations of the additives in the powders, individual particles, and residues were calculated based on the internal standard method, using peak area counts of a gunpowder additive standard, internal standard (MNA), and the powder samples to determine the concentration in mg/L. The mass of each sample and any dilutions were incorporated into the final calculation to produce concentrations in mg/g. Average mg/g concentrations (for 10 measurements), standard deviations of the mean (where n equaled the number of independent samples), and relative standard deviations of the mean (in %) were determined.

## Capillary Electrophoresis

A capillary electrophoresis system (Beckman P/ACE 5510, Brea, CA) with fixed wavelength detection at 214 nm and chromatography data collection software was used. Extended path length (200  $\mu$ m window diameter) capillaries were used (Agilent Technologies, San Fernando, CA) with a 75  $\mu$ m inner diameter and an overall length of 77 cm. The micellar CE run buffer was 25 mmol/L SDS and 10 mmol/L sodium borate at pH 9.1. Sample vials and holders, with a working volume of 200  $\mu$ L, were purchased from the manufacturer. 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. The internal standard for the CE, MNA, was chosen from a variety of compounds investigated and is described elsewhere (6,10).

## **Results and Discussion**

Our study of the characteristic features of gunpowder and handgun-fired residues began with a thorough characterization of the seven commercial reloading powders in Table 1. Compositional measurements were made on "bulk" samples of 10 mg, representing more than 50 individual powder particles, and also for "singleparticle" grains.

#### Bulk Powder Composition

Of the seven powders analyzed, one was a single-base (N320) and the other six were double-base powders. None of the powders contained any of the DNT isomers. All of the powders, including N320, had a single stabilizer, which was either DPA or EC. Two of the powders, HP-38 and Winchester 231, contained DPA and significant amounts of NnDPA, a decomposition product. It is useful to consider the combined DPA and NnDPA concentrations as a measure of the stabilizer composition in such powders.

The two powders produced by Primex, Winchester 231 and HP-38, had similar compositions of NG, DPA, and NnDPA. However, the two powders manufactured by Alliant, Bullseye and Universal Clays, used different stabilizers and amounts of NG. For example, Bullseye powder contained approximately 364 mg/g  $\pm$  7 mg/g NG and 10 mg/g  $\pm$  0.1 mg/g EC, while Universal Clays had 70 mg/g  $\pm$  4 mg/g NG and 7.1 mg/g  $\pm$  0.3 mg/g DPA. The remainder of the bulk powder compositional measurements are presented as part of Table 2 (vide infra). All unfired powder values represent measurements on ten samples.

## Individual Gunpowder Particle Composition

Individual powder particles were weighed and their compositions were determined. Masses of single particles ranged from 0.031 mg to  $0.706 \text{ mg} (\pm 0.002 \text{ mg})$ . The results obtained indicated that there was no strong correlation between the mass of the particle (lower bars, Fig. 2) and the concentration of the additives in each particle, shown in the upper bars of Fig. 2.

Compositional variations between the particles was significant

Powder	Additive	Unfired (mg/g)	9 mm (mg/g)	.38 Special (mg/g)	.45 ACP (mg/g)
Accurate No. 2	NG	$104 \pm 5$	$102 \pm 7*$	$118 \pm 2$	
	DPA (NnDPA)	15.2 + 0.0		11.5 + 0.2	
	EC	$15.2 \pm 0.8$	$10.8 \pm 0.9^{*}$	$11.5 \pm 0.3$	<b>2</b> 04 + 4
Bullseye	NG	$364 \pm 7$	$380 \pm 12$	$340 \pm 7$	$306 \pm 6$
	DPA (NnDPA)				
	EC	$10.0 \pm 0.1$	$10.0 \pm 0.3$	$7.0 \pm 0.1$	$6.9 \pm 0.3$
Hi-Skor $700 \times$	NG	$251 \pm 9$	$261 \pm 29^{+}$	$279 \pm 12 \ddagger$	$282 \pm 148$
	DPA (NnDPA)			·	
	EC	$7.9 \pm 0.3$	$8.6 \pm 1.1 \dagger$	$8.3 \pm 0.4 \ddagger$	$9.9 \pm 0.5$ §
HP-38	NG	$200 \pm 1.4$	$176 \pm 19$	$210 \pm 4$	$205 \pm 6 \P$
	DPA (NnDPA)	$5.6 \pm 0.1 (6.1 \pm 0.1)$	$3.8 \pm 0.4 (5.8 \pm 0.7)$	$3.3 \pm 0.1 (7.8 \pm 0.2)$	$4.3 \pm 0.2 (7.0 \pm 0.3)$ ¶
	EC	5.0 = 0.1 (0.1 = 0.1)	5.0 = 0.1 (5.0 = 0.7)	5.5 = 0.1 (7.6 = 0.2)	1.5 = 0.2 (7.6 = 0.5)
N320	NG	$8.2 \pm 0.4$		$4.7 \pm 0.3 (1.5 \pm 0.2)^{**}$	
18320	DPA (NnDPA)	$8.2 \pm 0.4$		$4.7 \pm 0.3 (1.5 \pm 0.2)$	
U	EC	$70 \pm 4$	51 + 4+	$40 \pm 2$	$102 \pm 5$
Universal Clays	NG	$70 \pm 4$	$51 \pm 6^{\dagger \dagger}$	$40 \pm 2$	$123 \pm 5^{++}_{++}$
	DPA (NnDPA)	$7.1 \pm 0.3$	$6.5 \pm 0.5 (1.2 \pm 0.1)$ ††	$7.2 \pm 0.2 \ (2.6 \pm 0.1)$	$9.8 \pm 0.4 (3.0 \pm 0.2)$
EC					
Winchester 231	NG	$200 \pm 2$	$221 \pm 15$ §§	$207 \pm 3$	$208 \pm 6 \parallel \parallel$
	DPA (NnDPA) EC	$5.8 \pm 0.1 \ (6.5 \pm 0.1)$	$4.5 \pm 0.4 \ (7.0 \pm 0.5)$	$2.5 \pm 0.1 (7.5 \pm 0.2)$	$3.9 \pm 0.1 \ (6.7 \pm 0.3)$

 TABLE 2—Concentration of additives in bulk unfired powder and handgun-fired residues. Data represent the mean and standard deviation of the mean for ten independent samples, unless otherwise noted.

\* SD of mean with number of independent measurements, n = 4.

 $\dot{\$} n = 5.$  $\parallel n = 5.$ 

 $\prod n = 4.$ 

\*\* n = 5.

- \$\$ n = 5.
- |||| n = 5.

in some cases, although the ball powders manufactured by Primex (HP-38 and Winchester 231) appeared to have more consistent particle-to-particle compositions than the extruded powders. For some powders, such as Universal Clays, the composition of individual particles varied by as much as 8.8 mg/g to 241 mg/g for NG and 6.3 mg/g to 15 mg/g for DPA. A comparison of the concentration of additives based on the average of the ten individual particles to the values as determined for the bulk powder is shown in Table 3. Although many of the measured averages fall somewhat outside one standard deviation of the mean, reasonably good agreement was found for the concentrations of propellants and stabilizers for six of the seven powders. The concentration measurements for bulk and single particle Bullseye were in poor agreement, differing by nearly a factor of 2. Substantial compositional heterogeneity can occur in some gunpowder samples. During the manufacture of smokeless gunpowder some of the nitrocellulose feed may be recycled from previous batches or surplus military ammunition (7). Finished powders may also be blended to achieve target performance characteristics. Thus, care should be taken when only analyzing the composition of a single or a limited number of particles, as individual particles may not be sufficiently representative of the bulk powder to associate the residue uniquely with the powder. This heterogeneity would also be expected in individual OGSR particles. Additional identification information, such as particle morphology or the observation

of colored product identification particles (7), can help to associate particle samples with bulk powder.

Since absolute quantitative compositional analysis of the individual additives appeared to be somewhat unreliable as the sole method of matching particle composition to the bulk, we evaluated the calculation of the ratio of NG propellant to stabilizer for the six double-base powders. This P/S ratio, defined as the concentration of nitroglycerin in mg/g divided by the sum of all the concentrations of stabilizers and stabilizer decomposition products in mg/g, might be a more robust means of associating particle to bulk composition. The P/S values and their variabilities for individual particles are also presented in Table 3. For these six powders, either DPA or EC was the primary stabilizer (noted as D or E), but commercial gunpowders may contain both DPA and EC, and occasionally, methyl centralite (4,11). The P/S ratio seemed to be a more reliable identifier for individual particles than the absolute concentration. For five of the six powders, the P/S ratio of all ten particles was in good agreement with the bulk powder measurement as evidenced by the low standard deviations of the mean and narrow range of values. The range for the Accurate No. 2 powder measurements was increased by a single outlier P/S value of 5.7, with the remaining nine P/S measurements having a range of 9.4 to 11.5. The composition of the Universal powder was significantly more heterogeneous, and the P/S ratio failed to compensate reliably for the disparate additive measurements and had a range of 0.7 to

 $<sup>\</sup>dagger n = 2.$ 

 $<sup>\</sup>ddagger n = 8.$ 

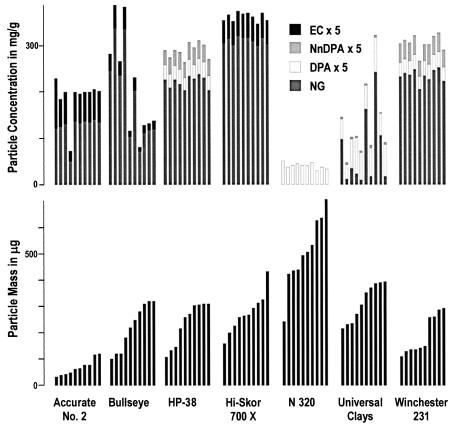


FIG. 2—Single-particle extractions of the unfired reloading powders. Lower bars: mass of individual particles. Upper bars: measured concentration of additives in individual particles corresponding to lower bars.

Powder	Additive	Bulk (mg/g)	Particle (mg/g)	P/S for Bulk	P/S for Particles
Accurate No. 2	NG	104 ± 5	124 ± 8	6.8 ± 0.5 (E)*	10.1 ± 0.5 (E)
	DPA (NnDPA)				range: 5.7–11.5
	EC	$15.2 \pm 0.8$	$13 \pm 1$		
Bullseye	NG	$364 \pm 7$	$187 \pm 31$	$26.4 \pm 0.9$ (E)	37.7 ± 0.8 (E)
	DPA (NnDPA)			36.4 ± 0.8 (E)	range: 34.9-42.5
	EC	$10.0 \pm 0.1$	$5.1 \pm 0.9$		Tunge: 5119 1215
Hi-Skor 700 $\times$	NG	$251 \pm 9$	$309 \pm 2$		30.9 ± 0.2 (E)
	DPA (NnDPA)			31.8 ± 1.7 (E)	20 C 20 D
	EC	$7.9 \pm 0.3$	$10.0 \pm 0.1$		range: 29.6–32.2
HP-38	NG	$200 \pm 1.4$	$221 \pm 4$		$16.7 \pm 0.3$ (D)
	DPA (NnDPA)	$5.6 \pm 0.1 \ (6.1 \pm 0.1)$	$6.1 \pm 0.1 (7.1 \pm 0.1)$	$17.1 \pm 0.2 \text{ (D)}^{\dagger}$	
	50				range: 15.2–18.3
N320	EC NG	$8.2 \pm 0.4$	$8.0 \pm 0.4$		
N320	DPA (NnDPA)	0.2 - 0.4	$8.0 \pm 0.4$		
	EC				
Universal Clays	NG	$70 \pm 4$	$72 \pm 25$	$0.0 \pm 0.7$ (D)	$6.0 \pm 2 (D)$
	DPA (NnDPA)	$7.1 \pm 0.3 (\text{ND})$	$11 \pm 1 \ (0.7 \pm 0.1)$	$9.9 \pm 0.7$ (D)	range: 0.7-15.3
	EC				Tunge: 0.7 15.5
Winchester 231	NG	$200 \pm 2$	$234 \pm 4$		$17.0 \pm 0.2$ (D)
	DPA (NnDPA)	$5.8 \pm 0.1 \ (6.5 \pm 0.1)$	$6.0 \pm 0.1 \ (7.8 \pm 0.2)$	16.3 ± 0.2 (D)	155 150
	EC				range: 15.5–17.8

 TABLE 3—Comparison of measurements on bulk unfired powder to the single particles. P/S is the ratio of the propellant concentration (P) to the total amount of stabilizer (S) in the samples.

\* E represents the EC stabilizer and any EC decomposition products.

† D represents the DPA stabilizer and its decomposition products (such as NnDPA).

15.3. This is in contrast to the success of the ratio to characterize correctly the Bullseye powder, where the variability of absolute composition caused the average for the ten particles to provide a poor association to the bulk measurements, differing by nearly a factor of 2 for both NG and EC. The P/S ratio values of 36.4 for bulk and 37.7 (range 34.9 to 42.5) for single particles of Bullseye were in good agreement.

Thus, overall, the P/S ratio gave reliable identification information for 49 of the 60 particles evaluated using comparison to bulk powder measurements. Although our calculation of the P/S ratio was determined for weighed particles, the ratio is a unitless parameter independent of mass. The P/S ratio can be determined on quantities of powder too small to weigh conveniently by quantitatively determining the ratio of the solution concentrations of the extracted additives.

#### Residue Analysis and Comparison to Unfired Powder

OGSR obtained by firing 100 rounds of ammunition were collected in 21 boxes. Insufficient residues for analysis were found in three of the boxes used for the 9 mm and .45 ACP loaded with N320 and the .45 ACP loaded with Accurate No. 2. The quantity of OGSR obtained in the remaining 18 boxes ranged from 1.4 mg to 938 mg. Differences in the amount of OGSR collected were likely due to differing powder combustion efficiency, cartridge caliber and loading, and weapon type, as well as the effectiveness of the collection protocol. Significant losses of collected residues were attributed to backscattering from the force of the muzzle blast in subsequent firings.

Figures 3 and 4 show two examples of the data from the large data set (presented in Table 2) comparing the unfired powder composition with the OGSR collected from the three weapons. The height of the bar is the mean, and the error bars represent two standard deviations of the mean. Although the quantitative data for all of the residue NG and NnDPA for Winchester 231 were in agreement within two standard deviations of the unfired powder, the values for DPA were much lower in the OGSR for all calibers. For

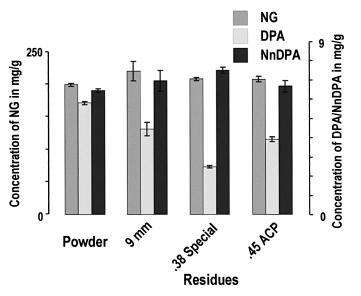


FIG. 3—Comparison of Winchester 231 unfired powder and handgunfired residues. Error bars represent two standard deviations of the mean.

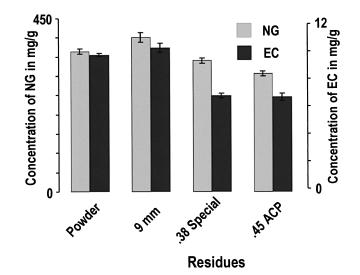


FIG. 4—Comparison of Bullseye unfired powder and handgun-fired residues. Error bars represent two standard deviations of the mean.

powders like Winchester 231, which contained DPA as the stabilizer, it was useful to consider the sum of the concentration of DPA and its primary powder decomposition adduct, NnDPA, as a measure of the stabilizer content. With this summing approach, the total stabilizer concentration of the OGSR (9 mm: 11 mg/g, .38 Special: 9.9 mg/g, .45 ACP: 11 mg/g) was a much closer match to the results for the unfired powder (12 mg/g) than was the concentration of DPA only (4.5, 2.5, 3.9, 5.8 mg/g, respectively). Powders and OGSR stabilized with EC did not show any measurable decomposition products. Figure 4 shows typical results for an EC-containing powder, Bullseye. NG measurements of the 9 mm OGSR of Bullseye were slightly higher than those of the unfired material, and the .38 and .45 were slightly lower. The opposite was true for HP-38, where the NG concentration was slightly lower in the 9 mm and slightly higher in the .38 and .45 relative to that of the unfired powder.

Since we could find no clear trend in the OGSR compositional data as a function of weapon caliber, we thought it would be instructive to sum together the OGSR data from all weapon calibers and compare that to the unfired powder compositions. Figure 5 shows this "averaged" OGSR data relative to the unfired powders for the individual additives. In general, the composition of the OGSR largely reflected the composition of the unfired powder, with some variability in the OGSR values, but had no universal trends of any additive being consistently higher or lower in concentration. Some of this variability in the quantitative concentrations of individual additives can be reduced by calculating the P/S ratio (see Fig. 6). The P/S ratio appears to be a useful tool for comparing the composition of different powders. Although the P/S ratio is not absolutely unique for each of the seven powders tested, it is a relatively robust factor that can be used in conjunction with other identification information. The compositional analysis also vields qualitative information wherein powders can be categorized into single- and double-base, and into those with DPA or EC or other additives (4,11). The qualitative and quantitative compositional information can be combined with other observations, such as particle shape, color, and size, to help associate unknown powders or OGSR with known samples.

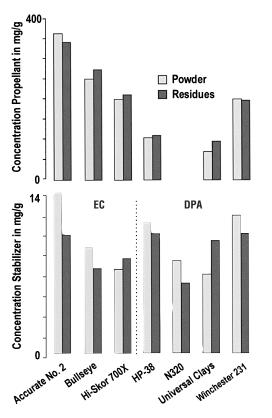


FIG. 5—Comparison of the composition of unfired powder to the cumulative residue results from three weapon calibers.

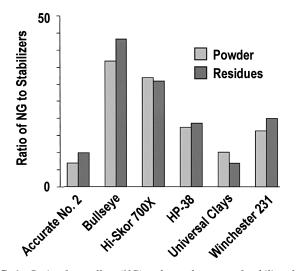


FIG. 6—Ratio of propellant (NG) to the total amount of stabilizer found in the unfired powders and handgun-fired residues.

#### Conclusions

Determining the additive composition of smokeless gunpowders and handgun-fired residues can provide useful information that may aid in associating an unknown sample of powder or residue to known samples. The additive composition of OGSR, determined for seven reloading powders in three different handgun calibers, was generally quite similar to the unfired powder and was not highly dependent on weapon caliber. Heterogeneity in individual powder grains and in the composition of OGSR relative to the unfired powder will contribute to uncertainty in making unique powder identifications based solely on additive composition, especially if the amount of test sample is limited. The ratio of nitroglycerin to stabilizer in double-base propellants appears to be a useful and robust characteristic of a powder or OGSR sample that can be quantitatively determined to aid in powder identification and that will lower some of the variability found with single-additive compositional comparisons.

#### Acknowledgments

The authors would like to acknowledge the financial support for this research that was provided by the National Institute of Justice through the NIST Office of Law Enforcement Standards. The technical assistance of National Technical Systems (Camden, AR) for handgun residue collection is also acknowledged. 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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