

TECHNICAL NOTE

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A Quantitative Comparison of Smokeless Powder Measurements*

ABSTRACT: Measurements of the type and concentration of propellant and stabilizer additives in smokeless gunpowder are used by forensic scientists investigating the source of explosives and by military laboratories assuring the safety and efficacy of munitions. The National Institute of Standards and Technology recently assessed the state-of-the-practice of smokeless powder measurements through an international measurement comparison exercise. We here present results provided by the five participants (of 20 total) reporting quantitative as well as qualitative values for two handgun reloading powders. All five of these participants reported values for nitroglycerin (NG), ethyl centralite (EC), diphenylamine (DPA), and N-nitrosodiphenylamine (NnDPA). Several participants additionally reported the concentrations of secondary stabilizer decomposition products. The unstable NG propellant additive appears to be more susceptible to method-specific calibration biases than are the stabilizer additives. All results from one participant were strongly biased relative to those of the other four. The within-participant measurement uncertainties for all analytes were self-reported to be 1 to 5% relative; among the four concordant participants, the measurement ranges are 5 to 10% relative. There was little consistency among the participants as to what components of measurement variance were included in their uncertainty statements. A discussion of the certainties in these measurements and factors that affect the accuracy of gunpowder additive determinations is presented.

KEYWORDS: forensic science, measurement comparison, smokeless powder, gunpowder additives, propellant, nitroglycerin, diphenylamine, ethyl centralite

The accurate determination of additive ingredients in smokeless gunpowder is important to two groups of analysts: forensic scientists investigating the source of explosives in pipe-bombing incidents and military laboratories assuring the safety and efficacy of munitions. Determination of the specific additives present in a residue sample recovered from a pipe-bomb crime scene can provide forensic investigators with information to categorize the smokeless powder. The U.S. Bureau of Alcohol, Tobacco, and Firearms maintains a database of information about commercial gunpowder that combines qualitative additive composition with dimensional measurements of the powder grains. Evidentiary residue samples may then be compared to entries in the database for categorization (1,2). Recent investigations have also explored the idea of using quantitative analysis of the gunpowder additive composition as a means of associating handgun-fired residues with unfired powders (3–5). Given that addition of identification taggants to gunpowder was recently considered impractical and unnecessary

by a national commission (6), categorizing powders based on quantitative additive analysis is an idea that merits increased investigation.

Military laboratories are also keenly interested in accurately determining the remaining stabilizer content of stored munitions as a predictor of propellant stability (7–9). Subsamples from every lot of gunpowder propellant procured by the U.S. military are stored and evaluated periodically for safety. As many as 5000 samples per year are subjected to quantitative compositional evaluation by individual military laboratories to determine the remaining stabilizer content. The usefulness of gunpowder compositional measurements for forensic and military application depends greatly on accurate measurements.

The accuracy of quantitative determination of gunpowder/propellant composition can suffer as a result of a number of factors. Gunpowder is a blend of nitrocellulose (NC) and several possible additives that may include an additional propellant, nitroglycerin (NG), and stabilizers, diphenylamine (DPA) and ethyl centralite (EC—N, N'-diethyl, N, N'-diphenylurea). These additives have been extracted from the bulk NC matrix with organic solvents (9–12) or supercritical fluids (13–16). However, achieving a quantitative extraction can be a balancing act between extracting sufficiently long to recover the additives from the polymer yet averting decomposition losses and/or cross-reaction between the nitro propellants and the amine stabilizer analytes. Generally, the bulk dissolution of the nitrocellulose powder complicates a chromatographic determination of the additives and requires either precipitation with water before injection (9) or cyclical back-flushing of the analytical column to remove accumulated NC. Obtaining high-

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purity primary materials and maintaining stable calibration solutions of the additives can also prove challenging, especially in the case of NG and also for N-nitrosodiphenylamine (NnDPA), the initial nitration product of DPA.

Despite the difficulty of determining these unstable analytes and the significance of their determination, there have been no reported studies comparing analytical determination of smokeless powder additives. To evaluate powder additive measurement practice, the National Institute of Standards and Technology (NIST) conducted a measurement comparison in Spring 2000. The test materials for this study were two samples of commercial smokeless reloading powders used for handgun ammunition that had been evaluated by NIST measurements. Twenty laboratories participated in the qualitative identification component of this study; we describe these results elsewhere (17).

In addition to providing qualitative results, the five laboratories listed in Table 1 also quantitatively evaluated the concentrations of the NG propellant and stabilizer additives. Participants used their own choice of analytical measurement technique(s) for the determination and laboratory-defined calibration and value-assignment

TABLE 1—List of gunpowder measurement comparison participants.

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National Institute of Standards and Technology Analytical Chemistry Division Stop 8394 Gaithersburg, MD 20899-8394
US Army Aviation and Missile Command AMSAM-RD-PS-R Redstone Arsenal Huntsville, AL 35898
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approaches. In this paper, the quantitative results from the interlaboratory study are evaluated.

Experimental

Smokeless Gunpowder Test Material

Two commercial smokeless reloading powders, 231, distributed by Winchester and manufactured by Primex Technologies (St. Marks, FL), and HiSkor 700X, from IMR Powder Company (Washington, PA), were obtained in 227 g (1/2 lb) or 454 g (1 lb) canisters. Originally, the Hi-Skor 700X contained both yellow and black particles in a proportion of approximately 1 to 100. The yellow particles were composed of NG, DPA, NnDPA, and EC, but the black particles contained only NG and EC. Since the yellow particles were smaller in number and in size, a majority of the particles could be separated from the black particles by screening with different-sized sieves. The few remaining visible yellow particles were then removed by hand leaving the black component of the HiSkor 700X for testing. Canisters of the HiSkor 700X powder were then blended by tumbling in a large metal can for 0.5 h to insure homogeneity. Canisters of the 231 were similarly blended, providing approximately 1.8 Kg of each powder for testing. Each laboratory received 5 g of Hi-Skor 700X (labeled "Powder 1") and 5 g of 231 (labeled "Powder 2"). Careful measurements on small subsamples were made by NIST to confirm reasonable homogeneity. Five-gram samples in amber bottles were sent by next-day shipment to participants with a form to report results and comment on the method(s) used for the measurements.

Gunpowder Measurements

The analytical methods used by all quantitative measurement participants are summarized in Table 2. The NIST method for determination of primary additives used ultrasonic solvent extraction (USE) followed by micellar capillary electrophoresis (CE) (17). For the extraction, a 10-mg portion of gunpowder was weighed into a 15-mL centrifuge tube. A total of 1 mL of solvent, 250 μ L of 2-butanol, and 750 μ L of methanol, was added and the tube was capped. The solution was vortex-mixed for 10 s and agitated in the ultrasonic bath for 15 min. Samples were vortex-mixed again for

TABLE 2—Participant analytical methods.

Lab	Extraction	Separation	Detection
1	2.5 g dissolved in 100 mL acetonitrile, stir 3 days, 0.45 μ m filter	LC, Restek "Allure" C-18 column, 250 mm \times 4.6 mm, 60°C, mobile phase: acetonitrile/water	Diode array absorbance, internal standard: dimethyl phthalate
2	50 mg ultrasonically extracted in 5 mL methanol 15 min, 5 μ L dissolved in 50 μ L CE buffer	CE, 25 mmol/L SDS, 10 mmol/L borate buffer pH 8.5, 30 kV	Diode array absorbance at 200 nm, internal standard: 2-naphthol
3	0.5 g dissolved in 45 mL methanol overnight, filter, dilute to 50 mL	LC, Brownlee SPHERI-5RP-18, 5 μ m, 220 mm \times 4.6 mm, 50% acetonitrile/water, 1.25 mmol/L triethylamine/acetic acid pH 6.0	Diode array absorbance at 254 nm for EC and DPA, 215 nm for NG
4a	100 mg dissolved in 4 mL of methanol overnight, centrifuge, 0.45 μ m filter	LC, Zorbax ODS column, 250 mm \times 5.7 mm, methanol/water at 1.5 mL/min	Diode array absorbance at 214 nm, internal standard: diethyl phthalate
4b	100 mg dissolved in 4 mL of acetonitrile overnight, 0.45 μ m filter		
NIST	10 mg–50 mg ultrasonically extracted with 1 mL 25% 2-butanol/methanol 15 min, 40 μ L extract dissolved in 500 μ L CE buffer	CE, 40 mmol/L SDS, 10 mmol/L borate buffer pH 9.2, 22 kV	Filter UV absorbance at 214 nm, internal standard: quinazoline

10 s and finally centrifuged for 5 min. A 40- μ L portion of the extract was removed and added to 500 μ L of the CE run buffer/internal standard solution, and then 200 μ L of the solution was transferred to a CE sample vial.

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 μ m window diameter) capillaries with a 75- μ m inner diameter and an overall length of 77 cm were used (Agilent Technologies, San Fernando, CA). A temperature-controlled bath kept the samples at 20°C during analysis on the CE. Instrument conditions were as follows: a 1-s pressure injection with a separation voltage of 22 kV, a column temperature of 30°C, and 20 min analysis time. The column was subjected to a reverse pressure rinse each morning using Gradient-grade Milli-Q™ (Millipore, Bedford, MA) water for 5 min and the CE run buffer for 20 min. Before and after each sample analysis, the column was reverse pressure rinsed for 2 min with the run buffer.

The CE run buffer was 40 mmol/L sodium dodecylsulfate (SDS) (Pierce, Rockford, IL) and 10 mmol/L sodium borate buffer (boric acid, Mallinckrodt, Paris, KY, and sodium borate, J. T. Baker, Phillipsburg, NJ), pH 9.2. The internal standard (IS), quinazoline (Aldrich, St. Louis, MO), was prepared in concentrations of approximately 140 μ g/mL in an aqueous solution with the CE run buffer. The standard solution of NG (10 000 μ g/mL), DPA (1000 μ g/mL), NnDPA (1000 μ g/mL), and EC (1000 μ g/mL) in HPLC grade methanol (J. T. Baker, Phillipsburg, NJ) was diluted with the CE run buffer/IS solution and 2-butanol (Aldrich, Milwaukee, WI) for calibration. Previous evaluation of this commercial standard against individual gravimetric standards of NG, DPA, and EC, all from Picatinny Arsenal, and NnDPA from Fluka (Milwaukee, WI) and Sigma (St. Louis, MO), confirmed the accuracy of this calibrant. The volume of organic solvent in the calibration standard was kept proportional to the volume of organic solvent in the samples to achieve the correct viscosity during injection on the CE. Therefore, the calibration standard was prepared by using 278 μ L of the standard methanolic solution and 92 μ L of 2-butanol and diluting to 5 mL in a volumetric flask with the run buffer/IS solution. Ideally, less than 20% of the solution should be organic solvent. With this method of preparation, organic solvents comprised only approximately 7% of the total solution. Studies of the calibration standard and IS in run buffer indicated that these solutions were stable for one week when stored in a refrigerator. At the beginning of each week, fresh solutions were prepared for the week's analyses.

Results and Discussion

Quantitative NG Measurements

The quantitative measurements and uncertainties of NG reported for each of the five participants in the quantitative component of the measurement comparison study are summarized in Table 3. Laboratory 4 submitted two different analytical results for each powder, based on extraction of the samples with methanol (result "4a") and with acetonitrile (result "4b"). Since the two results are equivalent within analytic certainty, the average of the results was used to characterize this participant's results. The data provided by Laboratory 2 were reported as "semi-quantitative" and fall significantly outside other laboratories' measurements. Combining results of Laboratories 1, 3, 4, and NIST, the grand mean NG value (± 1 standard deviation) for Samples 1 and 2 was determined to be 300 ± 22 mg/g and 203 ± 22 mg/g, respectively.

The NIST-reported NG values represent the evaluation of two sets of five independent samples. The range of uncertainties reported by the other participants varied from 0.1 to 8.2 mg/g. It appears likely that the various laboratories use different definitions of "measurement uncertainty." Taking into account the interlaboratory standard deviation, the number of independent reported values, and the NIST-reported measurement repeatabilities (18), we estimate the 90% confidence interval for Powders 1 and 2 to be 266 to 334 mg/g and 171 to 236 mg/g, respectively.

Figure 1 is a modified Youden plot (19) for NG determinations displaying the joint distribution between measurements of Powders 1 and 2. In addition to displaying how the interlaboratory data cluster, this type of plot can reveal within-lab systematic bias and problems with calibration. The measurements of the four laboratories cluster closely around a line drawn through the grand mean and the

TABLE 3—Reported propellant (NG) values and uncertainties in mg/g for Powders 1 and 2.

Lab	Nitroglycerin		Notes on Measurements
	Powder 1	Powder 2	
NIST	298 (5)	205 (3)	10 samples, 3 injections each 1 sample: certainties estimated from similar samples
1	328.8 (0.1)	233.8 (0.1)	
2	393.0 (45)	315.0 (30.0)	
2*	260 (30)	231.0 (19.0)	3 samples, 3 injections each 3 samples, 3 injections each
3	274.8 (8.2)	190.8 (5.7)	
4a	297.52 (3.43)	186.2 (2.5)	2 samples: certainties estimated from evaluation of control propellants and standards 3 samples, 3 injections each 1 sample, 4 injections
4b	298.18 (3.28)	180.7 (1.3)	

* Subsequent data submission.

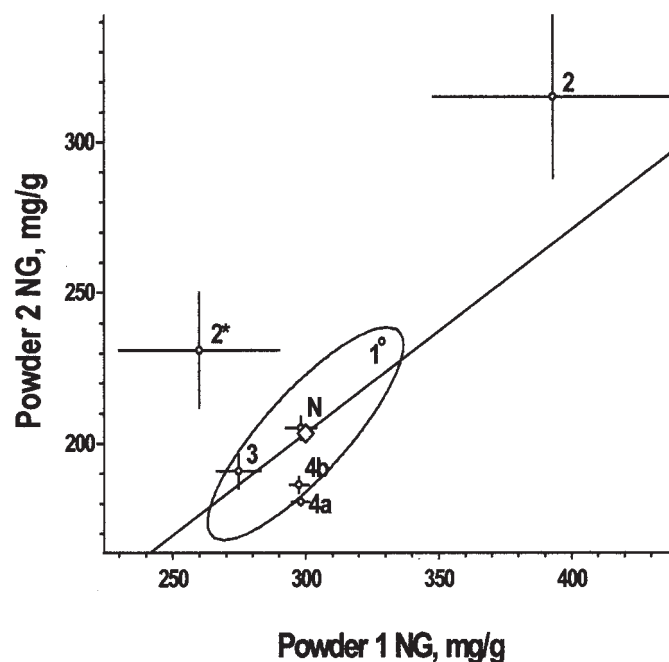


FIG. 1—Youden-type plot of nitroglycerin determinations in two gunpowders. Error bars represent reported uncertainties; ellipse encompasses the 90% bivariate tolerance interval of the measurements.

origin. The ellipse represents an estimate of the 90% bivariate tolerance interval for this limited dataset (20,21). The measurements of NG in the two powders are strongly correlated, i.e., laboratories report results that are consistently “high” or consistently “low” relative to the grand mean. Such a trend is consistent with calibration being the primary source of NG measurement bias between laboratories. Accurate standards for nitroglycerin can be troublesome to prepare and maintain. Neat nitroglycerin is hazardous to handle, so a solvent-diluted material (typically a mass fraction of 5%) is further diluted for calibration standards. One laboratory reported allowing the acetone solvent from a 5% NG solution to evaporate on the balance so that a gravimetric measurement of neat NG could be made for calibration.

It was immediately clear that the initial NG results submitted by Lab 2 (denoted “2”) for both powder samples were significantly different from the other laboratories. On investigation, we found that the solution Lab 2 used for calibration of NG was five years old and likely suffered some decomposition, affording incorrectly high values for the unknown samples. Recalibration with a fresh standard solution resulted in the NG values denoted 2* on the figure. Although recalibration of NG improved the general agreement, some as-yet-unidentified discrepancy remains in the NG measurements reported by this lab.

Quantitative Stabilizer Measurements

Table 4 summarizes the reported quantitative measurements and uncertainties of the stabilizers, DPA and EC, as well as their decomposition products. The “Notes on Measurements” of Table 3 also apply to this table. The grand means for the stabilizers are for EC in Powder 1 and DPA + NnDPA (Σ DPA) in Powder 2, which was combined on a molar basis and converted to mg/g DPA equivalent. Calculation based on Σ DPA helped address the possibility of the formation of NnDPA and loss of DPA during the analytical extraction step (10). The minor decomposition products, 4-NEC, 2-NDPA, and 4-NDPA, were not included in the data analysis as they were not consistently reported by all participants and were sometimes reported with high relative uncertainties. As with NG, we did not include Laboratory 2 results in the statistical evaluation. The grand mean and standard deviation for Powder 1 was 9.9 ± 0.4 mg/g EC and 10.8 ± 0.4 mg/g for Σ DPA. An estimation of the certainty of the NIST measurements, in terms of the standard deviation of the mean, was 0.28 and 0.35 mg/g, respectively. As for NG, we estimate the 90% confidence interval for EC in Powder 1 to be 9.2 to 10.7 mg/g and for Σ DPA in Powder 2 to be 9.9 to 11.8 mg/g.

Although the two powders contained different stabilizers, EC, DPA, and NnDPA have some common properties that influence their analytical measurement. All are derived from aromatic amines, have molecular weights between 168 and 268, and are incorporated into a common matrix (nitrocellulose) in a similar man-

ner. This is likely to result in some correlation of the measurements of EC with Σ DPA. Figure 2 shows a Youden-type plot for Powders 1 and 2, with each axis representing the measurements of the respective stabilizer. The line intersects the grand mean and the origin. The envelope is the 90% bivariate tolerance interval. The NIST values are somewhat lower than the grand mean for both stabilizer analytes.

Analytical Methods/Bias

A summary of the analytical methods used by participants is presented in Table 2. There is no correlation between the order of listing Table 1 and the numbers in Table 2 designating the laboratory

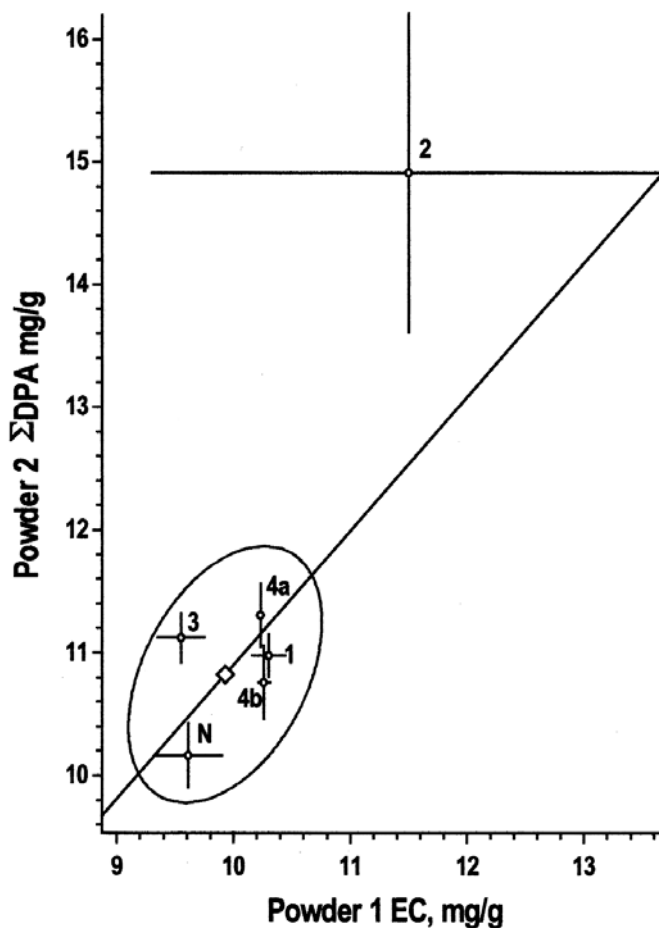


FIG. 2—Youden-type plot of stabilizer determinations in two gunpowders. Error bars represent reported uncertainties; ellipse encompasses the 90% bivariate tolerance interval of the measurements.

TABLE 4—Reported values and uncertainties in mg/g of stabilizers in Powders 1 and 2.

Lab	Powder 1		Powder 2			
	EC	4-NEC	DPA	NnDPA	2-NDPA	4-NDPA
NIST	9.6 (0.3)	...	5.0 (0.1)	6.1 (0.3)
1	10.3 (0.1)	0.1	5.0 (0.1)	7.0 (0.1)	0.3 (0.1)	0.2 (0.1)
2	11.5 (2.2)	...	7.4 (0.9)	8.8 (1.1)
3	9.5 (0.1)	...	5.4 (0.1)	6.7 (0.2)	0.3 (0.01)	0.3 (0.01)
4a	10.23 (0.03)	...	5.26 (0.03)	7.08 (0.05)	0.61 (0.32)	...
4b	10.26 (0.05)	...	4.85 (0.04)	6.92 (0.06)	0.77 (0.37)	...

results. All laboratories used solvent extraction of the additives from the nitrocellulose gunpowder matrix with solvents such as methanol or acetonitrile using either overnight dissolution or 15 min of ultrasonic agitation. Most laboratories used isocratic reverse-phase liquid chromatography with an acetonitrile wash, to remove nitrocellulose, and diode array absorbance detection at a compromise wavelength for the measurements. The two laboratories that used CE had sodium dodecylsulfate as the micellar agent with a borate buffer with absorbance detection. All participants, except Laboratory 3, used an internal standard in their calibration. Laboratory 3 used external calibration against a single calibration solution.

Within the scope of this study, it is impossible to quantitatively assess the contribution of the analytical methods to bias and long-term precision. In addition to method biases, calibration and operator skill contribute to measurement differences found in this comparison. The self-reported, within-laboratory precision for these additives, estimated from analyses of multiple samples, is of the order of 1 to 5%. This is much smaller than the 5 to 10% among-laboratory variation noted. Although not directly determined, other larger interlaboratory studies have shown that the among-laboratory variability is a good estimate of long-term within-laboratory reproducibility (22,23). Development of a reference material would facilitate a more complete accounting of the relative contributions of the analytical method, calibration, and operator skill to the bias and precision of smokeless powder additive measurements.

Acknowledgments

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