

TECHNICAL NOTE

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A Qualitative Comparison of Smokeless Powder Measurements

ABSTRACT: In the spring of 2000, the National Institute of Standards and Technology and nineteen participants conducted a comparison of smokeless powder additive compositional measurements. The purpose of this exercise was to determine the state-of-the-practice for forensic smokeless powder determinations. For the comparison, two handgun reloading powder samples were mixed and were compositionally evaluated for homogeneity by NIST. Participant laboratories included military, academic, and state/local, federal, and international forensic agencies. We solicited qualitative data identifying nitroglycerin, diphenylamine, *N*-nitrosodiphenylamine, and ethyl centralite. In addition, some laboratories provided identification of additional ingredients, such as nitrocellulose, or reported other measurements, such as dimensional morphology.

In this paper, laboratory identification of the smokeless powder additives and a summary of the measurement methods used for the evaluations are presented. All laboratories correctly identified NG and the major stabilizers. Some disparity between laboratories was noted for the identification of minor (<0.01% by weight) stabilizers and stabilizer decomposition products.

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

The accurate determination of the ingredients in smokeless powder provides forensic investigators with information that can be used to identify smokeless powder as the source of collected residues. With the use of exemplar databases, based on qualitative composition and morphology, investigators can often associate the partially burned particles collected in pipe bomb incidents with specific powders (1,2). Also, recent investigations have explored the use of quantitative analysis of the smokeless powder additive composition as a means of associating handgun-fired residues with unfired powders (3–6). Given that addition of identification taggants to smokeless powder was recently considered impractical and unnecessary by a U.S. national commission (7), categorizing powders based on the native additive content 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 (8–10). All of these applications depend on the accurate measurement of smokeless powder additive content.

To assure consistently accurate forensic laboratory measurements, most major law enforcement agencies in the United States participate in voluntary quality assurance activities, such as achieving performance certification from the American Society of Criminal Laboratory Directors' Laboratory Accreditation Board (ASCLD-LAB). An important component in accreditation is per-

formance verification using independently produced test samples. Specific analytical methods may also be validated through accurate measurements on certified reference materials. Currently, there are no available certified reference or test materials for forensic explosive evaluation.

To evaluate the practice of forensic analysis of low explosives, a preliminary laboratory-performance study was conducted by NIST in the spring of 2000. The test materials for this study were two samples of commercial smokeless reloading powders typically used for handgun ammunition. The 19 participants included international and U.S. national, state, and city/regional crime laboratories. We solicited measurements on the following analytes: nitroglycerin (NG), ethyl centralite (EC), diphenylamine (DPA), and its initial nitration product, *N*-nitrosodiphenylamine (NnDPA). All laboratories provided qualitative additive identifications, and five laboratories submitted quantitative measurements that are detailed elsewhere (11). Participants were permitted to utilize any method for their compositional evaluations and were encouraged to provide additional data from other measurements that they use in their smokeless powder evaluations.

Experimental³

Smokeless Powder Test Materials

Two commercial smokeless reloading powders, 231, distributed by Winchester and manufactured by Primex Technologies, and Hi-

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Received 9 Jan. 2001; and in revised form 14 May 2001, 10 July 2001, and 13 Dec. 2001; accepted 26 Feb. 2002; published 17 July 2002.

³ 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.

Skor 700X, from IMR Powder Company, were obtained in 1/2 lb (227 g) or 1 lb (454 g) canisters, which were combined to provide a total of approximately 1.8 kg of each powder. 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 through different sizes of sieves. The remaining visible yellow particles were then removed by hand. Each of the two powders was blended in a large metal can by tumbling for 30 min. Each laboratory was sent 5 g powder samples labeled: Powder 1 (processed Hi-Skor 700X) and Powder 2 (231).

NIST Smokeless Powder Measurements

The two test materials were evaluated by an ultrasonic solvent extraction/capillary electrophoresis measurement technique (USE/CE) (12). Powder 1 was found to have (298 ± 5) mg/g (mean \pm one standard deviation of the mean) of NG, (9.6 ± 0.3) mg/g EC, with DPA/NnDPA < 0.5 mg/g. Powder 2 contained (201 ± 3) mg/g of NG, (5.0 ± 0.1) mg/g DPA, (6.1 ± 0.3) mg/g NnDPA with EC < 0.5 mg/g (11). The NIST-reported values represent the evaluation of two sets of five independent samples using USE/CE, with one set using a 10-mg sample size and the second using 50 mg.

Participant Measurements

Participants were given the option of describing their measurement techniques for this smokeless powder examination. The analytical methods that were used by participating laboratories are summarized in Table 2. Details and descriptions of these methods are presented directly as reported by the participants. In order to resolve seemingly disparate results reported for certain minor additives on the two powder samples, we solicited additional documentation (chromatograms, mass spectra) of the measurement results from selected participants.

Results and Discussion

Identification of Smokeless Powder Additives

The 19 participants in this comparison (see Table 1) represent a broad cross-section of laboratories interested in smokeless powder measurements. In general, the forensic laboratories are making two primary goals with smokeless powder compositional measurements: (1) unequivocally identifying a questioned powder or residue sample as being smokeless powder, and (2) using the pattern of components to categorize a questioned sample into subclasses, followed by additional evaluations to limit associations/exclusions to specific exemplar powder samples. As a vehicle for the first goal, many laboratories identify the major component, nitrocellulose (NC). NC is also used as a base material for photographic film. Additional identification of NG with NC confirms that a questioned sample is double-base powder, despite its other uses in dynamite and blood pressure medications. Finding EC in a questioned sample is unique, with its use limited to a smokeless powder/propellant stabilizer. Identifying DPA/NnDPA is a useful characteristic of smokeless powder, but these analytes also occur in stabilized rubber products. Thus, unambiguous forensic smokeless powder confirmations/classifications may require the simultaneous identification of a number of potential additive components.

For the measurement comparison, two double-base smokeless powders were selected. Based on extensive evaluations by NIST

TABLE 1.—List organizations participating in the measurement comparison.

Agency	Laboratory Type
Bureau of Alcohol, Tobacco and Firearms Atlanta, GA	Federal Forensic
Bureau of Alcohol, Tobacco and Firearms San Francisco, CA	Federal Forensic
Centre of Forensic Sciences Canada	International Forensic
Division of Identification and Forensic Science Israel	International Forensic
Forensic Explosives Laboratory United Kingdom	International Forensic
Forensic Science Agency of Northern Ireland	International Forensic
Geo-Centers, Inc./US Army ARDEC Picatinny Arsenal, NJ	Military
New York City Police Department Laboratory	Regional Forensic
Ohio University	Academic
San Diego Police Department Crime Laboratory	Regional Forensic
South Carolina Law Enforcement Division	State Forensic
U.S. Army Aviation and Missile Command Redstone Arsenal, AL	Military
U.S. Army Criminal Investigation Division Forest Park, GA	Military Forensic
U.S. Naval Surface Warfare Center Indian Head, MD	Military
Virginia Division of Forensic Science Richmond, VA	State Forensic
Washington State Patrol Crime Laboratory	State Forensic

measurements, Powder 1 (disk powder) contained NG and EC, and Powder 2 (flattened ball powder) contained NG and DPA/NnDPA. Both powders exhibited good homogeneity in all measurements. The participants were sent 5 g samples of each powder in amber vials. One state forensic system, with four regional laboratories, used participation in this measurement comparison as partial fulfillment of proficiency testing requirements for ASCLD-LAB accreditation in explosive analysis. For these participants, the NIST test materials were sent to a system coordinator, who sent blind subsamples to individual regional laboratories as proficiency test materials. The reported measurements from four of these laboratories are discussed as independent results.

The methods used for the smokeless powder additive determinations, as reported by the participants, are listed in Table 2. In general, Fourier Transform Infrared Spectroscopy (FTIR) was used to identify NC, and various chromatographic methods (TLC, LC, GC, CE) were used for the other additives. A number of laboratories used highly selective detectors such as mass spectrometric (MS) or nitrogen-specific chemiluminescent (EGISTM) detection.

Each laboratory was asked to identify the four target analytes (NG, DPA, NnDPA, EC), as well as any additional analytes determined. A description of the analytical technique was also solicited. Table 3 is a graphical summary of the submitted additive results presented in a manner uncorrelated with the order in the participant list. Filled boxes in this table signify that the additive was qualitatively identified. Laboratories 1, 2, 3, and 4 also submitted quantitative results, which are discussed in detail elsewhere (11). In addition to the core additives, some laboratories provided identification of additional ingredients, such as dibutyl phthalate and nitrocellulose, which are also noted in the chart. For the purposes of this comparison, "major" additives are those analytes present in concentrations of 0.1 to 100% by weight. Additives with concentrations less than 0.1% by weight are referred to as "minor." All 19 laboratories iden-

TABLE 2—Participant methods.

Lab ID	Method
NIST	10 to 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 \approx 2.5 g dissolved in 100 mL acetonitrile, stir 3 days, spike internal standard (dimethyl phthalate), filter with 0.45 mm into autosampler LC vial; LC: HP 1100 with diode array detector, Restek "Allure" C-18, 250 mm \times 4.6 mm column, 60°C oven, acetonitrile/water mobile phase, wavelengths used and ratio of mobile phase are different for each stabilizer and NG
1.	Visual stereoscopic exam, ultrasonic solvent extraction 15 min for 0.05 g in 5 mL methanol; extract put in 50 μ L buffer on CE, methanol extract examined by GC/MS, TLC, FTIR; solid residue in methanol
2.	0.5 g dissolved in 45 mL methanol overnight, filter, dilute to 50 mL; LC: Brownlee SPHERI-5RP-18, 5 μ m, 220 \times 4.6 mm; 50% acetonitrile/water, 1.25 mmol/L TEA/HAc pH 6.0, Diode Array Absorbance at 254 nm for EC and DPA, 215 nm for NG
3.	Extraction: 100 mg powder and 4 mg diethyl phthalate (internal standard) in 4 mL methanol (overnight), centrifuged, supernatant filtered (0.45 mm); LC: Zorbax ODS 25 cm \times 5.7 mm column, methanol/water at 1.5 mL/min, Waters 996 PDA, calibration at 214 nm
4.	Acetone and water (NC precipitate), add ethanol, GC/MS for EC/NG, evaporate for FTIR of NG
5.	TLC, FTIR, GC/MS
6.	Bulk SEM-EDS (measurements), stereomicroscopy (morphology), FTIR extraction with acetone for NC; GC/MS: injection 100°C, program 40°C/hold 1°C, ramp 25°C/min to 275°C, hold for 9.6 min (extract with dichloromethane, sonicate)
7.	FTIR, GC/MS
8.	Spot test (3% potassium hydroxide in ethanol, Griess reaction), TLC, FTIR, GC/MS
9.	FTIR: Nicolet, 100 scans, 1–2 disks/particles; GC: HP5890 II, 3 m Restek RTX-1 column, FID, 6 mg/mL acetone
10.	TLC: stabilizers—eluent: 30% petroleum ether (60–80°C), 65% toluene, 5% ethyl acetate, visualization: vanillin and Maraour's Reagent; explosives—eluent: 90% toluene, 10% ethyl acetate, visualization: sodium hydroxide, Greiss Reagent
11.	GC/MS of sample dissolved in acetone
12.	TLC and GC/MS with electron impact
13.	TLC of NG, FTIR of NC, GC/MS of EC and NG
14.	GC/MS, extract in dichloromethane
15.	Visual/stereomicroscopic exam; acetone extract—EGIST TM explosive detector, TLC, FTIR; IST—burning characteristics; computer database search; comparison with known standards
16.	Gradient HPLC with PDA detection (230 nm) and GC/MS; sample extraction in dichloromethane, supernatant dried and reconstituted in methanol
17.	Visual exam/stereoscope, DPA microchemical test, FTIR, GC/MS, ignition test
18.	Dissolved in acetone/methanol, GC/MS with EI and NCI modes, dissolved in ethanol, GC/MS with EI mode; NG also identified by EGIST TM
19.	

ACRONYMS: EGISTTM = nitrogen-specific chemiluminescent detection, EI = electron ionization, FID = flame ionization detection, FTIR = fourier transform infrared spectroscopy, GC = gas chromatography, HAc = acetic acid, IST = Ignition Susceptibility Test, LC = liquid chromatography, MS = mass spectrometry, NCI = negative chemical ionization, PDA = photodiode array, SEM-EDS = scanning electron microscopy–energy dispersive spectrometry, TEA = triethylamine, TLC = thin layer chromatography, UV = ultraviolet.

tified propellant nitroglycerin in both samples and were also in good agreement on the identification of the major stabilizers (DPA and EC) in each powder. Not every laboratory normally identifies selected stabilizers as part of their smokeless powder evaluations, and these are noted with asterisks in the table.

In order to consider the similarities and differences noted for the results identifying additional stabilizers and stabilizer decomposition products, we must consider the processes contributing to the additive measurements of these two commercial powders. Commercial smokeless powder products are commonly blended to achieve propellant specifications using two or more manufactured batches (7). Each batch may be comprised of as much as 30% "rework" of powder that did not meet specifications and/or was recycled from retired munitions. Contamination of each batch by residuals on the processing equipment is also possible. Thus, in addition to the major (intended) additives, minor amounts of additional additives may be present in the final product. Particle-to-particle heterogeneity may also be expected for these minor additives. Thus, it is likely that additional additives might be detected in minor amounts by participants, particularly if their method sensitivity is better than the NIST measurement method (approximately 0.5 mg/g or 0.05% by weight). To address this issue, we may consider the measurements of NIST and Laboratories 1, 3, and 4 in the table, which reported quantitative additive values, as a reference group. The results from Laboratory 2 were reported to be only semi-quantitative and will not be included in this reference group. Laboratories 1, 3, and 4 report detection of the additional stabilizers and decomposition products

down to 0.1 mg/g, are highly familiar with the determination of stabilizer decomposition products as an indicator of propellant stability, and make as many as 5000 smokeless powder measurements per year. Consideration of measurements by these laboratories serves to extend the level of detection of minor additives by another factor of 5 (0.01% by weight) with good confidence.

Based on the reference group's measurements, we believe Powder 1, with EC as the major stabilizer, contained no more than 0.1 mg/g of DPA as a minor additive. Laboratory 19 reported the identification of DPA in Powder 1 using GC/MS. They reported a "tiny peak" with the retention time of DPA in the total ion mass chromatograms (TIC). The presence of DPA was confirmed by evaluating the full mass spectrum. The presence of minor amounts of DPA in this EC powder might be expected based on the preparation of this test sample. All visible yellow marker particles stabilized with DPA were physically removed from the sample. However, traces of DPA might have been transferred from the commingled yellow and black particles. A recent study of mixtures of compositionally different smokeless powders found transfer of additives, including DPA, after 270 days of contact (13).

Laboratory 9 found methyl ethyl centralite (MEC) in addition to EC in Powder 1. From the TIC submitted, the peak area for MEC is estimated to be orders of magnitude smaller in area than the EC peak. However, there was sufficient MEC in the GC/MS determination to achieve confirmation by the mass spectrum. The detection of this minor stabilizer by only one participant is inconsistent with all 18 of the other participant's observations on this powder.

TABLE 3—Qualitative identification of smokeless powder additives. Filled blocks denote smokeless powder additive identified.

Powder 1												
Lab ID	NG	DPA	NnDPA	2-NDPA	4-NDPA	EC	MEC	4-NEC	2,4-DNT	DBP	DEP	NC
NIST												
1												
2												
3												
4												
5												
6												
7												
8			*			*						
9												
10												
11												
12												
13			*			*						
14												
15												
16		**				**						
17												
18												
19			***									

Powder 2												
Lab ID	NG	DPA	NnDPA	2-NDPA	4-NDPA	EC	MEC	4-NEC	2,4-DNT	DBP	DEP	NC
NIST												
1												
2												
3												
4												
5												
6												
7												
8			*			*						
9												
10												
11												
12												
13			*			*						
14												
15												
16		**				**						
17												
18												
19			***									

Acronyms: NG = nitroglycerin, DPA = diphenylamine, NnDPA = N-nitrosodiphenylamine, 2-NDPA = 2-nitrodiphenylamine, 4-NDPA = 4-nitrodiphenylamine, EC = ethyl centralite, MEC = methyl ethyl centralite, 4-NEC—4-nitroethyl, centralite.

* Does not analyze for NnDPA or EC.

** Does not analyze for DPA or EC.

*** Does not analyze for NnDPA.

Three issues need to be considered when reconciling such isolated observations: (1) higher relative heterogeneity of minor additive levels in the test samples, (2) differences in measurement method sensitivity, and (3) trace contamination by the participant during handling, extraction, solution manipulation, or analysis, contributing additives that were not present in the original sample. More thorough study of the contributions of these issues with a carefully designed test sample is needed to resolve the importance of these factors in forensic additive evaluations.

Powder 2 contained DPA as the major stabilizer as well as substantial amounts of the DPA nitration product, NnDPA. EC was not detectable above 0.1 mg/g in the reference group's measurements. However, nine laboratories (Numbers 6, 7, 9, 10, 14, 15, 17, 18, 19) reported qualitative detection of EC in Powder 2. Examination of the raw chromatograms from these laboratories using GC/MS (Laboratories 6, 9, 14, 15, 19) and LC/UV (Laboratory 17) showed

a clearly defined peak at the retention time expected for EC. In addition, Laboratories 6, 9, 14, 15, and 19 reported confirmation of EC in Powder 2 by mass spectral analysis. Examination of the TIC reveals that the peak for EC might be coarsely estimated to be orders of magnitude smaller in peak area than DPA. Based on the submitted documentation, finding minor levels of EC by nine laboratories is considered persuasive.

It should be noted that the reference group and two laboratories that routinely measure EC using GC/MS did not report finding this additive in Powder 2. These disparate results can be reconciled by two possible factors: (1) The nine laboratories used methods that were more sensitive for EC determination than the reference group's 0.1 mg/g and (2) Powder 2 may have exhibited much poorer homogeneity for this minor level of EC than for the major additives. Either factor alone or in combination could easily account for the lack of consensus on detecting EC in Powder 2.

Some laboratories reported the detection of additional stabilizer decomposition products such as 4-nitroethyl centralite (4-NEC), 2-nitrodiphenylamine (2-NDPA), and 4-nitrodiphenylamine (4-NDPA). One of the reference laboratories (Laboratory 1) reported 4-NEC at 0.1 mg/g in Powder 1, and seven laboratories found 2-NDPA in Powder 2. Reference Laboratories 1, 3, and 4 reported a range of values that differed by more than a factor of 2 (0.3 to 0.77 mg/g for 2-NDPA in Powder 2), which could be considered “trace” levels (11). Thus, sample heterogeneity at the trace level and differences in method sensitivity can account for a lack of consensus in participant identification of 2-NDPA in Powder 2.

One laboratory also identified 2,4-dinitrotoluene in Powder 2 from a small bump in the LC/UV baseline, no thicker than a pen width, with no additional confirmation. Given that none of the reference or other laboratories found 2,4-dinitrotoluene, contamination of the powder extract by a calibrant solution might also explain this anomalous identification in Powder 2.

Other Smokeless Powder Measurements

Several laboratories use the morphology of the smokeless powder grains as a means of categorizing powder samples. Their dimensional measurements are included in Table 4. Some differences in dimensional measurements are noted. For example, Laboratory 2 found significantly larger values for the thickness and diameter of Powder 1 particles than did the other participants.

To categorize smokeless powders, Laboratory 16 combines their qualitative determination of NG with measurements of morphological characteristics and compares that information to a smokeless powder database. The laboratory used this approach to render a guess at the identities of the brand names of the two powder samples. Powder 1 (Hi-Skor 700X) was correctly identified as being consistent with IMR HiSkor 700X, but “curiously without the yellow particles.” Powder 2 (231) was incorrectly identified as Hodgdon HP-38. However, since both 231 and HP-38 have been known to be manufactured by Primex Technologies by an identical process, it is possible that the submitted Powder 2 sample (231) was indistinguishable from an archived sample of HP-38.

TABLE 4—Morphology measurements submitted by participant laboratories. Mean values (1 standard deviation) are given.

POWDER 1		
Lab ID	Thickness (mm)	Diameter (mm)
2	0.2	2
7	0.1	1.33
14		1.48 (0.03)
16	0.11	1.49
18	0.11	1.37
19	0.15	1.50
POWDER 2		
2	0.3	<1 to 1.5
7		
14		1.08 (0.03)
16	0.26	1.13
18	0.25	1.03
19	0.28	1.17

Conclusions

The forensic laboratory’s primary goal of detecting smokeless powder additives as a means of confirming the propellant nature of a questioned powder/residue sample appears to be well served by this comparison of measurements on two powder samples. Participants made accurate identification of the major additives NG, DPA, and EC in these NIST-characterized test samples. In addition to the major stabilizer, a number of participants identified a minor stabilizer that was not noted in the NIST powder evaluation or by a reference group of participants that submitted quantitative measurements. However, identification of a minor stabilizer (EC in Powder 2) could be rationalized by further evaluation of supporting chromatographic and mass spectral documentation solicited for this report. In all follow-up discussions, responding participants characterized the second stabilizer as present at “trace” levels relative to the major stabilizer. Based on the reference group measurements, these second stabilizers were likely to be present at levels less than or equal to approximately < 0.1 mg/g (0.01%) in both submitted powder samples.

The forensic laboratory’s second goal for compositional evaluation as a means of differentiating questioned smokeless powder samples may not be optimally served by the simple qualitative identification of all ingredients. A lack of consensus in the identification of minor additives was noted throughout this study. Possible problems with contamination in the analysis, greater uncertainty that attends trace/minor component analytical measurements, differences in method sensitivity, and difficulties with additive heterogeneity of questioned samples and exemplars indicate that care should be exercised in interpreting qualitative identification of minor additives (<0.01% by weight), particularly between laboratories sharing measurement results. Perhaps noting these additives as minor components in the evaluation might be more appropriate. Further comparisons of smokeless powder measurements by a larger set of laboratories would be needed to determine if this is a significant issue in forensic examinations.

It was clear that a number of the participants valued highly the opportunity to test their laboratory’s smokeless powder methods and operator performance on evaluated test samples. Routine quality assurance testing would be facilitated by the availability of a reference smokeless powder. Development of a reference material for smokeless powder additive composition is a goal of our current NIST program.

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