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Feasibility of Gunshot Residue Detection Via Its Organic Constituents. Part II: A Gas Chromatography-Mass Spectrometry Method

This is the second in a series of publications to examine the feasibility of tests for gunshot residue based on volatile or volatilizable organic constituents (VOC), either present in the original smokeless powder formulation or produced during firing.

In the first paper [1], the VOC from 33 samples of smokeless powder from various brands and types of cartridges, and from one reloading powder, were characterized by combined gas chromatography-mass spectrometry (GC-MS). These VOC were found to contain combinations of nitroglycerin (NG), 2,4-dinitrotoluene (DNT), diphenylamine (DPA), dibutyl phthalate (DBP), and ethyl centralite (EC, 1,3-diethyl-1,3-diphenylurea). In addition, some powders contained trace constituents such as mononitroglycerin, 2,6-DNT, and 2- and 4-nitrodiphenylamine [1].

The object of the present work is to characterize the organic residue that results from the firing of a gun. This residue may consist of flakes of smokeless powder, a film, or dust of certain characteristic VOC which were originally present in the powder or were formed during firing. Once the analytical problems imposed by the nature and amount of residue are known, it may be possible to develop an analytical method suitable for routine application.

A second topic concerns the GC analysis of the VOC. If powder residues can be individualized by their VOC analysis, it might be possible to link the residues found as clue materials to the specific type of cartridge suspected of having been used in a crime. Tests were performed to see whether VOC remain in pieces of the intact powder residue (referred to as "flakes") and whether these flakes are subject to contamination from previous firings with a different type of ammunition.

The principal analytical technique used in this study is GC-MS with methane chemical ionization and subsequent computer reduction of the data. Although this method does not satisfy the criminalist's need for a low-cost, simple test for gunshot residue, it was used to obtain a quantitative description of the amount of VOC that could be anticipated in residue and to see whether interferences from hand samples posed a serious analytical problem. In a typical GC-MS run, masses from 46 to 500 were scanned. The output from the computer is a reconstructed gas chromatogram, analogous to a conventional GC trace but with the intensity proportional to the sum of only those ions counted during the scan. All or part of each mass spectrum can be recalled from the computer memory after the run is over. The original reconstructed gas chromatogram is the recall of all the mass spectral ions versus scan number data. However, it is possible to recall one

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mass ion versus scan number, effecting a limited mass scan for that particular ion. For example, a GC-MS run can be scanned for the ion at m/e 170 (the diphenylamine methane chemical ionization parent ion). A peak of the mass 170 ion at the correct GC retention time would then be a strong indication of the presence of DPA.

The second technique, mass fragmentography, uses the MS to monitor up to four ions of different masses, neglecting other species. Because the MS detection limit (that is, signal-to-noise ratio) is proportional to the square root of the time the mass spectrometer counts a particular ion and because each ion is monitored for a longer time in the mass fragmentography mode (typically 250 ms) than the reconstructed gas chromatogram (4 ms) mode, mass fragmentography results in an increased detection limit toward the selected ions compared to the conventional reconstructed gas chromatogram/limited mass scan means of gathering data.

At the outset, it must be stressed that the major thrust of the present work was not to develop a GC-MS test for gunshot residue, but rather to use a very sensitive analytical instrument available to characterize the VOC found on the hand of a person who has recently fired a gun. With this background, it might then be possible to develop a low-cost analytical procedure for use by most crime laboratories. Specifically, the following points were addressed in this study.

1. Are flakes of partially burned smokeless powder found on the hand of a person who has fired a gun and, if so, do they retain their original VOC?

- 2. Are these flakes contaminated by VOC from previous firings?
- 3. Is a film, or dust, of VOC deposited on the hand?
- 4. How efficient is the acetone swab VOC collection technique?
- 5. Are characteristic analyzable derivatives formed during firing?
- 6. Are the VOC retained on the hand for any significant period after the firing of a gun?

Experimental Procedure

Handguns and Ammunition

The following handguns were used in the experiments:

- (1) .22 H&R Model 929, 9-shot revolver;
- (2) .32 Llama pistol;
- (3) .380 Browning "Standard" pistol;

(4) .38 Smith & Wesson revolver, Special, either Model 10-4 (10-cm barrel) or Model 10-2 (5-cm barrel); and

(5) .45 Colt pistol, Model 1911-A1, U.S. military issue.

The following ammunition was used:

(1) .22 long rifle-Remington-Peters Pistol Match, 40-grain, round nose lead; Federal Hi-Power, 40-grain, hollow point; and Winchester-Western Super X, Lubaloy, 40-grain, round nose lead;

(2) .32 A.C.P.-Remington-Peters, 71-grain, full metal jacket;

(3) .380 A.C.P.—Winchester-Western, 95-grain, full metal jacket and Remington-Peters, 95-grain, jacketed hollow point;

(4) .38 Special—Norma 158-grain, round nose lead or 125-grain, jacketed hollow point; Super Vel, 110-grain, jacketed hollow point; Winchester-Western Super-X, Lubaloy, 158-grain, round nose lead; and Remington-Peters Hi-Speed, 158-grain, round nose lead or 125-grain, jacketed hollow point; and

(5) .45 A.C.P.—Norma, 230-grain, jacketed hollow point or round nose lead and Winchester-Western, 230-grain, full metal jacket.

Analysis

Samples were analyzed on a Finnigan Model 3200 computerized GC-MS with methane chemical ionization. Studies on the flakes of smokeless powder and the search for subparticulate deposits were done by using a 50-cm by 0.318-cm outside diameter stainless steel column packed with 10% UC W-98 on 80-100 Chromosorb W/AW, operated at $175 \,^{\circ}$ C with methane carrier gas at a flow rate of 20 ml/min. The remainder of the work employed a 1.5-m by 2-mm inside diameter glass "U" column packed with 3% OV-17 on 80-100 Chromosorb W/HP. Various temperatures and program rates were used for different parts of the work. In general, mass fragmentography was done isothermally, but some of the reconstructed gas chromatogram/limited mass scan runs were temperature programmed. The original VOC were easily separable under isothermal GC conditions. The GC-MS of VOC derivatives presented a more difficult separation problem; hence, these analyses were temperature programmed.

Sensitivity

Before we attempted to draw conclusions about whether or not VOC are present in the samples, we performed tests to determine the sensitivity of the GC-MS to selected components.

Figure 1 shows the reconstructed gas chromatogram of a 5- μ g smokeless powder sample that was obtained from 1.0 μ l of a 0.5% acetone solution of powder from a Western .22 long rifle, round nose lead cartridge. Because DPA is present in smokeless powders at around 1% [2], GC-MS sensitivity to DPA in the reconstructed gas chromatogram/limited mass scan mode was much better than 50 ng.

The data from the mass fragmentography runs show the detection limit of DPA to be approximately 20 pg (20×10^{-12} g) at twice the background noise level.



FIG. 1—Reconstructed gas chromatogram of 5 μ g of smokeless powder from a Western .22 long rifle, round nose lead cartridge. Time base: 50 scans = 2.00 min, 10% UC W-98 on 80-100 Chromosorb W/AW at 175°C, methane carrier at 20 ml/min.

Sampling

Hand samples taken by using acetone-soaked cotton swabs or tape lifts were analyzed directly; no effort was made to separate the VOC from skin oils or other possible contaminants. Solvents other than acetone were not used either because they would not efficiently remove the VOC or because of their toxicity (for example, pyridine or carbon disulfide) [3].

Results and Discussion

Flakes of Smokeless Powder

An H&R .22 revolver was loaded with Winchester-Western .22 long rifle cartridges containing NG, DNT, DPA, EC, and DBP. The handgun was encased in, and fired through, a plastic bag. Several powder flakes were found after each firing and these were analyzed by GC-MS with the reconstructed gas chromatogram/limited mass scan technique. Of 15 separate flakes analyzed, NG and DPA were found in all 15 cases, DNT and EC in 13, and DBP in 3. The poor results for DBP were not considered serious because this material, a rather ubiquitous plasticizer, would not be unique to gunshot residue.

Persistence tests on gunshot residue for this study, and for The Aerospace Corporation's scanning electron microscope program in inorganic particle analysis [3], indicate that large flakes of smokeless powder are usually gone from the hand within 1 h. Effective analysis of flakes should be possible in suicide cases because the gunshot residue remains in place. Moreover, the persistence of these flakes on clothing has not yet been explored; they might remain in place on certain kinds of fabrics and weaves much longer than on the hand.

Contamination of the Flakes

Once it was determined that the powder flakes maintained their chemical composition fairly well, attention was turned to the problem of contamination: Do the flakes become contaminated by VOC left in the gun from previous firings? Five calibers of handguns and 15 brands of cartridges were used, and the results of the analyses are shown in Table 1. The tests summarized in Table 1 were performed with handguns cleaned, before the series of firings, in the normal manner with patches and solvents. Several shots of each type of ammunition were then fired in an attempt to clear any previous residue before the flakes were collected. In addition, cartridge types were loaded sequentially from the simpler to the more complex powders to further minimize cross-contamination effects. Of all the VOC, DPA was the one most consistently detected, and NG was the next most reliable. Serious cross-contamination occurred in the case of the single-base (no NG) Norma rounds. With the .38 Special handgun these Norma rounds could be mistaken for the Super Vel ammunition, based solely on VOC analysis. Dibutyl phthalate was a common interference, but, as pointed out before, its use as a gunshot residue indicator is not pivotal.

Because the flakes show significant amounts of cross-contamination, additional tests were conducted with a Smith & Wesson Model 10-2 .38 Special revolver and even greater care was taken in cleaning it between different firings. This weapon was cleaned with conventional solvents and patches before it was fired. Five brands of ammunition were used in sequence: Norma jacketed hollow point, Remington-Peters round nose lead, Super Vel jacketed hollow point, Remington-Peters round nose lead. Except for the Remington-Peters round nose lead

Brand ^b	NG	DNT	DPA	EC	DBP
.22 Long Rifle					
Remington-Peters, RNL	1/1	0/1	1/1	0/1	0/1
Federal, HP	0/1	0/1	0/1	0/1	1/1
Winchester-Western, RNL	1/1	0/1	1/1	0/1	0/1
.32 A.C.P.					
Remington-Peters, FMJ	1/1	0/1	1/1	0/1	0/1
380 A.C.P.					
Winchester-Western, FMJ	4/4	4/4	4/4	0/4	4/4
Remington-Peters, JHP	1/1	0/1	1/1	0/1	0/1
38 Special					
Norma, JHP	1/2	1/2	2/2	0/1	1/2
Super Vel, JHP	1/1	0/1	1/1	0/1	0/1
Winchester-Western, RNL	0/1	1/1	1/1	0/1	1/1
Remington-Peters, JHP	2/2	2/2	2/2	0/1	2/2
45 A.C.P.					
Norma, JHP	2/2	0/2	2/2	2/2	1/2
Norma, RNL	1/1	0/1	1/1	1/1	1/1
Winchester-Western, FMJ	0/1	0/1	1/1	0/1	0/1

TABLE 1—Gas chromatographic-mass spectrometric analysis for VOC in flakes of smokeless powder.^a

^aSamples were analyzed on the UC W-98 column at 175 ∞ . Italicized entries indicate that the materials were present in the original smokeless powder. The table entries show "number of analyses showing this compound/number of particles analyzed." See Experimental Procedures for detailed cartridge descriptions.

^bAbbreviations: NG = nitroglycerin; DNT = 2,4-dinitrotoluene; DPA = diphenylamine; EC = ethyl centralite; DBP = dibutyl phthalate; RNL = round nose lead; HP = hollow point; FMJ = full metal jacket; JHP = jacketed hollow point.

and the Super Vel, the order of firing was chosen to minimize possible contamination of the powder residues by previously deposited VOC.

Four rounds of the Norma cartridges were fired in these tests and the powder residues were collected in a fresh plastic bag, as outlined previously. The inside of the cylinder and barrel, as well as the external parts, were then wiped with a clean rag. To further reduce the chances of cross-contamination, a fresh, rubber surgical glove was worn on the firing hand for each type of cartridge and four rounds of the next cartridge were fired to clear the gun of powder residue before the new residue was collected. The results of the reconstructed gas chromatogram/limited mass scan analyses of the residue are shown in Table 2; the VOC that were present in the original, unfired powder are italicized.

The data for the first, second, and third type of cartridge show that contamination of the flakes by VOC in a "clean" gun can lead to serious errors in identifying a powder as having come from a particular cartridge. Based solely on the VOC analysis (neglecting morphology and color), the flakes from the Norma jacketed hollow point and the Super Vel cartridges appear to be identical in one analysis. Both contain only NG and DPA. This occurs even though the Norma ammunition was single-base and contained no NG. The Remington-Peters round nose lead flakes did not show EC, although it was present in the virgin powder. Finally, flakes from both the Remington-Peters jacketed hollow point and the Winchester-Western Lubaloy round nose lead cartridges were found to contain all of their original VOC.

These studies show that the flakes can be characterized as smokeless powder by GC-MS analysis for their VOC. The VOC data, combined with morphological observations (size, color, and form) and other tests (solubility and thin-layer chromatography, for example) should increase the reliability of designating these small flakes as smokeless powder.

Brand	NG	DNT	DPA	EC	DBP
Norma, JHP	3/3	0/2	3/3		0/2
Remington-Peters, RNL	3/3	0/3	2/3	0/3	2/3
Super Vel, JHP	3/3	0/3	2/3		1/3
Remington-Peters, JHP	4/4	4/4	4/4		4/4
Winchester-Western Lubaloy, RNL	2/2	2/2	2/2	•••	2/2

 TABLE 2—Gas chromatographic-mass spectrometric analysis for VOC in flakes of smokeless powder collected from a .38 Special revolver.^a

^aSee Table 1 for experimental details and explanation of nomenclature.

Subparticulate Deposits

There seems to be little doubt that a flake of smokeless powder can be identified as such by its morphology and by chemical analysis for VOC and nitrocellulose. It was recognized that these flakes rapidly disappear from the hand if there is any subsequent hand activity. Consideration was next given to whether characteristic VOC, apart from those in the visible smokeless powder flakes, are deposited on the hand during the firing of a handgun. Does the cloud of smoke from the handgun contain subparticulate deposits of VOC that will remain on the hand for several hours and that can form the basis of a simple test for gunshot residue? To answer this question, tests were performed in which handguns were fired several times inside plastic bags; subsequently, the inside surfaces of the bags were sampled and analyzed for VOC exclusive of large flakes. With the Western .22 long rifle cartridges, which had all of the five principal VOC, no VOC was recovered from acetone swabs or rinses of the inside of the bags.

A second sampling method involved the tape lifts used to gather gunshot residue material for analysis by The Aerospace Corporation's scanning electron microscope technique [4]. Aluminum sample disks, 25 mm (1 in.) in diameter, were coated with double-sided tape, and gunshot residue (generally from the firing of one round) was removed from the hand by patting it with the adhesive surface of the disk. After the visible powder flakes were picked off and discarded, the adhesive surface was suspended over boiling acetone. The solvent condensed on the surface and, it is hoped, washed off the accessible organic materials. These tests were done on several types of cartridges and handguns. Applying reconstructed gas chromatography/limited mass scan or mass fragmentography techniques, no VOC other than an occasional trace of DBP was ever found.

The next phase of this work was the analysis of acetone swab hand samples. Several handguns (including .22 and .38 Special revolvers and a .380 pistol) were employed and several rounds of different types of cartridges were fired. Data gathered for Aerospace's scanning electron microscope program show that the amount of residue found on the hand does not increase as a linear function of the number of rounds fired. However, it was felt that multiple firings would still increase the likelihood of finding detectable amounts of VOC on the hand.

At the high sensitivity of the GC-MS analysis (typically ~ 1 ng or less), interferences from skin oils, constituents of the cotton swab, and problems with nitrocellulose contamination of the column led to severe problems with GC ghost peaks, that is, components from a previous injection adsorbed on the GC column or septum and then partially desorbed by the next solvent injection, whether or not it contained any VOC. Similar results for NG analysis were obtained by Twibble [5]. This is understandable given the nature of the interferences, many of which are polar compounds (such as skin oils) resembling GC liquid phases. Because of the low levels at which it was anticipated VOC would be present, clean-up methods were not employed; adsorption or decomposition of some VOC during these procedures would, it was felt, reduce them to undetectable levels. Even after care was taken to clean up the GC column between injections, no detectable VOC were found in any of the acetone extract samples.

Sampling Studies with Diphenylamine

It had not yet been determined whether the original VOC were present on the hand and not efficiently detected or whether they were not there in the first place. Consequently, control experiments were performed to see if DPA was somehow lost in the acetone swab technique. Recognizing that DPA could be lost at any of several stages of the sampling method, we postulated the following questions.

1. How efficiently is DPA collected from the hand by wiping with an acetone-soaked swab?

2. Is DPA absorbed in the swab?

3. Is DPA adsorbed by the glass vial used to contain the sample?

These studies complemented the tests carried out by Twibble [5] on the sampling of NG from hands by using acetone-soaked swabs.

Hand Tests—A solution of 100 ppm (w/v) DPA in ethanol was applied to the hand with a 10- μ l syringe. Samples were taken with acetone-soaked wooden-stemmed cotton swabs. To remove the DPA from the swabs, 1 ml of acetone was boiled in a small vial and the cotton tip eluted by the boiling acetone condensate. Excess acetone was removed from the swab by pressing it against the side of the vial. The acetone was evaporated at room temperature and the DPA residue was dissolved in a few microlitres of fresh acetone. Analysis showed that no DPA was recovered at low levels (8 ng applied), but using 80 ng of DPA results in a 6.5% recovery, at best (Table 3).

Swab Control Test—A sample of 8 ng of 100 ppm (w/v) DPA in ethanol was applied to a cotton-tipped swab. After the solvent evaporated, the DPA was concentrated as in the hand tests. Analysis showed that 16% of the original DPA was recovered (Table 3).

DPA Evaporation Test—A sample of 8 ng of 10 ppm (w/v) DPA in ethanol was placed in either a 3.6-ml (1-dram) glass vial or a capillary vial made by sealing the end of a shortened disposable pipet. After the solvent evaporated, acetone was added and the DPA content was assayed; it showed a 15 to 20% recovery (Table 3).

DPA Control Experiment—A sample of 8 μ l of a 1-ppm (w/v) DPA solution in ethanol was placed in a capillary vial and allowed to stand for 5 to 10 min with occasional shaking. Analysis showed about a 100% recovery of the DPA (Table 3).

Test	Recovery, %
Hand test	
8 ng applied	0
20 ng applied	0–1
80 ng applied	1-6.5
Swab test	
8 ng applied	16
Evaporation test	
8 ng applied	15-20
Diphenylamine control test 8 ng in solution	~100

TABLE 3-Recovery tests with DPA.^a

^aSamples were analyzed on the 3% OV-17 column at 175 °C.

Discussion—Clearly, this preferred collection technique is not efficient in recovering DPA at very low levels (<30 ng) for a number of as yet unknown reasons. The DPA might be absorbed into the skin when either ethanol or acetone is used. Allowing the sampling solution to go to dryness also has a deleterious effect which even subsequent elution in boiling acetone does not fully rectify.

If we assume a GC-MS detection limit of 20 pg for DPA, these data indicate that if any subparticulate VOC deposits are left on the hand after firing, they are present in amounts much less than 1 ng. (Remember that these tests were done with multiple firings to enhance the amount of possible residue.)

The Search for Derivatives—Although VOC originally present in the smokeless powder were not found in the swab sampling tests, there may have been combustion products or other derivatives formed during firing that were deposited on the hand and that could provide the basis of a test for gunshot residue. To test this hypothesis, a large number of rounds were fired from two handguns; we collected hand samples with acetone-soaked cotton swabs, taking care to avoid visible particles of smokeless powder.

Figure 2a is the reconstructed gas chromatogram of an acetone extract of an unused wooden-stemmed cotton swab. The mass spectral base peak or protonated parent ion (underlined, if known) is listed above several of the more prominent peaks.

Figure 2b is the reconstructed gas chromatogram of swabs from hands that had not fired a weapon ("handblanks"); again, mass spectral information is shown on several peaks. Figure 2c is the reconstructed gas chromatogram of a hand sample from the firing of 24 rounds of .38 Special Remington-Peters round nose lead ammunition, and Fig. 2d was derived from firing 36 rounds of .22 long rifle Winchester-Western round nose lead. For comparison of retention times, a sample of smokeless powder from the Western ammunition is shown in Fig. 2e.

No compounds were seen in the firing samples that were not present in the handblanks despite the use of computer search techniques to make buried peaks visible. The same situation would prevail, of course, with just GC.

Although the reconstructed gas chromatograms in Figs. 2a through 2e are quantitatively different, mass spectral analysis of the peaks shows that the same components are present in most of the scans. Except for some DBP the original VOC from smokeless powder (Fig. 2e) are not present in Samples 2a through 2d. Most of the interferences come from the swab extract, with the hands contributing peaks at scans 282, 320, and 363. Plastic-handle swabs were tried, but these also contributed a background; the overall result was similar to the case of the wooden-stemmed swabs.

Persistence of VOC on the Hands—During the course of the tests for VOC, hand samples were taken immediately after a firing and were analyzed for VOC either as the original components or as derivatives formed during firing. Except for the analysis of flakes of smokeless powder, we did not find detectable amounts of VOC using the acetone swab technique. Because the amounts of VOC will decrease with time as the person who fired the gun goes about his usual activities, VOC will decrease from "below" detectable levels to "well below" detectable levels, that is, the VOC will never be detected. This fact holds for hand swabs after multiple firings with visible flakes removed before sampling and, of course, would hold for single firings.

Smokeless powder flakes are useful if they can be found on hand or clothing, but nonflake deposits of VOC or derivatives cannot be detected with the current method. The persistence of smokeless powder flakes on clothing is now being explored in this laboratory.

Conclusions

Based on the data gathered in this study, we can now answer the questions posed at the beginning of this report.



FIG. 2—Reconstructed gas chromatograms of acetone extracts: (a) clean cotton-tipped swab, (b) sample from hand that had not fired a gun, (c) swab sample from hand after firing 24 rounds of Remington .38 Special, round nose lead cartridges, (d) swab sample from hand after firing 36 rounds of Western .22 long rifle, round nose lead cartridges, and (e) smokeless powder from Western .22 long rifle, round nose lead cartridge. Time base: 100 scans = 3.57 min, 3% OV-17 on 80-100 Chromosorb W/HP, programmed from 80 to 200°C at 10°C/min, methane carrier at 20 ml/min. Prominent mass spectral ions are shown by the major peaks; where known, the parent ion is underlined.

1. In general, flakes of partially burned smokeless powder are found on the hand if collected promptly. These flakes generally retain their original VOC.

2. Smokeless powder flakes are sometimes found to be contaminated with VOC, either from previous firings or from unknown sources. Serious errors can arise from attempting to determine solely from VOC analysis that a smokeless powder flake came from a certain cartridge.

3. Any subparticle film or dusting of VOC deposited on hands must be present at less than 50 ng.

4. The acetone swab technique, at least for the collection of DPA from hands, results in a very low recovery efficiency when very small amounts of DPA are present (6.5% recovered for 80 ng DPA on the hand). The situation gets worse as the amount of DPA on the hand decreases.

5. No characteristic derivatives were detected on hands that had recently fired a handgun.

6. No VOC, aside from flakes of smokeless powder, were detected on a person's hand immediately after having fired a gun. In addition, the flakes are not found on the hand after an hour. Thus, no VOC would be detected at any time after that point.

Summary

Combined GC-MS has been used to test for the presence of gunshot residue on hands by measuring either (1) traces of the original VOC present in the smokeless powder or (2) characteristic organic derivatives formed during the firing process. Our results show that partially burned flakes of gunpowder are frequently found on a person's hand immediately after firing a gun, but these flakes quickly fall off with hand activity. The results further show that there is no other form of organic gunpowder residue on the hand above our combined collection-detection limit of about 50 ng. Characteristic derivatives, if they are formed, are either present at similarly low levels or else obscured by volatiles from skin oils and other sources present on the hand. The GC-MS computer techniques, such as mass fragmentography or limited mass scans, helped to eliminate interfering species that could have obscured the specific VOC; however, the problem of too little material being deposited on, and sampled from, the hand still remained.

Flakes of smokeless powder collected from a gun firing could be analyzed for VOC by GC-MS. However, extreme caution should be exercised in the designation of a powder particle as coming from a specific brand and type of cartridge based on this analysis because of possible cross-contamination from previous firings of different types of cartridges. Cross-contamination was even detected after a previously cleaned and oiled pistol was used.

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