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Feasibility of Gunshot Residue Detection Via Its Organic Constituents. Part I: Analysis of Smokeless Powders by Combined Gas Chromatography-Chemical Ionization Mass Spectrometry

A quick, convenient, and reliable test for detecting gunshot residue is needed to cope with the increasing number of crimes involving handguns. Chemical color tests for nitrates [1] and for antimony, barium, and lead [2] have been used, but these methods lack the required sensitivity and specificity for forensic purposes. Two instrumental techniques, neutron activation analysis and atomic absorption, have found some acceptance by crime laboratories. Neutron activation analysis has been used to detect antimony and barium in hand samples [3], but it suffers from the drawbacks that samples must be sent out for analysis and that it is insensitive to lead. Atomic absorption has been used to detect lead by flame atomization [4], and antimony and barium have been detected with a flameless atomizer [5]. This approach has extended the availability of gunshot residue analysis. However, the costs still limit the number of laboratories with the required equipment. Perhaps a more serious limitation of any technique that is based on the bulk amounts of antimony, barium, or lead on a person's hands is the possibility of environmental or occupational sources of these elements on the hands. This limitation requires setting a threshold often higher than the amount deposited by firing. Furthermore, the amount of residue on the hand declines very rapidly with time.

In 1974, we prepared a survey and technical assessment [6] of the present status of gunshot residue detection techniques and identified promising new approaches to a more reliable test. We are now developing three new methods for the detection and identification of gunshot residue. The first of these is a particle analysis procedure using a scanning electron microscope (SEM) with an energy-dispersive X-ray analyzer [7]. When a handgun is fired, micrometre-sized spheroids derived from the primer and bullet are given off and deposited on the firing hand. Analysis of these particles by SEM shows them to have a characteristic size range, morphology, and elemental composition. This method seems to satisfy the requirement of a definitive test for gunshot residue but obviously has limitations because of the cost of equipment.

The second method takes advantage of the fact that gunshot residue contains high concentrations of lead and sometimes antimony. By a low-temperature luminescence technique, we are able to detect these metals down to the low- and sub-nanogram region [8]. This method is similar in capability to atomic absorption; its advantage is the reduced cost of the equipment.

The third approach, reported here, is the detection and identification of organic compounds characteristic of gunshot residue: materials either originally present in the smokeless powder or formed during its combustion. This method, it was hoped, could satisfy all of the goals of our program, namely, be definitive, rapid, and inexpensive.

Smokeless powders commonly used in small arms ammunition are blends of nitrocellulose and small amounts of stabilizers and plasticizers as well as various modifying agents. "Double-base" smokeless powders contain nitroglycerin (up to 40% by weight), while "single-base" smokeless powders do not. In addition, these materials contain additives to achieve desirable burning rates and increased storage life. The analysis of smokeless powders and similar propellants has been accomplished by using thin-layer chromatography [9,10], gas chromatography [11-16], high-performance liquid chromatography [17,18], and mass spectrometry [19-24].

This paper is the first in a series on the efficacy of the detection and identification of organic compounds characteristic of gunshot residue, and it reports gas chomatographic-mass spectrometric (GC-MS) analysis of the volatile (by GC) organic components in 33 smokeless powder samples. Thirty-two of the powders were obtained from commercial cartridges, and one was a reloading powder. Subsequent papers will deal with the characterization of actual gunshot residue samples and with sampling and analysis schemes using thin-layer chromatography and GC.

Experimental Procedure

Apparatus

Samples were analyzed by using a Finnigan 3200 GC-MS with a differentially pumped chemical ionization (CI) source and a Model 6000 data system. The total GC effluent was introduced into the CI source; no separator was used. Two GC columns were used. The first was a 0.5-m by 3-mm outside diameter (20- by $\frac{1}{8}$-in.) stainless steel tube packed with 10% UC W-98 on Chromosorb W/AW/DMCS, 80-100 mesh; the temperature was held isothermally at 175°C. The second was a 1.2-m (4-ft) by 2-mm inside diameter silane-treated glass "U" tube packed with 3% OV-17 on Chromosorb W-HP 80-100 mesh; the temperature was programmed from 160 to 250°C at 15°C/min. The injector temperature was 200°C, and the separator oven/transfer line temperature was 175°C. The carrier gas was methane at 20 ml/min. The mass spectrometer was set at 1 s/scan.

For solid probe runs, the temperature was set to increase from ambient to 350°C in about 1 min. The methane CI runs were scanned from 32 to 39 and 42 to 320 atomic mass units (AMU), and the helium CI and electron impact runs were scanned from 10 to 310 AMU.

Procedure

Reference Mass Spectra—Samples were introduced into the mass spectrometer by using the heated solid probe inlet. Electron impact (EI) spectra were run at a source pressure of 13 MPa (10^{-4} torr), while methane and helium CI spectra were run at pressures of 133 and 53 Pa (1.0 and 0.4 torr), respectively. The solid probe was ballistically programmed at a heater setting of 950. The MS ion source was maintained at 130°C for both the probe and GC runs. While this temperature is 65°C lower than the transfer lines and might lead to sample condensation and memory effects, higher temperatures were not used because of the possibility of sample decomposition by ion-molecule reactions in a hot source [22].

Because methane CI mass spectra show very intense reagent gas ion peaks at m/e 28 to 31 and 40 to 41, these mass ranges were not scanned. Therefore, the NO$^+$ ion ($m/e = 30$), if it was formed, was not seen in the methane CI runs.

In some cases, the methane CI spectra show adduct ions at M + 29 and M + 41 arising from addition of the reagent ions $C_2H_5{}^+$ and $C_3H_5{}^+$, respectively, to the sample molecules.

Smokeless Powders—For GC analysis, powder samples were dissolved in reagent-grade acetone and analyzed by GC-MS. The entire GC effluent was routed to the MS ionizer to yield the methane CI mass spectrum.

For solid probe analysis, particles of smokeless powder were slowly heated in the MS solid probe inlet to see whether any of the volatile organic components outgassed from the nitrocellulose matrix.

Results

GC Analysis

The results of the GC-MS analysis of 33 sources of smokeless powder are given in Table 1, and the reconstructed gas chromatograms of the Western .22 powder (Table 1) analyzed on the UC W-98 and OV-17 columns are shown in Fig. 1. Five major components were found: nitroglycerin (NG), 2,4-dinitrotoluene (2,4-DNT), diphenylamine (DPA), dibutyl phthalate (DBP), and ethyl centralite (EC, 1,3-diethyl-1,3-diphenylurea). It is noteworthy that the NG was detected without a great deal of difficulty with either the stainless steel UC W-98 or glass OV-17 columns. The crucial factor in NG analysis by GC might be its residence time in the heated parts of the system rather than the analysis temperature, although they are inversely related. In our analyses, the column and glass-lined GC-MS transfer lines were kept around 175°C. The short residence time of NG, about 20 s, might prevent substantial decomposition. Analysis on the OV-17 column reveals the presence of minor components. Preceding the NG peak, at Scan 15, is what appears to be mononitroglycerin (MNG, m/e 128, MH$^+$). A small amount of 2,6-DNT is seen at Scan 59, before the main peak for 2,4-DNT at Scan 72. The 2- and 4-nitrodiphenylamines (NDPA) elute at Scans 174 and 260, respectively.

With the exception of Powder 1, all of the other samples were analyzed on the UC W-98 column, and only the five major volatile components, NG, 2,4-DNT, DPA, EC, and DBP, are reported. The MNG and 2,6-DNT are probably impurities in the NG and 2,4-DNT, respectively, used in the manufacture of the powders. The 2- and 4-NDPA arise from the reaction of DPA with the decomposition products of NG and nitrocellulose formed during storage. Although some workers [17] report that 2-NDPA is used as a stabilizer in the manufacture of cast explosives, it was never found to be present in more than trace amounts.

The other powders were not analyzed for the minor components because (1) they would be present in amounts too low to be of practical value in a test for gunshot residue, (2) they might vary unpredictably from batch to batch of the same powder because of changes in the starting materials, and (3) the presence of some of them, such as 2- and 4-NDPA, is a function of both the age and storage history of the powder.

Except for the Federal .32 (No. 8), 9-mm Lapua (No. 15), and the Norma rounds (Nos. 13, 26, 27, and 32), all of the powders examined were double-base and contained substantial amounts of NG. Diphenylamine was the most common additive, being present in all except the .32 Winchester-Western (No. 6), 9-mm Federal (No. 14), .38 Special Remington (No. 22), .45 Remington (No. 3), and the IMR 4875 reloading powder (No. 33). The rarest component was EC; methyl centralite was not detected in any of the samples. Dibutylphthalate and 2,4-DNT were present in about half of the powders. In cases where the reconstructed gas chromatogram did not clearly show one of the five major volatiles to be present, a limited mass scan of the base peak versus scan number was performed. This is a useful way to increase the apparent sensitivity of the analysis, since the methane CI base peak generally accounts for a least 50% of the total ions and since ions of other molecular weights do not appear in the limited mass scan. For example, small amounts of DPA are confirmed by the appearance of an m/e 170 peak at the appropriate retention time.

TABLE 1—*The GC-MS determination of the compositions of smokeless powder from 33 sources.* [a]

Source	NG	DNT	DPA	DBP	EC
.22 Long Rifle					
1. Western Super X, 40 grain, Lubaloy RNL	x	x	x	x	x
2. Western Super X, 37 grain, Lubaloy HP	x	x	x	x	—
3. Remington Pistol Match, 40 grain, RNL	x	x	x	—	—
4. Federal Hi-Power, 40 grain, copper-coated, RNL	x	—	x	—	—
5. Federal Power-Flite, 40 grain, RNL	x	—	x	—	x
.32 Automatic					
6. Western, 71 grain, FMJ	x	—	—	x	—
7. Remington, 71 grain, FMJ	x	—	x	—	—
8. Federal, 71 grain, FMJ	—	x	x	—	—
9. Hirtenberg, 71 grain, FMJ	x	x	x	x	x
.357 Magnum					
10. Western Super X, 158 grain, Lubaloy FNL	x	x	x	x	—
11. Remington Hi-Speed, 125 grain, JHP	x	x	x	x	—
12. Super Vel, 110 grain, JSP	x	—	x	x	—
13. Norma, 158 grain, FNL	—	—	x	—	—
9-mm Parabellum (Luger)					
14. Federal, 123 grain, FMJ	x	—	—	—	x
15. Lapua Patrunnatehdas, FMJ	—	—	x	—	—
16. Speer, 100 grain, JHP	x	—	x	—	—
.380 Automatic					
17. Remington, 95 grain, FMJ	x	—	x	—	—
18. Western, 95 grain, FMJ	x	x	x	x	—
19. Super Vel A.C.P., 88 grain, JHP	x	—	x	—	x
.38 Special					
20. Remington, 158 grain, RNL	x	—	x	—	x
21. Remington Hi-Speed, 125 grain, JHP (April 1974)	x	x	x	x	—
22. Remington Hi-Speed, 125 grain, JHP (Dec. 1975)	x	—	—	—	x
23. Western, 158 grain, Lubaloy RNL	x	x	x	x	—
24. Western, 158 grain, RNL	x	x	x	x	x
25. Federal, 158 grain, RNL	x	—	—	—	x
26. Norma, 158 grain, JHP	—	—	x	—	—
27. Norma, 158 grain, RNL	—	—	x	—	—
28. Super Vel, 110 grain, JHP	x	—	x	—	—
.45 Automatic					
29. Western, 230 grain, FMJ	x	x	x	x	x
30. Remington Targetmaster, 185 grain, WC	x	—	—	—	x
31. Super Vel A.C.P., 190 grain, JHP	x	—	x	—	—
32. Norma A.C.P., 230 grain, JHP	—	—	x	—	—
Reloading powder					
33. IMR 4875	x	x	—	—	—

[a] Abbreviations
DBP = dibutyl phthalate
DNT = 2,4-dinitrotoluene
DPA = diphenylamine
EC = ethyl centralite
FMJ = full metal jacket
FNL = flat nose lead
HP = hollow point
IMR = improved military rifle
JHP = jacketed hollow point
JSP = jacketed soft point
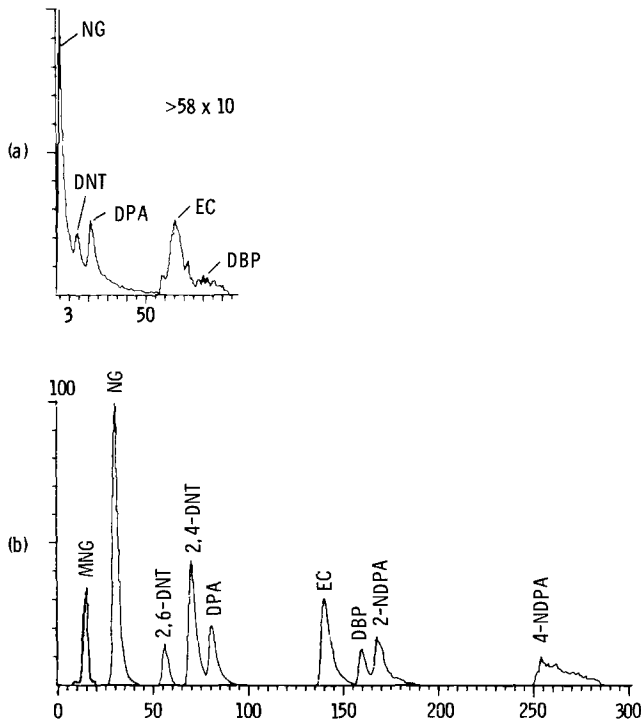NG = nitroglycerin
RNL = round nose lead
WC = wadcutter

FIG. 1—*Reconstructed gas chromatogram of smokeless powder from a 5.6-mm (.22-caliber) Western Super-X 40-grain Lubaloy RNL Cartridge;* (a) *UC W-98 column, 100 scans* = *5.22 min and* (b) *OV-17 column, 100 scans* = *3.57 min.*

Standard Mass Spectra

To achieve chemical ionization, the MS must have a so-called tight source in which a relatively high pressure of the reagent gas can be maintained. This is to ensure a large number of collisions between the sample molecules and the reagent gas ions. Because of this design, samples run in the EI or helium CI mode in a CI source often exhibit protonated parent ions from reactions between the sample and residual water or other proton sources, or ions resulting from the reaction of the neutral parent molecule and one of its decomposition ions. Thus, the EI spectrum of 2,4-DNT shows a protonated parent ion at m/e 183 and a $(DNT + NO)^+$ addition ion at m/e 182 + 30 = 212. While EI spectra run in a CI source are generally, with the exception of the protonated and addition ions, qualitatively identical to EI spectra run in an "open" EI source, they can be quantitatively different in terms of relative ion abundancies.

The EI, helium CI, and methane CI mass spectra of the five major volatile organic components of the smokeless powders examined are presented in Figs. 2 to 6.

Nitroglycerin (Fig. 2)—Both the EI and helium CI spectra show no ions above m/e 76, that is, $CH_2ONO_2^+$. The base peak is NO_2^+ (m/e 46), and some NO^+ (m/e 30) is formed in each case. The mass spectra are essentially identical to those reported by Fraser and Paul [23]. The methane CI spectrum shows a small (1.7%) protonated molecular ion at m/e 228, an ion at m/e 165 (12%) arising from the loss of nitric acid from the protonated molecular ion, and, as before, a base peak of NO_2^+. Saferstein et al [24] have examined NG by isobutane CIMS and report a base peak at m/e 117 and other fragment ions at m/e 73 and 88. Because isobutane is a gentler ionizing gas than methane, it is curious
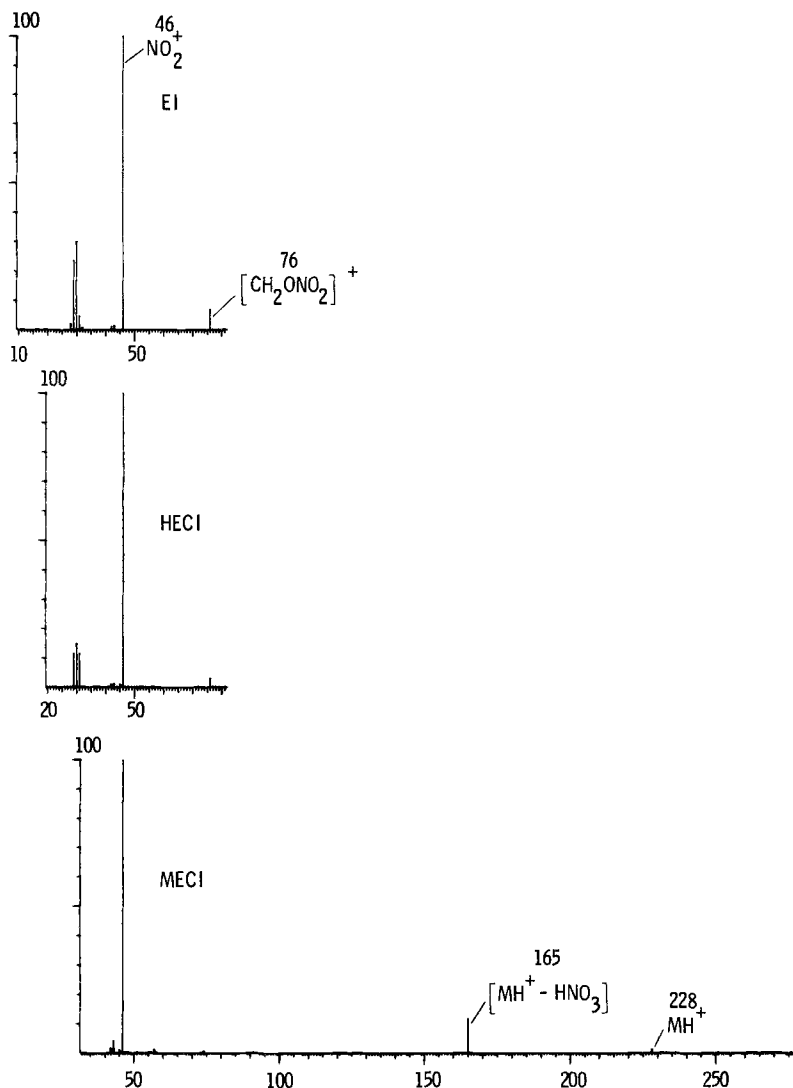
FIG. 2—*Mass spectra of nitroglycerin.*

that we see a parent ion at m/e 228, while the isobutane CIMS shows no ions higher than m/e 117. Slight differences in source temperature and ion residence times could account for this discrepancy.

2,4-Dinitrotoluene (Fig. 3)—The molecular ions in both the EI and helium CI spectra are accompanied by the (M + 1)⁺ ions arising from protonation by residual water or other proton donor in the MS source. The base peak at m/e 166 in the EI spectrum probably arises from loss of OH from the protonated molecular ion, while the peak at m/e 212 represents addition of NO⁺, formed during sample fragmentation, to the parent molecule. Except for the (M + 1)⁺ ion, the EI mass spectrum is identical in all major respects to that reported by Juhasz et al [19].

The methane CI spectrum shows only the protonated molecular ion at m/e 183, along with the CI adduct ions at M + 29 and M + 41.
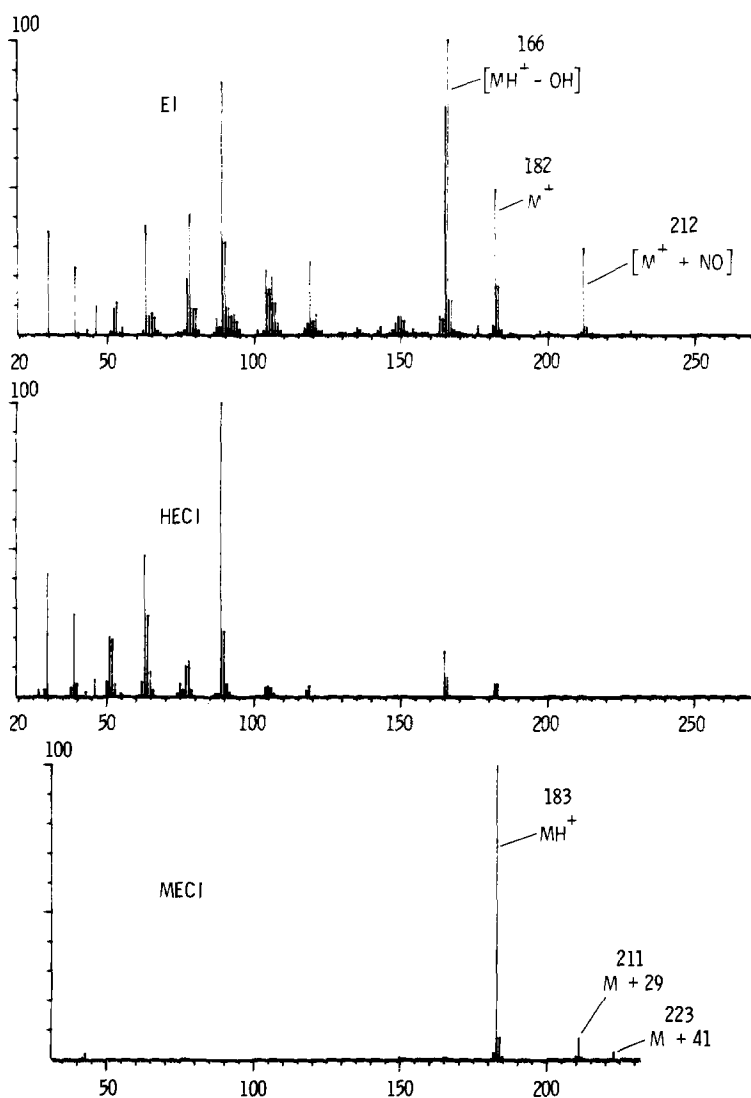
FIG. 3—*Mass spectra of 2,4-dinitrotoluene.*

Diphenylamine (Fig. 4)—Both the EI and helium CI spectra show the parent ion to be the base peak at *m/e* 169. The principal fragment is the *m/e* 77 ion $(C_6H_5)^+$. Methane CI shows the protonated molecular ion at *m/e* 170 plus adduct ions at *m/e* 198 and 210.

Ethyl Centralite (Fig. 5)—The principal fragments in the EI and helium CI spectra are at *m/e* 29, 77, 120, and 148, with a small (5%) parent ion at *m/e* 268. The *m/e* 29 ion is probably the ethyl or CNH_3 cation. The EI mass spectrum is qualitatively identical to that reported by Juhasz et al [*19*]. Methane CI gives, as the base peak, the protonated molecular ion at *m/e* 269, the M + 29 adduct ion at *m/e* 297, and fragments at *m/e* 77 (3%), 120 (14%), and 148 (30%).

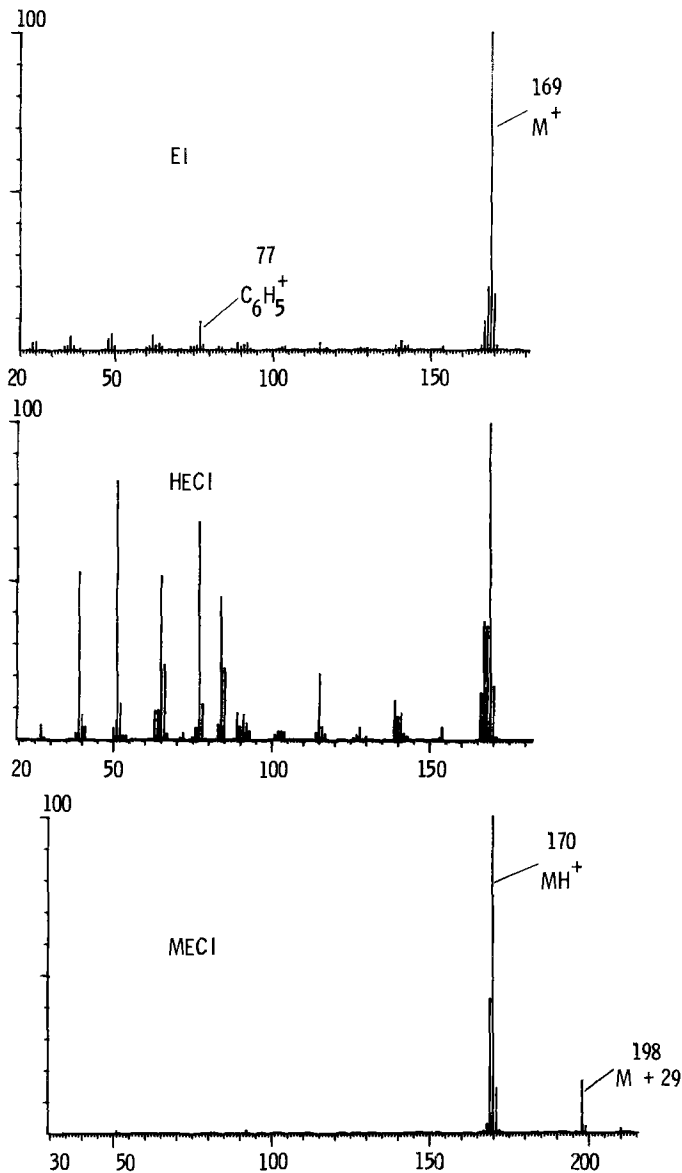*Dibutyl Phthalate (Fig. 6)*—In all cases, the base peak is *m/e* 149: protonated phthalic

FIG. 4—*Mass spectra of diphenylamine.*

anhydride. Only methane CI yields a parent ion, $(MH)^+$, at m/e 279 (23%). The ion at m/e 205 results from the loss of a butoxy group.

Solid Probe Analysis

Slow heating of smokeless powder particles in the MS solid probe inlet did not result in the detection of any of the five major volatile components from the nitrocellulose matrix. Even using mass fragmentography, in which the MS was set to detect only the most intense methane CI ion of four of the five components (NG, 165; 2,4-DNT, 183; DPA,
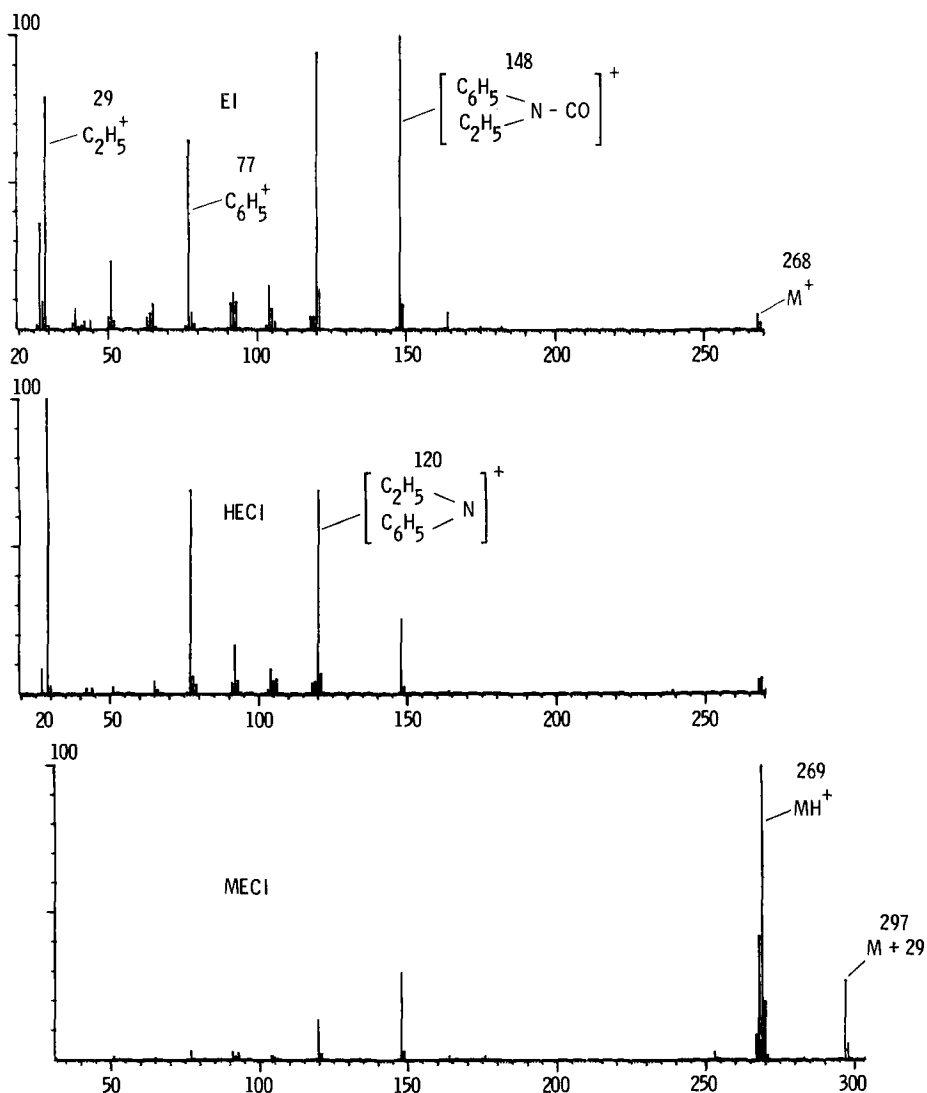
FIG. 5—*Mass spectra of ethyl centralite.*

170; and EC, 269), failed to reveal their presence. These components have been detected by the solid probe technique with a field ionization MS [20]. The detection of these types of compounds with a CIMS has been reported to occur only when the reagent gas is swept through the solid probe sample [25]. When water, generated in situ by the thermal dehydration of a mixture of inorganic hydrates, was passed over a sample of glutathione (an involatile tripeptide) a water CIMS was obtained. Conversely, no ions were seen when the water was not swept over the sample but was instead introduced through a separate port.

In the present case, the solid probe assembly would have to be modified to permit the CI reagent gas to be introduced coaxially with the probe sample. While we have not performed calibration runs to determine sensitivity values (A/torr) for these components, a detectable amount of any of these compounds would be in the neighborhood of
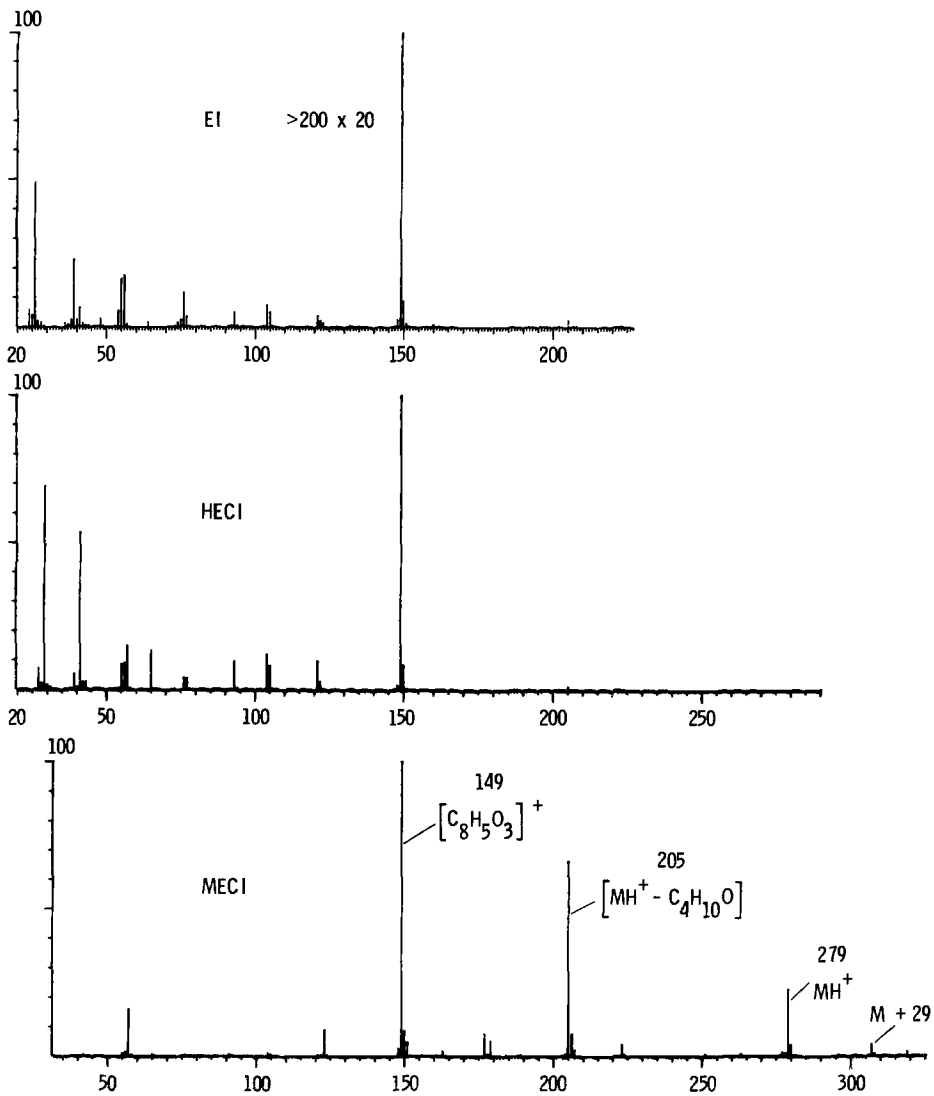
FIG. 6—*Mass spectra of dibutyl phthalate.*

0.13 Pa (10^{-3} torr). Rapid surface depletion of these volatiles, coupled with the inter-molecular forces involved in the gel-like nitrocellulose matrix, could account for these negative findings.

Discussion

The results in Table 1 show that there is no simple way to predict the composition of a smokeless powder based on caliber or bullet type. The cartridge manufacturers employ different powders, even in the same caliber, to achieve optimum performance and stability characteristics. For example, Remington-Peters uses NG, DPA, and EC in their lead-nose .38 Special cartridges but NG, 2,4,-DNT, DPA, and DBP in their same caliber hollow point.

It is tempting to interpret the data in Table 1 to mean that one can sometimes distinguish between cartridges that have been used in various shootings on the basis of the analysis of residual powder. A piece of smokeless powder residue could probably be linked to the powder from a specific cartridge on the basis of morphology, that is, its shape (ball, flake, or rod), color, and size. Additional corroboration might then be gained by finding that the two powders have the same qualitative chemical composition as shown by GC-MS. However, several precautionary points must be made:

1. Powder residue found after firing must be shown to be chemically identical to virgin powder and not lose one or more components by selective volatilization. This topic will be addressed in a forthcoming publication.

2. The powder particles might be contaminated with residues from a previous firing of the gun with a different ammunition. For example, contamination of .22 long rifle Federal powder (No. 4) by the 2,4-DNT in .22 long rifle Remington (No. 3) would make them appear chemically equivalent. The problem of cross-contamination will be explored further in a subsequent publication.

3. It must be known whether the manufacturer regularly uses only one kind of powder for each particular caliber/bullet combination. This is not always the case. For example, the powder from the Remington .38 Special 125-grain Hi-Speed jacketed hollow point, purchased in April 1974, was a dark gray ball powder, approximately 0.45 mm in diameter, containing NG, 2,4-DNT, DPA, and DBP. The identical ammunition, purchased in December 1975, was light gray and gray-green flake powder about 1.25 mm in diameter and contained only NG and EC. Such radical changes both in appearance (Fig. 7) and composition of powders from the same source complicate their forensic identification.

Recently manufactured smokeless powders are sometimes reworked from older batches by adding additional explosive components and stabilizers to achieve desirable ballistic properties. If Company X reworks a powder from Company Y, there will probably be no
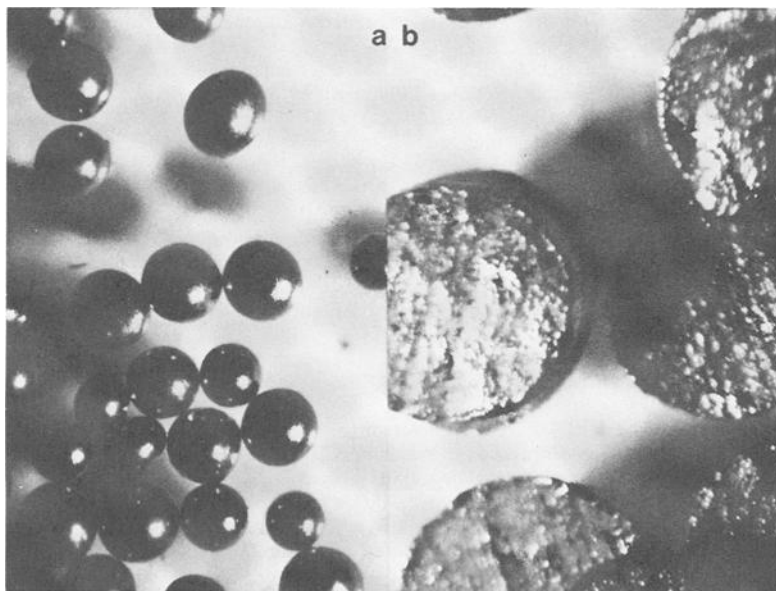

FIG. 7—*Comparison photomicrograph of the powders from .38 Special Remington Hi-Speed 125-grain JHP Cartridges; (a) No. 21, purchased April 1974 and (b) No. 22, purchased December 1975 (magnification, both × 20).*

sure way to ascertain the manufacturer by GC-MS analysis for volatile organic components. Furthermore, manufacturers are not constrained to continue using a specific type of smokeless powder in a specific type of cartridge.

This situation is analogous to trying to determine the brand of a sample of gasoline by a comparison of GC curves. With high resolution GC techniques, very slight differences between gasoline samples become apparent. However, the problem lies not with the accuracy of the GC method but with the variability of supposedly identical samples. Midkiff [26] has explored in great detail the refining, distribution, and marketing factors which can result in the fact that gasoline sold at a station owned by oil company A might be bought from oil company B, refined by oil company C, and have come from a well run by oil company D. Thus, the idea that brands of gasoline have certain characteristics like, for example, a Chevrolet or a Ford, is not valid.

Conclusions

Analysis by GC-MS of smokeless powders reveals the presence of combinations of up to five organic compounds: NG, 2,4-DNT, DPA, DBP, and EC. In conjunction with morphology, the presence of one or more of these compounds can corroborate assignment of a suspect particle as smokeless powder. Caution must be exercised, however, in drawing conclusions about cartridge caliber and type from these data alone.

Based on the limited data from our GC-MS analysis, EC would seem to be the most characteristic, although the rarest, material found in smokeless powders, followed by 2,4-DNT, and then DPA. The possibility of using these materials as the basis for a simple and reliable test for gunshot residue will be explored in Part II of this series.

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