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TECHNICAL NOTE

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Differentiation Between Single-Base and Double-Base Gunpowders

One aspect of our continuing investigation of smokeless gunpowders in the forensic science laboratory has been the differentiation between single-base and double-base gunpowders. Nitrocellulose is the basic ingredient in single-base gunpowder. Nitroglycerine added to nitrocellulose in quantities varying from 1 to 40% w/w is the distinguishing ingredient of double-base gunpowders.

This paper describes a rapid and definitive method to differentiate between single- and double-base smokeless gunpowders. A simple quadrupole mass spectrometer with electron impact (EI) source and direct inlet solid probe was used. In addition to differentiating between single- and double-base gunpowders, the positive identification of nitroglycerine can rule out many false positives encountered in chemical spot tests for nitrates and nitrites used for gunpowder identification.

Materials

The mass spectrometer used in this work was a Finnigan Model 4000 quadrupole sector with a direct insertion probe. The EI ionization mode was used, and the data were recorded and manipulated by a Finnigan Model 6110 data system.

The 18 canister smokeless gunpowders examined in this study are listed in Table 1 along with the average weight of an individual powder particle. The average weight per particle varied from about 0.05 to about 2.3 mg. The sample size in this study was about 0.5 mg of each powder, which was placed in the bottom of a glass capillary tube about 1 mm inside diameter by 8 mm long. This necessitated cutting some of the powder particles into smaller pieces in some cases and using several particles with a combined weight of about 0.5 mg in other cases.

Method

Other workers [*1*] have recently reported no success in this type of analysis using the direct inlet solid probe. Therefore, it is urged that the method of headspace analysis outlined here be closely followed to reproduce these results.

Instrument conditions for EI ionization are these: mass range, 28 to 80 AMU; integration time (per mass unit), 2 ms; 1 s per scan; 0.3 mA filament; 1450 V electron multiplier; filter, 10^3 AMU/s; and sensitivity, $\times 10^{-7}$ A/V. Source pressure before analysis is about 67 μPa (5×10^{-7} torr), and source temperature is 250°C.

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TABLE 1—*Smokeless canister gunpowders examined. The weight given is the average of ten randomly selected powder flakes* (SB = *single base and* DB = *double base*).

Powder Name	Average Weight per Particle, mg	Manufacturer's Designation
Hodgdon BL-C	0.138	DB
Hodgdon H870	0.349	DB
Hodgdon H380	0.156	DB
Heters 102	0.787	SB
Du Pont 3031	1.328	SB
Du Pont 4198	1.070	SB
Du Pont IMR 4064	1.591	SB
Hercules Reloader 21	2.333	DB
Hodgdon 4895	1.143	SB
Du Pont 4320	0.970	SB
Hodgdon 4831	2.253	DB
Alcan 5	0.095	SB
Hercules Red Dot	0.169	DB
Hercules 2400	0.278	DB
Hodgdon 162	0.346	SB
Hercules Bullseye	0.047	DB
Du Pont Hiskor	0.062	SB
Hercules Reloader 7	0.434	DB

The filament and electron multiplier should be turned on and the data acquisition activated. The probe and sample are inserted into the pump-down chamber and evacuated to about 6.66 Pa (0.05 torr) (this takes about 5 or 6 s). The Huntington bellows isolation valve is then cracked open to sample the headspace of the pump-down chamber. The mass spectrum is recorded for 30 to 60 s, after which time the isolation valve is closed, the sample is removed, the filament and electron multiplier are turned off, and the data acquisition is stopped. All 18 of the gunpowders were run in this manner. A blank was run between each sample to check for cross-contamination.

The powders were reexamined with the same techniques but with methane chemical ionization (CI) instead of EI ionization. The source pressure was about 33 Pa (0.25 torr) and the source temperature about 200°C during these analyses. A blank was run between each sample. The mass range was 100 to 250 AMU, the integration time was 3 ms, and all other conditions were the same as for the EI analysis.

Results

A typical mass spectrum corrected for background of a single-base gunpowder is shown in Fig. 1. This EI ionization spectrum lacks peaks at *m/e* 46 (NO_2^+) and 76 ($CH_2NO_2^+$), which are indicative of the presence of nitroglycerine. A typical mass spectrum of a double-base powder is shown in Fig. 2. Here both the indicative peaks are present in the EI ionization spectrum, and they are present in the same ratio (*m/e* 46/*m/e* 76) as the known EI ionization spectra of nitroglycerine.

The EI ionization and the CI mass spectral determinations agreed in every case with the data supplied by the powder manufacturers (see Table 1). No attempt was made to determine the actual sensitivity limits of this method. Most manufacturers of double-base powders add at least 1% w/w nitroglycerine to the nitrocellulose base, so a minimal weight of nitroglycerine per sample would be about 5 μg.

The methane CI spectra of double-base powders clearly showed a typical nitroglycerine spectrum with an M+1 ion at 228 AMU. This M+1 ion was absent in all of the single-base powders. Typical spectra are shown in Figs. 3 and 4.

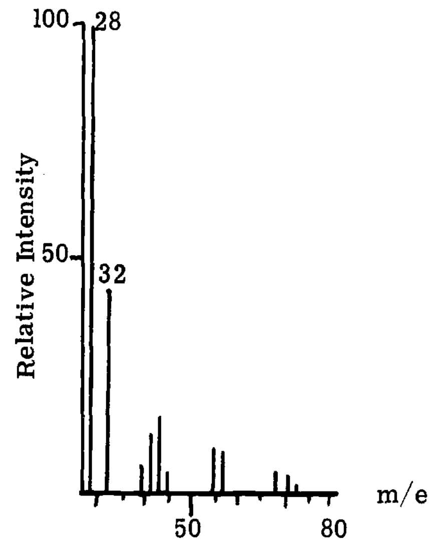

FIG. 1—*Typical EI ionization mass spectrum of single-base powder headspace analysis (Du Pont 3031).*

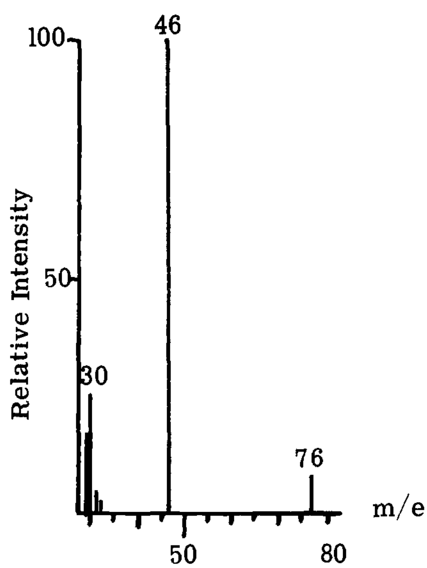

FIG. 2—*Typical EI ionization mass spectrum of nitroglycerine from double-base powder headspace analysis (Hercules Bullseye).*

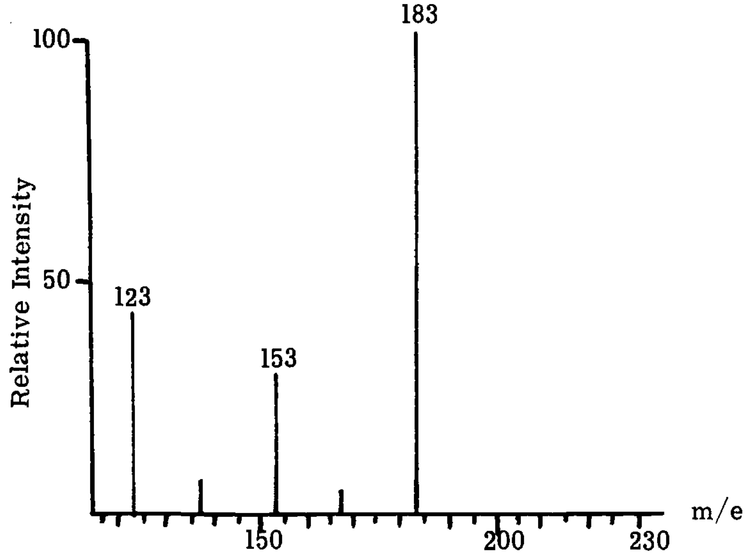

FIG. 3—*Typical CI mass spectrum of single-base headspace analysis (Hodgdon 4895).*

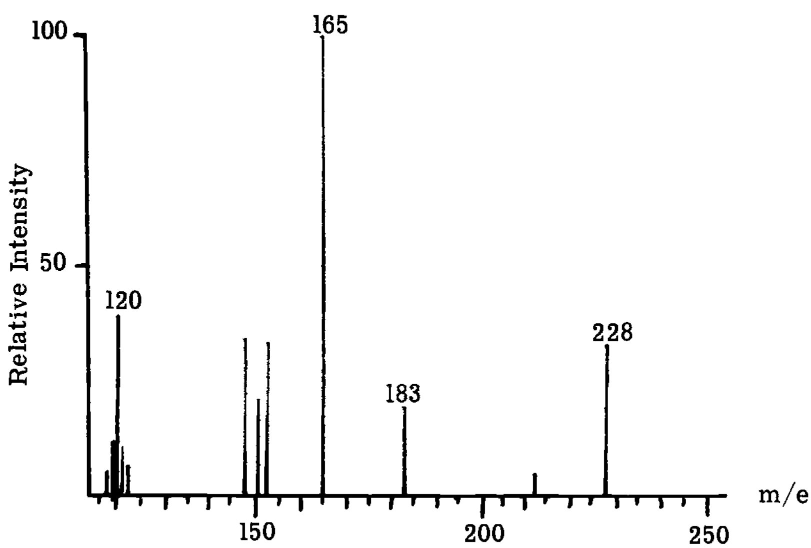

FIG. 4—*Typical CI mass spectrum of headspace nitroglycerine from double-base powder headspace analysis (Hercules Reloader 7).*

Reference

[1] Mach, M. H., Pallos, A., and Jones, P. F., "Feasibility of Gunshot Residue Detection Via Its Organic Constituents. Part I: Analysis of Smokeless Powders by Combined Gas Chromatography-Chemical Ionization Mass Spectrometry," *Journal of Forensic Sciences,* Vol. 23, No. 3, July 1978, pp. 433–445.

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