International Journal of Forensic Science & Pathology (IJFP)
ISSN 2332-287X

A Method for the Differentiation of Single-Base and Double-Base Smokeless Powders using the Hanging Drop Technique

Research Article

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Abstract

Bulk smokeless gunpowder is manufactured in a large variety of shapes, sizes, and textures, many different types look similar, making it challenging to differentiate between types of powders. One chemical discriminating parameter is the presence of nitroglycerin, which often requires expensive instrumentation to be detected. By using the Hanging drop technique, whereby a sample is induced to react inside a drop hanging over it after, it is possible to differentiate between single and double-base smokeless powders. The method resulted in a successful, time effective, and non-destructive result for the detection of nitroglycerin.

Keywords: Hanging Drop; Smokeless Powders; Nitroglycerin; GCMS.

Introduction

Smokeless powders are typically utilized for sport and recreational purposes. However, they are also used in the construction of improvised explosive devices (IEDS) [1-4], and may be encountered in any crime scene that involves a firearm. Smokeless powders are traditionally analyzed in a laboratory setting and require time-consuming protocols and expensive confirmatory instrumentation. Previous studies cite the use of gas chromatography-mass spectrometry (GC-MS), high performance liquid chromatography (HPLC), ion chromatography, and micellar electrokinetic capillary electrophoresis [5-10]. Though the confirmatory result is advantageous, the potential drawbacks for many laboratories are a drain on the economic and time resources. Presumptive tests can offer an opportunity for pre-screening and information gathering. In order to mitigate these potential disadvantages, an approach to screen evidence using presumptive tests may prove beneficial, although lacking in specificity, gain in reduced costs and time.

Smokeless powders are commercially available in the single-base and double-base form. Triple-base ammunition; however, is typically reserved for military ordinance. Single-base powders contain nitrocellulose (NC) while double-base powders contain nitrocellulose and nitroglycerin (NG) [11, 12]. Differentiation and identification between the single-base and double-base powders using presumptive tests maybe more practical than performing extensive confirmation techniques at the onset. The most efficient way to differentiate between these two classes is by detecting the active ingredient nitroglycerin in double-base powders, because single-base powders lack it. In order to create a fast, effective, non-destructive, and economical presumptive test this particular hanging drop method was applied to differentiate between single-base and double-base smokeless powders [13]. The presumptive test results were supported by confirmation of the presence or absence of nitroglycerin using GC-MS.

Methods

Presumptive Test

The method's foundation is based on the hanging drop method. The test reagent mixture is composed of 2.0 mg of diphenylbenzidine (DPB) in 10 mL of sulfuric acid. This would be an excellent place for the author to expand on the prior uses in the literature of DPB, including references. Also to explain how the reaction works. This is chemistry and explainable. It is not enough to show

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Received: August 12, 2016
Accepted: September 29, 2016
Published: September 30, 2016

Citation: Gittings M, Petraco N, Roberts M (2016) A Method for the Differentiation of Single-Base and Double-Base Smokeless Powders using the Hanging Drop Technique. *Int J Forensic Sci Pathol.* 4(9), 266-270. **doi:** http://dx.doi.org/10.19070/2332-287X-1600063

that it works, how it works needs to be explained. Ten smokeless powders were obtained for tentative identification. This method was performed with five single-base and five double-base smokeless powders. Table 1 lists the smokeless powders analyzed using the method along with their classification as single-base or double-base. The classification was determined by the ammunition's MSDS. A single pellet, of known origin was placed onto a clean microscope slide and covered by a glass sublimation ring with an outer diameter of 22mm (Image 1). A 20µl of DPB test reagent was placed onto a circular cover slip with a 22mm diameter (Image 2). The coverslip holding the DPB test reagent was inverted and placed on top of the ring such that the DPB test reagent acted as a hanging drop (Image 3). The set-up was placed onto a preheated 70°C hotplate and allowed to warm for three minutes. (Image 4).

Image 1.

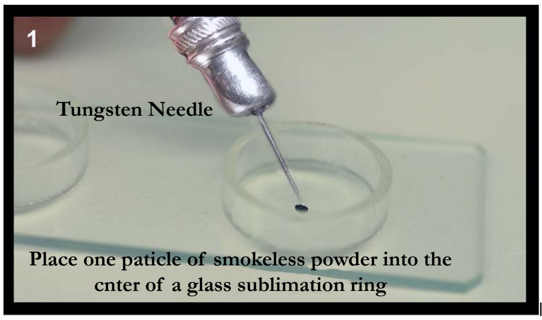


Image 2.

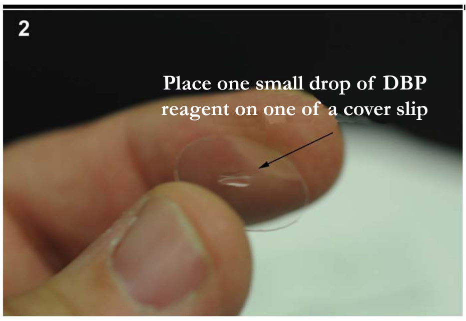


Image 3.

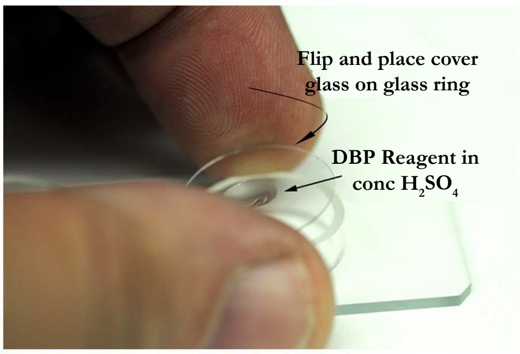


Image 4.

Confirmatory Test

In order to demonstrate the validity of the presumptive method, confirmatory tests were performed alongside. The individual smokeless pellets were subsequently dissolved in 500µL of acetone, then the supernanent diluted to 750µL. The solution was analyzed on an Agilent 7890B gas chromatograph coupled with an Agilent 5977A mass spectrometer. The GC column was an Agilent HP-5MS 5% Phenyl Methyl Silox. The injector temperature was 150°C. The column temperature was held at 100°C for 3 minutes, and heated to 250°C at a rate of 10°C/min and held for 5 minutes at 250°C. The scan range was 25 to 500m/z. The scar rate was 31.scans/sec. The carrier gas was helium. Injections were carried out on a split mode with a ratio of 150:1. The injected sample volume was 1µl. NG was prepared traditionally, dissolved in 750µL of acetone and analyzed using the GC-MS with the specifications stated above.

Results

Presumptive Test

The hanging drop over the single-base pellet produced a colorless ring. The drop hanging over the double base pellet produced a ring with a purple hue. The purple hue also developed within 15 seconds. The reagent control did not produce a hue of any kind. The positive control, nitroglycerin, produced a consistent purple hue that was observed in the reaction for the double-base powder. The reaction time for the positive control was immediate. Figure 1 depicts the results observed in four reactions.

The top left corner sample is a single-base powder, the top right corner sample is nitroglycerin (positive control), the bottom left corner sample is a double-base powder, and the bottom right corner sample is diphenyl amine (negative control).

Confirmatory Test/GC-MS Data

Figure 2 displays the GC for a single pellet of Alliant Unique double-base powder that was recovered after the hanging drop method was performed to test for the presence of NG. In addition to the presence of a ring with a purple hue that was observed in the hanging drop method, NG was still detected in the pellet when it was analyzed by GC-MS. Figure 3 is GC for the bulk positive control of Alliant Unique.

Figure 4 displays the GC for a single pellet of IMR 4350 single-base powder that was recovered after the hanging drop method was performed to test for the presence of NG. In addition to the colorless ring that was observed in the hanging drop method, NG was not detected in the pellet when it was analyzed by GC-MS. Figure 5 is a GC for the bulk positive control of IMR 4350.

Discussion

The hanging drop approach to detect nitroglycerin in smokeless powders was successfully developed. The method is reproducible, cost effective, and nondestructive. A positive reaction produces a purple drop, when NG is present. It is necessary to note that the concentration of NG in bulk-manufactured pellets varies wildly, ranging as much as 4-40%. This results in small variations of time and intensity of purple in positive results.

Table 1. Experimental Smokeless Powders.

Smokeless Powder	Classification
Alliant Unique	Double-base
Hercules 2400	Double-base
Hercules Bullseye	Double-base
Winchester Western Ball	Double-base
Hercules Blue Dot	Double-base
IMR 4350	Single-base
IMR 3031	Single-base
Dupont SR 4759	Single-base
Hogdon H110	Single-base
Hogdon H4831	Single-base

Figure 1. Photographs of Presumptive Hanging Drop Method of Single-Base Powder (Top Left); Double-Base Powder (Bottom Left); Nitroglycerin (Top Right); Diphenyl Amine (Bottom Right).

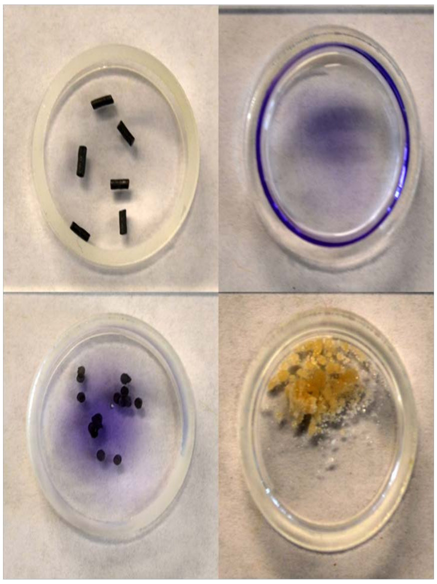


Figure 2. Gas Chromatogram of the Supernatant from a Single Pellet of Double-Base Powder Ammunition (Alliant Unique) from the Hanging Drop Method.

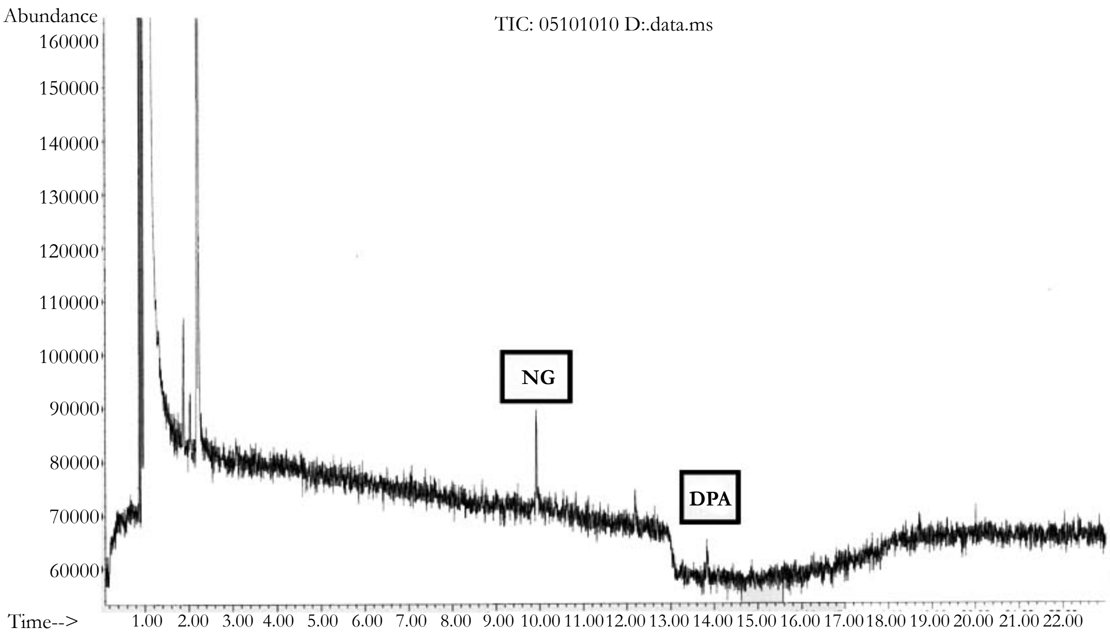


Figure 3. Gas Chromatogram of the Supernatant from Bulk Double-Base Ammunition (Alliant Unique).

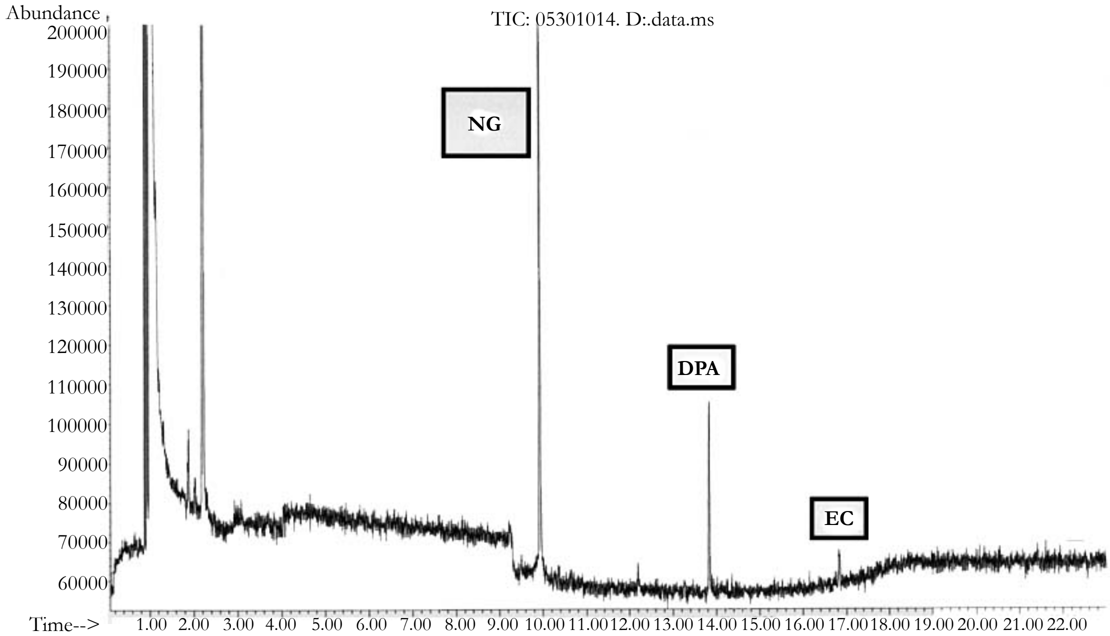


Figure 4. Gas Chromatogram of the Supernatant from a Single Pellet of Single-Base Powder Ammunition (Imr 4350) from the Hanging Drop Method.

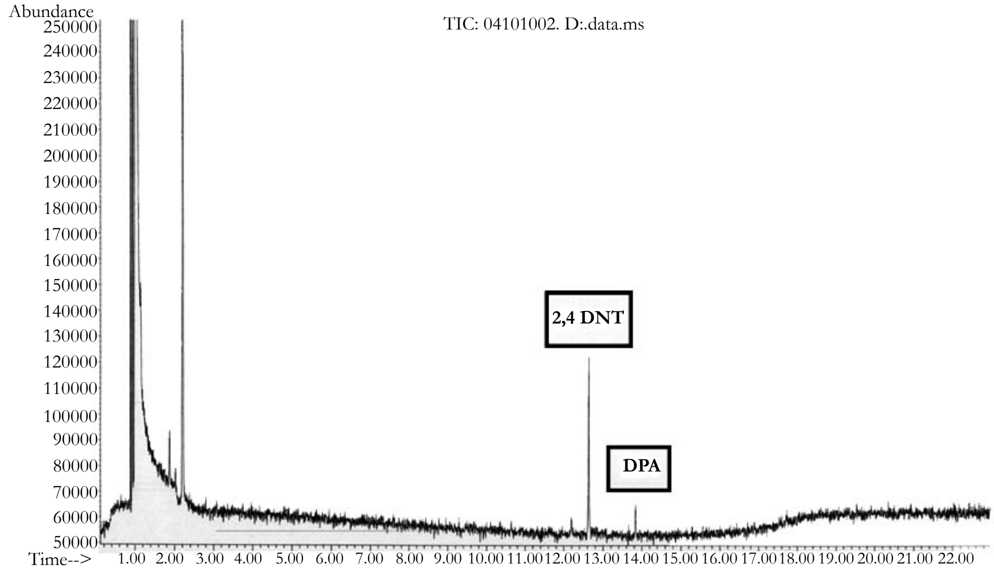
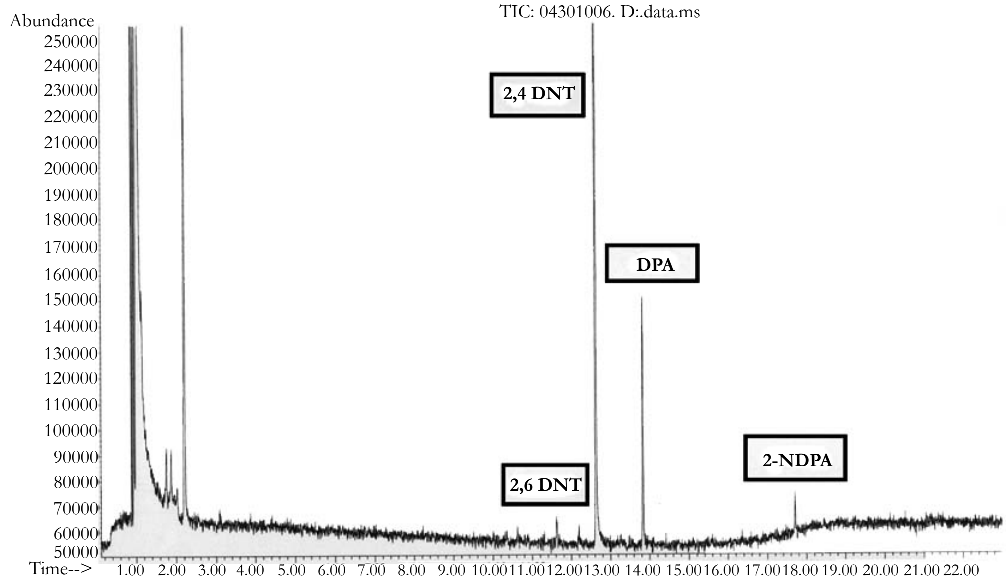


Figure 5. Gas Chromatogram of the Supernatant from Bulk Single-Base Powder Ammunition (Imr 4350).



Acknowledgments

We would like to thank Dr. Peter Diaczuk for his unfathomable knowledge of firearms and gunpowder and his assistance in obtaining smokeless powders.

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