B85 The Effect of Water Immersion on the Analysis of the Organic Additives in Smokeless Powder

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Attendees will learn about what organic additives are present in smokeless powders, about how these additives may be analyzed by GC/MS and about how the analysis of the organic additives may be adversely affected by environmental conditions.

This presentation will impact the forensic community by encouraging forensic chemists to explore the effects of environmental conditions on post-blast explosive residues.

Forensic chemists frequently analyze post-blast debris from pipe bombs for traces of smokeless powder. If smokeless powder grains are found they may be analyzed by Fourier transform infrared (FTIR) spectrometry to demonstrate the presence of the energetic compound nitrocellulose. Organic additives in the smokeless powder grains (for example, stabilizers such as diphenylamine and ethyl and methyl centralite) may also be analyzed by gas chromatography, gas chromatography-mass spectrometry or micellar electrokinetic capillary electrophoresis (MECE). The organic additive suites in smokeless powders are useful for determining the brand and lot of smokeless powder from which the explosive filling of the bomb came. Environmental conditions may adversely affect such analyses. This research examines the effect of exposure of smokeless powder grains to water on the quantities of diphenylamine and methyl dinitrobenzene isomers extracted from the grains.

Samples of eight different smokeless powders were immersed in distilled water for up to four weeks. Four of the powders were reload powders from IMR Powder Company (Plattsburgh, NY): SR 4759 (lot # L11208), IMR 4895 (lot # L7927), IMR 7828 (lot # L8208) and PB (lot # L14547). Three were reload powders from Hodgdon Powder Company (Shawnee Mission, KS): Hodgdon HS-7, Hodgdon BL-C(2) and Hodgdon Titewad. The remaining powder was Solo 1000 reload powder from Accurate Arms Company, Inc. (McEwen, TN). Small (4-6 mgm) samples of each powder were carefully weighed and then immersed in 1 mL of distilled water for two and four weeks. At the end of the immersion periods, the water was carefully removed from each sample vial; the smokeless powder grains were then briefly rinsed with 100 µL of methanol; and finally 100 µL of a 0.1% (w/v) methanolic solution of octadecane were added to each sample vial. After overnight extraction, 1-µL aliquots of the methanolic extracts were analyzed by gas chromatography-mass spectrometry using a Varian Saturn 2000 GC/MS system equipped with a Varian CP-SIL 8 capillary column. The helium carrier gas flow rate was 1.0 mL/min. The injector temperature was 250°C and a 1:20 split ratio was used. The following oven temperature program was used: The start temperature was 90°C; the temperature ramp was 16°C/min; and final temperature was 230°C. The mass spectrometer scanned the mass-to-charge ratio range from 20 to 330. The ratios of the peak areas of diphenylamine and the energetic plasticizers 2-methyl-1,3-dinitrobenzene, 1-methyl-2,4-dinitrobenzene, 4methyl-1,2-dinitrobenzene and 2-methyl-1,3,5-trinitrobenzene to the peak area of the octadecane internal standard were determined.

The quantities of the energetic plasticizers and diphenylamine extracted from the smokeless powder grains generally declined as the period of immersion in water increased. However, for some of the smokeless powders the quantities of organic additives extracted first declined and then increased. This may be a real effect: the smokeless powder grains may break down, exposing more surface area to the methanol extractant. Because the method for quantitating the organic additives in the smokeless powder samples may have been subject to significant error, the ratios of the peaks representing the methyl dinitrobenzene isomers to the diphenylamine peak were also examined. For two of the powders (IMR 4895 and IMR 7828) the ratio of the aggregate areas of the peaks representing the energetic plasticizers to the diphenylamine peak fell significantly over the course of the experiment (in the case of the IMR 4895 powder by more than a factor of two). Most of the decline occurred in the first week of the experiment. The decline in the quantity of the methyl dinitrobenzene isomers extracted from the powder particles after water immersion may be due to the fact that the solubilities of the methyl dinitrobenzene isomers in water are higher than the solubility of diphenylamine. Changes in the relative amounts of organic additives detected in smokeless powder grains would have a negative impact on the determination of the brand and lot of smokeless powder used in a pipe bomb.

Explosives, GC/MS, Smokeless Powder

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