

Rapid Identification of Smokeless Powders

Introduction

Smokeless powders (Figure 1) are often used in improvised explosive devices. The formulations for smokeless powders vary between manufacturers and between brands from a given manufacturer; ingredients include energetics, stabilizers, plasticizers and deterrents. Both chemical composition and morphology are important in characterizing smokeless powders.

Chemical analysis of smokeless powders can provide valuable forensic evidence. Observation with the SEM can reveal morphological information to help with identification. Here we show how SEM-EDS analysis can be used to identify inorganic components, and how the AccuTOF-DART mass spectrometer can rapidly identify the organic components and provide a chemical fingerprint that can be used to identify individual powder particles.



Figure 1. Several smokeless powders viewed under an optical microscope.

Experimental

Mass Spectrometry

The AccuTOF™ mass spectrometer was operated in positive-ion mode. Polyethylene glycol (PEG-600) was measured as a reference standard within each data file, but separate from the smokeless powder particle measurements. Individual particles sampled with vacuum tweezers were positioned in the DART gas stream for analysis (Figure 2). Disposable vacuum tweezers were constructed by passing a glass capillary through an Eppendorf pipette tip and inserting the pipette tip into a rubber hose connected to a low-vacuum pump. Data were acquired by using JEOL Mass Center software and mass spectra were processed by TSSPro3 software (Shrader Software Solutions, Detroit, MI).

The DART ion source was operated with a gas heater temperature setting of $\leq 200^{\circ}\text{C}$ to avoid damaging or detonating the smokeless powder particles. Individual samples could be analyzed within seconds. As long as the gas heater temperature did not exceed 200°C , the samples were not consumed and could be examined or reanalyzed at a later time.

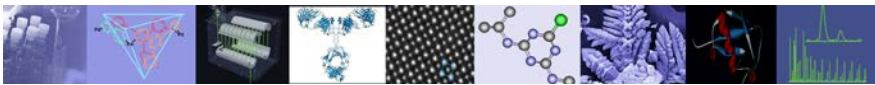


Figure 2. AccuTOF-DART analysis of a smokeless powder particle.

A list of target compounds commonly used in smokeless powders was created in a Microsoft Excel spreadsheet. The spreadsheet contained the names and elemental compositions of the target compounds. The target compound spreadsheet was used by Mass Mountaineer software (www.shop.mass-spec-software.com) to identify compounds in the smokeless powder mass spectra based upon exact masses and isotopic data. The Mass Mountaineer program also allowed us to create and search mass spectral databases created from the mass spectra of smokeless powder standards.

Scanning Electron Microscopy

Examination by SEM-EDS (Figure 3) is a non-destructive test method that can provide additional insight to complement data obtained with the AccuTOF-DART. SEM images not only provide information on size and shape but also can give clues to chemical makeup when imaged with backscatter electron detector. Energy-dispersive X-ray Spectroscopy (EDS) analysis provides additional information on the elemental composition and in some instances spatial distribution. Where AccuTOF-DART readily identifies the organic components, SEM-EDS can provide information on the presence of inorganic components. The information coupled together can provide valuable information in forensic investigations.

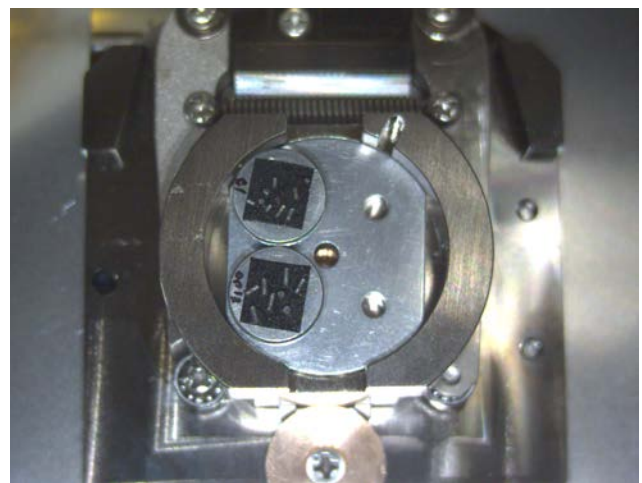
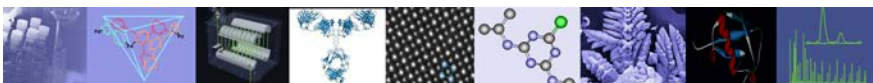


Figure 3. InTouchScope SEM used to examine a smokeless powder particle.



Results

The AccuTOF-DART mass spectra for several smokeless powders are shown in Figure 4a and 4b. The DART mass spectra show unique patterns of organic components typically detected as protonated molecules $[M + H]^+$. Because some samples from the same manufacturer showed the same organic components, but had different morphology, it appears that both morphology and DART analysis will be required for a unique identification of a specific powder.

Because labeled compounds were tentatively identified solely by exact mass and isotopic data, the presence of isomers cannot be ruled out. Regardless, the mass spectra are reproducible and distinctive, making it possible to identify unknown particles by comparison against database spectra.

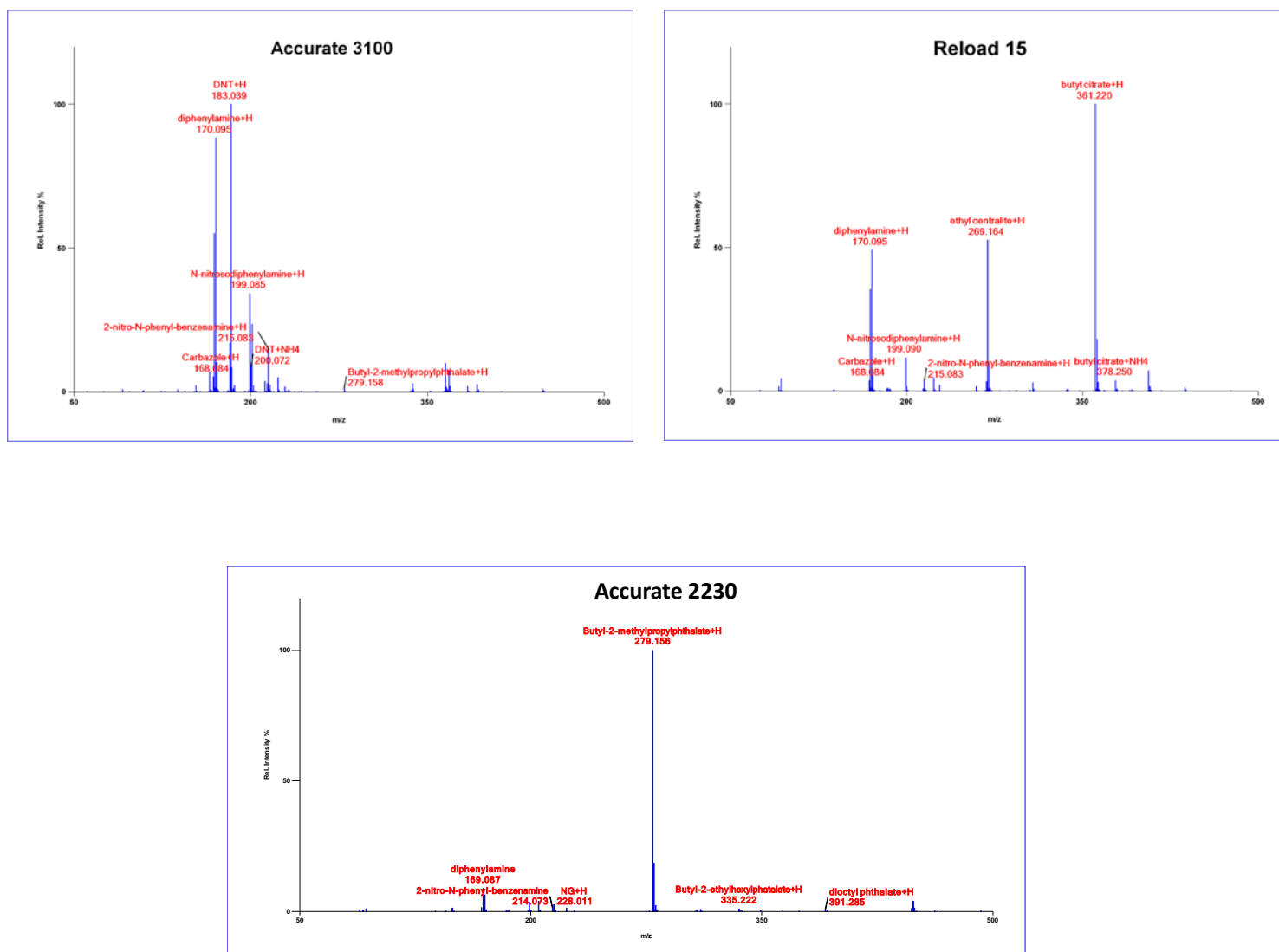
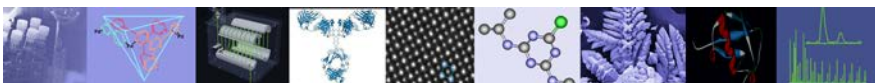


Figure 4. (a) AccuTOF-DART mass spectrum of an Accurate 3100 smokeless powder particle; (b) AccuTOF-DART mass spectrum of a Reload 15 particle, c) AccuTOF-DART mass spectrum of an Accurate 2300 smokeless powder particle.



InTouchScope SEM imaging of the smokeless powder particle identified as Accurate 2230 (Figure 5) revealed a brighter area when the image mode was switched from secondary electron to backscattered electron detector. The bright area observed was indicative of a component with higher average atomic number. Subsequent analysis by JEOL EDS indicated that the bright areas were high in potassium.

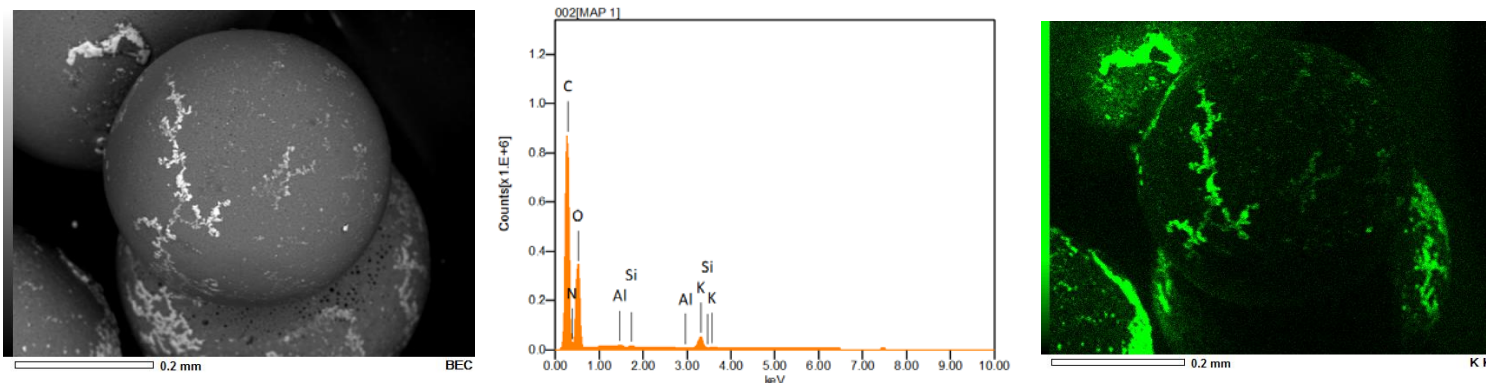


Figure 5. (a) Backscatter Electron SEM image of Accurate 2230 smokeless powder particle; (b) EDS analysis indicating bright areas are high in potassium; (c) X-ray intensity map for potassium.

Conclusions

The AccuTOF-DART mass spectrometer is capable of rapid fingerprinting of organic compounds in smokeless powders. Although further work needs to be done to expand the database and validate the method, the AccuTOF-DART mass spectra (in combination with particle morphology) offer a rapid and potentially nondestructive approach for the identification of individual smokeless powder particles.

The SEM provides not only high magnification imaging for morphological identification, but analytical techniques such as EDS.

These complementary techniques can be used for rapid positive identification of trace evidence such as smokeless powders.

Acknowledgement

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