X-Ray Fluorescence Helps Identify Peaks in DART Mass Spectrum of Electrical Tape - ElementEye JSX-1000S and AccuTOF-DART

LC and Ambient Ionization HRTOF Mass Spectrometer JMS T100LP AccuTOF-DART™

X-ray Fluorescence Spectrometer JSX-1000S ElementEye

Introduction

The identification of electrical tapes is important for forensic investigation of improvised explosive devices [1]. Pyrolysis mass spectrometry [2] and X-Ray Fluorescence (XRF) [3] are among the methods that are used for the forensic analysis of electrical tapes.

Direct Analysis in Real Time (DART) can be operated with a high gas temperature as an alternative to conventional pyrolysis GC/MS methods [4,5]. A sample of electrical tape analyzed with the AccuTOF-DART™ showed distinctive peaks in the negative-ion DART mass spectrum. No reasonable elemental compositions could be determined by assuming the presence of only the common organic elements: C, H, N, O, P, S, Cl, Si, and Br. X-ray fluorescence (XRF) data obtained with the ElementEye™ indicated the presence of Zn and Sb, allowing us to correctly assign the elemental compositions for the peaks observed in the DART mass spectrum.

Experimental

A piece of electrical tape was placed in the DART gas stream with the DART gas heater set to 500°C (pyrolytic DART conditions). A sample of poly(perfluoropropyl ether) was measured in the same data file as a mass reference standard for exact mass measurements. Elemental compositions with isotope matching were determined by using Mass Mountaineer™ software. The XRF spectrum of the sample was measured with the Element Eye by using the Quick and Easy Organic Analysis method and the ElementEye reporting program. The collimator was set to 2 mm and the total analysis time was 60 seconds.

Results and Discussion

The negative-ion DART mass spectrum in Figure 1 shows distinctive peaks with isotope patterns that suggest the presence of multiple halogens (chlorine and/or bromine). This is not unexpected considering that electrical tape can be made of polyvinyl chloride (PVC). However, no reasonable elemental composition assignments could be made by assuming only elements present in common organic polymers. To assign the elemental compositions, we needed additional information about which other elements might be present.

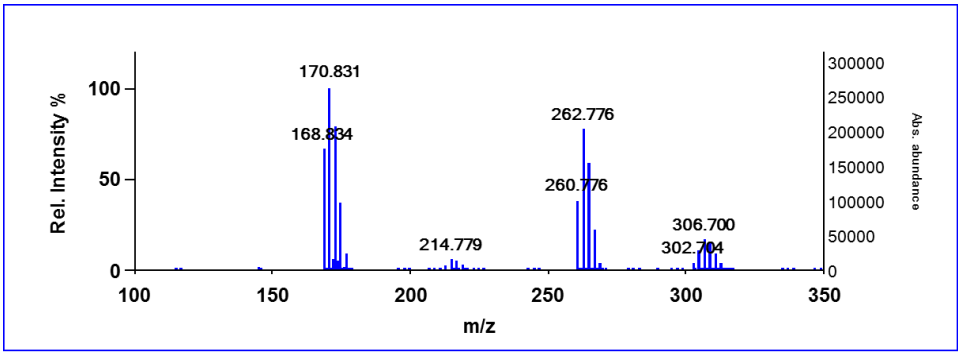

Fig. 1 Negative-ion AccuTOF-DART mass spectrum of a piece of electrical tape.

Among the elements detected in the XRF spectrum (Figure 2, Table 1) are antimony, zinc, chlorine and bromine. Adding these elements to the constraints for the elemental composition calculation for the AccuTOF-DART data allows us to correctly assign the elemental compositions for these peaks (Figure 3). The measured isotope peaks show excellent agreement with the calculated isotope patterns (Figure 4).

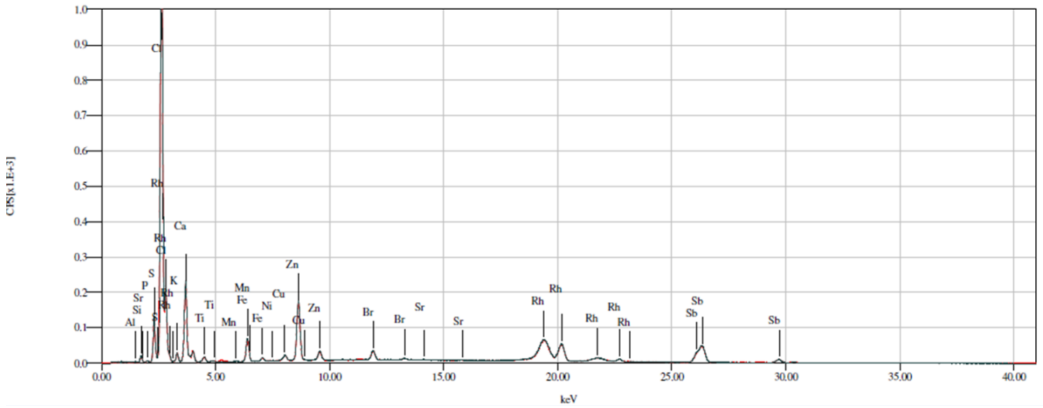

Fig. 2. XRF Spectrum of the electrical tape obtained with ElementEye

Analysis Target	Result	Unit	3sigma
Aluminium(Aluminu	1.01	%	0.30
Iron	0.18	%	0.00
Manganese	0.00	%	0.00
Nickel	0.00	%	0.00
Potassium(Kalium)	0.43	%	0.01
Silicon	1.38	%	0.05
Strontium	0.00	%	0.00
Titanium	0.10	%	0.00
Antimony(Stibium)	1.87	%	0.02
Copper	0.03	%	0.00
Zinc	0.27	%	0.00
Calcium	2.58	%	0.02
Sulfur	1.13	%	0.01
Phosphorus	0.09	%	0.01
Bromine	0.03	%	0.00
Chlorine	19.21	%	0.06

Table 1. Elements detected by the ElementEye

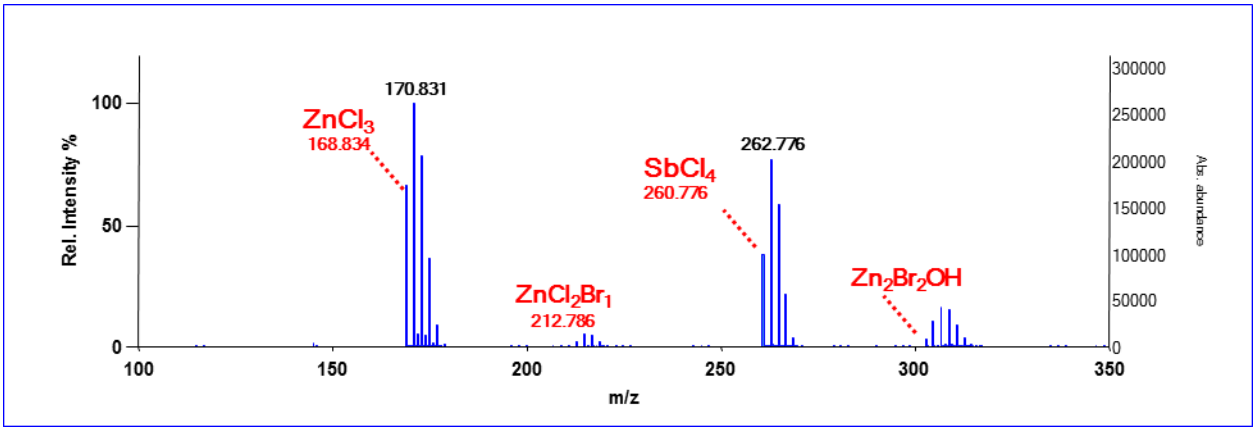

Fig. 3. Elemental composition assignments for the peaks in the mass spectrum from Figure 1 calculated after determining the presence of Zn, Sb, Cl, and Br from the XRF data.

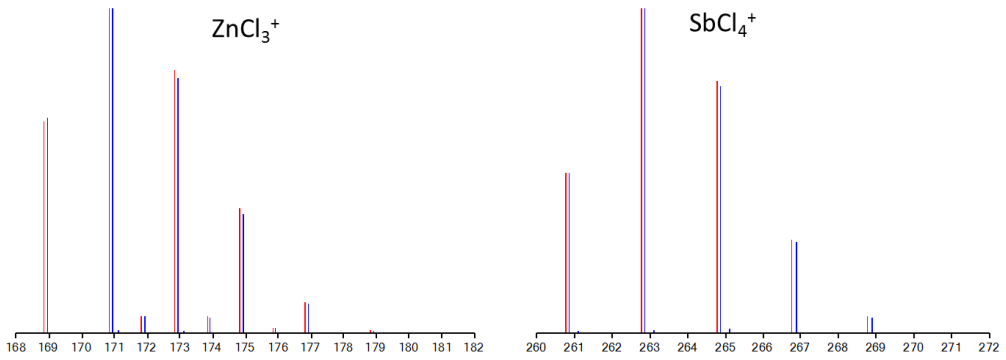

Figure 4. Comparison of calculated (red) and measured (blue) isotope peaks for $ZnCl_3^+$ and $SbCl_4^+$

Conclusion

Elemental composition assignments based on accurate mass and isotope measurements by mass spectrometry require the operator to provide a list of elements that may be present and their limits. If an element is omitted from the list, the correct assignment will not be reported. If too many elements are added to the list, the number of possible compositions becomes uninterpretable. The ElementEye is a rapid and convenient tool that provides complementary elemental information allowing us to assign the unknown peaks in the AccuTOF-DART data for the electrical tape sample.

References

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5. Lancaster, C.; Espinoza, E., Analysis of select Dalbergia and trade timber using direct analysis in real time and time-of-flight mass spectrometry for CITES enforcement. *Rapid Communications in Mass Spectrometry* **2012,** *26* (9), 1147-1156.