Polymer and adhesive tape analysis by thermal desorption and pyrolysis combined with Direct Analysis in Real Time (DART) mass spectrometry

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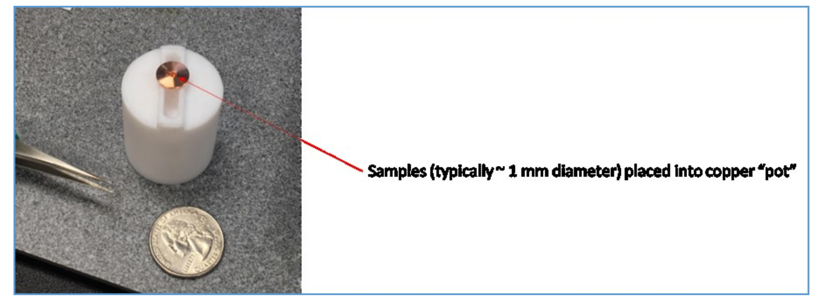
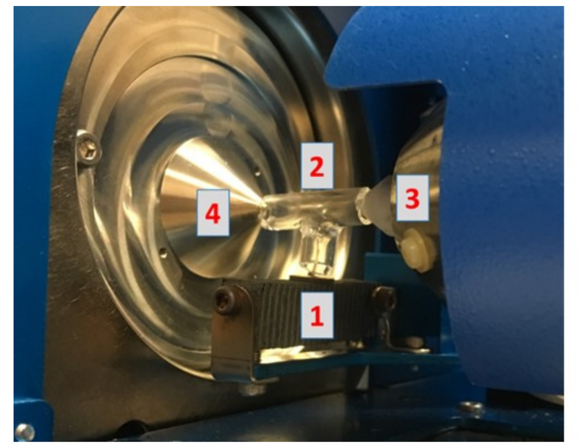
Introduction

Direct Analysis in Real Time (DART) ionizes materials through interactions between long-lived excited-state species and atmospheric gases and analytes. The DART gas stream can be heated to facilitate desorption of low-volatility compounds, or to induce thermal degradation and pyrolysis. However, controlled heating of the sample rather than the gas provides much better temperature control than using a heated gas stream. By using a sample heater with a well-regulated and reproducible temperature profile, we recorded thermal desorption profiles of materials in complex samples and separated components with different desorption profiles. Here we report the analysis and identification of polymers and adhesive tapes (electrical tapes and duct tapes) from their thermal desorption/pyrolysis profiles and the corresponding DART mass spectra.

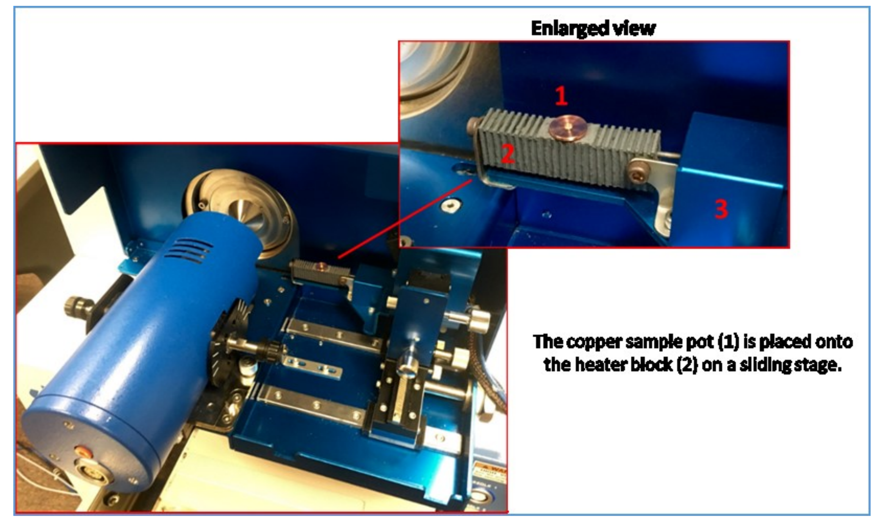
Methods

Mass spectra were obtained by using an AccuTOF-DART (JEOL USA, Inc.) time-of-flight mass spectrometer equipped with a DART ion source (IonSense LLC) and an ionRocket thermal desorption and pyrolysis unit (Biochromato, Inc.). The ionRocket was mounted directly between the DART ion source and the mass spectrometer sampling orifice both with and without using a Vapur gas transfer interface (IonSense LLC). A ceramic "chimney" was used to guide sample gases into the gas stream with the Vapur installed. A glass tee was used to sample gases when the Vapur was omitted. Mass spectra were measured in positive-ion mode at a resolving power of 10,000 (FWHM) as the samples were heated from ambient temperature to 600°C at a rate of 100°C min^{-1}.

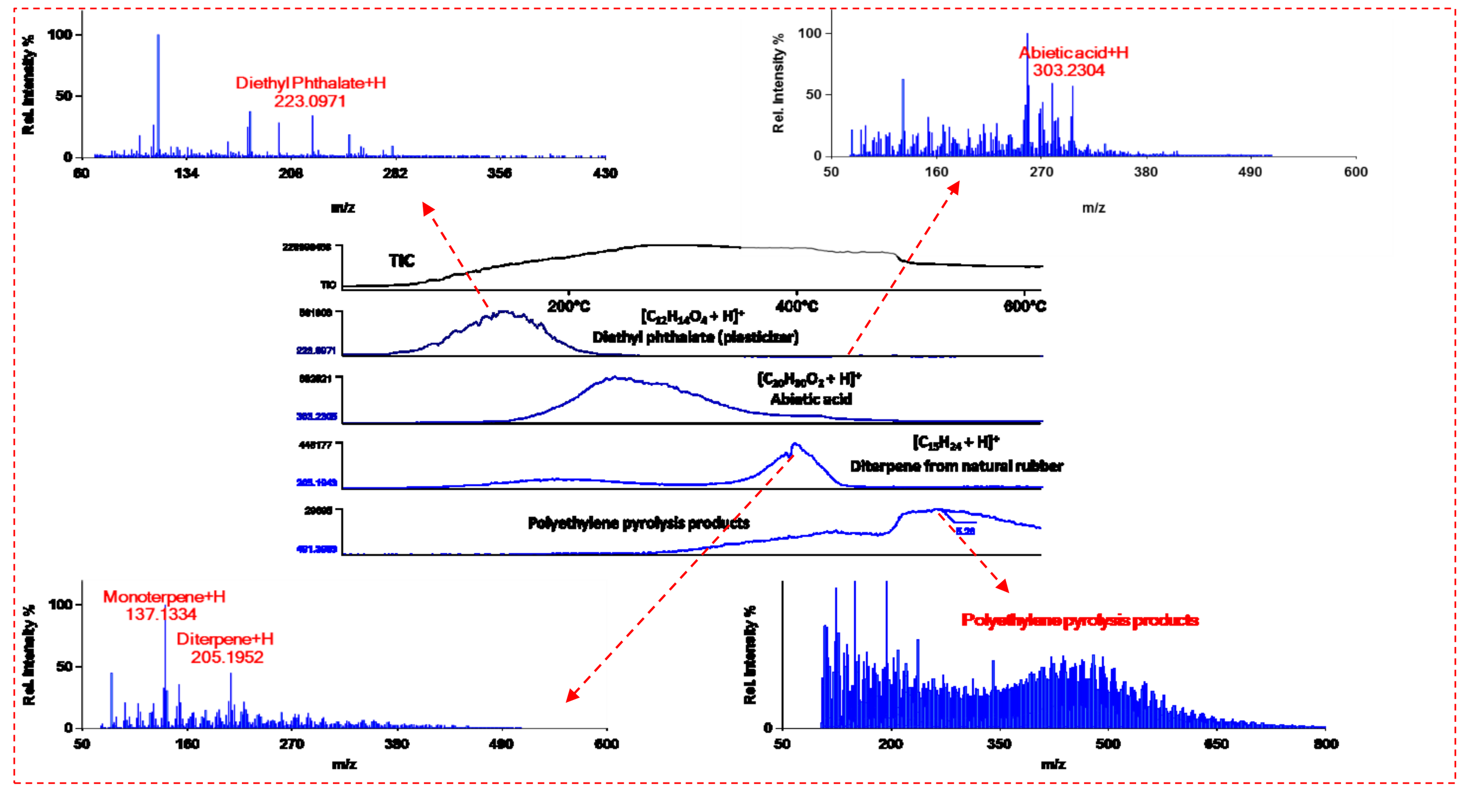




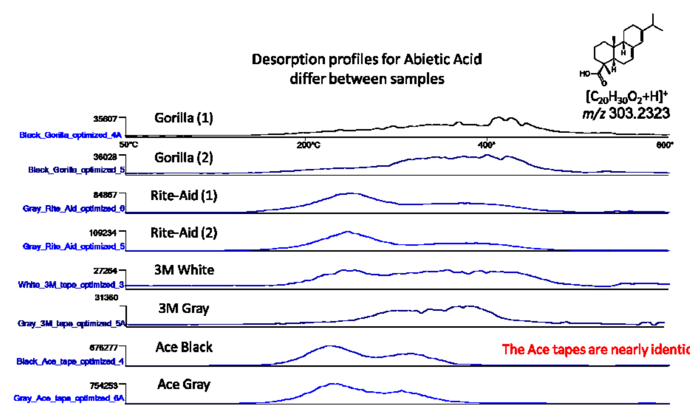
The sample pot and heater block (1) slide into position below a glass tee (2) mounted between the DART exit (3) and the mass spectrometer sampling orifice (4).

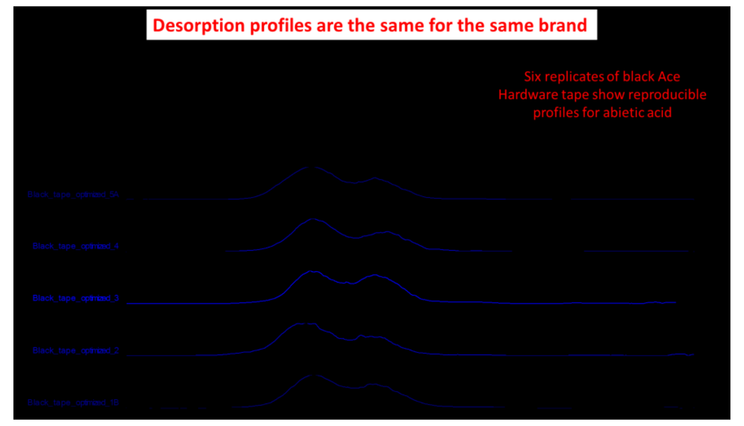


Results



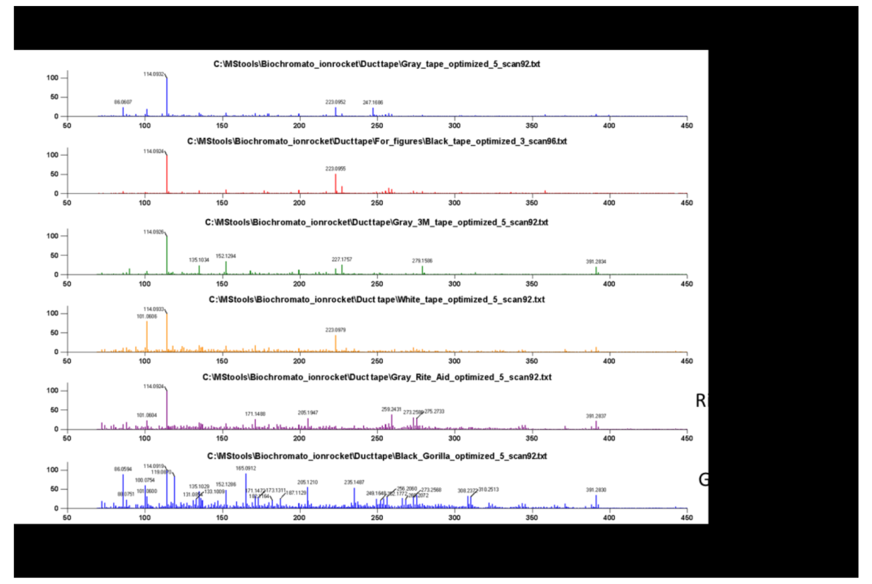
TDP/DART RIC's for selected components and mass spectra at different temperatures

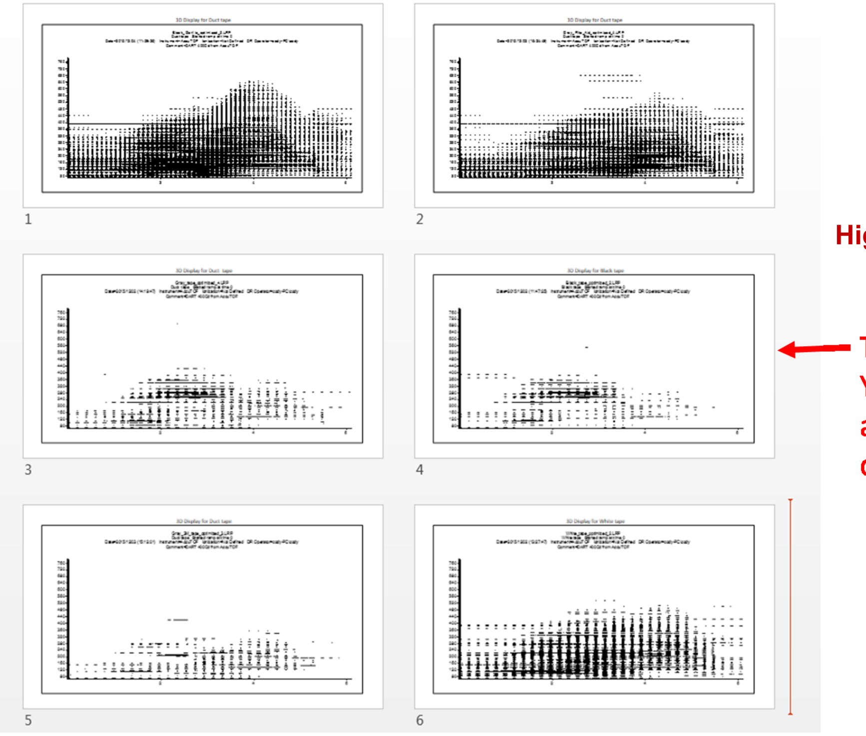




Thermal desorption profiles for abietic acid in different tape brands (left) and the same tape brand (right).

Mass spectra for volatile components in duct tapes

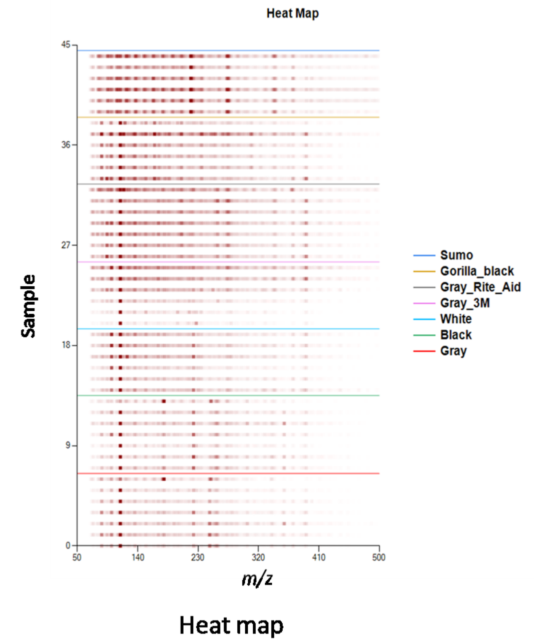




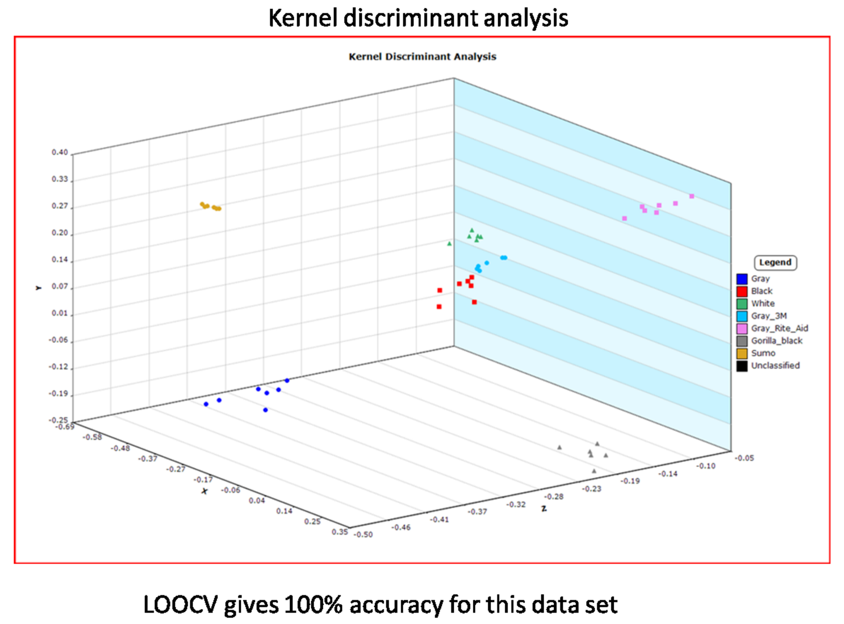
3D plots
x-axis: time/temperature
y-axis: *m/z*
Higher abundance = darker spots.

These two are the same tape. You can see that they look alike, but the other plots look different.

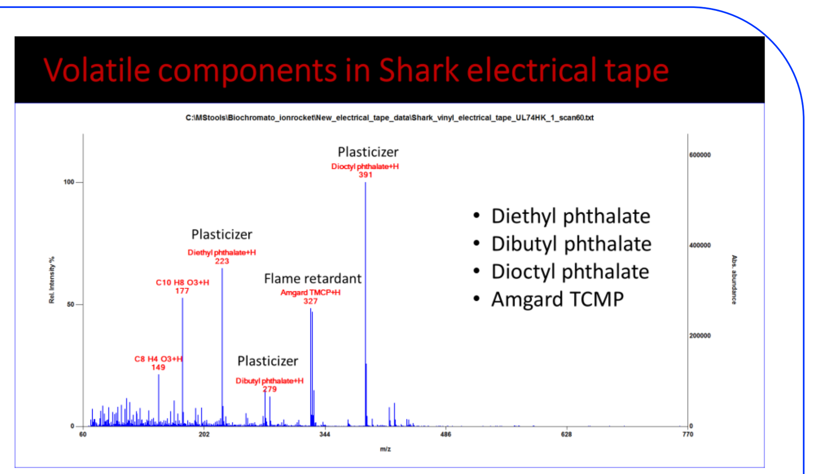
Chemometrics identify the tape manufacturer and brand

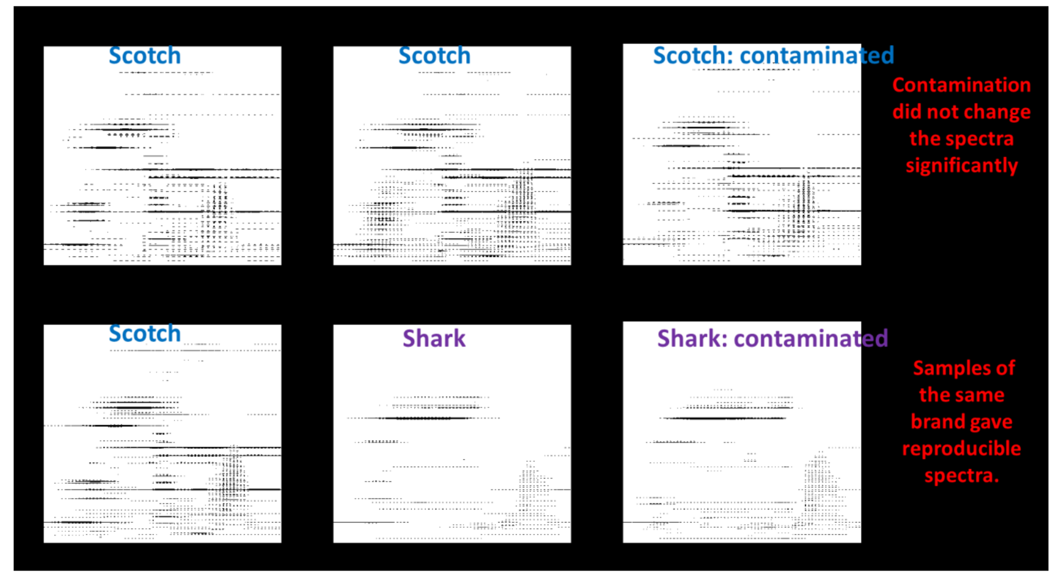


Heat map



LOOCV gives 100% accuracy for this data set





3D plots for TDP/DART analysis of electrical tapes
One sample of each tape was deliberately exposed to lab surfaces and human skin.

Conclusions

- Thermal desorption profiles for TDP/DART with the ionRocket showed excellent reproducibility
- Thermal desorption permitted temperature-dependent separation of plasticizers and additives, resins, adhesives, and the base polymer
- The pyrolysis mass spectra allowed us to identify the base polymers and minimized suppression from additives with high proton affinities.
- Nonpolar polymers like polyethylene, polypropylene, polyvinyl chloride could be identified with the glass tee interface, but ion-molecule reactions cause a loss of polymer signal with the Vapur interface.
- 3D plots provided a convenient means to visualize the total TDP/DART analysis
- Chemometric analysis allowed us to identify individual manufacturers and brands for the duct tapes tested
- Deliberate contamination of the electrical tapes by exposing them to lab surfaces and human skin had minimal effect on the 3D plots for electrical tapes.