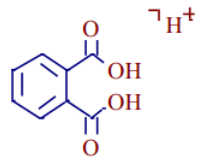
ionSense®
Your source for open air mass spectrometry.

Phthalate Fragmentation with DART-MS

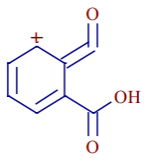
Introduction

Plasticizers have become a staple in the production of consumer products, home improvement supplies, and almost any synthetic plastic polymer manufactured throughout the past century. With any product made of plastic, plasticizers are almost always involved. One class of plasticizers, called phthalates, is what allows polyvinyl chloride (PVC) to become malleable and soft; however, studies conducted in recent years have put these phthalates into the spotlight. Controversy over the potential health effects of these chemicals has caused worry for some consumers, and governments have begun placing restrictions on manufacturers as a result.

In this study, we have used DART-MS (Direct Analysis in Real-Time – Mass Spectrometry) to analyze eight phthalates, including DINP, DOTP, DIBP, DBP, BBP, DEP, DIDP. Four of these, DINP, DBP, BBP, and DIDP, are restricted or prohibited in some products and children's toys in Europe and the state of California. Applying high voltages in the form of CID (Collision Induced Dissociation) to fragment the phthalates allowed us to learn the fragmentation patterns created by these chemicals and apply the patterns to the identification of phthalates in products. Two commonly formed phthalate fragments are:



167.03 m/z



149.02 m/z

However, we have focused on the fragments that vary from phthalate to phthalate, which is exactly what gives each its own identity with DART-MS.

Results

Each phthalate was made into 0.1% and 1% solutions in hexane. DIBP and DOTP (two structurally different molecules) were analyzed with a thermal profile to determine an optimal temperature of 300˚C.

Each 1% solution was then analyzed with DART-MS at the optimal temperature, under various voltages. This analysis was performed using DART-QDa, a small, compact MS capable of easily detecting a variety of molecules. The instrument is pictured below.

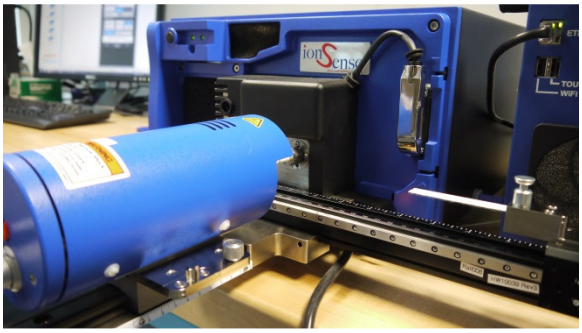


Fig.1: DART-QDa

The mass spectra below display BBP unfragmented, and then with increasing collision energies until only 147 m/z remains as a peak.

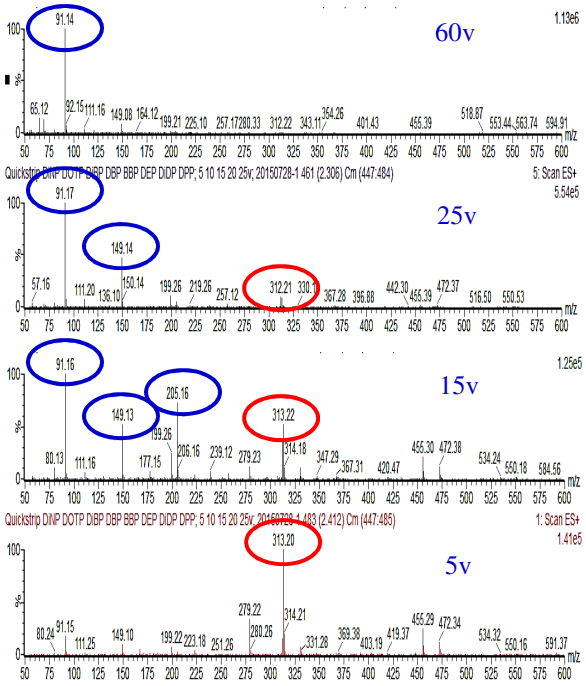


Fig.2: DART-MS spectra of 1% BBP fragments

The second set of spectra below show the fragmentation of DIDP as the collision energy increases in increments of 5v.

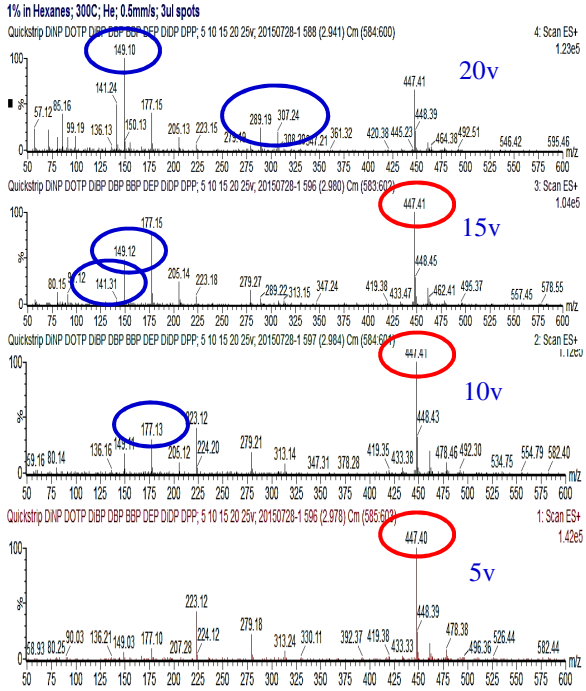


Fig.2: DART-MS spectra of 1% DIDP fragments

Analysis of Plastics

Various products, such as plastic bags, paint samples, and dated plastic children's toys, were analyzed with DART-MS. Various phthalates were detected in these samples, and we determined that one product actually contained a restricted phthalate. The most abundant peak in the mass spectrum of the plastic microscope in a children's C.S.I. kit was DINP, an antiadrogen known to cause liver effects. Below are the spectra from the sample and the DINP standard.

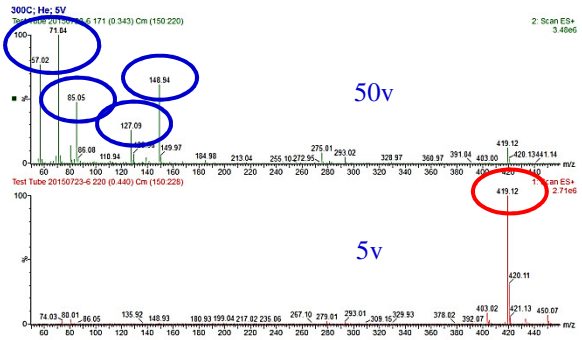


Figure 3: DART-MS of Microscope Base, both fragmented (top) and unfragmented (bottom)

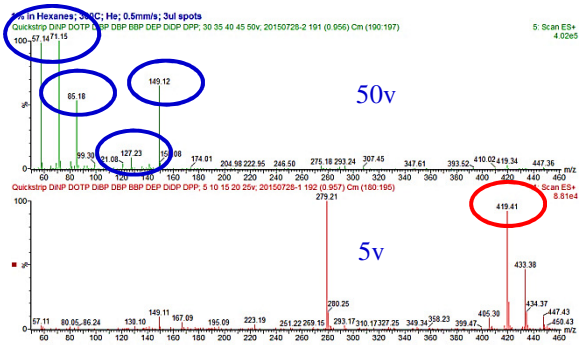


Figure 4: DART-MS of 1% DINP Standard, both fragmented (top) and unfragmented (bottom)

Experimental

Procedure:

- Solutions containing a phthalate (DINP, DOTP, DIBP, DBP, BBP, DEP, DIDP, DPP) in hexane at 1% and 0.1% were created
- Quick Strip cards of 1% DIBP and 1% DOTP analyzed with DART-MS thermal profile in positive-ion mode, applying 5 V, using helium gas heated from 150 - 300˚C in increments of 50˚C
- All phthalates were analyzed on a Quick Strip card with DART-MS in positive-ion mode, using helium gas heated to 300˚C, applying every increment of 5 V from 5 - 75 V

Conclusion

DART-MS was successfully able to detect phthlates at both 1% and 0.1% concentrations, as well as several of their fragments, with the application of high voltages. Through studying the fragmentation patterns of these chemicals with DART-MS, we were able to identify the presence of these phthalates in other samples and products. The ability to detect chemicals such as these in various types of samples is key to ensuring the purity and safety of consumer products.

Doc # 7.5.124 Rev 1