Fiber analysis by thermal desorption/pyrolysis DART®

Introduction

Analyzing fiber samples has always been difficult by DART®. The problem has been that there is no easy way to hold the fiber in the gas stream without losing it into the vacuum system. A fiber can be secured in the DART gas stream with forceps or other means, but if the DART gas is too hot, the fiber can break off and be lost into the mass spectrometer vacuum system through the atmospheric pressure interface.

A thermal desorption/pyrolysis stage (The Biochromato, Inc. "ionRocket™") designed for use with DART produces highly reproducible thermal desorption profiles that show outgassing, additives, and high-quality pyrolysis DART mass spectra for materials. Because fiber samples placed in the disposable copper sample "pots" are not positioned directly in the DART gas stream, a single fiber can be analyzed without risk of loss into the vacuum system.

Experimental

Mass spectra were acquired by using a JEOL *AccuTOF™-DART® 4G* mass spectrometer (Figure 1) equipped with a Biochromato, Inc. *ionRocket* thermal desorption and pyrolysis system (http://biochromato.com/ionrocket/).

Figure 1. The ionRocket thermal desorption/pyrolysis system mounted on the AccuTOF-DART 4G mass spectrometer

A single fiber or a small clump of fibers was placed into a disposable copper sample stage (or "pot") for the *ionRocket* (Figure 2).

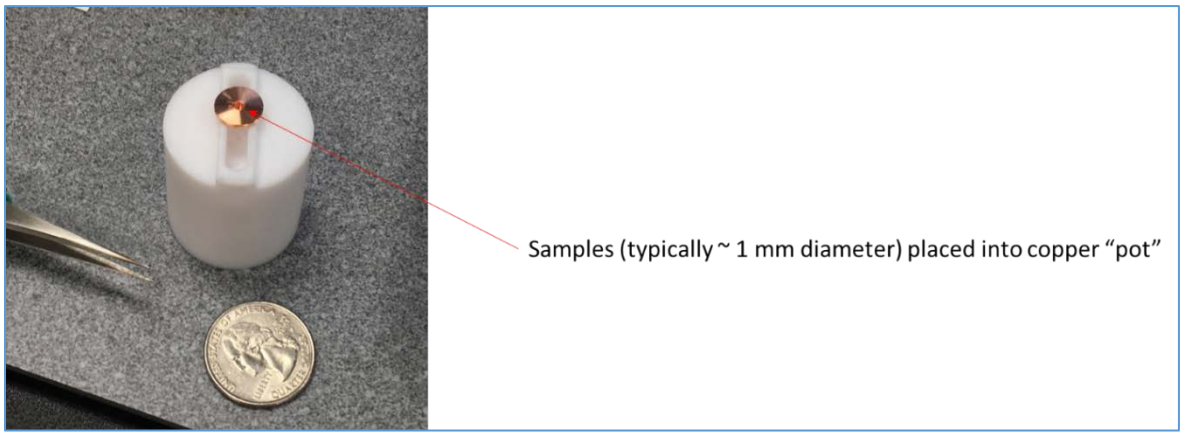


Figure 2. A copper pot used as a sample holder for the ionRocket

The copper sample stage was placed onto the *ionRocket* heater (Figure 3) and moved into position between the exit of the DART ion source and the sampling orifice of the *AccuTOF-DART 4G* mass spectrometer. A glass tee positioned above the sample (Figure 4) guides the thermal desorption and pyrolysis products into the DART gas stream.

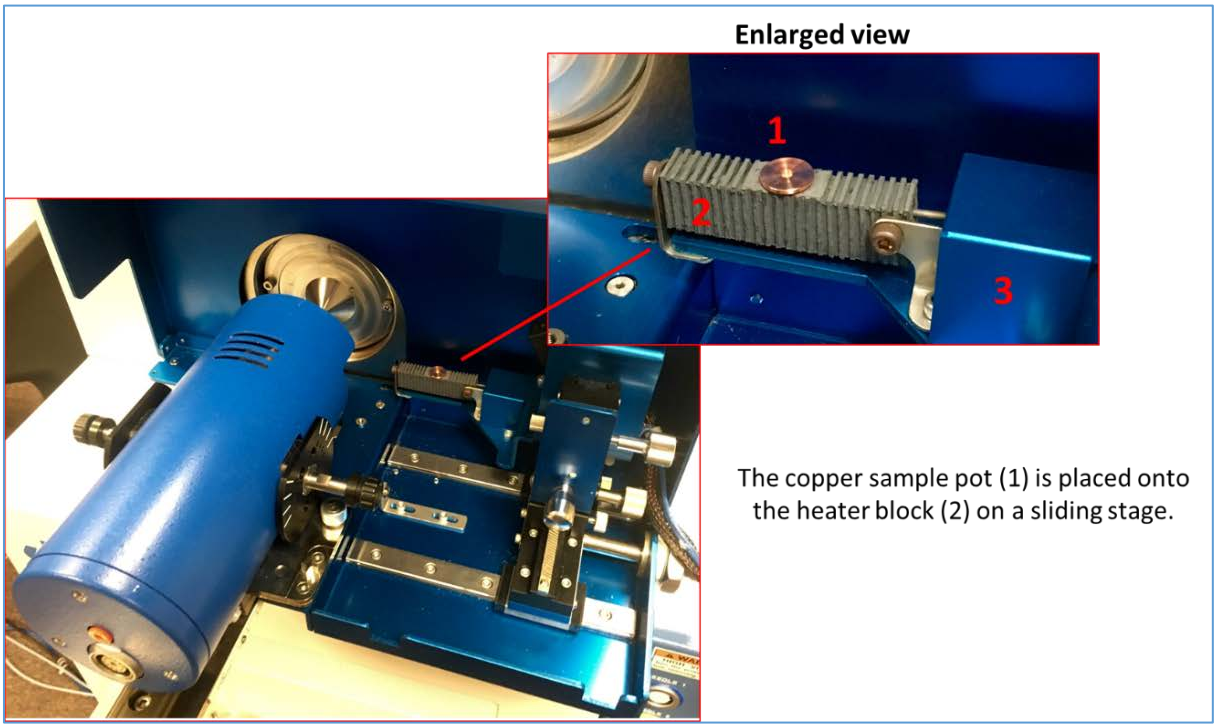
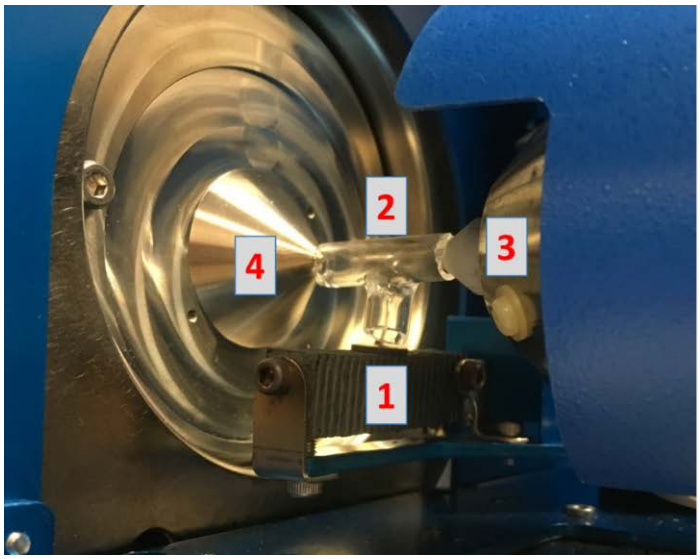


Figure 3. A sample mounted onto the ionRocket heater block.



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).

Figure 4. A sample mounted on the heater block, positioned below a glass tee.

The temperature ramp was programmed from ambient temperature to 600°C at a rate of 100°C min-1. Mass spectra were acquired at a resolving power of 10,0000 in positive-ion mode at a spectral acquisition rate of 1 spectrum per second for the *m/z* range 50-1000.

Results

Carpet fiber

A small tuft of fibers from the carpet in the JEOL booth at PittCon 2016 was placed onto a copper sample holder for the *ionRocket* and analyzed by the *AccuTOF-DART.* The thermal desorption profiles of selected components are shown in Figure 5. Oleamide, a common stabilizer shows a maximum abundance at temperatures below 200°C. The stabilizer 2,5-di-*t*-butylhydroquinone maximizes at temperatures just above 200°C, and high-*m/z* pyrolysis products from the polyolefin fiber maximize at temperatures above 400°C.

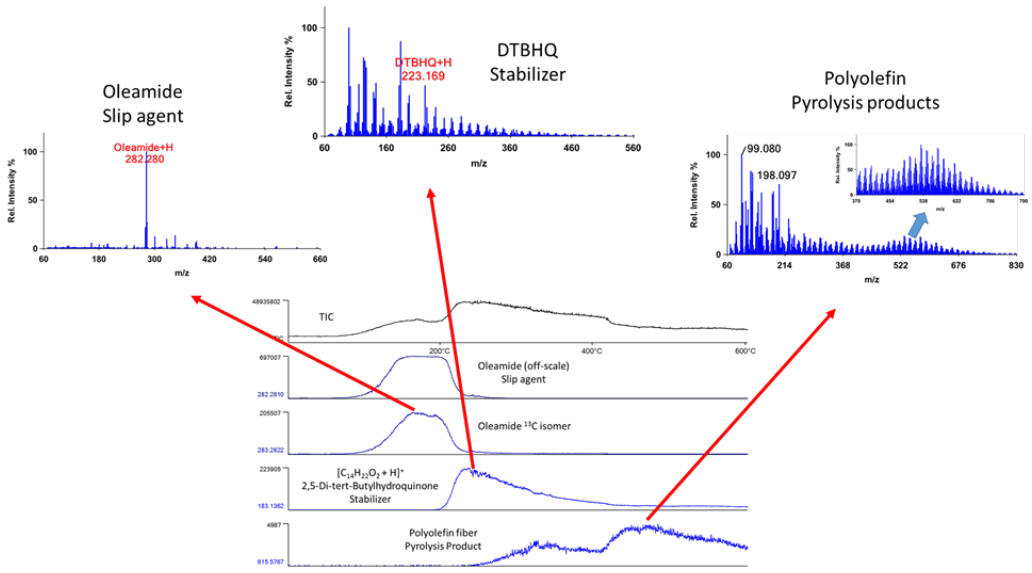


Figure 5. Thermal desorption profiles and mass spectra for components in carpet fiber.

Fiber from a badge holder lanyard

A single fiber from the badge holder lanyard from PittCon 2016 was placed onto a copper sample holder for the *ionRocket* and analyzed by the *AccuTOF-DART* (Figure 6). At temperatures below 300°C, we detect additives such as dibutyl phthalate plasticizer. At higher temperatures, we detect pyrolysis fragments from the polyethylene terephthalate (PETE) polymer.

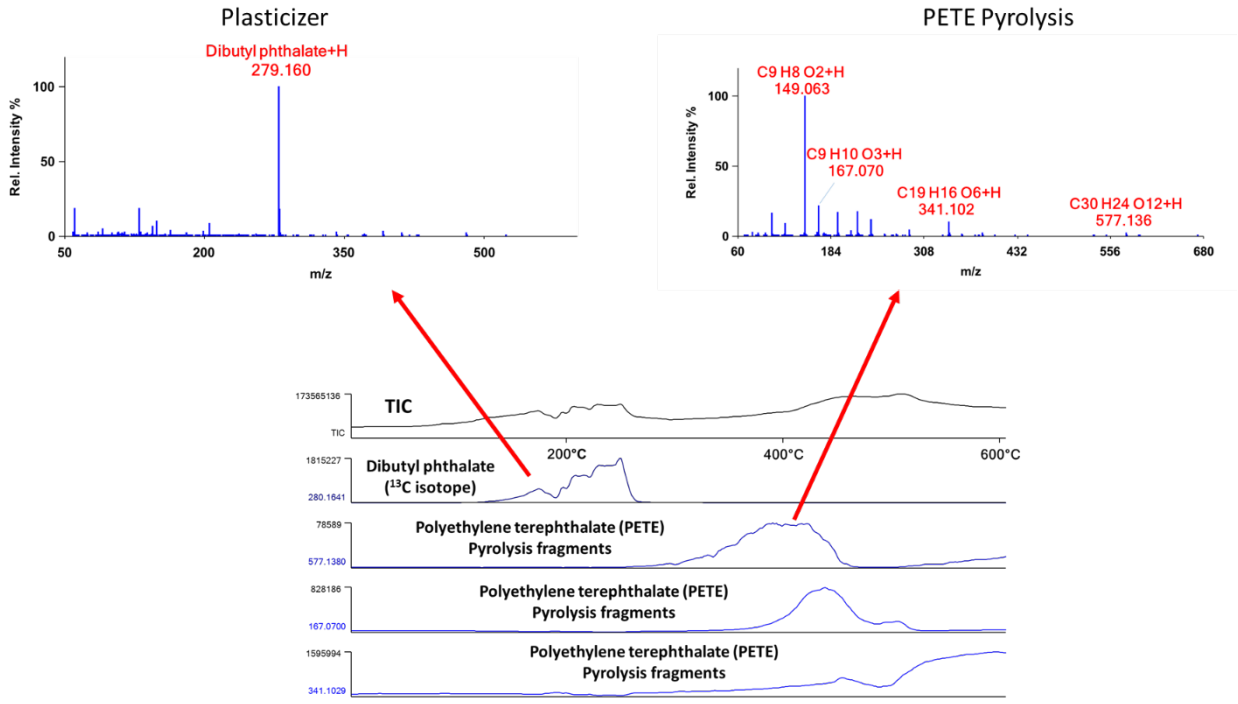


Figure 6. Thermal desorption profiles and mass spectra for components in a badge holder lanyard.

Conclusion

Thermal desorption/pyrolysis with the *ionRocket* offers a convenient solution for the analysis of single fibers with the *AccuTOF-DART*. Accurate-mass and isotopic data measured by the *AccuTOF-DART* system allows us to detect additives and identify the base polymer. Because the fiber is not held directly in the DART gas stream, there is no risk of losing the fiber into the mass spectrometer vacuum system if the fiber decomposes upon heating.

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