Temperature-regulated Thermal Desorption and Pyrolysis Device for DART®-MS System ~ ionRocket ~

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ionRocket

Direct analysis in real time-mass spectrometry (DART®-MS) enables nearly instantaneous determination of sample composition using mass spectrometry. Therefore, DART®-MS is a powerful method for analysis of sample mixtures. However, this method is not suitable for polymer analysis because many polymers are difficult to volatilize.

For polymer analysis using DART®-MS, we developed a peripheral device to DART®-MS, called "ionRocket" (Fig.1, 2), which induces thermal desorption and pyrolysis of samples. The vapor phase of polymer samples was generated by applying a temperature-controlled heating gradient, then ionized and introduced into the mass spectrometer. In this poster, we describe the analysis applications using the ionRocket combined with DART®-MS.

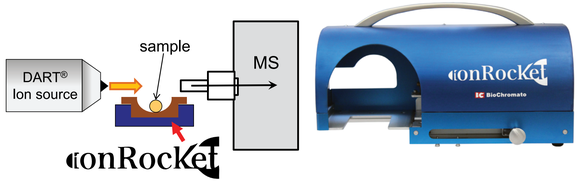


Fig.1 ionRocket

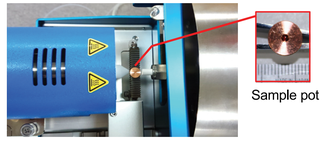


Fig.2 ionRocket combined with DART®-MS system

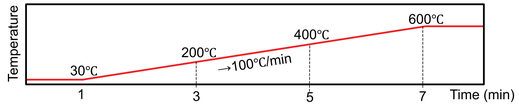


Fig.2 Example of heating condition of ionRocket

Application 1

SYNTHETIC FIBERS

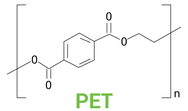
● INTRODUCTION

In order to analyze condensation polymers containing ester linkages, such as polyester fiber, we usually use reaction pyrolysis-GC/MS. However, this method requires a methylating agent to suppress thermal decomposition products.

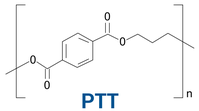
In this application, we used the ionRocket with DART®-MS to analyze polyester fibers without the use of a methylating agent.

● SAMPLES

Polyester fibers:
- polyethylene-terephthalate: PET
- polytrimethylene-terephthalate: PTT



Repeat structure: $C_{10}H_8O_4$ (Mw 192)



Repeat structure: $C_{11}H_{10}O_4$ (Mw 206)

● METHOD

Samples were analyzed with the ionRocket connected to the DART®-MS system. Polyester fibers were cut to 10 mm lengths and then placed into the sample pot (Fig.2). The samples were heated from room temperature to 600°C at a rate of 100°C/ min (fig.3).

● RESULT

Total ion current chromatograms of PET and PTT are shown in Fig.4. The total current profiles are different between PET and PTT. Mass spectra measured at 450°C are shown in Fig.5. In Fig.5, most of the detected peaks were due to species related by intervals of 192, which were derived from the repeated structure of PET, and species related by intervals of 206 derived from the repeated structure of PTT. Thus, it was determined that this method can identify and differentiate different polyester fibers without the use of a methylating agent.

In summary, ionRocket combined with DART®-MS enables rapid and easy identification of polyesters. Therefore, this method is useful for the field of research and development, as well as quality control. Moreover, this method should contribute to further investigation in the field of polymer chemistry.

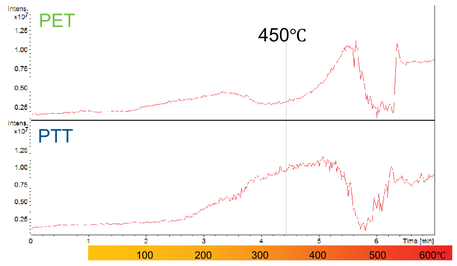


Fig.4 Total ion current chromatograms of PET and PTT

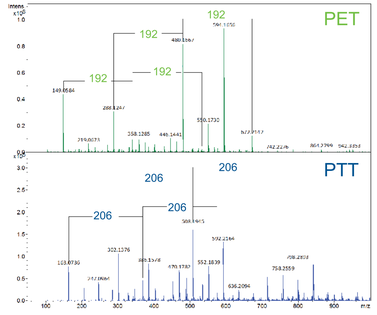


Fig.5 Mass spectra of PET and PTT

Application 2

TWO-PART EPOXY ADHESIVE

● INTRODUCTION

The hardening reaction of a two-part epoxy adhesive is initiated by blending an epoxy resin and a hardening accelerator. The analysis of hardened materials is often limited due to the difficulty in dissolving hardened materials. In this application, we describe the analysis of two-part epoxy adhesive without any pretreatment.

● SAMPLES Two-part epoxy adhesive (marketed production)

● METHOD

Samples were analyzed with the ionRocket, connected to the DART®-MS. The liquid epoxy adhesive solutions A and B were each put into the sample pot (Fig.2) and analyzed. The resulting hardened material after blending solution A and solution B was shaved off with a razor. Small quantities of hardened materials were also put into the sample pot and analyzed. The samples were heated from room temperature to 600°C at a rate of 100°C minute[-1] (Fig.3).

● RESULT

Mass spectra of solution A and solution B are shown in Fig.6. Bisphenol A diacetate and bisphenol A diglycidyl ether were detected as main compounds of solution A, tris(dimethylaminomethyl) phenol was detected as main compound of solution B.

Extracted ion current grams of 15 minutes and 45 minutes after blending are shown in Fig.7. In the sample of 15 minutes after blending, two main compounds of solution A (bisphenol A diacetate and bisphenol A diglycidyl ether) were detected around 130°C, whereas in the sample of 45 minutes after blending, no peaks were detected around 130°C. Therefore, it was determined that these two main compounds detected at 130°C were the reaction residuals. In addition, it was presumed that the sample from 45 minutes after blending was completely cured. Moreover, these two main compounds also detected at 200°C to 300°C were presumed to have been derived from the pyrolysis products.

In summary, ionRocket combined with DART®-MS is useful for analyzing insoluble materials such as hardened materials of adhesives. Therefore, we suggest that it is a useful method to evaluate curing conditions of adhesives such as curing time and temperature by directly and rapidly measuring hardened materials after blending or curing.

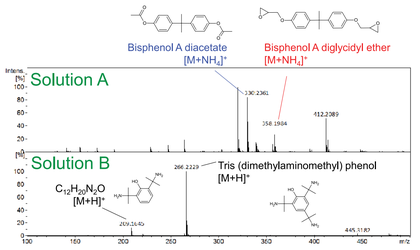


Fig.6 Mass Spectra of solution A and solution B

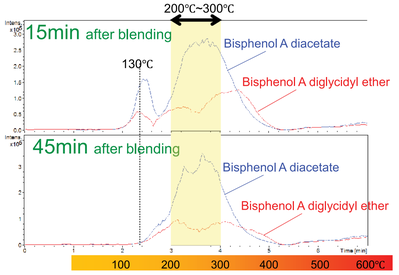


Fig.7 Extract ion chromatograms of hardened samples;
15 min and 45 min after blending.

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