Direct analysis in real-time mass spectrometry (DART®-MS) enables nearly instantaneous determination of sample composition using mass spectrometry. Therefore, DART®-MS is particularly powerful 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), which induces thermal desorption and pyrolysis of samples. The vapor phase of polymer samples was generated by applying a temperature-controlled heating gradient, then introduced and ionized into the mass spectrometer. In this poster, we describe the analysis applications using the ionRocket combined with DART®-MS.

**Application 1**

**SYNTHEIC FIBERS**

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

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

- **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 600ºC at a rate of 100ºC/min (Fig. 3).

- **RESULT**
  Total ion current chromatograms of PET and PTT are shown in Fig. 4. The total ion current profiles are different between PET and PTT. Mass spectra measured at 450ºC 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 repeat structure of PET and species related by intervals of 206 derived from the repeat structure of PTT. Thus, it was determined that this method can identify and differentiate different polyester fibers without the use of a methylating agent.

- **APPLICATION**
  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.

**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**
  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 and then analyzed. Small quantities of hardened materials were also put into the sample pot and analyzed. Samples were heated from room temperature to 600ºC at a rate of 100ºC/min (Fig. 3).

- **RESULT**
  Mass spectra of solution A and solution B are shown in Fig. 6. Bisphenol A diacate and bisphenol A diglycidyl ether were detected as main components of solution A, [(CH₃)₂NCH₂]₂C₆H₄OH was detected as main component 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 B (bisphenol A diacate and bisphenol A diglycidyl ether) were detected around 130ºC, whereas in the sample of 45 minutes after blending, no peaks were detected around 130ºC. Therefore, it was determined that these two main compounds were reaction residuals. In addition, it was presented that the sample from 45 minutes after blending was completely cured. Moreover, these two main compounds also detected at 200ºC to 300ºC 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.

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**REFERENCES**

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