Application Note TA6013

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TA6013 - Comparison between TG-DTA/GC-MS and Thermomass Photo

Introduction

Polyphenylene sulfide (PPS) is a crystalline polymer having a symmetrical, rigid backbone chain consisting of recurring p-substituted benzene rings and sulfur atoms. PPS is well known polymer because of resistance to heat and chemical resistance, widely used in several applications such as automotive, healthcare, office equipment, engineering part, textile, and so on.
To evaluate the applications above, thermal analysis using mass spectrometry (MS) covers a large number of related and analytical powerful techniques such as evolved gas analysis using mass spectrometry (EGA-MS) including termogavimetry-mass spectrometry (TG-MS).

In conventional TG-MS, TG is connected by the capillary interface to MS, thus, the evolved gaseous products from TG, which are introduced rapidly to MS. However, the capillary interface has many problems; (1) high boiling point gases become massed together because the capillary interface can't heat the temperature same as the furnace, (2) the evolved gases transubstantiate due to contact between inside wall of the capillary and evolved gases itself, and (3) the residual product of evolved gases emerge from inside of the capillary.

Our newly developed Thermomass Photo adopts the skimmer interface which is an orifice tube connected TG to MS, it has a good benefits such as direct measurement that it is available to study in detail about the evolved gas behavior for time without less contamination problem. In the present work, we study the comparison between Thermomass Photo and conventional TG-MS.

Instrument:

ThermoMass Photo

Thermomass Photo is an evolved gas analytical system designed for real-time simultaneous measurements of thermogravimetry – differential thermal analysis (TG-DTA) coupled with electron impact ionization (EI) and the fragment-free photoionization (PI) mass spectrometry (MS) that performs measurements as a function of temperature or time.

TG/GC-MS

Thermogravimetric-differential thermal analysis, TG-DTA, was performed using a Rigaku Thermo Plus 8121 system. The spectra of the gaseous products evolved from the specimen in the TG-DTA were simultaneously monitored with a quadropole mass spectrometer (Q-MS). The Q-MS system is connected to the TG-DTA via a gas transfer tube consists of stainless steel capillary whose internal surface is inactivated.

Measurement and analysis

The result of TG/GC-MS measurement for PPS using the cold trap method is shown in Figure 1. In the cold trap method, it detects many components such as benzene, toluene and benzenethiol etc. Although for example of components of toluene and biphenyl, these are hard to believe with pyrolysis products estimated from PPS molecular structure, hence the results, all detected components of pyrolysis products are open to question.

The results of the Thermomass Photo are shown in Figure 2. Both EI and PI method, H2S is detected which didn't detect at the GC-MS, on the other hand, the components detected at the GC-MS, those components didn't detect at the Thermomass Photo. Also, there are the detected components at the cold trap method which didn't appear at the Themomass Photo too.

We listed the detected gas components at the cold trap and the Thermomass Photo in Table 1. These are hard to believe with the structure as the pyrolysis product of PPS that the components detected at the cold trap method but it didn't detect at the Thermomass Photo.

Hence the results, the molecules reacted in the cold trap and generated newly molecules, and there is possibility of reflection inability to original evolved gas.

At the Thermomass Photo, the thermal behavior of each component can be observed for temperature and obtained as the valuable real-time temperature information which can't observe at the cold trap method. The Thermomass Photo can detect the actual evolved gas in real-time, which the cold trap method can't obtain (see Figure 3).

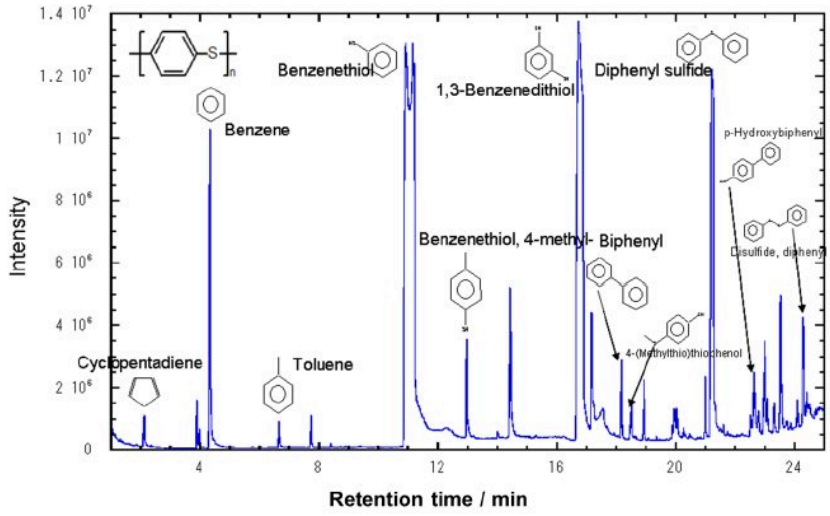

Figure 1: The result of GC/MS for PPS using the cold trap method

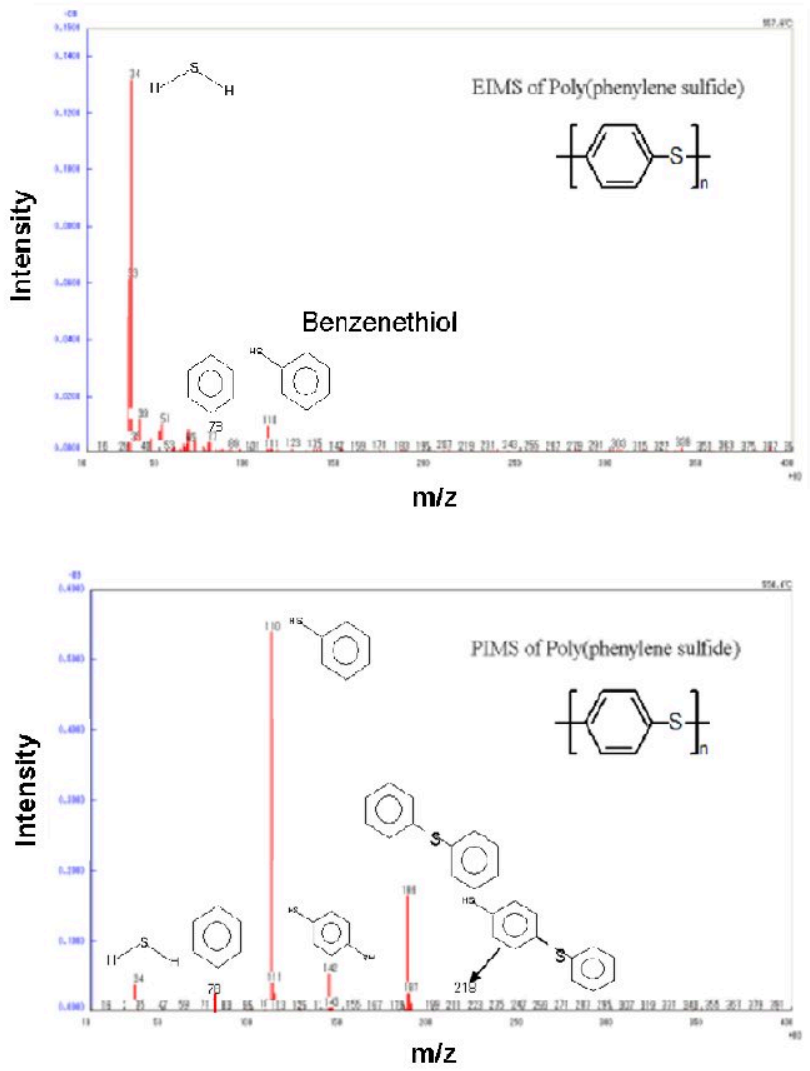

Figure 2: The result of mass spectra of Themomass, EI method (top) and PI method (bottom) at around 555℃.

Table 1: Comparison in each apparatus between predictable and un-predictable of detected evolved gases for the pyrolysis product of PPS

	Evolved gas components	**TG-DTA/GC-MC cold trap method**	**ThermoMass Photo**
Predictable evolved gas components of pyrolysis product of PPS	Hydrogen sulfide	X	○
	Benzene	○	○
	Benzenethiol	○	○
	Benzenedithiol	○	○
	Diphenyl sulfide	○	○

Unpredictable evolved gas components of pyrolysis product of PPS	Toluene	●	N.D.
	Benzenethiol, 4-methyl-	●	N.D.
	Biphenyl	●	N.D.
	p-Hydroxybiphenyl	●	N.D.
	Disulfide, diphenyl	●	N.D.

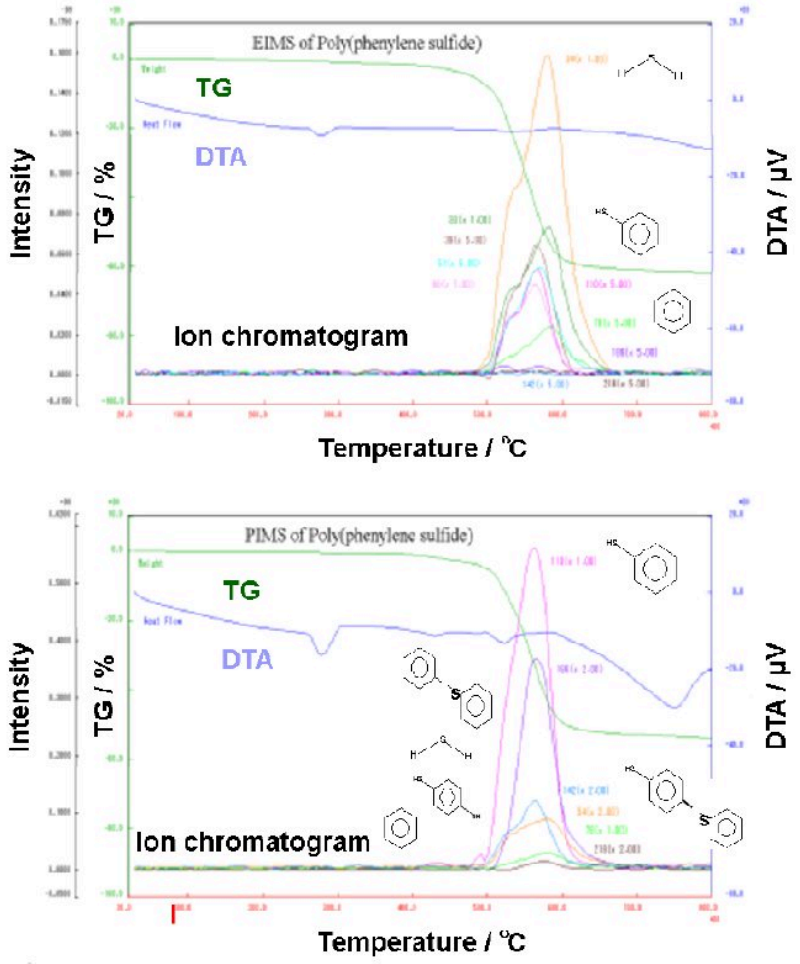

Figure 3: The result of TG-DTA/MS of ThermoMass, EI method (top) and PI method (bottom)

Related products

ThermoMass Photo

An integrated thermal analysis instrument capable of high-p
recision mass analysis of evolved gases without breaking t
he molecules, allowing direct measurement.