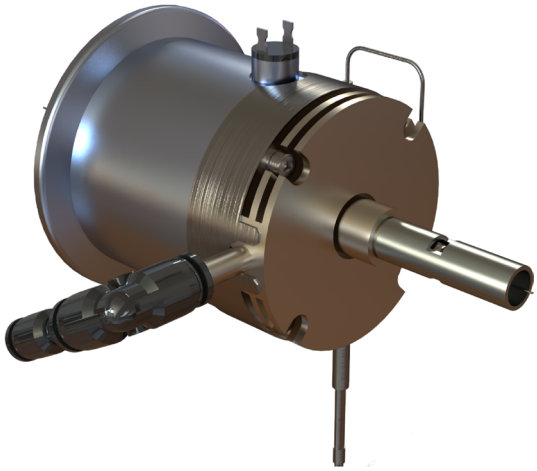
Thermo Mass Photo Capillary Interface

—New Interface Without Cold Points for High Boiling Point Gases Detection—



Abstract

Rigaku has developed a new capillary interface for the TG-DTA/Photoionization Mass Spectrometer 'Thermo Mass Photo.' This new interface enables high-sensitivity detection of gases and improves the detection capability of high-boiling-point gases. Furthermore, its design allows it to be interchangeable with the conventional skimmer interface, providing flexible adaptation to different measurement purposes. It is also characterized by its ease of maintenance and versatility in handling various samples, making it suitable for a wide range of applications.

1. Introduction

Thermal analysis is used in a wide range of fields. This method allows users to understand physical or chemical thermal changes of samples at a macro level. However, in order to obtain information on what exactly is happening at a micro level, complex measurements in combination with other methods are necessary. One of these complex analysis methods is the thermogravimetric differential thermal analysis and mass spectrometry method (TG-DTA-MS), which combines thermogravimetric differential thermal analysis (TG-DTA) and mass spectrometry (MS). As an instrument for the TG-DTA-MS method, Rigaku offers Thermo Mass Photo (TG-DTA/photoionization mass spectrometer) with a skimmer interface.

TG-DTA-MS requires an interface for gas transport in order to accurately introduce the gas generated from the sample section of the TG-DTA analyzer into the MS instrument.

Two typical interfaces are capillary and skimmer types. In the conventional capillary type, the two instruments are connected by a 1 to 2 m long narrow tube (capillary), and the capillary is heated at a constant temperature. This method requires uniform heating at the connection points between the capillary and both instruments, but the long route and complicated connections cause cold points, which lead to accumulation of high-boiling point gases. The skimmer type, on the other hand, incorporates a differential evacuation unit based on the jet separator principle inside the electric furnace of the thermal analyzer. This method enables the gas generated from the sample section at atmospheric pressure to be introduced into the MS chamber in a vacuum atmosphere over a short distance of approximately 160 mm. However, the connection on the MS side is difficult to heat due to its configuration so, although the path is short, there are cold points just as in the capillary method.

In this new interface for Thermo Mass Photo, Rigaku succeeded in developing a capillary interface without cold points, enabling the detection of high boiling point gases at a wider range than ever before.

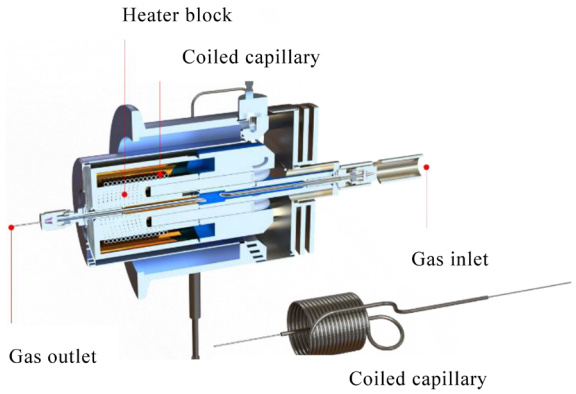


Fig. 1. Cross section of the capillary interface

2. Features

2.1. Configuration capable of detecting high boiling point gases

We have developed a compact and efficient heating interface by encapsulating a coiled capillary in a heater block. The temperature can be set up to 350℃.

Using state-of-the-art simulation technology, we have created a configuration that enables uniform heating of the heater block that encapsulates the capillary, allowing the gas to pass through the interface from the gas inlet to the gas outlet without accumulating on the inner walls of the capillary.

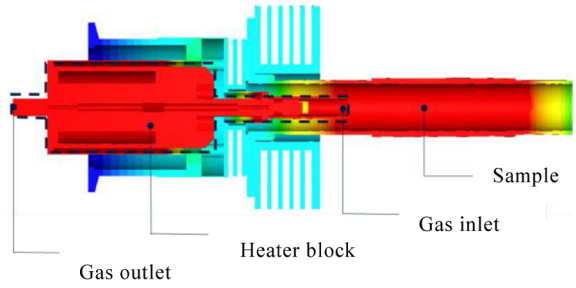


Fig. 2. Simulation diagram

2.2. Interchangeability with skimmer interface

The newly developed capillary interface is designed to be interchangeable with the skimmer interface, so the interface can be replaced depending on the measurement purpose. As mentioned above, the skimmer interface is based on the jet separator principle. This means that the concentration ratio of the sample gas exiting the outlet is larger than the concentration of the sample gas entering the gas inlet with respect to the carrier gas. Thanks to this configuration, the skimmer interface has the advantage of higher sensitivity for gases with low boiling points compared to the capillary interface.

2.3. Simple maintenance

Depending on the type of generated gas and the frequency of use, the gas may adhere to the capillary and clog it. The capillary must be replaced when the sensitivity deteriorates. Coiled capillaries for replacement are available as consumables, so the customers can easily replace the capillary themselves.

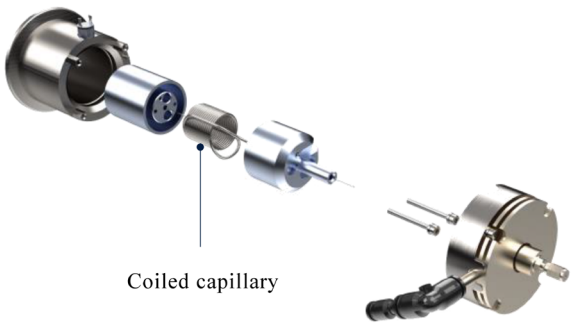


Fig. 3. Coiled capillary replacement diagram

3. Measurement Examples

3.1. Bisphenol A in polycarbonate

Figure 4 shows the results of electron ionization (EI) measurements of 2 mg of polycarbonate in He atmosphere heated from room temperature to 700℃ at a 20℃/min heating rate. TIC (total ion current) indicates the overall behavior of the generated gas. The generation of peaks at m/z 213, 228, the main ions of bisphenol A, are clearly detected, confirming that bisphenol A was generated during the pyrolysis of polycarbonate (500 to 600℃).

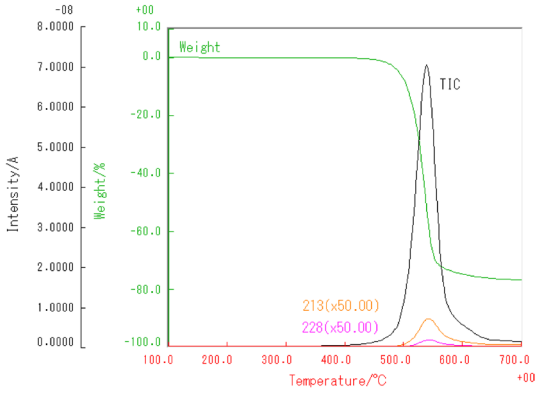


Fig. 4. TG and MS results of polycarbonate

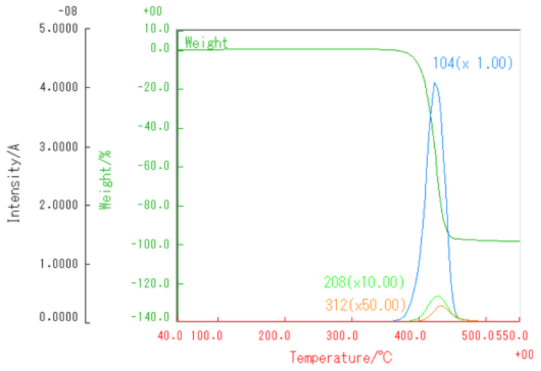


Fig. 5. TG and MS results of polystyrene

3.2. Detection of polystyrene dimers and trimers

When polystyrene is heated in an inert atmosphere, styrene monomer is mainly generated, but styrene dimers and trimers are generated as well.

Figure 5 shows the results of EI measurements of 2 mg of polystyrene in He atmosphere heated from room temperature to 550°C at a 20°C/min heating rate.

The generated gas mainly contained styrene (m/z 104), but dimeric and trimeric molecular ions, m/z 208 and 312, respectively, were also detected.

4. Conclusion

This interface has been newly added to the Thermo Mass Photo lineup. The features of both these interfaces can be implemented to measurements, making the instrument suitable for a wider range of applications than ever before.