Gas Analysis
Application Note 270/1

TA-MS
Thermal Analysis – Mass Spectrometry

TA-MS Overview

Evolved gas analysis techniques can be used to provide important information about decomposition or desorption processes when coupled to thermal analysis techniques such as DSC, TGA, and DTA

Mass spectrometry (MS) offers a number of advantages for evolved gas analysis. As well as being of high sensitivity, modern mass spectrometers can provide simultaneous, unequivocal and fast detection of several gaseous species within the available mass range. MS therefore compliments TA techniques with speciation as a function of time and/or temperature.

The key requirements of the TA-MS Interface are:

- Minimum dead volume.
- Controllably Heated sample inlet - no cold spots.
- Inert materials.
- High performance gas handling for operation with low molecular weight gas components (H_2, He) and for flow matching with the TGA.

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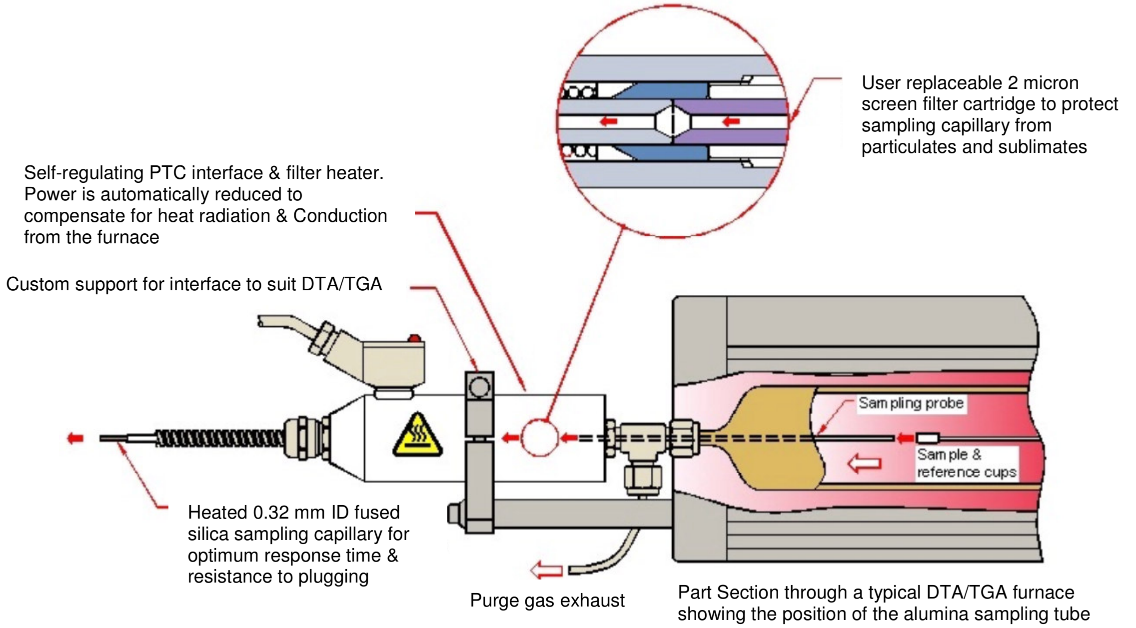

Figure 1 Example of a Hiden TGA-MS Interface

HPR-20 Mass Spectrometer

The HPR-20 is a compact, bench top gas analysis system (Figure 2) which features Hiden's heated *QIC* (Quartz Inert Capillary) inlet with controlled by-pass for rapid and continuous sampling in the process pressure range 100 mbar to 2 bar absolute. The unique *QIC* inlet design allows for a range of custom adaptors, flow and temperature matched, to be interfaced with specific TA/TGA instruments.

Figure 2 HPR-20 QIC Mass Spectrometer

For ease of use the HPR-20 is configured for automatic start/stop from an external trigger signal (from the TGA) allowing synchronization of weight, temperature and MS data. Additionally, two 0-10V analogue inputs read weight and temperature data directly into the MS.

The TA- MS Interface

Thermal Analysis equipment operates at near atmospheric pressure whereas Mass Spectrometers operate at high vacuum, some nine decades lower. The TA-MS interface is therefore critical for accurate measurement of both evolved gases and vapours.

Hiden Analytical has worked closely with major TA instrument manufacturers and end users to produce a comprehensive range of TA-MS interface adapters for the *HPR-20 QIC*. These adapters are interchangeable to suit different instruments and can be specified at point of sale (with new systems) or as simple retro-fits (to existing apparatus). Figure 1 shows a typical Hiden interface design.

QIC Response

The combination of low dead volume sampling with fully heated transfer line, inert material construction and superior gas handling/inlet dynamics mean that the *HPR-20 QIC* offers unrivalled accuracy and speed of response – to both gases and vapours.

Figure 3 shows typical response curves for the *QIC* inlet compared with conventional capillary inlets. Response times of less than 150 milliseconds are possible with the *QIC* inlet.

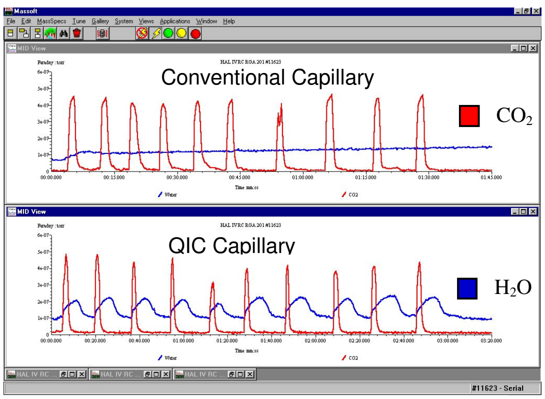

Figure 3 QIC Inlet Response

TA- MS Examples

1. Calcium Oxalate Decomposition

Coupled TG-MS analysis data reveals i) water desorption, ii) partial oxalate decomposition with CO / CO_2 evolution and iii) full decomposition of oxalate with CO_2 evolution. One can also see the perfect correlation between mass loss and MS signals.

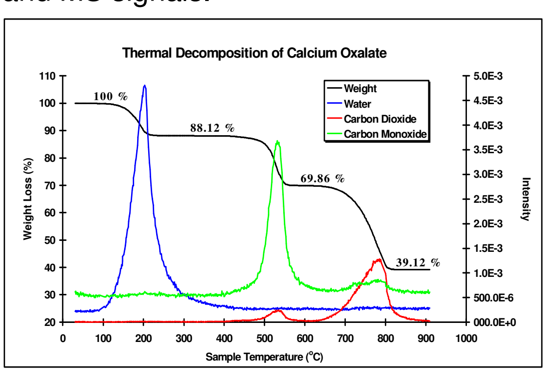

***Figure 4**. Calcium Oxalate Decomposition*

2. Decomposition of an Organo-Sulphur Compound

Figure 5 below shows there to be only one major weight loss feature shown by the TGA instrument. However, the MS data shows that this weight loss is due to the coincident loss of several species including H_2S, formaldehyde, carbonyl sulphide, isopropyl alcohol and CS_2.

This example demonstrates the advantages of attaching the HPR-20 mass spectrometer to a TGA system.

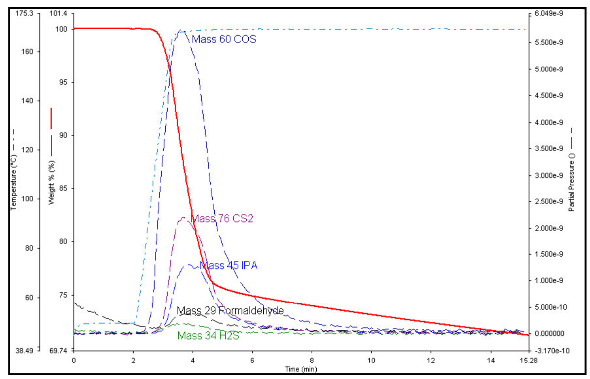

Figure 5 TG-MS Plot of Organosulphur Compound Decomposition

3. Dehydration of Lactose Monohydrate

Lactose is widely used in the pharmaceutical industry as a filler or binder in tablet manufacture. Therefore, the adsorption of water during prolonged storage is of interest. Figure 6 shows a TG-MS plot of the dehydration of freshly prepared lactose monohydrate. Here, one distinct weight loss can be seen. The mass spectrometer trace shows that this is due to the loss of water probably from the dehydration of the lactose.

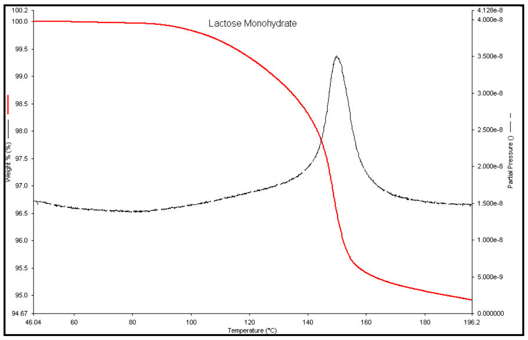

Figure 6 TG-MS Plot of Lactose Monohydrate Dehydration

Figure 7 shows the TG-MS plot of a sample of lactose monohydrate that was exposed to the atmosphere for 24 hrs.

Here, it can clearly be seen that there is an additional loss of water detected in the mass spectrometer trace at lower temperature, approx 70 ˚C. This is due to desorption of water adsorbed on the lactose during exposure to the atmosphere.

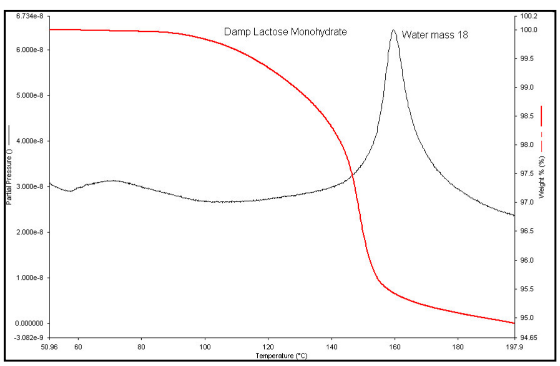

Figure 7 TG-MS Plot of Lactose Monohydrate Dehydration after atmospheric exposure for 24 hrs

Conclusion

The examples shown demonstrate that coupling a mass spectrometer to TA equipment can provide invaluable information about the identity of desorbed species or decomposition products formed during a thermal analysis experiment. This information is complimentary to that obtained by the TGA to give a complete understanding of the desorption/decomposition process.