



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 from TGA instruments over other techniques such as FTIR. As well as being of high sensitivity (100 % to ppb levels) modern mass spectrometers can provide simultaneous, unequivocal and fast detection of several gaseous species within the available mass range. However, the importance of the coupling between the MS and TGA instrument should not be underestimated.

Introduction

The coupling of TGA and MS is important if high quality results are to be achieved. 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.

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 (H₂, He) and also flow matching with the TA.

It is important that all of these factors are taken into account when designing a TA-MS interface to ensure accurate, reproducible results.

QIC Inlet

The QIC inlet is designed to give responses in gas/vapour concentration in less than 300ms. 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 1 shows typical response curves for the QIC inlet compared with conventional capillary inlets.

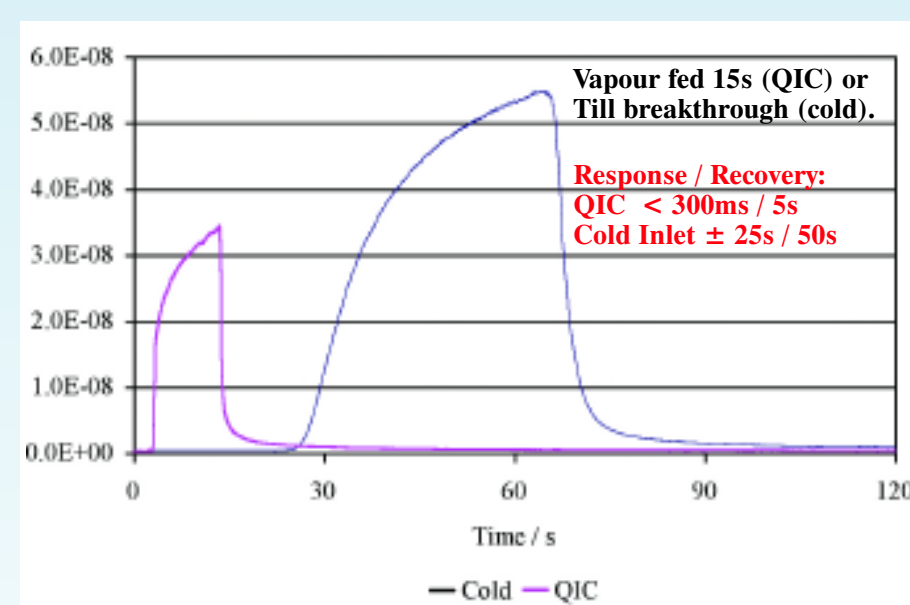


Fig 1. Effect of cold spot on capillary response - IPA

Gas Sampling

Rapid transfer of the gas desorbing from your sample to analysis is essential in TGA-EGA experiments if the desorption event is to be correlated with the desorption temperature. A high sampling flow rate allows dead volumes to be minimised therefore allowing close correlation between weight loss and evolved gas detection. The gas sampling arrangement of the Hiden HPR-20 QIC also allows a high flow rate of He as a carrier gas. Using He as a carrier gas is advantageous as He does not have spectral overlaps in common regions of interest, unlike other carrier gases such as N₂ (M/Z 28, 14) and Ar (M/Z 40, 36, 20, 18).

Figure 2 Hiden gas sampling arrangement - see opposite (top)

Hiden has worked closely with leading TGA manufacturers to further minimise dead volumes to provide the closest coupled sampling arrangement possible.

Figures 3a-3b PE instrument coupling - see opposite
Figures 4a-4b TA Inst. instrument coupling - see opposite

TA-MS Examples

1. Analysis of Calcium Oxalate Decomposition.

Coupled TG-MS analysis data reveals i) water desorption, ii) partial oxalate de-composition with CO / CO₂ evolution and iii) full decomposition of oxalate with CO₂ evolution. One can also see the perfect correlation between mass loss and MS signals.

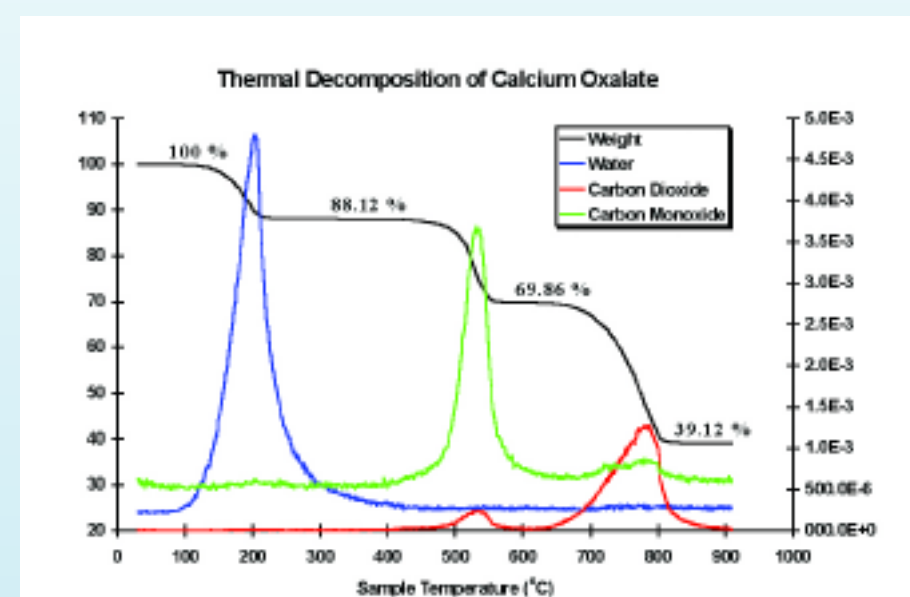


Fig 5. Calcium Oxalate Decomposition

2. Thermal decomposition of CuSO₄

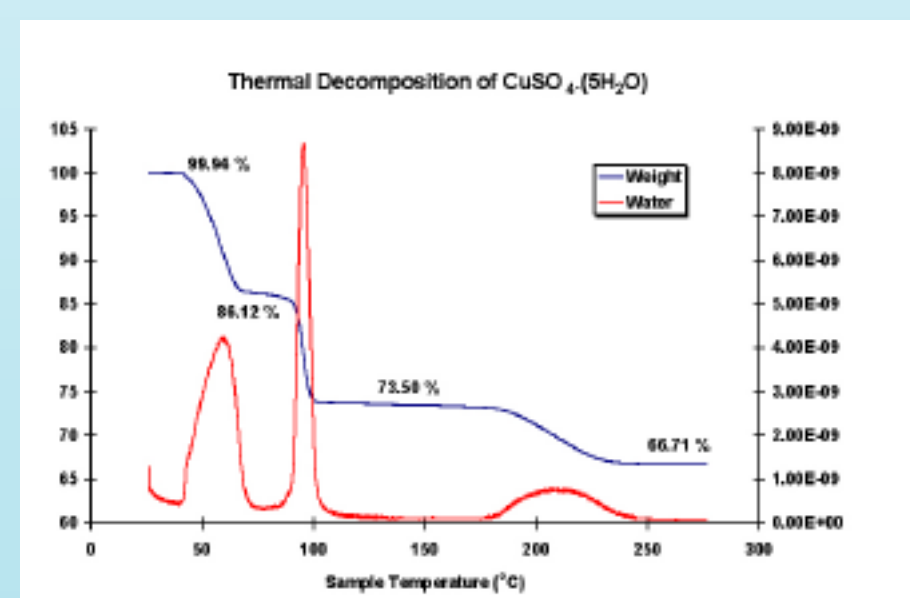


Fig 6. Coupled TGA/DTA-MS of this sample showed good agreement with expected values and again reflected exact correlation between mass loss and MS signals

Fig 2. Hiden gas sampling arrangement

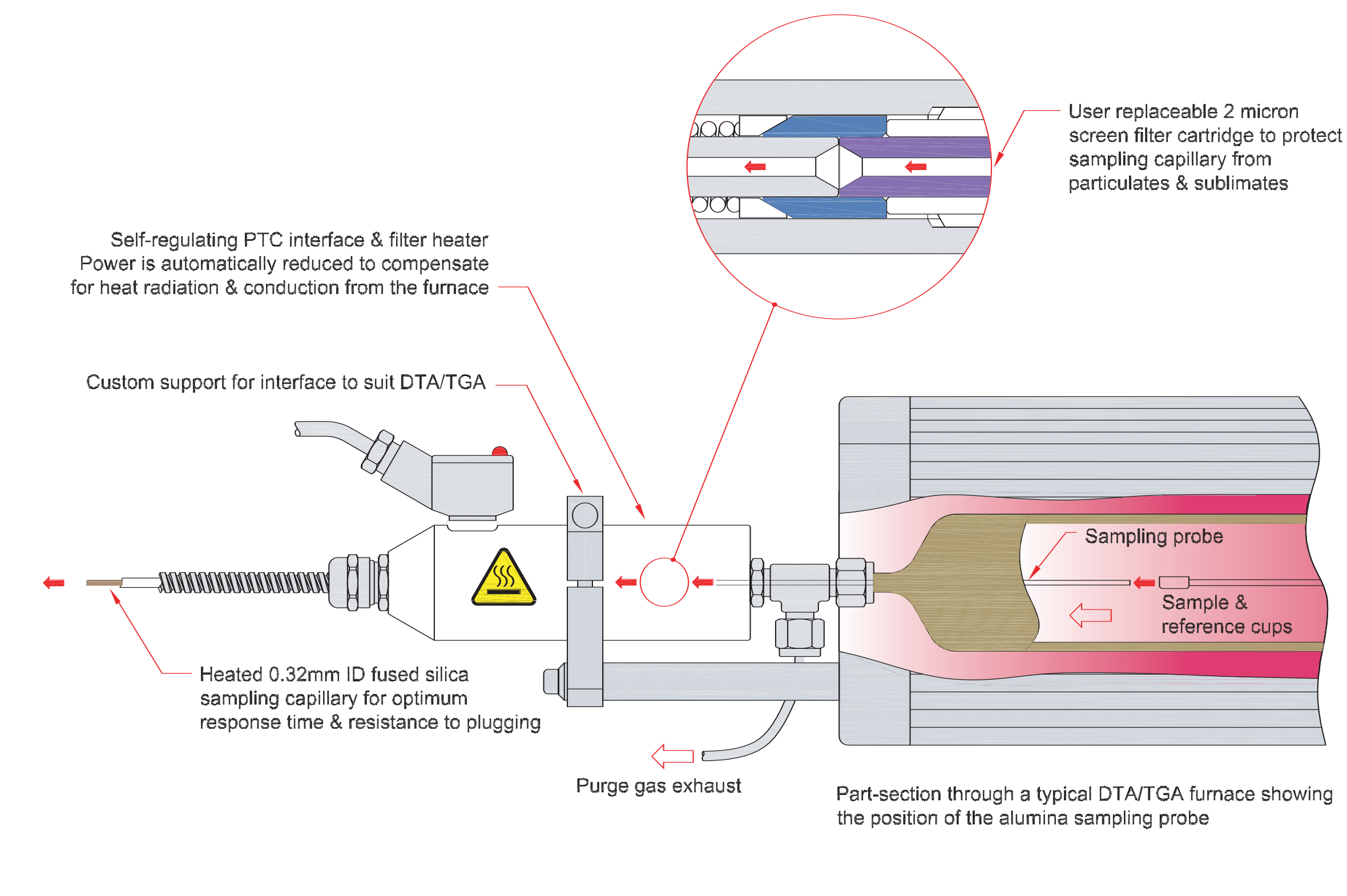


Fig 3a. TGA Interface for PE Pyris TGA
Custom designed Hot zone inlet for the PerkinElmer Pyris TGA system. Includes optimised 'Hot-zone' sampling connection



Fig 3b. TGA Interface for PE Diamond TGA
Custom designed Hot zone inlet for the PerkinElmer Diamond TGA system. Includes optimised 'Hot-zone' sampling connection and heated filter assembly.



Fig 4b. TGA Interface for TA Instruments Q600
Custom designed Hot zone inlet for the TA Instruments Q500 TGA system. Includes optimised 'Hot-zone' sampling connection and heated filter assembly and support.

Fig 4a. TGA Interface for TA Instruments Q500
Custom designed Hot zone inlet for the TA Instruments Q500 TGA system. Includes optimised 'Hot-zone' sampling connection and heated filter assembly.