

The Sniffer Interface

Product Description

The patented Sniffer interface was developed by Thermo Cahn to satisfy three objectives. First, the interface must sample the evolved gases before they are allowed to diffuse through the entire volume of the TGA reaction cell. Second, the interface must connect to both mass spectrometers and FTIR spectrometers. Third, each spectrometer must receive the optimum flow from the TGA without compromising the performance of the TGA, FTIR or MS.

As shown in Figure 1, the Sniffer interface employs separate tubes for MS and FTIR sampling. The tubes are constructed from a high temperature alloy and positioned at an angle so they do not interfere with the TGA hang-down wire.

The Sniffer tubes sit in the gas separation zone region, just above the sample, enabling them to immediately capture evolved gas, minimizing any dilution effects. Because the Thermo Cahn TGA system allows for a gas separation zone and reduced turbulence within its reaction tube, the quality of the acquired data is greatly enhanced.

The sample is pulled through the Sniffer interface by a vacuum. In the case of the MS, the vacuum of operation serves to pump the evolved gas. For FTIR operation, a pump is placed after the FTIR flow cell. The flow rate is adjusted for each spectrometer by the vacuum and pump speed respectively.

Due to the differing flow requirements of the three instruments: TGA, FTIR, and MS, there has been limitations on the quality of the real-time data acquisition via the simultaneous use of all techniques. Traditionally, it has been necessary to compromise the performance of the various components in order to ensure simultaneous operation.

The Sniffer interface allows continuous operation of all acquisition techniques without any compromise in the quality of the acquired data.

Experimental

Three experimental tests were constructed to display the quality of information provided by the Sniffer interface.

A low volume TGA, a high volume TGA, and a high volume TGA with the Sniffer interface were used.

Since the sample capacity of the low volume system limited the maximum allowable sample weight, small sample loads of 15 mg were used in all three experiments.*

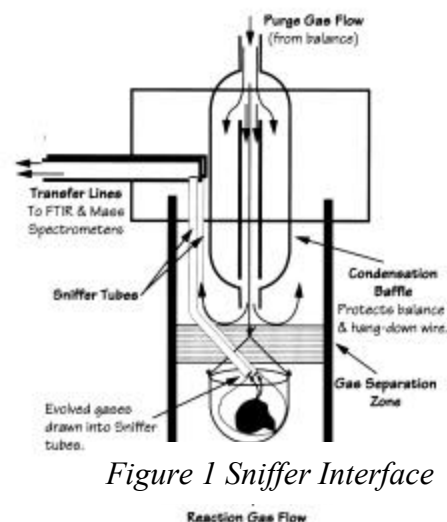


Figure 1 Sniffer Interface

Reaction Gas Flow

The sample used in the experiment was K resin (75% styrene, 25% butadiene), which was solution grafted with methacrylic acid, constituting 27% of the sample weight.

The experiment was a simple temperature ramp from 25 - 700 °C at 20 °C per minute, which was repeated on all three systems.

System 1 The low volume system was an Omnitherm TG-1000. This system was flowed at a rate of 35 ml/mm with approximately 15 mg of TGA sample.

System 2 The high volume TGA was a Thermo Cahn 131 system with a total flow output. The flow rate was 140 ml/mm.

System 3 The high volume Sniffer system was also a Thermo Cahn 131 fitted with a Sniffer interface; operated with a flow rate of 140 ml/mm.

Results

A sensitivity comparison of the peaks at 3000 cm^{-1} between the three experiments is shown in Figure 2. All the FTIR spectra are identical except for the absorbance amounts, which enabled us to contrast the three techniques.

The poorest performing experiment was the total flow, high volume TGA, which in the C—H stretching band provided an absorbance of 1.63.

The next best was the low volume TGA, which gave an absorbance of 2.92.

The best absorbance amount was from the high volume Sniffer experiment, which gave 13.20 and outperformed the low volume TGA by a factor of 4.5.

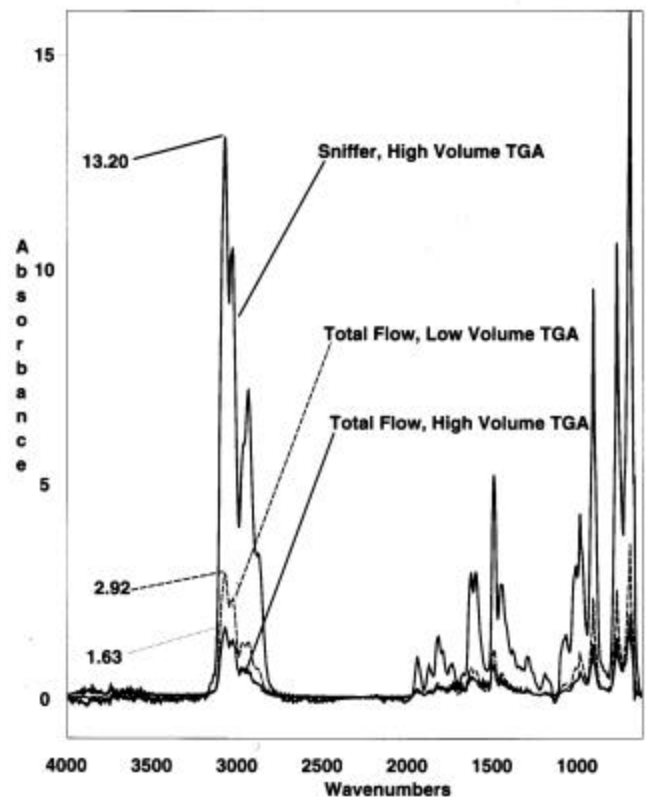


Figure 2 Sensitivity Comparison

Conclusions

The Sniffer interface provides four distinct advantages over any total flow designs for evolved gas transfer.

1. The Sniffer allows the use of a high-volume, high mass TGA without a compromise in sensitivity. This allows the use of large sample loads which will increase the percent of evolved sample gas.
2. Because the TGA allows high volume and high mass samples, whole components and fabricated parts can be analyzed in their entirety. Examples include disk-drive motors, turbine blades, and electronic components.
3. The Sniffer interface provides the capability of regulating both MS and FTIR detector flow rates. The TGA purge and reaction gas flows can be set to optimum experimental conditions without detrimental effect to the FTIR or MS data. This reduces the chances of evolved gas contaminating balance components and the TGA reaction chamber due to adequate reaction and purge flows. <
4. Finally, the Sniffer tube is quickly and easily disassembled for cleaning.

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