



TGA-MS for Evolved Gas Analysis

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Introduction

Thermogravimetric analysis (TGA) is a well-known method of quantitatively studying the loss of volatile components from a sample. This can be done isothermally to study weight loss during operation or with a temperature ramp to study pyrolysis. Recent changes in laws governing VOC, combustion products, and environmental discharges make identification of the evolved gases even more important. Several approaches are available including TG-IR and TG-GC directly from PerkinElmer



Figure 1. The TG-MS system is shown above. The high sensitivity of the Pyris 1 TGA with Pyris software is well known. With Pfeiffer's software running co-resident in Windows NT, only one computer is required for the system. The combined software, developed by companies with a long history of expertise in their respective technologies, offers the most complete data processing capability

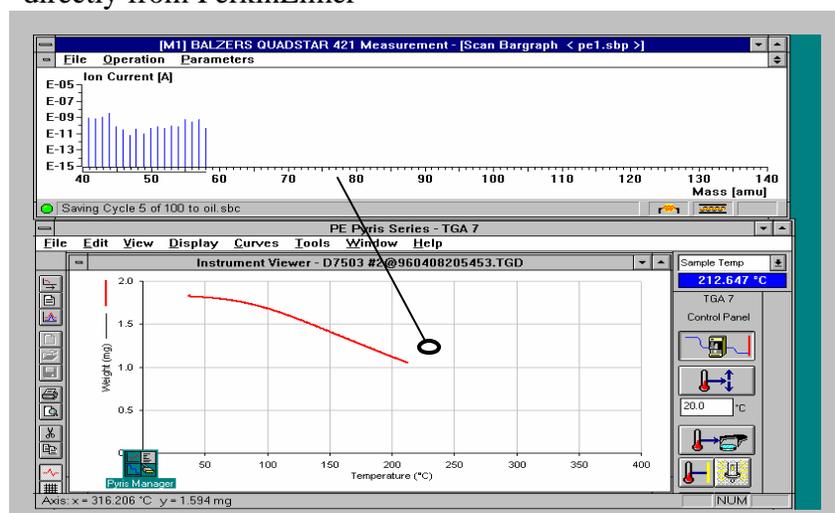


Figure 2. TGA/MS bring run on a sample of antibiotic ointment in the mass ion scanning mode, where a full mass spectrum is generated every 20 seconds during the scanning run. Heating rate was kept to 3 degrees per minute to allow one scan per degree. Sample mass was 26.234 mg.

Instruments. However, the combined TG-MS system combining the PerkinElmer Instruments Pyris 1 TGA and the Pfeiffer Vacuum ThermoStar Mass Spectrometer (figure 1) uses the strength of two industry leaders.

Applications

For this study, a manufacturer selected a commercial burn ointment. The sample can be

studied using any of the techniques of TGA including multiple step ramps, isothermals, auto-stepwise methods, special pans (e.g. laser pierced pans for controlling volatiles), etc. We choose a heating rate of 3 C/min in the Pyris 1 TGA to allow the collection of one mass spectrum per degree. The mass spectrometer and TGA are connected through a modified sample tube, through which the Pfeiffer MS “sniffs” the evolving gases for analysis. Unknowns are best run in two steps. The MS data can be first collected as a normal mass spectrum (e.g. every half minute, showing all masses). Figure 2 shows this type of data as it is collected in a TGA scan on an antibiotic ointment. The data can then be processed to show how certain ion concentrations vary with time or temperature.

Once certain mass ions are identified, evolved gases can be more rapidly labeled, with only a few mass ions being tracked. Figure 3 shows a run with these conditions on the same ointment.

Applications of TG-MS are very broad. Possible uses include the measurement of VOCs in pesticides or consumer products;

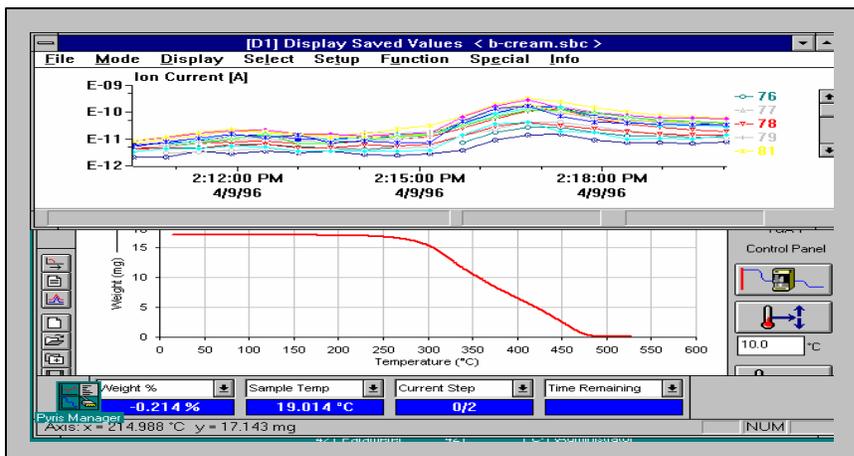


Figure 3. TGA/MS being used to track the mass ions of interest during the TGA run. Because both the PerkinElmer Pyris Software and the ThermoStar software can be run co-resident in Windows 2000, comparison of the data is very easy.

characterizations of gases released in reactions or burning, study of the volatiles in solid wastes, and identification of residual oils left on electronic components. In Figure 4, a 0.5 mg piece of

hard disk adhesive off-gases upon a slow heating ramp. Is the gas harmless like water or something that will redeposit and affect performance? TGA/MS can give the answer.

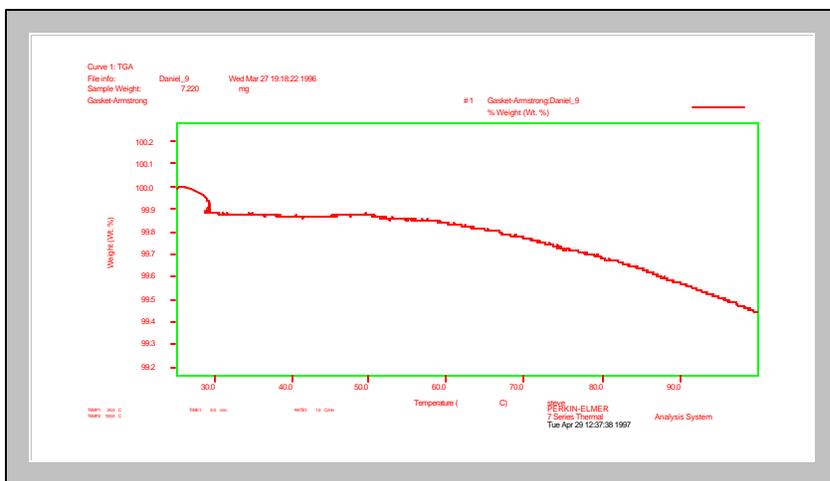


Figure 4. TGA run on a sample of hard disk drive gasket.

