Faster Results, Anytime, Anywhere

Combining a Thermogravimetric Analyzer (TGA) with a Gas Chromatography coupled with Mass Spectrometer (MS) allows separation and detection of very low levels of impurities. By heating a sample on the TGA, the sample will release volatile materials or generate combustion components as it burns. These gases are then transferred to the GC, where the components can be collected on a trapping media, in a gas sampling loop, or deposited on the head of a column. The sample can then be run by GC to separate the material, and the peaks identified by the MS. Because of its ability to detect very low levels of material in complex mixtures, the TG-GC/MS is a powerful tool for quality control, safety, and product development.

The PerkinElmer TL8500 transfer line allows you to couple our full range of Thermogravimetric Analyzers (TGA) and Simultaneous Thermal Analyzers (STA) to a PerkinElmer Clarus® SQ 8 GC/MS.

The TG-GC/MS System Uses:

- Our Full range of TGA and STA Systems such as the TGA 4000, STA 6000, STA 8000 or TGA 8000 can be connected depending on degree of precision and temperature range needed.
- The TL8500 transfer line runs at 350 °C and uses pumps and mass flow controllers to deliver a precise flow of gas to the GC/MS. Two sample collection loops are included.
- Clarus SQ 8 GC/MS provides maximum detection of low levels of contaminates which in-turn improves accuracy.

Figure 1. PerkinElmer TG-GC-MS

By using the PerkinElmer Clarus SQ 8 Mass Spectrometer, the same MS used in PerkinElmer's state of the art GC/MS systems, enjoy the advantages of:

- The detection of mass ions up to 1200 daltons
- Soft ionization (adjustable EI) to limit fragmentation of the mass ion
- The ability to add chemical ionization (CI) to decrease fragmentation
- Automatic triggering of the MS run at the start of the TGA run.
- SMART source™ (Simplified Maintenance And Removal Technology) in MS for Easy removal and Cleaning.
- Durable and Long-Lasting Marathon™ Filament which Works with both EI and CI sources and is highly resistant to demanding solvents and complex matrices.

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For the Better

Qualitative Analysis of Evolved Gases in Switchgrass by TG-GC/MS

Thermogravimetric analysis (TGA) measures the change in the weight of a sample as a function of temperature. A limitation of TGA is that it cannot identify what material is lost at a specific temperature. The analysis of gases evolved during a TGA experiment by gas chromatography mass spectrometry (GC/MS) provides laboratories with a way to identify the compound or groups of compounds evolved during a specific weight-loss event in a TGA analysis.

This application note discusses the utility of TG-GC/MS with an example application – the identification of specific organic acids evolved during TGA analysis of switchgrass.

A small quantity of dried and ground switchgrass was placed on the TGA pan and weighed using the Pyris software. A rapid TGA analysis based on heating the sample from 30 °C to 1000 °C at 100 °C/min in a nitrogen atmosphere was performed to determine which regions of the weight loss curve were to be further studied using the TG-GC/MS technique.

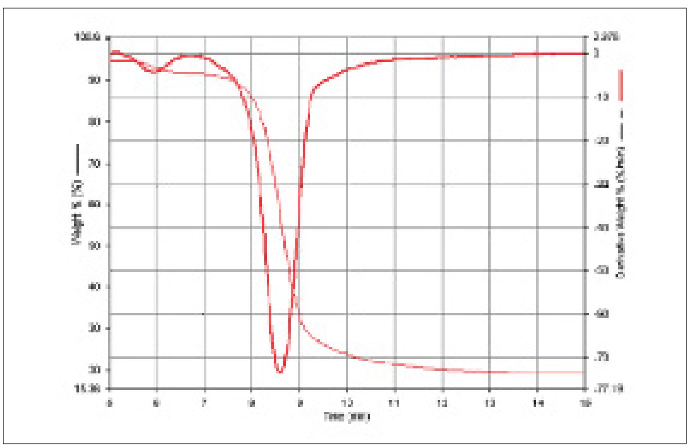

Figure 2. The TGA run of a sample of switchgrass shows most weight loss occurs in one temperature range.

After the sample was loaded onto the TGA and the furnace raised, the analysis was started immediately. The first step in the TGA heating program maintained the low initial furnace temperature for 5 to 10 min. During this time, the furnace environment is being purged with helium (or nitrogen/argon), and the carrier-gas pressure of 7.0 psig maintained at Aux 1 ensures that no sample can enter the analytical column. After this initial hold period, the TGA furnace begins to heat the sample, and simultaneously, the GC/MS run is started using an external start command.

A typical TGA weight-loss curve for the switchgrass is shown in Figure 2 and reveals a typical weight % loss curve for the sample of switchgrass that was tested. In addition, superimposed on the weight-loss curve is the derivative of this curve which greatly assists the analyst in setting up the GC timed events that will be used to sample the evolved gases onto the GC/MS column. Note that the TGA is held isothermal for the first 5.0 min at which point heating begins. Simultaneously, the GC/MS analysis is started.

Figure 3 illustrates the TG-GC/MS analysis of the switchgrass based on timed events that collect the evolved gases from the main transition shown in Figure 2. The smaller earlier transition, also seen in the same figure, was also sampled onto the GC/MS but preliminary findings indicate that this is simply evolved water. The major transition produced large numbers of oxygenated volatile organic compounds (VOCs), including some very polar species. Earlier work using a non polar capillary column had generated extremely smeared-out early eluting peaks. The chromatogram below was generated using a thick-film polar Elite WAX column.

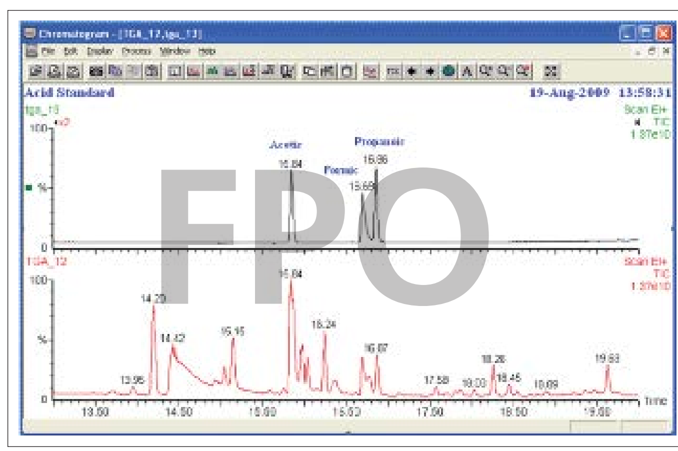

Figure 3. GC/MS on the gases evolved between 8 and 9 minutes and collected on the head of a GC column gave the chromatography seen on the bottom of the graph. MS analysis suggest that 15.8 is the acetic acid, which is confirmed above by running a standard of acetic, formic, and propanoic acids.

Conclusion

In this application note, we described the technique of TG-GC/MS through the analysis of switchgrass. TG-GC/MS is demonstrated to be a valuable technique in the separation and identification of complex mixtures of gas evolved during a TGA analysis.

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