

HUMAN HEALTH

ENVIRONMENTAL HEALTH

HYPHENATED TECHNOLOGY GUIDE





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HYPHENATED TECHNOLOGY GUIDE

Coupling, or hyphenating, two instruments can uncover results and insights not possible with individual techniques. The power of your analysis therefore becomes greater than the sum of its parts.

- Do you want to better understand how a material degrades or what gases evolve when it burns? The technique used to answer these questions is called Evolved Gas Analysis. Our Thermogravimetric (TG) instruments can be coupled to Fourier Transform Infrared (FT-IR), Mass Spectrometry (MS), or Gas Chromatography/Mass Spectrometry (GC/MS) to provide you with greater knowledge.

These technologies help advance the power of analysis in your laboratory. The more you know, the more you can do.

TG-IR



The combination of a Thermogravimetric Analyzer (TGA) with an Infrared Spectrometer (TG-IR) is the most common type of Evolved Gas Analysis (EGA) in use today. By heating a sample on the TGA, a sample will release volatile materials or generate combustion

components as it burns. These gases are then transferred to the IR cell, where the components can be identified. Because of its ability to detect functional groups, IR analysis allows greater understanding of the processes seen in the TGA.

The PerkinElmer TL8000 transfer line is a state-of-the-art system for TG-IR. Unlike simpler systems that simply move the gas to the TGA, the TL8000 is designed to make sure every component evolved in the TGA is transported to the IR.

Advantages of this System Include:

- Insulated heated transfer line with replaceable SilcoSteel® liner
- Heated zero-gravity-effect 'ZGCell' gas cell for the PerkinElmer FT-IR instrument incorporating automatic accessory identification, low volume, and efficient sample area purging
- Control unit incorporating a mass flow controller, particle filters, flow smoothing system, independent transfer line and gas cell temperature controllers, and vacuum pump with exhaust line
- Automatic triggering of IR data collection from the Pyris™ Software
- Spectrum Timebase™ Software for time resolved experiments

This design translates into some distinct advantages giving better data and greater ease-of-use:

- Constant gas flow giving optimum separation between time-resolved peaks
- Reduced mixing of IR signals
- No accumulations of heavy components in the IR cell due to ZGCell design

- Automatic importation of TGA data in Timebase Software

The TL8000 can be used to connect any of the PerkinElmer TGA/STA instruments to any of our FT-IR's. This offers you a range of options in terms of price and performance to fit your needs.

The TG-IR system is ideally suited for applications where one wants to identify materials evolved on heating, like residual solvents in pharmaceuticals, component identification in the analysis of plastics or rubbers, or the study of the combustion products from burning a sample. An example of the data one can get is shown below from a sample of switchgrass, a material being studied in North America as a possible source of biofuels.

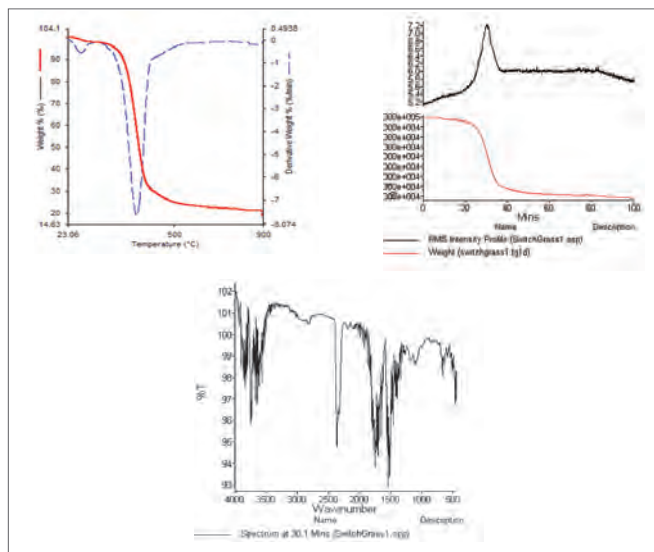


Figure 1. The data from the TGA run (top left) is transferred automatically to the Timebase Software and compared to the Gram Schmidt plot (top right). From this data, we can examine regions of interest as shown in the lower center image.

As you can see in the TGA, there is a small weight loss at low temperature and then a much larger one at approximately 250 °C corresponds to the burning of the organic matter. This data is imported into the Timebase Software where it can be compared to the total absorbance curve and the area of interest selected for analysis. Here we select 30 minutes in the midst of the burn and look at the spectra of the evolved gases.

Hidden Analytical MS Systems for TG-MS



Combining a Thermogravimetric Analyzer (TGA) with a Mass Spectrometer (MS) allows you to detect very low levels of impurities. By heating a sample on the TGA, it will release volatile materials or combustion components as it burns. These gases are then transferred to the MS for

identification. The sensitivity of TG-MS is a powerful tool for quality control, safety, and product development.

When working with a hyphenated instrument, it is important to understand how each instrument works and how the connection affects them both. PerkinElmer manufactures a wide range of products, from thermal to gas chromatography and from infrared and Raman spectroscopy to Inductively Coupled Plasma (ICP).

Hidden Analytical™ MS Systems:

Hidden Analytical has a reputation for making state-of-the-art mass spectrometers and SIMS. Our collaboration allows us to offer you a range of hyphenated solutions that can address your diverse laboratory needs.

- 200, 300, and 500 amu systems available
- Mass range is upgradeable post sales
- Operation in Helium
- Variable or Soft ionization to control fragmentation
- Easy to connect transfer line
- In-line filters with wide capillary end
- User friendly software
- Automatic triggering of the MS run at the start of the TGA run

Hidden Analytical systems can be coupled to a range of PerkinElmer products allowing you to configure a system with the type of TGA and MS unit needed to address their particular applications and budget needs. PerkinElmer's TGA systems which can be used with Hidden Analytical Systems are:

- TGA4000 – our rugged, low cost TGA solution

- STA 6000 – DTA and TGA results simultaneously to 1000 °C
- STA8000 - DTA and TGA results to 1600 °C

In pharmaceutical manufacture, small amounts of recrystallization solvents need to be removed before processing the powder. TG-MS allows the detection of low levels of residual solvents as shown below.

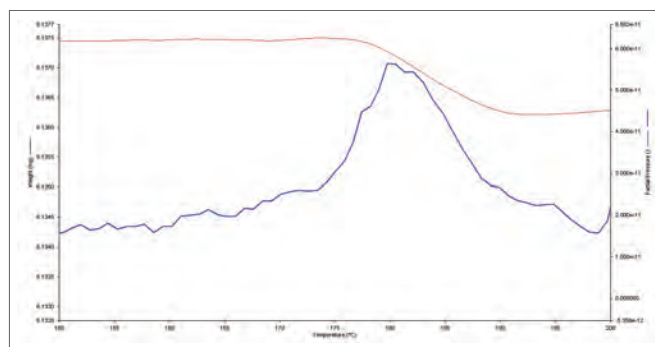


Figure 1. The combined TG-MS is ideal for detecting small traces of residual solvents in pharmaceuticals as shown in the detection of low levels of methylene chloride, above.

As you can see, the weight loss in the TGA is very small. Despite that, a clear identification of the material is obtained from the MS. In another case, a blend of solvents coming off in the same temperature range is identified. Shown below, the single weight loss in the TGA is seen to consist of water, ethanol, and acetone.

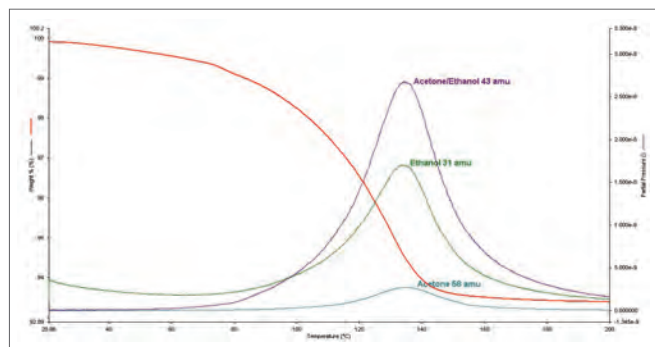


Figure 2. The combination of TGA and MS allows one to quantify the weight loss from a material and identify the components being evolved.

TG-MS



PerkinElmer's extensive product range means we can supply you with a complete system with one party service and support as well as the expertise and knowledge to help you use it effectively.

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-MS system uses:

- Our range of TGA and STA Systems, to optimize sensitivity to weight loss
- The TL8500 is equipped with a 350 °C transfer line, mass flow control, and pumps. The TL8500 allows connection to other brands of MS
- The Clarus SQ 8, for accurate identification

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

- Operation in Helium
- 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

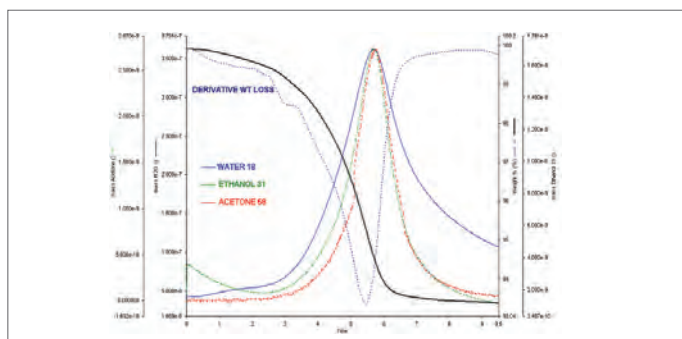


Figure 1. The combination of TGA and MS allow one to quantify the weight loss from a material and identify the components being evolved.

One of the advantages of TG-MS is it is real time and very sensitive. This has several applications in detecting residual solvents in pharmaceuticals and in measuring additives in polymers. In Figure 1, we see an example of a TGA curve, overlaid with the mass ion peaks for three common solvents.

Another example is the decomposition of rubber in the TGA while the evolved gases are tracked as a function of time (Figure 2).

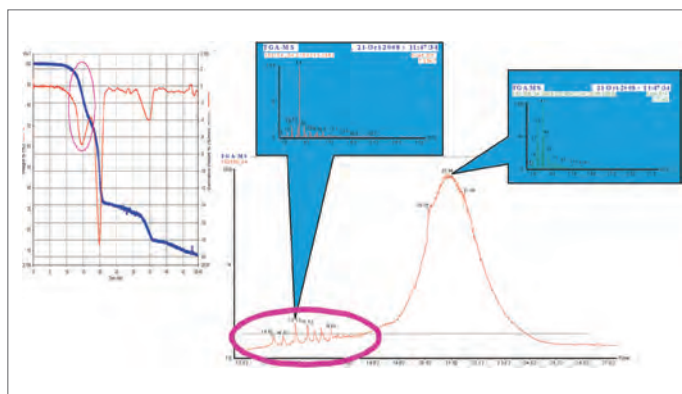


Figure 2. When burning a complex material like rubber, the TGA data is often confusing. MS on the evolved gas allows us to determine what comes off in the initial weight loss.

TG-GC/MS



Although TG-MS allows real-time monitoring, it can be confusing due to overlapping events and higher mass ions obscuring those of lower mass. By adding gas chromatography (GC) to the system, these events can be cleanly separated and very low levels of impurities detected.

Heating a sample on the TGA causes a sample to 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.

- Any of the TGA4000, STA6000, STA8000 or TGA 8000 can be connected depending on needed degree of precision and temperature range.
- 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 contaminants.

When working with a hyphenated instrument, it is important not only to understand how each of the instruments work, but also how the connection affects them both. Unlike many instrument companies, PerkinElmer makes a range of products, from thermal to gas chromatography and from Infrared and Mass Spectroscopy to ICP. Because of this experience, PerkinElmer is the only company capable of making, supporting, and servicing a combined TG-GC/MS system.

Coupling the PerkinElmer TGA 8000 to the Clarus SQ 8 GC/MS allows us to use several options to collect the evolved gases. Of these, the use of the chilled oven with the Swafer™ to collect the volatiles on the top of the GC column has been found to be most convenient.

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.

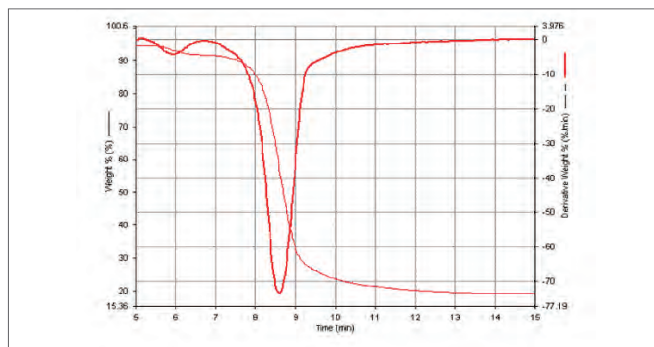


Figure 1. The TGA run of a sample of switchgrass shows most weight loss occurs in one temperature range.

TG-GC/MS

Collecting the material on a GC column and eluting it gives the following chromatogram. Using the data from the MS to identify the peaks, the progression of acids can be detected easily in the offgas. These components can not be isolated by TG-IR or TG-MS due to their low levels and the complexity of the mixture.

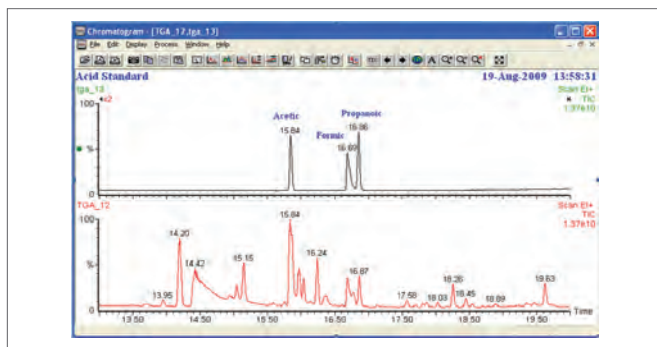


Figure 2. GC/MS on the gases evolved between 8 and 9 minutes and collected on the head of a GC column gave the chromatography seen on the bottom of the graph. MS analysis suggest that 15.8 is the acetic acid, which is confirmed above by running a standard of acetic, formic, and propionic acids.

TG-IR-GC/MS



Hyphenating TG-IR-GC/MS is a powerful approach for analysis of an unknown mixture to determine its primary components and identify additives or contaminants. This information may be needed, for example, to evaluate a competitor's product or to determine compliance with regulations.

The PerkinElmer TL9000 transfer line is used to allow TG-IR-GC/MS analysis on a sample by moving the off gases to the FT-IR and GC/MS after their evolution in the TGA. It acts as the interface between a TGA or STA, an FT-IR like the Frontier or Spectrum Two FT-IR, and a Mass Spectrometer or GC/MS, such as the Clarus SQ 8.

During the TGA thermal separation of a pigmented aqueous sample, the gases released by the sample were sent to the FT-IR for spectral analysis. The TG-IR data consists of a sequence of spectra, acquired at intervals of around 8 seconds. The standard presentation of the data is the adsorption versus wave number, and this spectral profile of the gases released by the sample is generated for each at roughly a two-degree interval. The TG-IR Spectrum Time Base Software provides a 3D graphical representation, consisting of stacked IR spectra, a feature that provides a snapshot of the entire TG-IR separation.

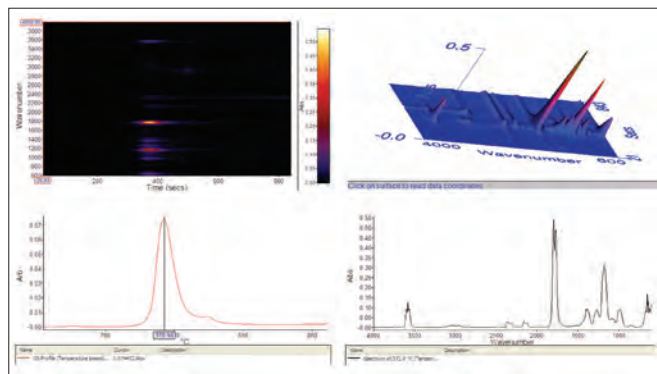


Figure 1. Spectrum Time-Based software outputs, which aid in interpreting the decomposition process. With experience, an operator will look at the stacked spectra (upper right plot) and see an “unexpected mountain range” that represents the transient presence of a particular species of off-gassing product of potential interest.

The TL9000 interface was used to perform a subsequent analysis to confirm the identity of the unknown substance in the aqueous sample. At the time of maximum concentration absorbance of the substance being analyzed, the gas in the IR gas cell was sent to a GC/MS.

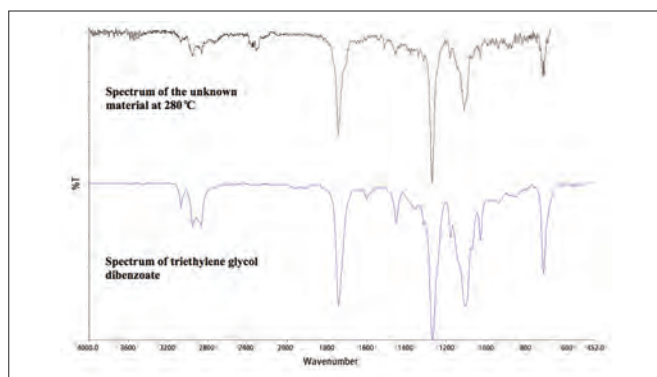


Figure 2. Best match spectra using PerkinElmer Spectrum Search software.

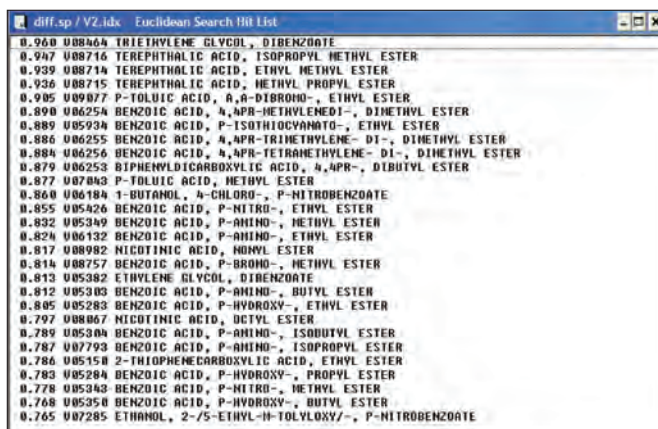


Figure 3. Output of Search software, showing match candidates.

Advantages

- TG-IR-GC/MS allows one to gain the advantages of two EGA techniques and compensate for their disadvantages.
- Unlike TGA-GC/MS, the TG-IR-GC/MS retains the relationship between temperature a gas is released at and its components by FT-IR.
- The sequentially measuring of the offgas components by GC/MS allow detection of trace levels too low to be seen in the FT-IR.

For more information, visit www.perkinelmer.com/hyphenation

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