PRODUCT NOTE

Thermogravimetric Analysis – Infrared Spectroscopy

PerkinElmer TG-IR EGA System.

- Complete system from a single supplier to measure evolved gases
- Allows IR measurement of gases evolved from the TGA
- Engineered solution for gas transfer, delivering high sensitivity and precise control

TL 8000 – Evolved Gas Analysis Using TG-IR

Introduction

Hyphenation, which is the connection of two analytical instruments to give more data than the single-component analyses, is vitally important to academia and industry due to the overriding need to fully characterize a material. Evolved gas analysis (EGA) is one such technique and can be carried out using thermogravimetric analysis combined with either mass spectrometry (TG-MS) or gas chromatography/mass spectrometry (TG-GC/MS) with the TL 2000 transfer line or combined with infrared spectrometry (TG-IR) with the TL 8000 transfer line.

The TL 8000 embodies PerkinElmer's many years of experience in both infrared spectroscopy and thermal analysis. Every aspect of the design of the TL 8000 is targeted at giving better performance and more reliable results. This is achieved by having a large operating temperature range, a short, robust transfer line, a balanced flow design and the unique zero-gravity-effect IR cell.

PerkinElmer®
For the Better

Large Temperature Range

While most TG-IR systems offer temperatures up to 300 °C, the TL 8000 boasts a maximum temperature of 350 °C for both the transfer line and the cell. The benefit of this is two-fold:

1. Enables heavy, high-boiling-point products such as flame retardants to travel through the system without the issues of condensation and deposition.

2. Gives peace of mind that in between runs a higher temperature can be used to burn off the system, preventing cross contamination.

The Transfer Line

Unlike some other transfer lines available which are easily deformed, the TL 8000 has a SilcoSteel®-coated copper tube wrapped with heating coils surrounded with insulation and a tough outer coating, ensuring robustness. A short line length of 1 m also means that the time lag between the two instruments is minimized, improving time resolution and dramatically reducing the probability of products condensing. The tube is also replaceable for cleaning, if required.

Balanced Flow

Balanced flow means that throughout the system, gas flow and temperature are both monitored and regulated. This is carried out by a mass flow controller and a filtration system contained in the controller box. Together with the constant gas temperature provided by the line and cell heaters, the resulting effect is that constant gas pressure and rate can be maintained throughout the system. The consequence of this is better separation of evolved products, leading to much clearer spectra and better time-resolved data. Figure 1 shows a Gram Schmidt profile (derived from IR plots) of ethylvinylacetate (EVA) heating at 20 °C/min from 50 to 650 °C, highlighting the sharp time-resolved data.

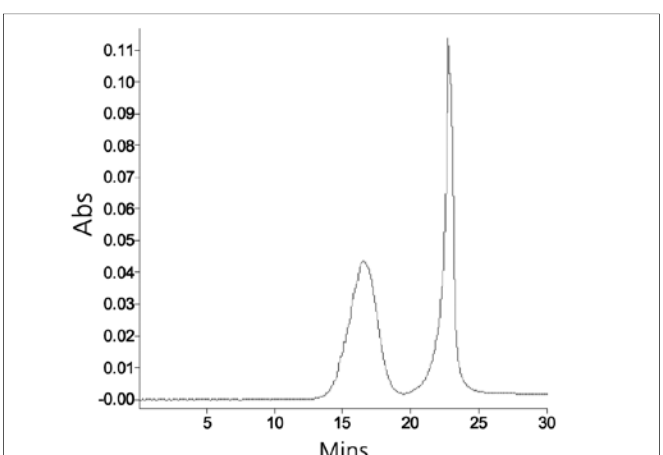

Figure 1. Gram Schmidt profile for EVA heated at 20 °C/min between 50 and 650 °C recorded using the TL 8000.

The IR Cell

The design of the IR cell is critical to the performance of the system. The cell has a path-length of 100 mm, which allows for high sensitivity without requiring a large volume. The small volume of 11.3 cm^3 improves time resolution and reduces the cleaning times between runs, therefore increasing productivity. The ability to purge the spectrometer sample compartment means that atmospheric absorption is minimized and, furthermore, the cell has the flexibility of using either standard KBr windows or ZnSe, if moisture resistance is required. The cell inlet and outlet ports are designed to ensure that heavy components are passed through the cell efficiently. With conventional cells, heavy fractions of evolved gas can accumulate in the cell (Figure 2B), thus compromising the time resolution of the signal. With the zero-gravity-effect cell (Figure 2A), the downward-facing outlet ensures that this does not occur.

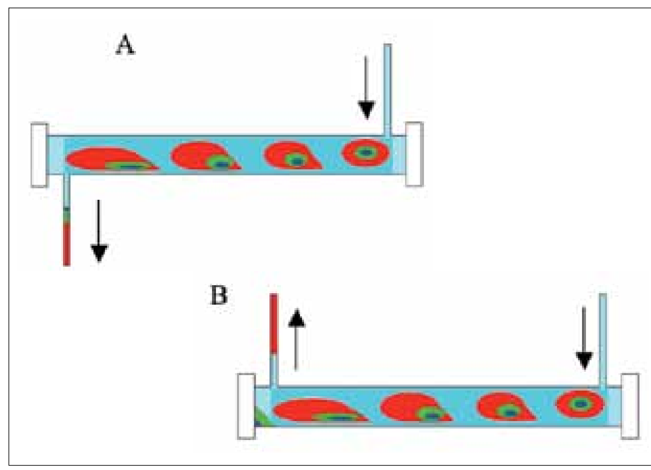

Figure 2. Graphical representation showing the zero gravity cell (A) and the build-up of heavy fractions in an ordinary IR cell (B).

Software

The TL 8000 system is controlled by two powerful pieces of software. Pyris™ Software controls the thermal analysis instrument, allowing desired thermal programs to be specified as well as automated triggering of the IR software (a representative method is shown in Figure 3 – Page 3).

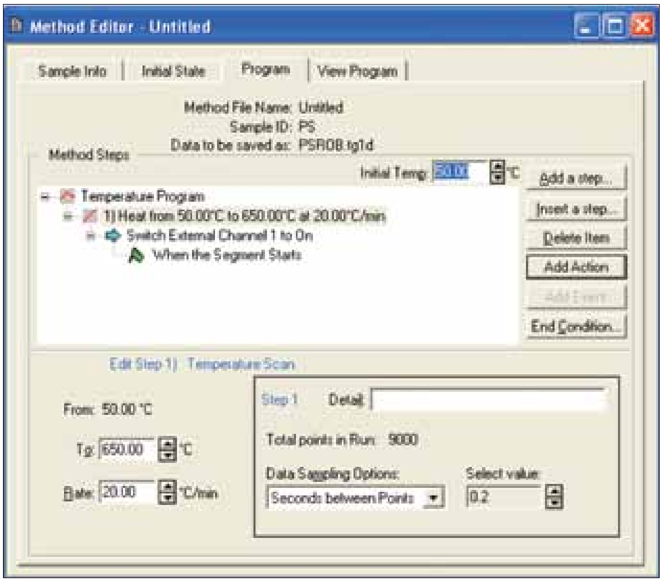

Figure 3. Method Editor window from Pyris Software showing a heating regime with the triggering functionality enabled.

The full manipulation of thermal curves produced is also available in Pyris Software with an example of the thermal data produced by the TL 8000 shown in Figure 4. The data was collected on heating ethylene vinyl acetate from 30 to 600 °C at 20 °C/min. The data in blue shows the weight change on heating, indicating two weight-loss events. The derivative weight loss is also presented in red.

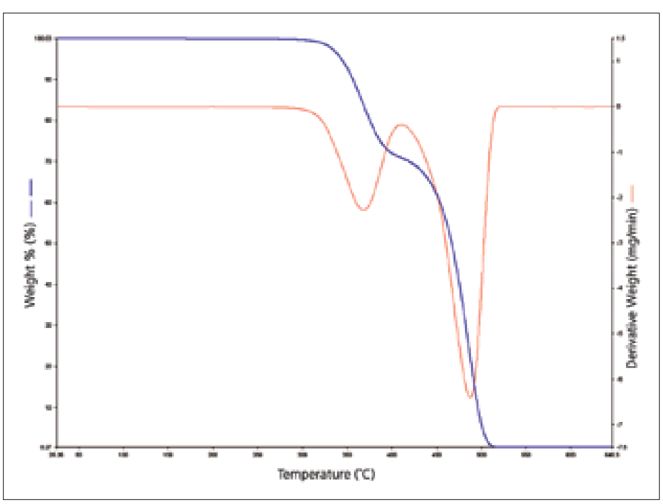

Figure 4. TGA plot showing EVA heating from 30 to 600 °C at 20 °C/min shown in blue with the derivative shown in red.

The second piece of software is Spectrum™ TimeBase™, which allows for time-resolved IR experiments. TimeBase waits for a trigger from Pyris (supplied via a relay box) by a simple tick box (Figure 5).

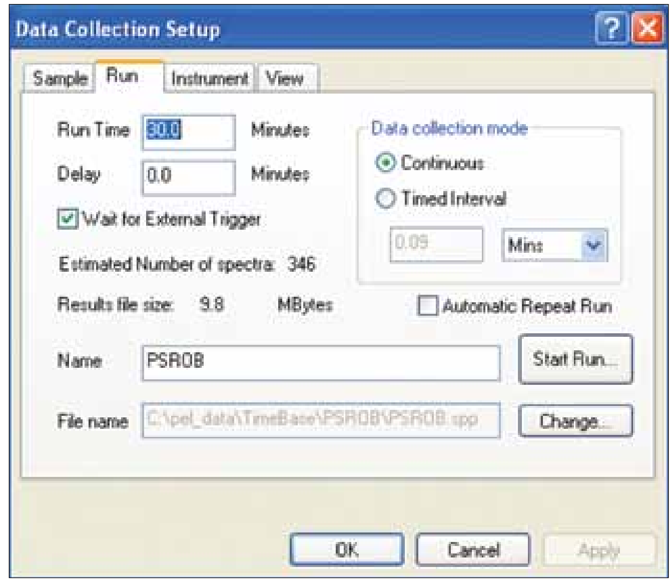

Figure 5. Data Collection Setup window for Spectrum TimeBase Software.

TimeBase allows for full manipulation of the IR data including Gram-Schmidt plots, individual functional group data, as well as individual spectra recorded during the run. Pyris data can also be exported into TimeBase as the raw data (Figure 6), or as the derivative plot. The plots in Figure 7 (Page 4) show the imported Pyris data for EVA (Figure 4) on the bottom in blue along with the Gram-Schmidt thermogram derived from the infrared absorbance and shown in red. This feature allows easy comparison of the data from the two instruments, and here it can be seen that the two periods of weight loss are associated with absorption by the evolved gases. TimeBase also allows construction of profiles corresponding to intensity of individual spectral bands and even quantitative Beer's Law or chemometric models.

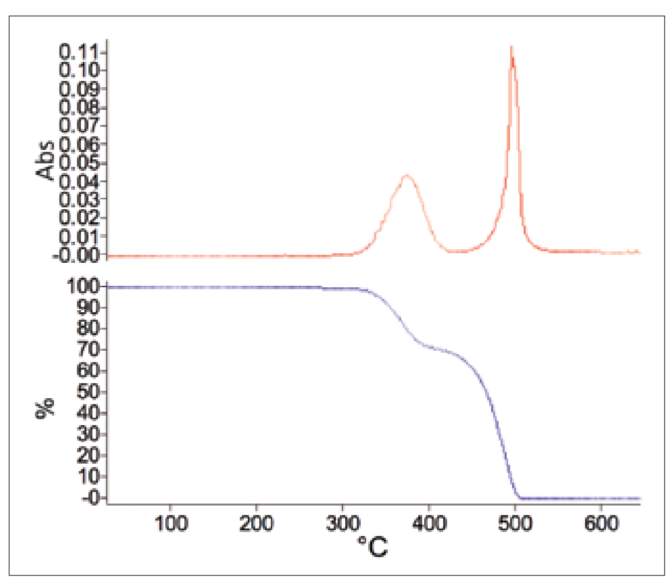

Figure 6. Weight-loss curve (blue) and Gram-Schmidt thermogram (red) for EVA recorded at 20 °C/min between 30 and 600 °C.

The system is also supplied with PerkinElmer® Spectrum 10 software, which allows sophisticated processing and analysis of the individual infrared spectra, such as library searching and comparison against reference spectra. As shown in Figure 7, the first peak in the Gram-Schmidt thermogram corresponds to acetic acid (and a small amount of CO_2). The second peak in the thermogram occurs as the polymer backbone breaks down, giving (in the nitrogen atmosphere) soot and aliphatic hydrocarbons (Figure 8).

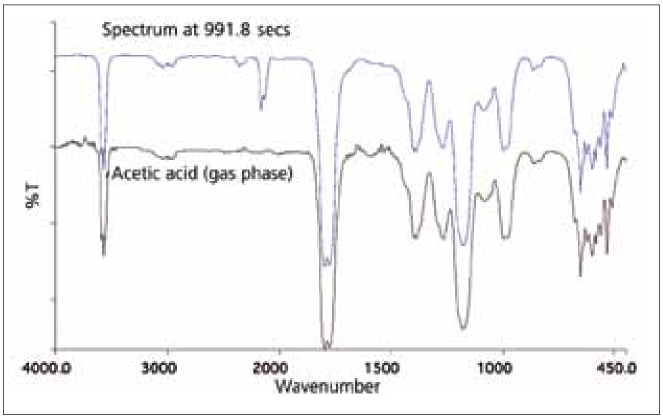

Figure 7. Comparison of the infrared spectrum recorded at 991.8 s (362.3 °C) with a reference acetic-acid spectrum. The identification is unambiguous, and the Compare function returns a correlation of 0.99, indicating excellent agreement. (The doublet around 2150 cm^{-1} is due to CO_2 in the evolved gas.)

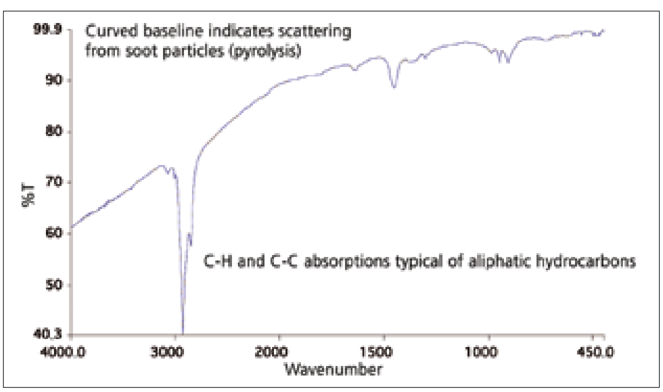

Figure 8. The infrared spectrum recorded at 1364.5 s (477.2 °C) shows that the polymer backbone degrades to give soot and long-chain hydrocarbons.

Compatibility

The TL 8000 system is compatible with the PerkinElmer Pyris 1 TGA, STA 6000 and TGA 4000 thermal analyzers and PerkinElmer's Frontier range of IR spectrometers. For further compatibility options please contact your PerkinElmer representative.

Conclusion

Design features such as balanced flow and the unique IR cell mean that the TL 8000 evolved gas analysis system gives the highest quality and most reliable results of any TG-IR system on the market.

To learn more about TG-IR or other hyphenated technology, visit www.perkinelmer.com/hyphenation

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