

TGA-FTIR for Material Decomposition Analysis

By Jenni L. Briggs, PhD, PIKE Technologies

Thermogravimetric analyzer (TGA) monitors sample weight loss caused by volatilization and degradation often as a function of temperature ramping using a high precision balance and furnace. Typical samples include polymers, epoxies, fibers and laminates for investigating deformation, thermal stability, or comparative study applications. TGA only measures the mass loss caused by evolved gas events. By coupling TGA with an FTIR, unique spectroscopic data as well as functional group information of the evolved gases may be now obtained.

The aims of this application note are to detail the [PIKE TGA-FTIR Accessory](#), and present sample data.

Methods

The heated gas cell was placed in the FTIR sample compartment. One end of the heated transfer line was interfaced to the inlet port of the gas cell and the other end to the TGA. The gas cell and transfer line was held at 300 and 280 °C, respectively. FTIR spectra were collected at 8 cm⁻¹. Ethylene vinyl acetate was loaded into the TGA furnace. TGA temperature was ramped from 30 – 550 °C at a rate of 40 °C/min.

Results and Discussion

The PIKE Technologies TGA-FTIR Accessory is designed to be an interface for evolved gas analysis from a TGA to an FTIR spectrometer. Evolved sample gases originating from the TGA pass through the heated transfer line into the beam-conforming heated flow cell placed in the FTIR sample compartment. As these evolved gases travel through the flow cell, FTIR spectra are collected and stored for further processing. Qualitative and quantitative measurements are doable from sample masses typically in the low milligram range.

In this application, ethylene vinyl acetate exhibits two weight loss transitions upon heating. The first is the release of acetic acid occurring near 600 s into the experimental run followed by degradation of the main chain ethylene observed near 725 s. The weight loss trace and corresponding IR spectrum are shown in Figures 2 and 3. The acetic acid exhibits a strong C=O bond at 1780 cm⁻¹. The C-H stretch band near 2950 cm⁻¹ is a prominent feature of the ethylene gas.

Conclusion

Using the PIKE TGA-FTIR Accessory allows unique chemical information to be obtained from traditional TGA decomposition experiments through the incorporation of IR spectroscopy. The PIKE transfer line and heated gas cell may be configured for most TGA ovens and FTIR spectrometers.



Figure 1. TGA-FTIR Accessory from PIKE Technologies

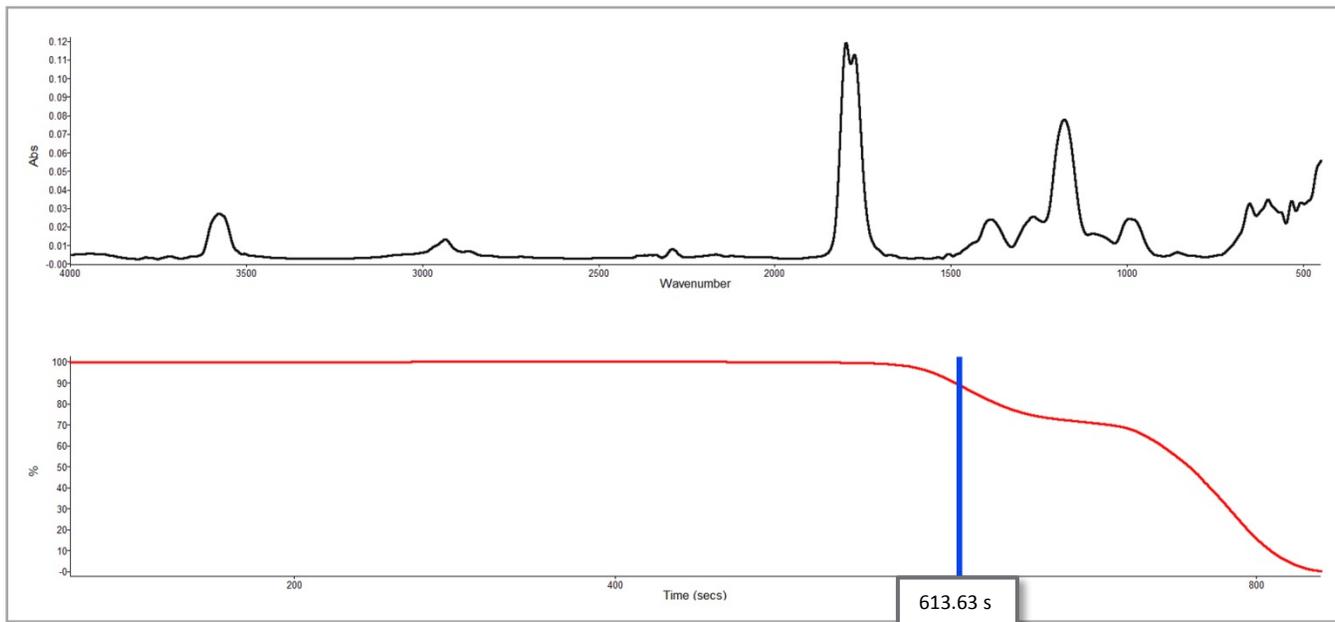


Figure 2. Weight loss trace and corresponding FTIR spectrum of acetic acid.

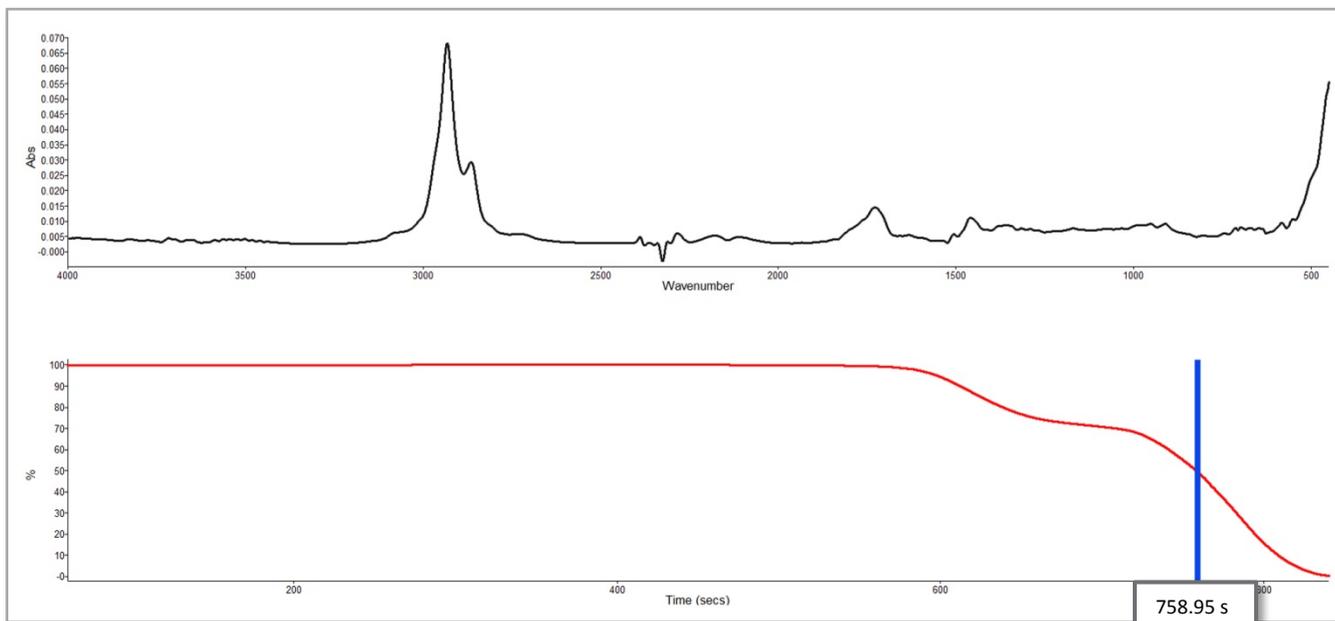


Figure 3. Weight loss trace and corresponding FTIR spectrum of ethylene.