Thermal Gravimetric-Infrared (TGA-IR) Analysis of Ammonium Acetate

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The TGA-IR combines the sample analysis tools of a TGA with the identification power of an FT-IR spectrometer to offer real-time monitoring of gases as they evolve from a sample.

The combination of thermal gravimetric analysis and infrared instrumentation allows the measurement of the change in weight of a sample as a function of temperature or time in a controlled atmosphere and the collection of the IR spectra of the evolved gas components. TGA-IR has many applications in today's world. It can be used in polymer research to determine the evolution of residual solvents, monomers, additives, and in the study of flame-retardant materials. In the electronics industry it can be used to study off-gassing by components and structural materials. In the study of composites and inorganic materials TGA-IR can be used as confirmation of small molecule loss.

The TGA-IR interface is connected to the TGA via a heated transfer line. As gases evolve during the TGA experiment, they pass into the flow cell of the TGA-IR interface where the infrared spectra are collected. The TGA-IR interface is beneficial in determining sample characteristics such as decomposition pathways, thermal stability or sample integrity. Its flexible interface design can be placed in the sample compartment of the FT-IR.

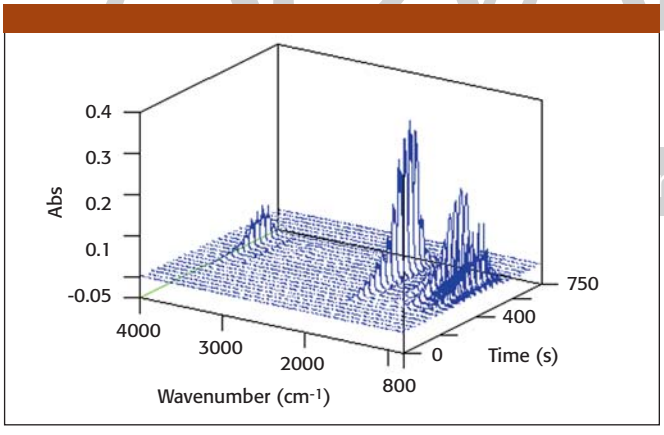

Figure 1. 3D FT-IR data of the evolution of gases from the ammonium acetate.

Experimental Conditions

The TGA-IR experiment was used to follow the thermal melt of Ammonium Acetate (CH_3COONH_4). The instrument consisted of a TA Instruments SDTQ-600 TGA unit attached to the Jasco FT/IR-660

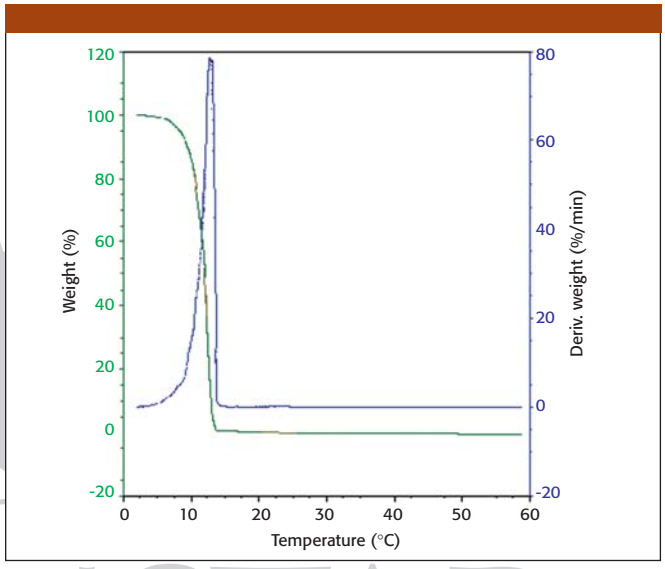

Figure 2. TGA curves. (Top) derivative curve (Bottom) weight loss curve.

instrument with a standard source and the DTGS detector via a heated gas line and 10 cm gas cell (TG-IR interface) from Pike Technologies.

A flow rate of 100 ml/min of nitrogen was used to sweep the evolved gases from the TGA furnace to the FT-IR gas cell. The temperature was varied from ~20 °C to 600 °C with a ramp rate of 20 °C/min. FT-IR scans were collected every 60 seconds, 12 scans at 4 cm^{-1} resolution used to follow the gases evolved from the thermal melt of the sample. The gases evolved included Ammonia and Acetic Acid. The interval software was used to collect and analyze the data contained in this note.

Results

Figure 1 shows the infrared spectra of the evolution of ammonia and of acetic acid over time. The evolved gas spectra corresponds to the weight loss of the sample in the TGA. Figure 2 shows the weight loss and 1st derivative curves for the sample generated by the TGA instrument.

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