TA Instruments

Thermal Analysis & Rheology

THERMAL SOLUTIONS

CLARIFICATION OF INORGANIC DECOMPOSITIONS BY TGA - MASS SPECTROMETRY

PROBLEM

Thermogravimetric analysis (TGA), which measures weight changes in a material as the material is heated, provides a convenient method for characterizing the decomposition process in inorganics. For example, calcium oxalate monohydrate ($CaC_2O_4 \cdot H_2O$), a standard material often used to verify TGA performance, exhibits three weight losses with temperature (Figure 1) in an inert atmosphere that appear to represent the loss of water, carbon monoxide and carbon dioxide via the process:

$$CaC_2O_4 \cdot H_2O \xrightarrow{-H_2O} CaC_2O_4 \xrightarrow{-CO} CaCO_3 \xrightarrow{-CO_2} CaO$$

Absolute confirmation of this process is possible when the gaseous by-products are identified as they evolve.

SOLUTION

Mass spectrometry (MS) is a sensitive, specific method for identifying gaseous materials. When coupled directly to the TGA, MS provides qualitative information which complements TGA and yields a more complete understanding of the decomposition process. In the case of calcium oxalate monohydrate, the MS histograms for water, carbon monoxide and carbon dioxide indicate that the actual decomposition process is more complex than shown by TGA alone (Figure 2). The second weight loss, in particular, results in both CO and CO_2 being evolved as off-gases. This seems hard to explain in the absence of oxygen (when an inert purge gas is used). However, it can be explained based on further reaction of some of the CO initially formed. Specifically, $2CO \rightarrow CO_2 + C$. The presence of this secondary reaction has been proposed in the literature (1) and is confirmed by examining the residue after the second weight loss has occurred. The presence of black carbon along with the remaining white $CaCO_3$ yields a gray residue.

Figure 1. CALCIUM OXALATE MONOHYDRATE (TGA Results)

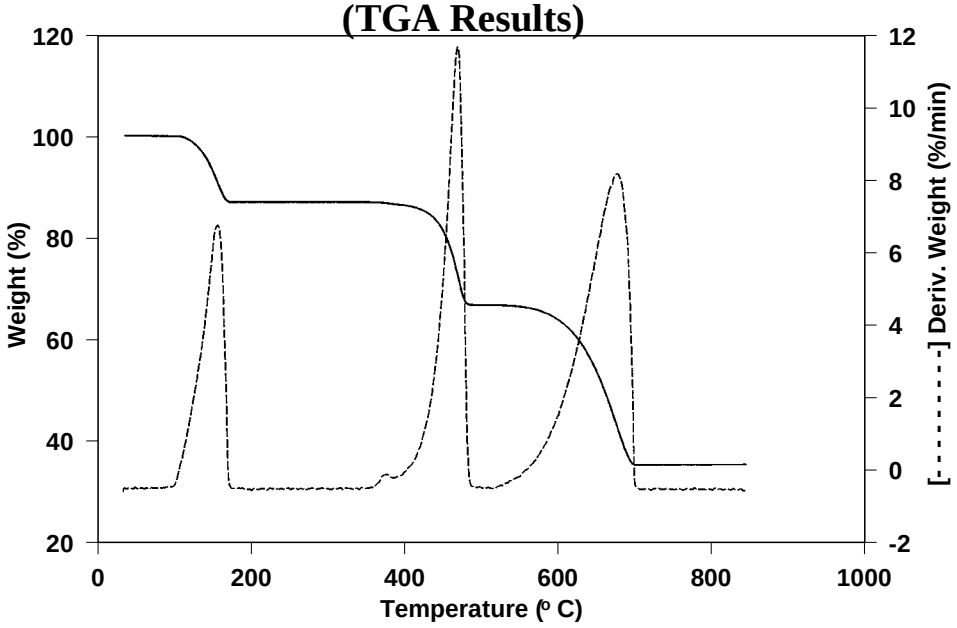
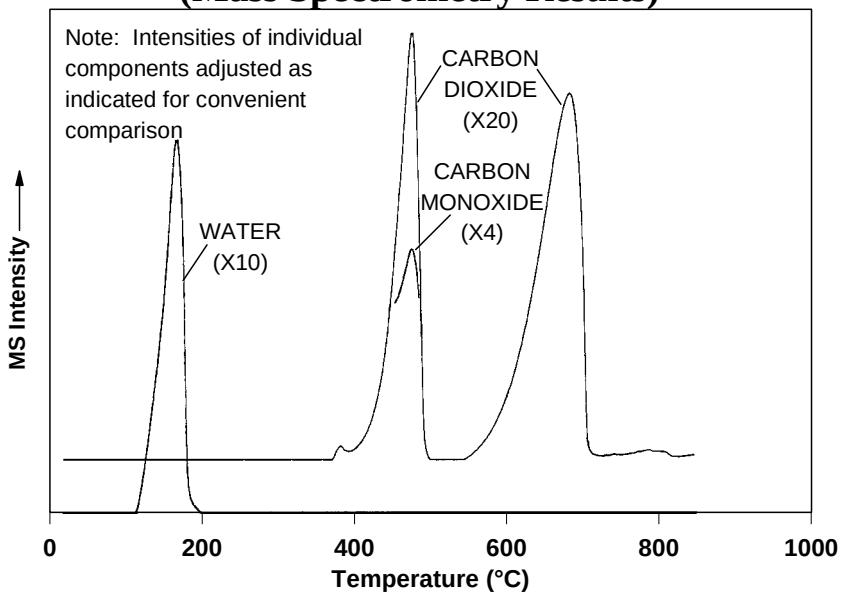


Figure 2. CALCIUM OXALATE MONOHYDRATE (Mass Spectrometry Results)



REFERENCE

1. E. Charsley et al., American Laboratory, January, 1990

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