APPLICATION**NOTE**

Acids – Simultaneous Thermal Analysis/ FT-IR

Citric Acid Decomposition Unveiled by TGA-DSC-FT-IR

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Introduction

Citric acid is a widely used tricarboxylic acid found in the food, pharmaceutical, and cosmetic industries, as well as in chemical applications such as chelating agents and bio-based polymer synthesis. Since it is frequently exposed to elevated temperatures during processing and storage, precise understanding of its thermal stability, dehydration behavior, and decomposition pathway is essential to avoid unwanted loss of active substances or the release of gaseous decomposition products. Simultaneous thermal analysis coupled with evolved gas analysis provides a powerful tool to characterize not only the mass loss and associated heat effects, but also the chemical identity of the volatiles released, allowing reliable and unambiguous interpretation of the underlying thermal processes.

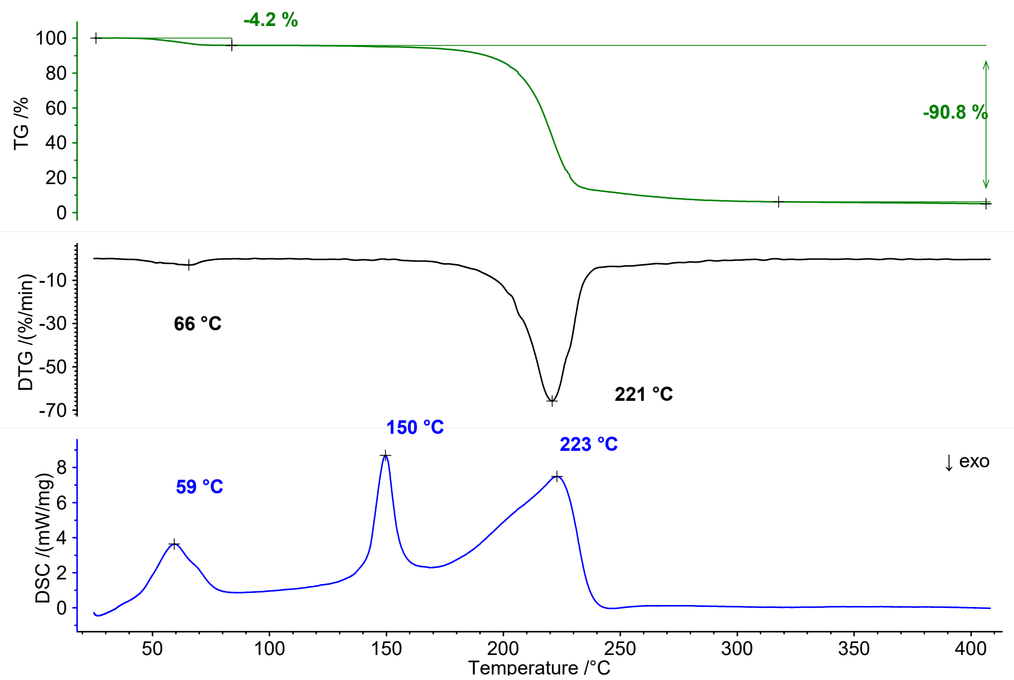
Mesasurement Conditions

Table 1 Masurement conditions

Instrument	STA *Jupiter*® FT-IR INVENIO
Sample	Citri acid
Sample mass	14 mg
Temperature range	30 - 400°C
Heating rate	20 K/min
Atmosphere	N_2, 70 ml/min
Crucible	Pt, 85 µl with pierced lid

Results and Discussion

The TGA-DSC curve in Figure 1 shows a multi-step thermal behavior of citric acid. A small mass loss of 4.2 %



1 Temperature-dependent mass change (TGA, green), rate of mass change (DTG, black) and heat flow curve (DSC, blue) of citric acid

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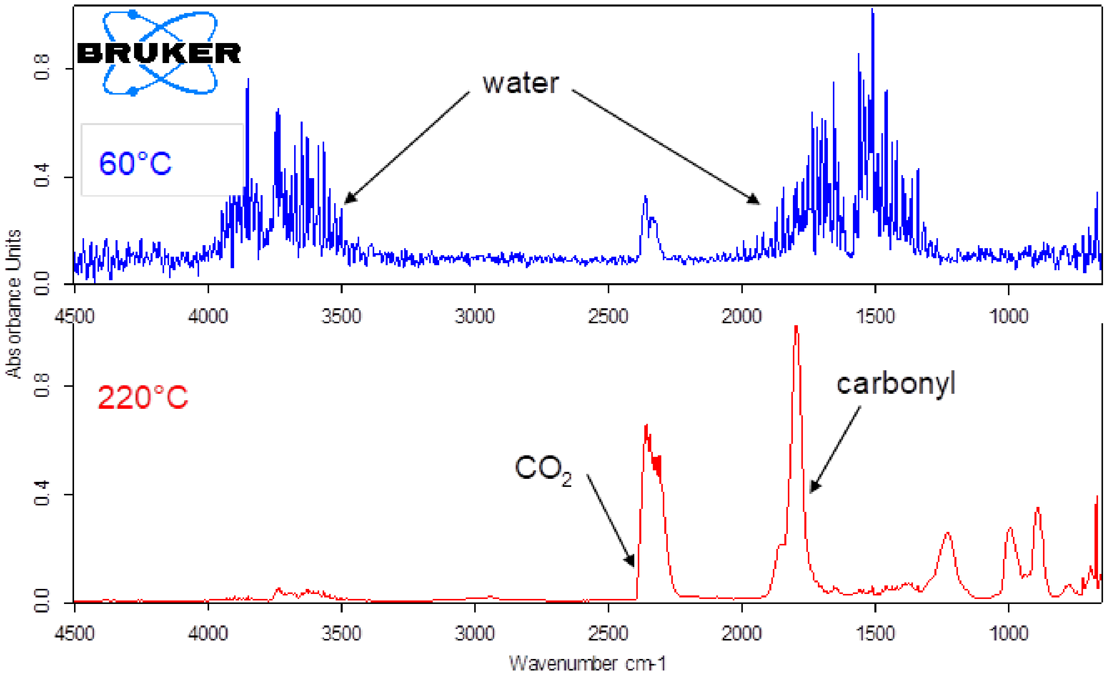

2 FTIR spectra of the gas phase evolved during the TGA-DSC measurement of citric acid, recorded at 60°C (top, blue) and 220°C (bottom, red).

occurs up to approximately 100°C, accompanied by a broad endothermic DSC signal with a peak temperature of 59°C and a DTG peak at 66°C. This step is attributed to the release of crystal water.

At 150°C, the DSC curve shows a sharp endothermic peak, which results from the melting of citric acid. Above this temperature, the material enters the thermal degradation region. The main decomposition step occurs between approximately 190°C and 240°C and results in a major mass loss of 90.8 %. The DTG curve shows the maximum decomposition rate at 221°C, while the DSC curve displays a strong endothermic peak at 223°C. This close agreement between DTG and DSC confirms that the largest mass-loss process is directly linked to an endothermic decomposition reaction.

The TGA-DSC data provide clear information about the temperature range, mass loss, and thermal character of the events. However, these data alone cannot identify the chemical nature of the released gases. For this reason, coupling thermal analysis with FT-IR spectroscopy of the evolved gas phase is essential. The FT-IR spectrum recorded at 60 °C, corresponding to the first mass-loss step, shows the characteristic rotational-vibrational fine structure of gaseous water in the regions around 3500-4000 cm^{-1} and 1300-2000 cm^{-1}, confirming that the initial low-temperature mass loss is due to physically or loosely bound water rather than to any decomposition reaction, see Figure 2. In contrast, the FT-IR spectrum recorded at 220°C, which corresponds to the major mass-loss step, reveals two dominant absorption features, a doublet band near 2300-2360 cm^{-1} characteristic of carbon dioxide, and a strong, sharp band near 1750-1800 cm^{-1} assigned to carbonyl stretching vibrations. These signals demonstrate that the main decomposition step is driven by the decarboxylation of the citric acid molecule, with simultaneous formation of carbonyl-containing fragments.

Summary

The thermal characterization of citric acid by simultaneous TGA-DSC combined with FT-IR gas-phase analysis reveals a three-step thermal profile consisting of an initial loss of residual moisture, melting and a major decarboxylation-driven decomposition above approximately 200°C. The coupling of thermal analysis and evolved gas data by FT-IR proved essential to correctly assign each thermal event to a specific physical or chemical process, providing a solid basis for defining safe processing and storage temperature limits for citric acid in industrial applications.

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