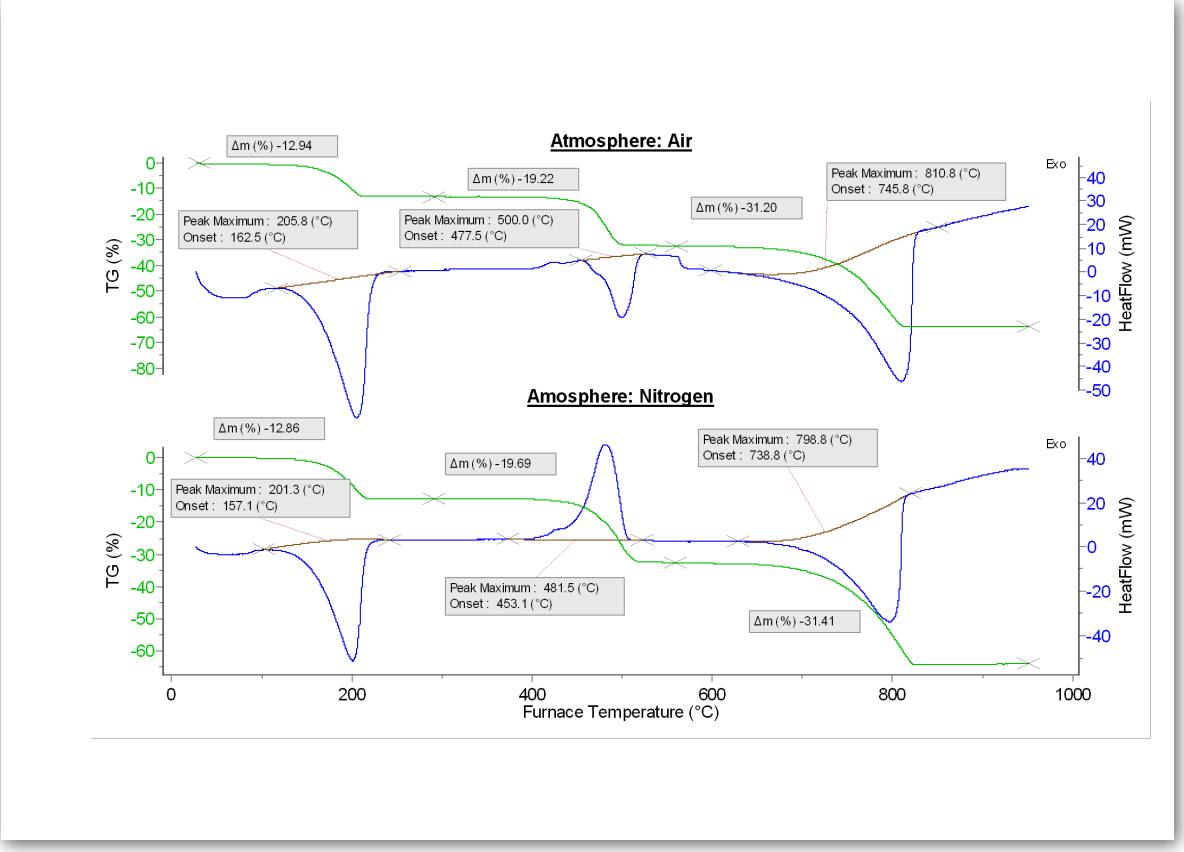
Thermal characterization of calcium oxalate

INTRODUCTION

The characterization of minerals involves the analysis of their composition. For instance, calcium oxalate is a calcium salt of oxalate whose monohydrate form occurs naturally as the mineral whewellite.

TGA allows for the characterization of minerals through their mass variation during heating and when different reactions like dehydration, dehydroxylation, or thermal dissociation occur. STA provides additional information about the thermal behavior of the mineral, like the endothermic or exothermic character of the detected reaction.



EXPERIMENT

SETLINE STA was used to characterize the thermal decomposition of calcium oxalate monohydrate. Its behavior under nitrogen and air were compared. A sample amount of 15 mg was weighed and inserted in an alumina crucible for each experiment. The following profiles were then applied:

- Heating from 30°C to 700°C at 10°C/minute
- Air or nitrogen flow at a rate of 30 ml/min

RESULTS AND CONCLUSION

The results of calcium oxalate monohydrate run under flowing air and under flowing nitrogen show three different mass losses.
For the two experiments, the first mass loss is linked to an endothermic effect (the peak on the Heatflow signal is down) due to the loss of the hydrate water:
- $CaC_2O_4, H_2O \rightarrow CaC_2O_4 + H_2O$

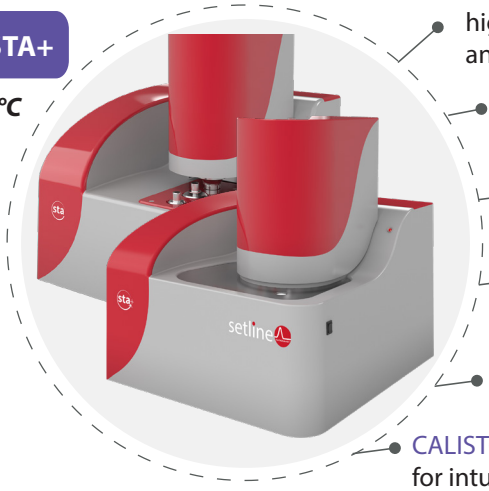
Under nitrogen, the second mass loss is associated to an endothermic peak corresponding to the decomposition of the oxalate with loss of carbon monoxide:
- $CaC_2O_4 \rightarrow CaCO_3 + CO$

Under oxidative atmosphere, the CO emitted is oxidized immediately and the effect linked to the second mass loss is exothermic (the peak on the Heatflow signal is up). Finally, for both gases, the last mass loss is due to the decomposition of calcium carbonate which is an endothermic effect:
- $CaCO_3 \rightarrow CaO + CO_2$

INSTRUMENT

SETLINE STA / STA+

Ambient to 1100°C



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