

Calcium oxalate decomposition under high pressure

INTRODUCTION

The influence of pressure over the thermal decomposition behavior of mineral materials is important in order to assess their reactivity and stability. Calcium oxalate monohydrate is a well characterized material frequently used to check the performance of a thermogravimetric analyzer.

EXPERIMENT

THEMYS HP was used for this experiment. A 56.1 mg calcium oxalate monohydrate sample was placed in a 1300 μ l crucible and heated from 30 to 1000 $^{\circ}$ C at a rate of 10 $^{\circ}$ C/min under a flow of nitrogen at 50 bar.

RESULTS AND CONCLUSION

The typical three step decomposition behavior of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ is observed, with the loss of one molecule of water per molecule of calcium oxalate monohydrate at low temperature (i), the loss of carbon monoxide at an intermediate temperature range (ii) and finally the loss of carbon dioxide at higher temperature (iii). The results fit well with the theoretical mass losses.

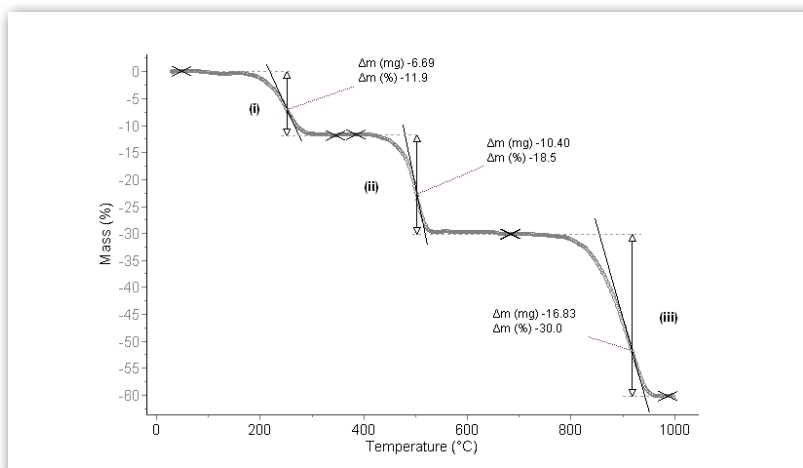


Figure 1 – Mass variations vs. temperature at 50 bar

| Mass loss | Theory | Experiment |
|-----------|---|------------|
| (i) | $M_{\text{H}_2\text{O}} / M_{\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}} = 12.3\%$ | 11.9% |
| (ii) | $M_{\text{CO}} / M_{\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}} = 19.1\%$ | 18.5% |
| (iii) | $M_{\text{CO}_2} / M_{\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}} = 30.1\%$ | 30% |

Table 1 – Comparison of experimental vs. calculated mass losses

INSTRUMENT

THEMYS HP



HIGH TEMPERATURE AND HIGH PRESSURE CAPABILITY
up to 1200 $^{\circ}$ C and 150 bar with a single furnace

High Accuracy and Versatile Hang-down Symmetrical Beam Balance

continuously measure sample mass variations, drastically limit the TGA signal background noise and reduce drift, improve gas/sample interaction and specially designed for TGA applications