

Chemistry 201

Gravimetric Analysis

NC State University

Mass measurement

The most basic measurement of mass can be carried out using an analytical balance with an accuracy 1 mg or perhaps a fraction of a milligram (with appropriate care).



An analytical balance.

Gravimetric analysis: precipitation

One can determine the mass of an ion in solution by causing a precipitation. The precipitant can then be weighed to provide a measurement of the mass.

We can use the stoichiometry to quantify, which species were in solution.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) measures changes in the physical and chemical properties of materials as a function of increasing temperature. Usually a constant heating rate is used as a function of time. TGA can provide information about physical phenomena, such as phase transitions, such as vaporization, sublimation, absorption, adsorption and desorption from materials. Likewise, TGA can provide information about chemical phenomena including chemisorption, dehydration and solid-gas reactions.

TGA instrumentation

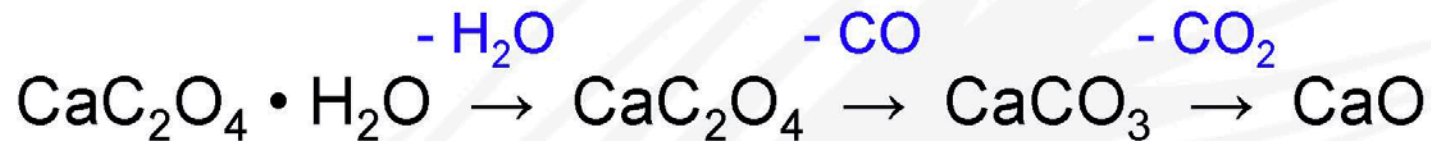
The TGA instrument uses a sensitively balanced sample pan. Mass changes on the sample pan can be detected with microgram accuracy. One places a small amount of sample on the pan and then the jacketed heating unit moves into place and surrounds the pan. Then the effects of heating on the mass of sample is measured.



TGA Example

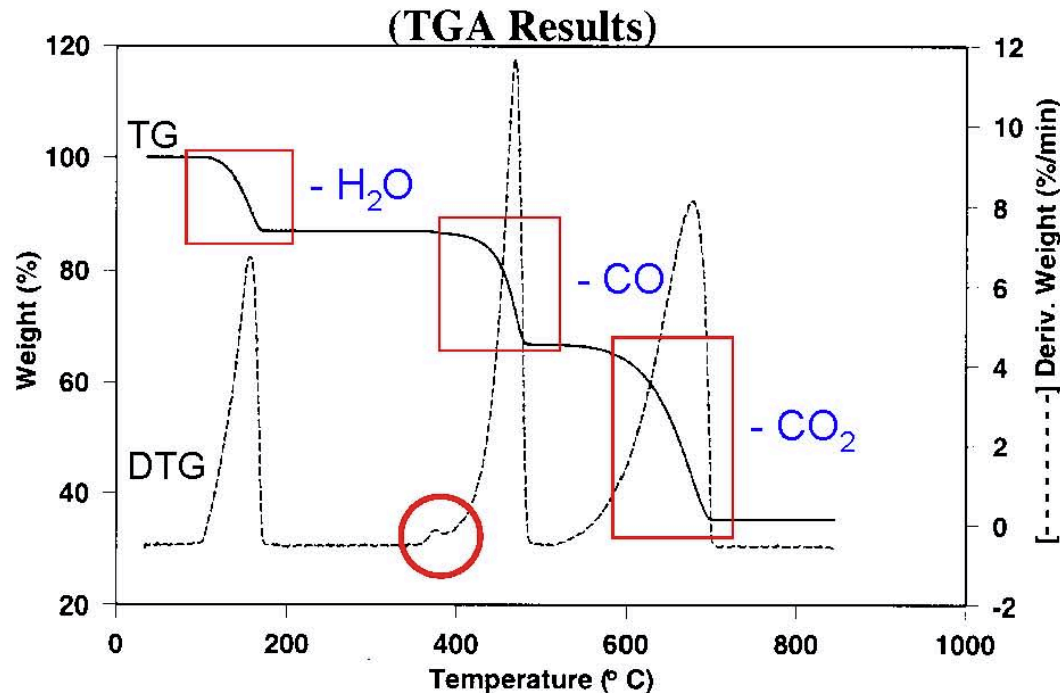
Decomposition of calcium oxalate monohydrate

- Calcium oxalate monohydrate, a standard material often used to demonstrate TGA performance.
- Exhibits three weight losses with temperature in an inert atmosphere (e.g. N₂).



TGA Example

Decomposition of calcium oxalate monohydrate



Absolute confirmation of the decomposition process is possible when the gaseous by products are identified as they evolve, eg. by mass spectrometry (MS).
