TGA evaluation

Thermogravimetry is a technique that measures the change of mass of a sample as a function of temperature or of time. Further analysis of the weight loss steps requires the evaluation methods included in the option 'TGA evaluation'.
The quantitative determination of the component of interest in a mixture is often possible if it exhibits a single isolated weight loss. Examples of this are the loss of water from hydrates, hydroxides and carbohydrates, the decomposition of organic or inorganic compounds (pyrolysis) or the oxidative decomposition (combustion) of organic materials. Evaporation, vaporization, desorption and sublimation are physical effects that are often used.
The quantitative determination of components that are removed completely during the measurement is easiest (e.g. in moisture determination, depolymerisation, rubber analysis).
Besides the determination of content, the conversion curve is also very important. It is used, for example, for different kinetic analyses and for the determination of stability.
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The evaluation possibilities include:
- **Content** (with a choice of tangents or horizontal lines):
  - percent
  - stoichiometric
  - empirical
- **Conversion** (based on the step or on the initial sample weight, total weight change)

The percentage content G is calculated from the weight loss Δm and the initial sample weight m_o as follows (a simple step can be evaluated with the standard software):

$$G = \Delta m / m_0 \cdot 100\%$$

The following equation applies for stoichiometric reactions with just partial weight loss such as dehydration (loss of water of crystallization) or decarboxilation (loss of CO_2):

$$G = \frac{\Delta m \cdot M}{n \cdot M_{Gas} \cdot m_0} \cdot 100\%$$

M = molar mass of the sample, M_{Gas} = molar mass of the gas liberated, n = number of molecules liberated per starting molecule.
In the empirical determination of content, the ratio of the weight loss of the sample to the weight loss of the pure known component is calculated.

$$G = \Delta m / m_0 \,/\, Gs \cdot 100\%$$

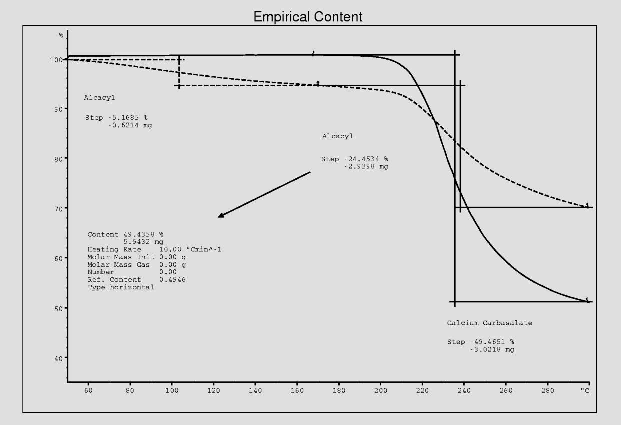
Gs = percentage loss of the reference substance

The conversion α is calculated as follows:

$$\alpha = \Delta m / \Delta m_{step} \quad \text{oder} \quad \alpha = \Delta m / m_0$$

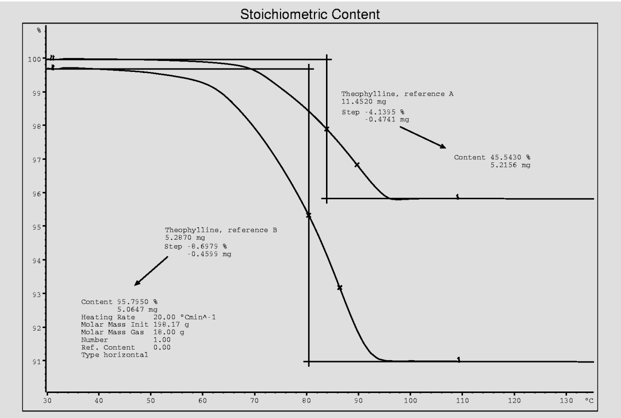
The calculation can be relative to the initial sample weight m_o or to the total weight loss of a step (the conversion is then from 0% to 100%).

Application examples



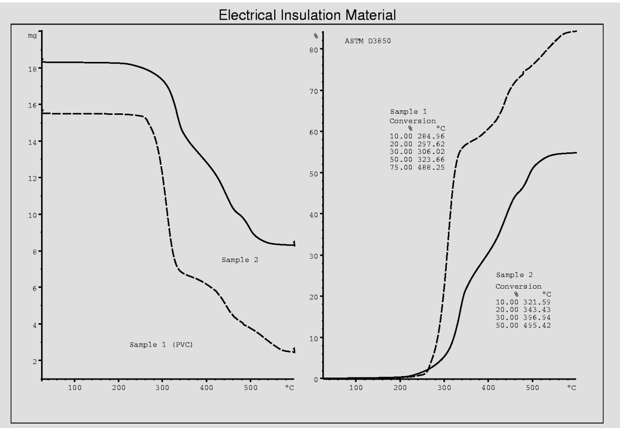
Empirical determination of content

The example shows the decomposition curves of a pure pharmaceutical active ingredient (calcium carbasalate) and of a preparation (Alcacyl) that contains this substance. The pure active ingredient shows a weight loss due to decomposition of 49.46% starting at about 210 °C. Under the same measurement conditions, the preparation shows a loss of moisture of about 5% followed by a weight loss step of 24.45%. The latter is due to the decomposition of the active ingredient. The value of 24.45% is divided by that of the decomposition step of the pure active ingredient 49.46%. This gives a result of 49.44% for the content of the active ingredient in the preparation.



Stoichiometric content

Many substances especially those used in the pharmaceutical industry can form hydrates (substances containing water of crystallization). These are more or less stable depending on the substance and the storage conditions. The hydrate content must therefore be determined before the substance can be used in pharmaceutical preparations. The example shows the TGA curves of two samples of theophylline, a substance that forms a monohydrate. The loss of water of crystallization occurs on heating and is already completed at 110 °C . A weight loss step of 9.1% is expected from a stoichiometric calculation for a sample of pure theophylline monohydrate (molar mass 198.2 g/mol). The theophylline samples investigated here have already lost water of crystallization; they still have contents of 95.79% (reference B: step = 8.69%) and 45.54% (reference A: step = 4.13%) of the theoretically expected values. Besides this, the sample B lost weight in the time between weighing out and the start of the measurement (‘deviation’, the curve starts at 99.7% and not at 100%). This is accounted for in the evaluation by performing the calculation on the basis of the original initial sample weight m_0.



Electrical insulation material

The investigation of the thermal stability of electrical insulation material (usually polymers) can be performed with thermogravimetry. The ASTM standard D3850 deals with the measurement of thermal stability and determination of the content of volatile components. In this connection it specifies the measurement of the weight loss of the insulation material in an air atmosphere at a heating rate of 5 K/min. The temperatures are determined at which weight losses of 10%, 20%, 30%, 50% and if appropriate 75% occur. The diagram shows the TGA curves of two different cable insulation materials (samples 1 and 2). Both samples exhibit decomposition that occurs in several steps and leaves a residue. For the evaluation according to ASTM D3850 the conversion curves for the whole of the decomposition process are calculated and the values required presented in tabular form. The temperatures obtained for the weight losses concerned can be used as quality control criteria or for identification purposes.

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