Application note No.11-004 2019-05-08

Differential Scanning Calorimeter

Thermal analysis with color changes

Chip-DSC 1

Introduction

Thermal analysis is a very useful tool for the analysis of various compounds. Differential Scanning Calorimetry (DSC) gives information about the energy change during phase transitions and chemical reactions. Some phase changes and reactions are accompanied by color changes which can't be observed in most DSC instruments. In contrast, Chip-DSC 1 and Chip-DSC 10 with their transparent cover are useful for observing the sample during measurements and thus gaining further information during the heating, cooling or at isothermal segments.

Methods

Using a DSC for analysing materials is a common technique. In this application, the Chip-DSC was used for measurements of powders changing their color during heating. The first example shows a reversible phase transition, the second one a chemical reaction; both include a color change.

The DSC signal in general is generated by heating a pan containing a sample and an empty reference pan using the same heat source. Subtracting the heat flow signals of the two pans from each other, results in endothermic or exothermic peaks if the sample temperature changes due to thermal effects.

The Chip-DSC integrates all essential parts of DSC, furnace, sensor and electronics, in a miniaturized housing. The chip-arrangement comprises the heater and temperature sensor in a chemically inert ceramic arrangement with a metallic heater and a temperature sensor. Therefore, the Chip-DSC allows a very fast heating and cooling speed combined with high resolution and accuracy as well as reproducibility. Due to the transparent cover, the sample can be observed during the measurement.

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Table 1. Experimental Conditions

Instrument	Chip-DSC 1
Heating rate	20 K/min
Sample mass	19,8 mg
Sample pan	open aluminum pans
Purge gas	none

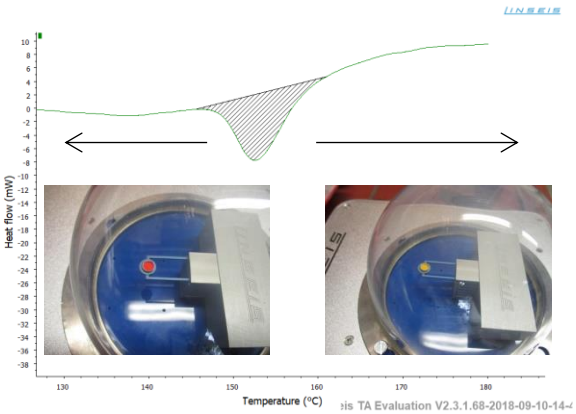

Figure 1. DSC diagram of HgI_2. Sample weight: 19,8 mg, heating rate: 20 K/min. Photos show the sample during the measurement (before and after the phase transition).

Example 1

Experimental

Mercury(II)iodide was measured on a single furnace, heat flux DSC under the conditions shown in Table 1. Open aluminum pans were used in order to observe the sample during heating and cooling.

Results

Mercury(II)iodide exists in at least two enantiotropic modifications, a red low temperature modification and a yellow high temperature phase. This phenomenon (reversible phase transitions with color change) is called « thermochromism ».

Figure 1 shows the DSC diagram and photos during the heating run. It can be clearly seen that the endothermic event is a solid-solid phase transition with color change.

Table 2. Experimental Conditions

Instrument	Chip-DSC 1
Heating rate	15 K/min
Sample mass	3,2 mg
Sample pan	open aluminum pans
Purge gas	none

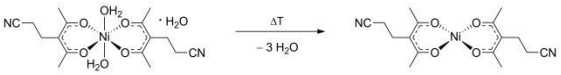

Figure 2: water loss of the Ni-complex

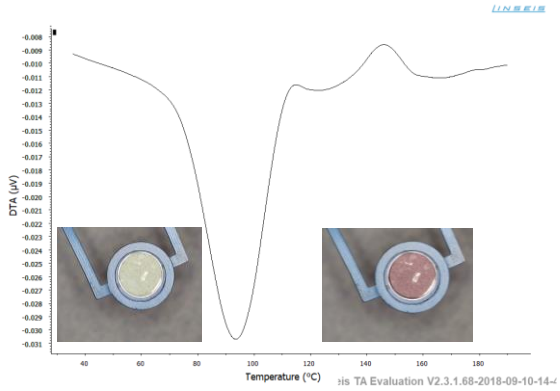

Figure 3. DSC diagram of the Ni-complex. Sample weight: 3,2 mg, heating rate: 15 K/min. Photos show the sample during the measurement (before and after the reaction).

Acknowledgement

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Example 2

Experimental

$Ni(acacCNen)_2$ was run on a single furnace, heat flux DSC under the conditions shown in Table 2. Open aluminium pans were used in order to observe the sample during heating and cooling.

Results

The Ni(II) complex of the bidentate ligand 4-acetyl-5-oxohexanenitrile (HacacCNen) can be obtained as a purple anhydrous complex with a square planar coordination sphere and as a green trihydrate with two aqua ligands that complete the octahedron and a co-crystallised water molecule. Upon heating, the green trihydrate loses all three water molecules in one endothermic event below 100 °C and is transformed to the purple anhydrous form. The peak at 140 °C is attributed to the exothermic recrystallisation of the anhydrous complex.

Summary

The Chip-DSC 1 with its transparent cover can be a useful tool to get further insight to phase transitions and chemical reactions. Forming of bubbles, fumes or, as shown in these examples, color changes can be easily observed and correlated to thermal events.

An optional video attachement allows for simultanous image acquisition.