

STA 449 C Jupiter[®] - Simultaneous Measurement of Caloric Effects and Mass Changes between -120°C and 1650°C



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1. Introduction

In general, the meaning of Simultaneous Thermal Analysis is the simultaneous application of two or more thermoanalytical methods on the same sample. This term, however, is in most cases used for simultaneous measurement of the mass changes and caloric effects. The advantages of such a system are obvious. First, the comparability of characteristic temperatures is ensured. Furthermore, there could be problems if two different samples must be prepared for the thermo-balance and DTA/DSC apparatus. A reproducible sample preparation (e.g. same density for powder samples) is only possible with a great deal of work. Regarding inhomogeneous materials, differences between the corresponding samples, complicating interpretation of the data, may occur.

The new STA 449C Jupiter[®] combines the advantages of an extremely accurate high sensitivity thermo microbalance and a highly reproducible, differential scanning calorimeter. Easily exchangeable furnaces and sample carriers allow the system to be adjusted to different temperature ranges (-120 to 1650°C) and applications (TG, TG-DTA and TG-DSC measurements). The vacuum-tight construction enables measurements in defined (e. g. pure inert) atmospheres.

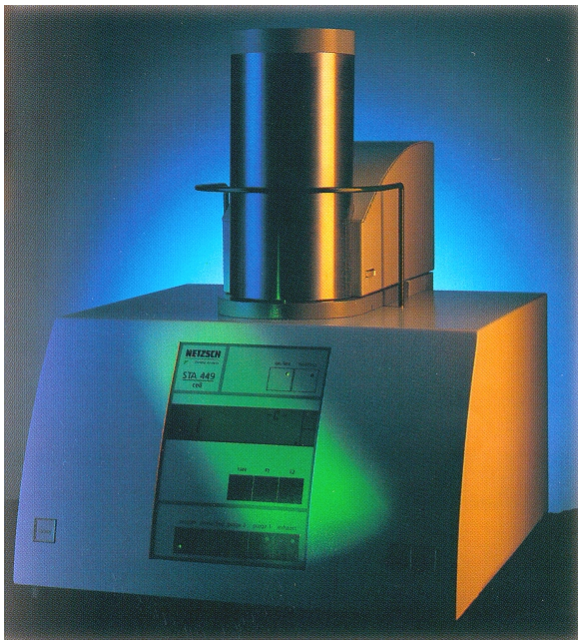


Fig. 1: STA 449 C Jupiter (1500°C-Version)

2. Experimental Results

The mass change and rate of mass change of Coppersulfate-Pentahydrate during heating are depicted in figure 2. The three-stage water evolution can clearly be seen. In the first two steps between room temperature and 130°C, from the $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ two water molecules are evolved. Around approx. 200°C, the last water molecule is removed. Between 600°C and 750°C, decomposition of the sulfate occurs. The last mass change step in the range of 1050°C is related to the reduction of residual copper oxide. Apart from the measured mass loss steps, the figure also shows the stoichiometrically evaluated values. Despite the small sample weights of 1.14 mg, the values are in good agreement with the measured data.

The mass change, rate of mass change as well as the transformation energetics of Iron(III)oxide (Fe_2O_3) are depicted in figure 3. At 678°C, a weak endothermal peak with an enthalpy of 4.6 J/g was measured. An enthalpy value of 4.2 J/g is given in [1]. At approx. 1050°C, a mass loss of 0.04% was detected. This can be explained by a small amount of impurities. At approx. 1100°C, the reduction of Fe_2O_3 to Fe_3O_4 begins, connected with a mass loss of 3.33% (calculated: 3.34%). The enthalpy of this transition was calculated as 354.8 J/g. The good agreement of the DSC peak and the course of the rate of mass change is obvious.

The thermal decomposition of Hydromagnesite is depicted in figure 4. Coupling of the STA 449 C to a mass spectrometer enables determination of thermal effects and mass changes as well as analysis of the evolved gases. During the different mass losses water and/or carbon dioxide evolves from the sample. The decomposition steps are connected with endothermal peaks in the DSC trace. A comprehensive characterization of the decomposition reactions is therefore possible.

High Accuracy - Measurement on $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

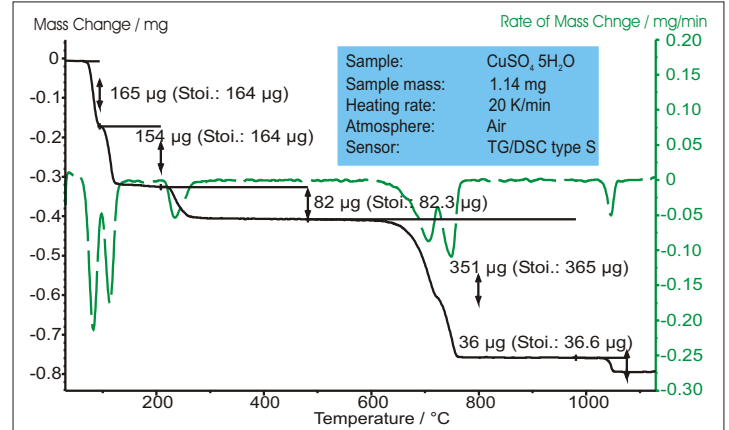


Fig. 2: Mass change and rate of mass change of Coppersulfate-Pentahydrate. Depicted in brackets are the stoichiometrically calculated mass changes.

Broad Temperature Range - Thermal Behavior of Fe_2O_3

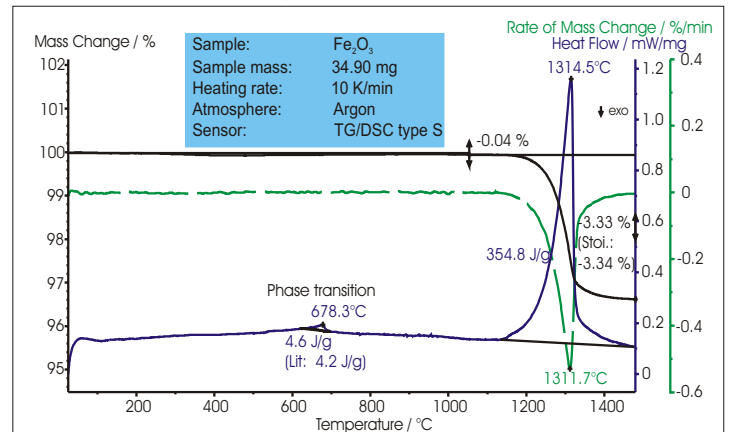


Fig. 3: Mass change, rate of mass change and heat flow of Iron(III)oxide. Additionally presented is the (stoichiometrically) calculated value for the reduction of Fe_2O_3 to Fe_3O_4 .

MS-Coupling Capability - Examination of Hydromagnesite

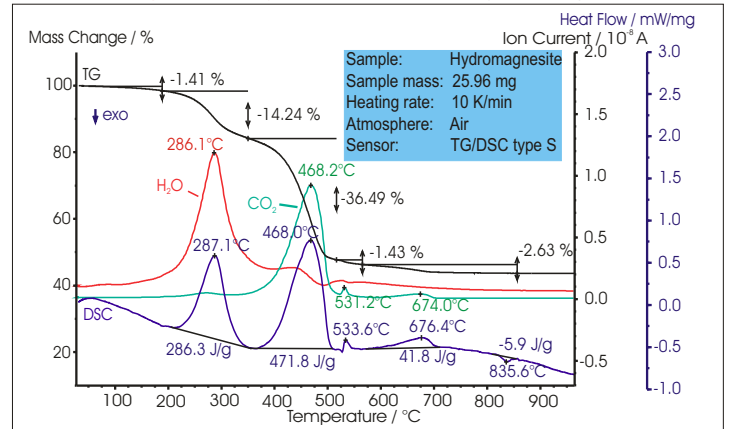


Fig. 4: Mass change, heat flow and ion currents (mass spectrometer-signals: carbon dioxide: —, water: —) of Hydromagnesite between room temperature and 950°C.

Literature:

[1] D'Ans, Lax, Taschenbuch für Chemiker und Physiker, 1. Band, Springer Verlag, 1967