Simultaneous Thermal Analysis of
Iron Hydroxy Sulfate

Fig. 1. The STA 449 *F1 Jupiter*® for simultaneous thermal analysis (depicted with a double furnace arrangement)

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Thermal analysis can yield valuable information on sample composition for a broad range of applications. Read on to see how simultaneous thermogravimetry and differential scanning calorimetry can help characterize minerals.

Simultaneous thermal analysis (STA) is an established method for material characterization which usually refers to the simultaneous measurement of mass changes and caloric effects of a single sample being subjected to a controlled temperature program. An STA apparatus offers several basic advantages:

- First of all, simultaneous thermal analysis allows for the determination of both temperature-dependent mass changes (TG) and caloric effects (e.g. phase transition temperatures and enthalpies) by means of DSC in a single measurement. The STA method therefore saves on time and also on sample material, which can be a great advantage if this material is expensive and/or difficult to produce.

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- In addition, the TG and DSC results of an STA measurement can truly be compared and correlated with each other, since the measurement conditions are identical and no possible differences in sample preparation need to be taken into consideration.

- Finally, exact knowledge of the current sample mass is also always given, for precise enthalpy determination by means of DSC.

Measurements between -150 and 2000°C

With the new STA 449 *F1 Jupiter*® (see figure 1), NETZSCH combines flexibility and performance in one instrument. A broad temperature range of -150°C to 2000°C ensures that nearly all possible applications from areas such as ceramics, metals, plastics and composites are covered. Temperature stability, decomposition, phase transitions, melting processes and compositions can be analyzed quickly and comprehensively. The easy-to-operate top-loading system features a balance resolution in the nanogram range (25 ng for a measuring area of 5000 mg) and high long-term stability. In addition, integrated sensors allow for sensitive DSC measurements with a high reproducibility and measurements of the specific heat capacity. These features make the STA 449 *F1 Jupiter*® to a very helpful tool for the thermal analysis of materials in research, development and quality assurance. The variety of optional accessories allows the system to be adjusted to many different circumstances: various furnaces which can be easily interchanged by the operator (or the optional swiveling double hoist for two furnaces),

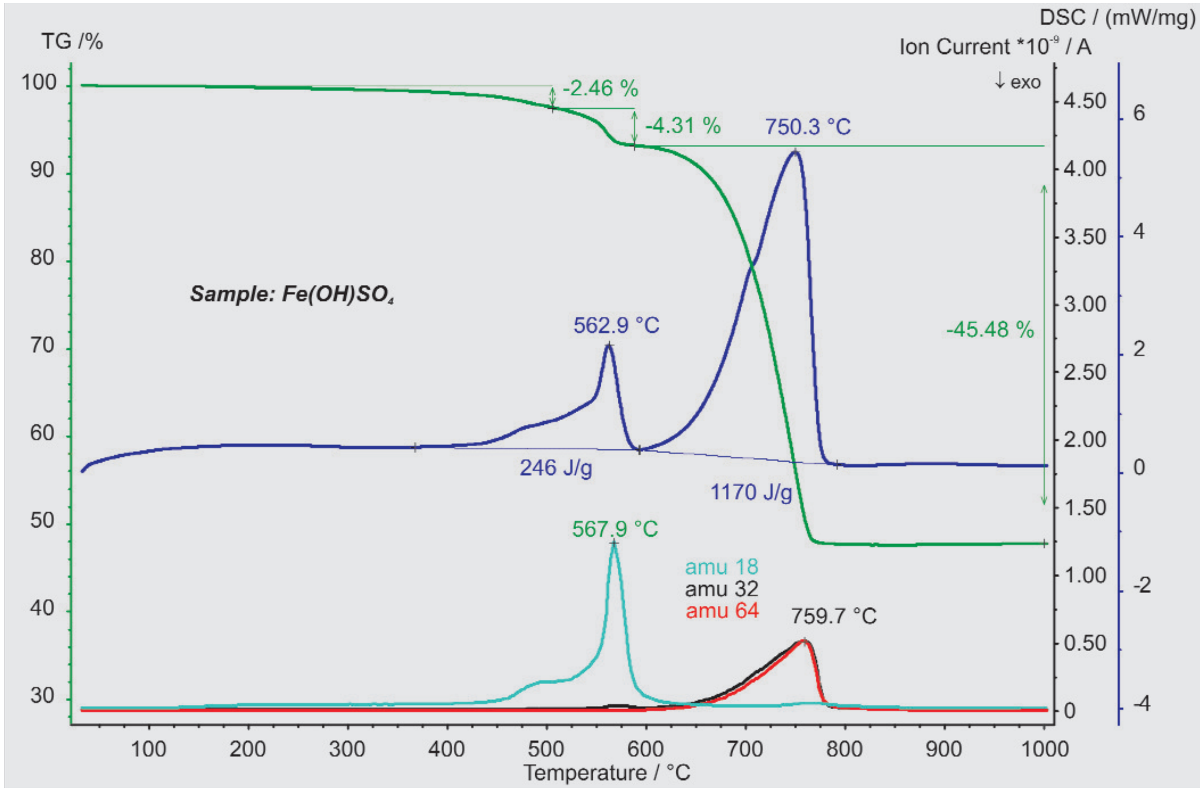

Fig 2. Mass change (TG), heat flow rate (DSC) and mass spectrometer curves (mass numbers 18, 32 and 64) of an $Fe(OH)SO_4$ sample

sample carriers (TG, TG-DSC, TG-DTA, etc.), an automatic sample changer (ASC) for up to 20 samples, an automatic evacuation and refilling system (Autovac), and other accessories such as crucibles in different forms and materials are available. Unique for STA is the temperature-modulated DSC (TM-DSC). By means of an additional MS- and/or FTIR-coupling, the STA 449 *F1 Jupiter®* can even be used to identify gases released from the sample.

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Iron hydroxy sulfate ($Fe(OH)SO_4$) is a possible base material for the production of iron oxide particles, which can be used as magnetic storage media or in ferrofluids. Figure 2 depicts the measurement results for an $Fe(OH)SO_4$ sample which were obtained with an STA coupled to a mass spectrometer. The sample (m_0 = 30.58 mg) was measured in a nitrogen atmosphere (70 ml/min) at a heating rate of 20 K/min. Below 600°C, the STA-MS measurement shows a two-step mass loss which can be attributed to the separation of water with a mass number of 18. Between 600°C and 800°C, the separation of sulfur dioxide with a mass number of 64 and oxygen with a mass number of 32 can be detected. The end product is Fe_2O_3 (hematite).

During the mass-loss steps, the DSC signal shows two endothermal effects with enthalpies of 246 J/g and 1170 J/g.

This example illustrates that STA-MS measurements yield important information about temperature behavior and composition, particularly for minerals.

Source: LaborPraxis LP March 2008