

STA 409 with FTIR Gas Analysis

Due to its universality, there is a wide range of applications for thermal analysis (TA). In particular, simultaneous TA methods such as TG/DTA or TG/DSC enable specific conclusions to be drawn concerning the material behavior as a function of temperature. If such preferred simultaneous TA methods (STA) are coupled with gas analysis,

the evolved gaseous products can be identified; the interpretation of the reactions occurring is thus simplified or even made possible to begin with. NETZSCH has more than 15 years experience with the coupling of thermobalances and quadrupole mass spectrometers for gas analysis.

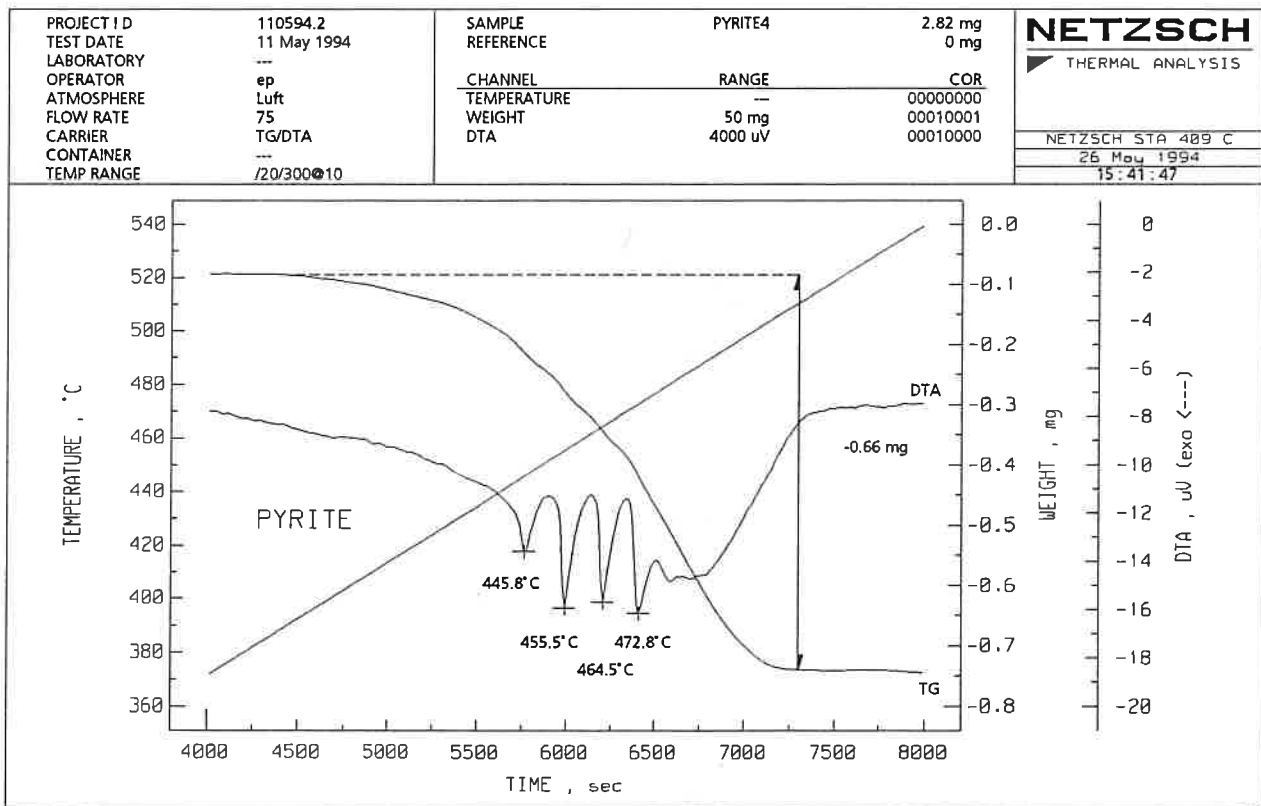


Fig. 1. TG/DTA Results for the Pyrite Sample

Another option for gas analysis is a Fourier transform infrared spectrometer (FTIR) for which a new coupling has been developed. The gaseous products evolving from the sample are continuously transported by a carrier gas to a gas cell, in which the infrared spectra can then be recorded in rapid succession. The transfer line and the connection to the STA - both easily detachable for cleaning and

transport - can be heated to 250°C in order to prevent condensation of the decomposition products to the greatest extent possible. The capability of the STA-FTIR coupling is demonstrated with a natural pyrite sample (FeS₂-ore). Figure 1 shows the TG and DTA curves for pyrite during heating in a dynamic air atmosphere.

The sample begins to decompose at approx. 390°C and releases SO₂ gas in the process. This release proceeds, in part, in "bursts", which is hardly recognizable in the TG curve, but becomes very clear through the exothermic peaks in the DTA curve. Here, the weight loss over the entire temperature range is only 0.66 mg. The high detection efficiency of the FTIR coupling is demonstrated in figure 2, where the intensity of the

SO₂ bands is plotted with respect to time. With the time axis, a direct correlation to the results of the STA measurement is possible. If the times of the DTA peaks are compared to those of the "FTIR peaks", retention times of approx. 10 to 15 seconds result. The short time constant for the transport of the gas to the gas cell affects a very good separation of successive reactions and, at the same time, a high detection efficiency.

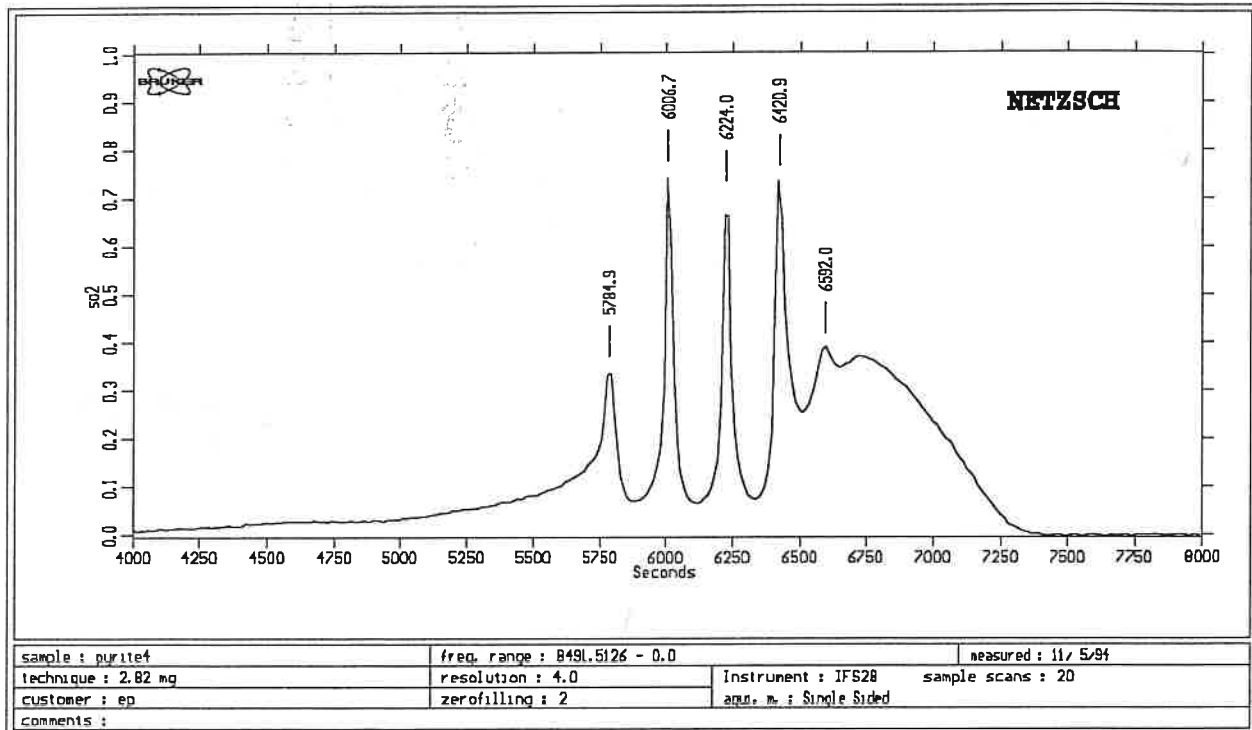


Fig. 2. Intensity progression of the SO₂ absorption bands with respect to time (=temperature)

The measurement demonstrates the high efficiency of the STA-FTIR coupling. The excellent resolution

and detection capability will open up new areas of application.

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