

# Simultaneous Measurement of Caloric Effects and Mass Changes of Inorganics and Polymers using the STA 409 PC Luxx<sup>®</sup>



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## 1. Introduction

Simultaneous thermal analysis refers to the simultaneous application of two or more thermoanalytical methods on one sample at the same time. This term is in most cases used for simultaneous measurement of the mass changes and caloric effects on a sample under thermal treatment. The benefits of such a system are obvious. Frequently the material available for testing is costly or difficult to produce. Using simultaneous thermal analysis one has the chance to get information on the transformation energetics and the mass change on one sample in one run under identical conditions. Of course, the time necessary for the tests is also reduced by a factor of two. Additionally, it can never be excluded that there are influences on the measurement conditions and/or sample preparation if two different instruments or samples are employed for the tests. Using an STA the comparability of characteristic temperatures measured on the TG and DSC runs is ensured. In case of inhomogeneous sample materials, problems resulting from differences in the sample composition for the TG and DSC measurements are excluded.



Figure 1: Simultaneous Thermal Analyzer STA 409 PC Luxx<sup>®</sup>

The new STA 409 PC Luxx<sup>®</sup> (figure 1) combines the advantages of a highly sensitive top-loading thermobalance and a high-temperature differential scanning calorimeter. The maximum sample mass as well as the measurement range of the balance are 18 g. The entire measurement range can be analyzed with a resolution of 2 µg (up to 0.00001%). Easily exchangeable sample carriers allow optimum adjustment of the system to different applications (TG, TG-DTA and TG-DSC measurements). The various sensors available are shown in figure 2. Different exchangeable furnaces for low temperature as well as for the high-temperature region (1550°C) are available. The vacuum-tight construction enables tests in defined (e. g. pure inert) atmospheres. Analysis of the measured data can easily be done with a standard PC system and a well-proven MS<sup>®</sup>-Windows<sup>™</sup>-based software package.

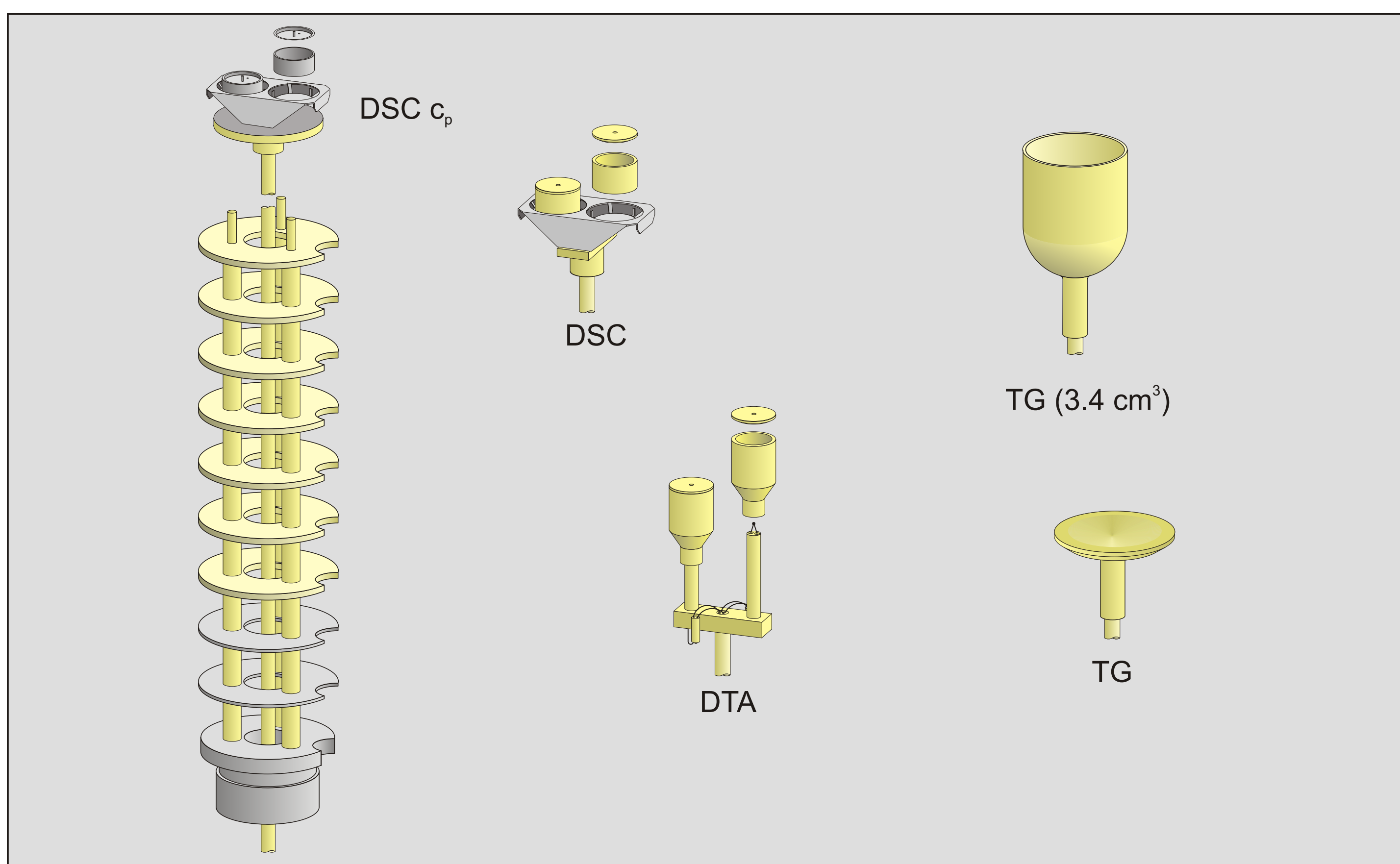


Figure 2: Sensors of the STA 409 PC Luxx<sup>®</sup>

## 2. Test results

Presented in figure 3 are the mass change and transformation energetics of a glass. It can clearly be seen that the glass shows no mass change over the entire temperature range. The measured heat flow rate (DSC curve) increases due to the increase in specific heat of the glass. Between 534°C (extrapolated onset) and 560°C (extrapolated end) the glass transition was detected. The glass transition temperature was at 547°C. At 572°C (peak temperature) a relaxation peak with an enthalpy of 1.53 J/g was found.

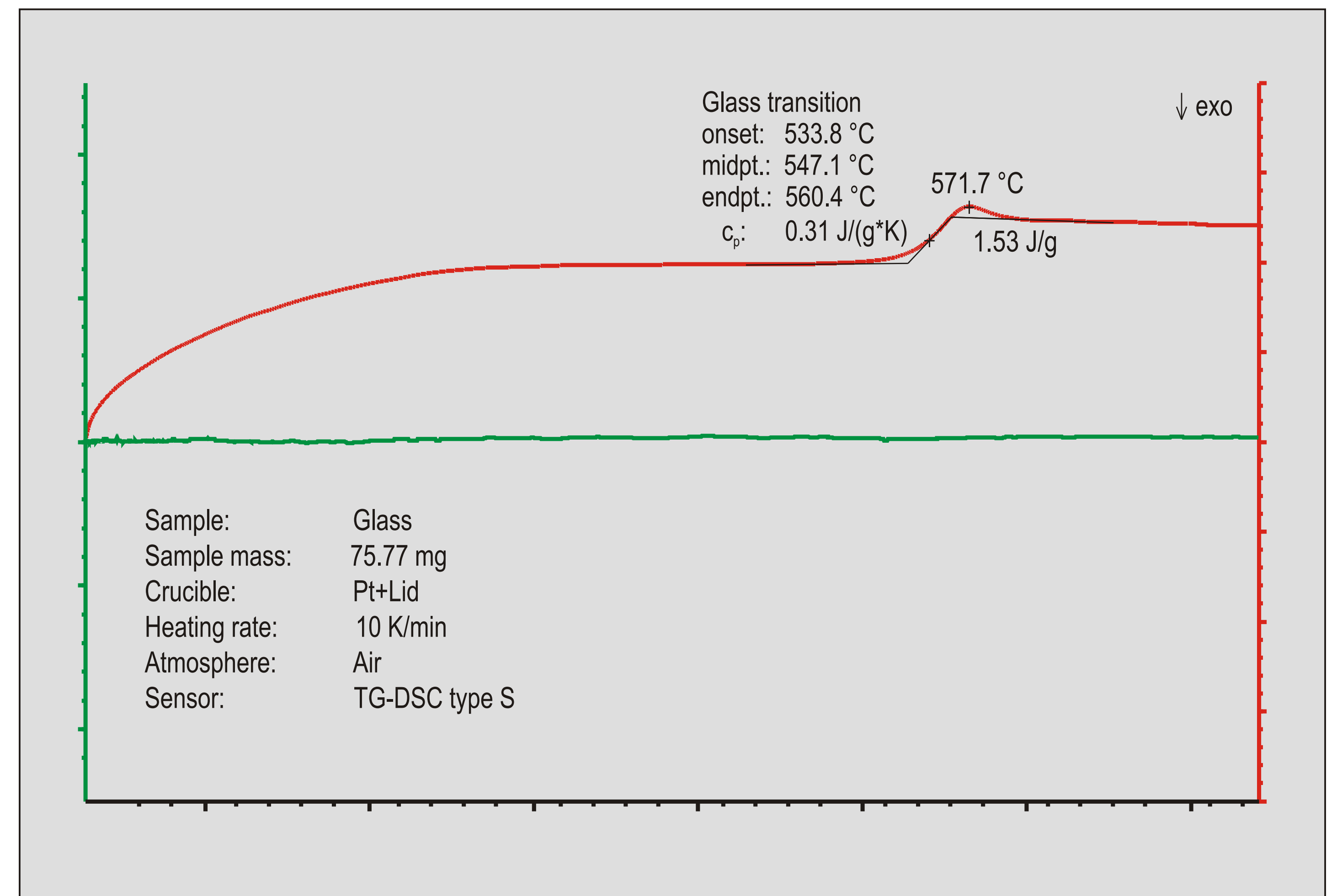


Figure 3: Mass change and transformation energetics of a glass sample

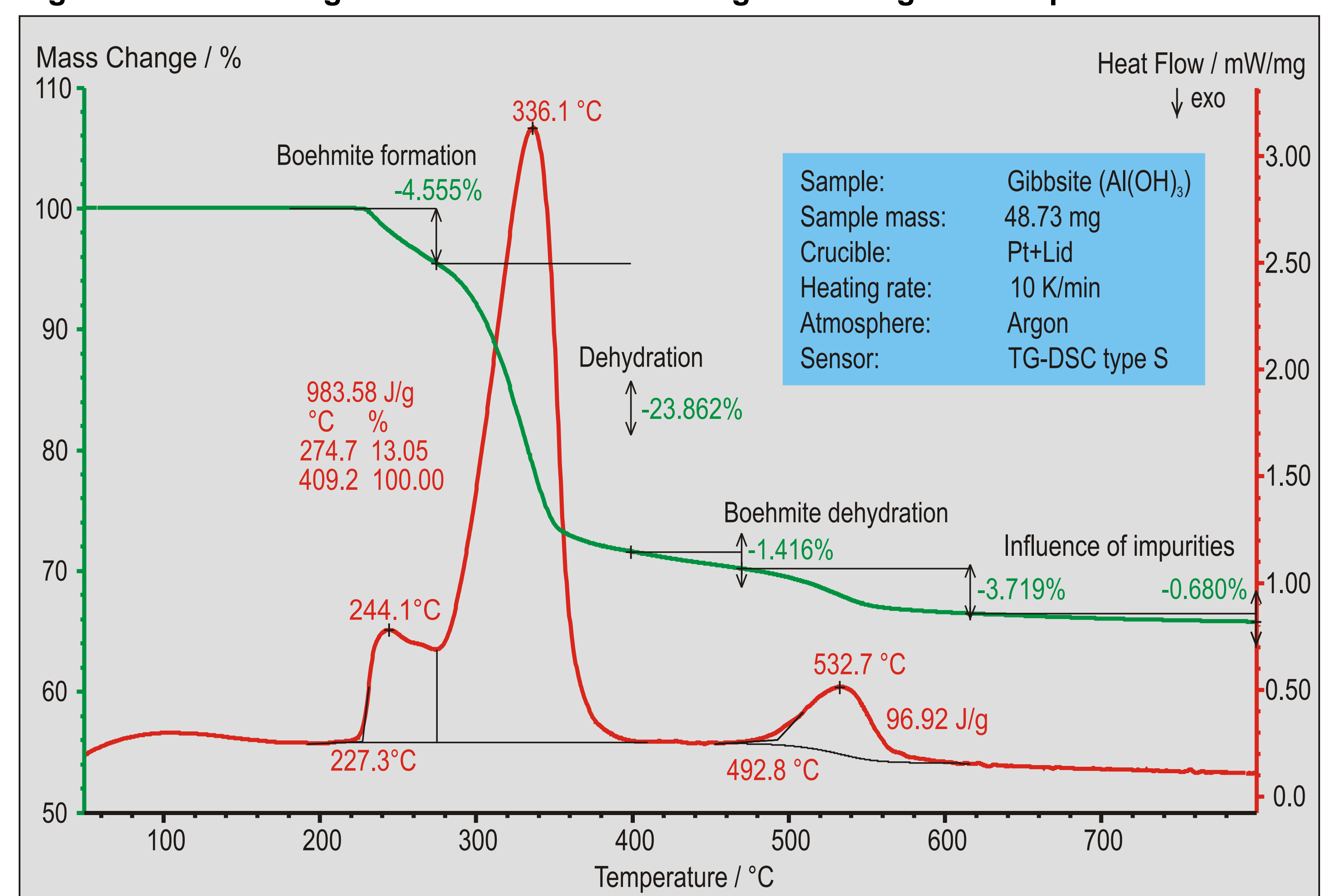


Figure 4: Mass change and transformation energetics of Gibbsite (Al(OH)<sub>3</sub>)

Presented in figure 4 are the mass changes and transformation energetics of Gibbsite. The different dehydration steps of Gibbsite are clearly visible in the mass change. The corresponding transition energies can be detected in the DSC result. The mass loss above 600°C indicates that the sample was not pure Gibbsite.

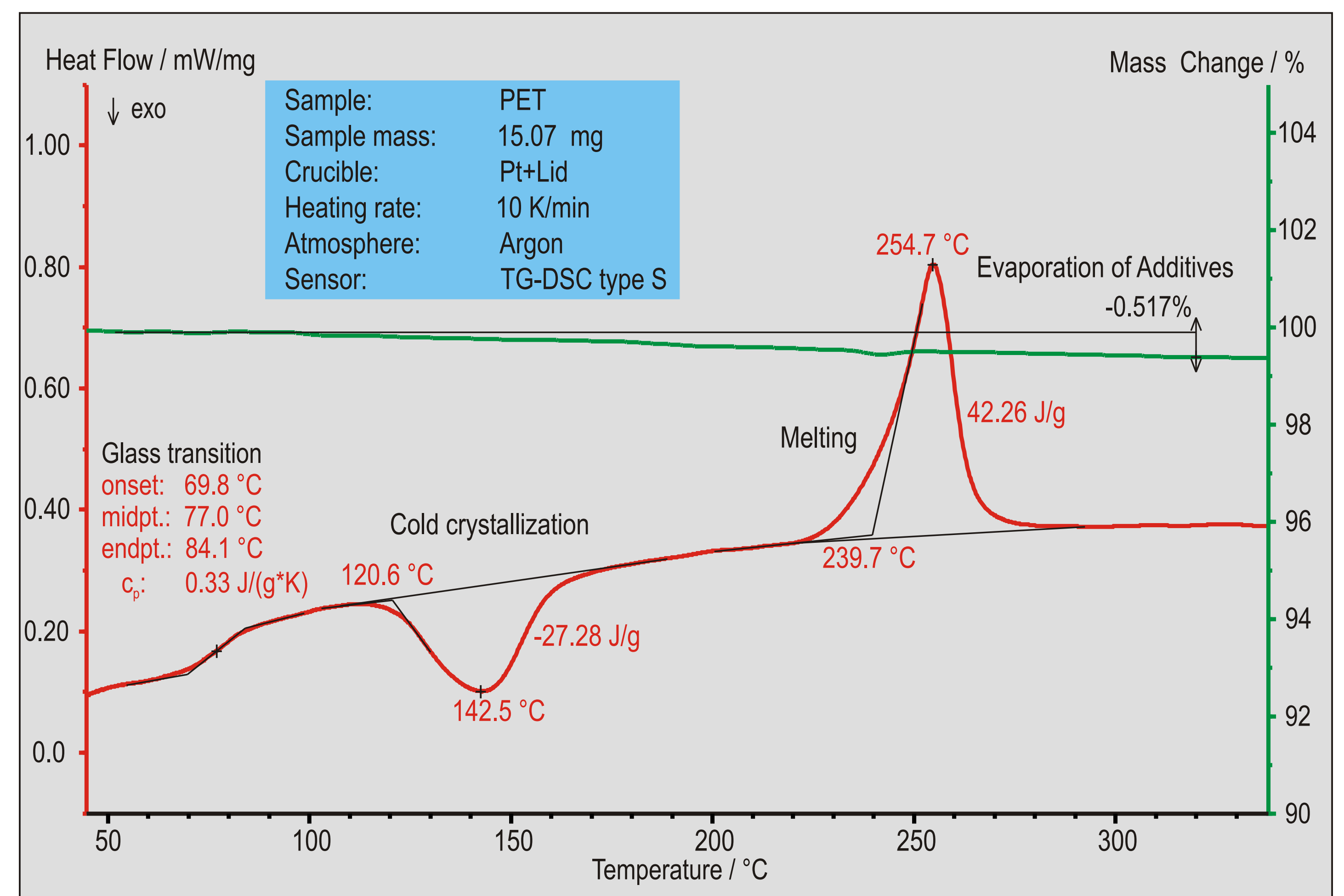


Figure 5: Mass change and transformation energetics of PET

Figure 5 shows the mass change and transformation energetics of PET. The DSC result clearly shows the glass transition (between 70 and 84°C), cold crystallization and melting. The small mass loss of 0.517% above the glass transition is most probably due to the evaporation of processing additives.