TA Instruments

Thermal Analysis & Rheology

# THERMAL SOLUTIONS

## EVALUATION OF INORGANIC PHASE TRANSITIONS

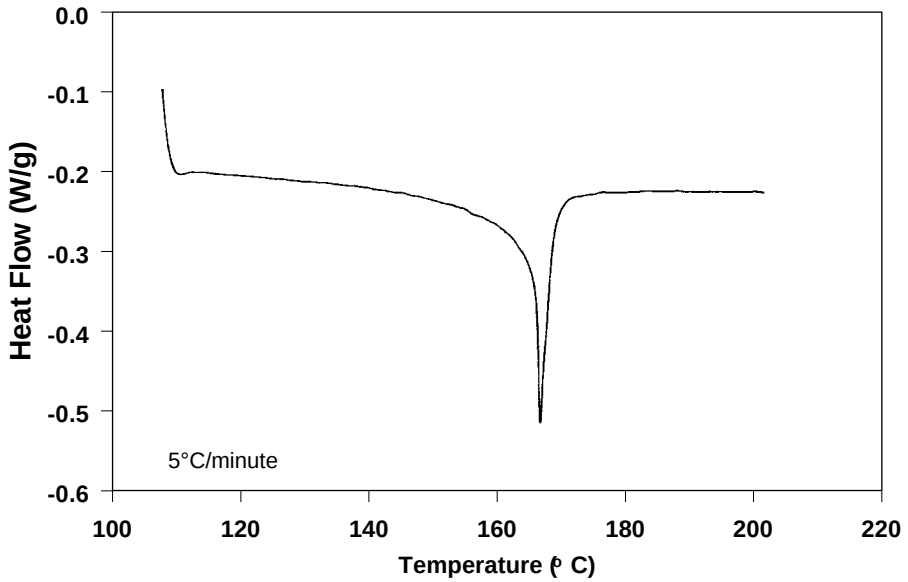
### PROBLEM

Conventional DSC is widely used to evaluate subtle transitions in materials. These transitions can be detected based on the absorption (endotherm) or evolution (exotherm) of heat, or a change in heat capacity. DSC, however, measures the sum of all thermal events occurring in the material at that particular temperature and time. Hence, if multiple transitions (or changes) are occurring, it is generally not possible to detect /differentiate the individual events. Sodium nitrite ($NaNO_2$), for example, is an inorganic salt known to undergo phase transitions related to changes in ferroelectric/antiferroelectric behavior between 160 and 165°C. (1) The presence of these two weak transitions which occur less than 2°C apart is not seen in conventional DSC (Figure 1) because the small changes in heat capacity that occur during the transitions are obscured by a large endotherm associated with the first transition.

### SOLUTION

Modulated DSC™ is a new technique which subjects a material to a linear heating method which has a superimposed sinusoidal temperature oscillation (modulation) resulting in a cyclic heating profile. Deconvolution of the resultant heat flow profile during this cyclic heating provides not only the "total" heat flow obtained from conventional DSC but also separates that "total" heat flow into its heat capacity-related (reversing) and kinetic (nonreversing) components. Thus, MDSC™ provides all the same information and benefits as conventional DSC plus several unique benefits including the direct measurement of heat capacity and heat capacity changes even in situations such as curing (2) where other thermal phenomena are occurring simultaneously. In the case of sodium nitrite, MDSC (see Figure 2) is able to separate the expected heat capacity changes from the endotherm associated with the

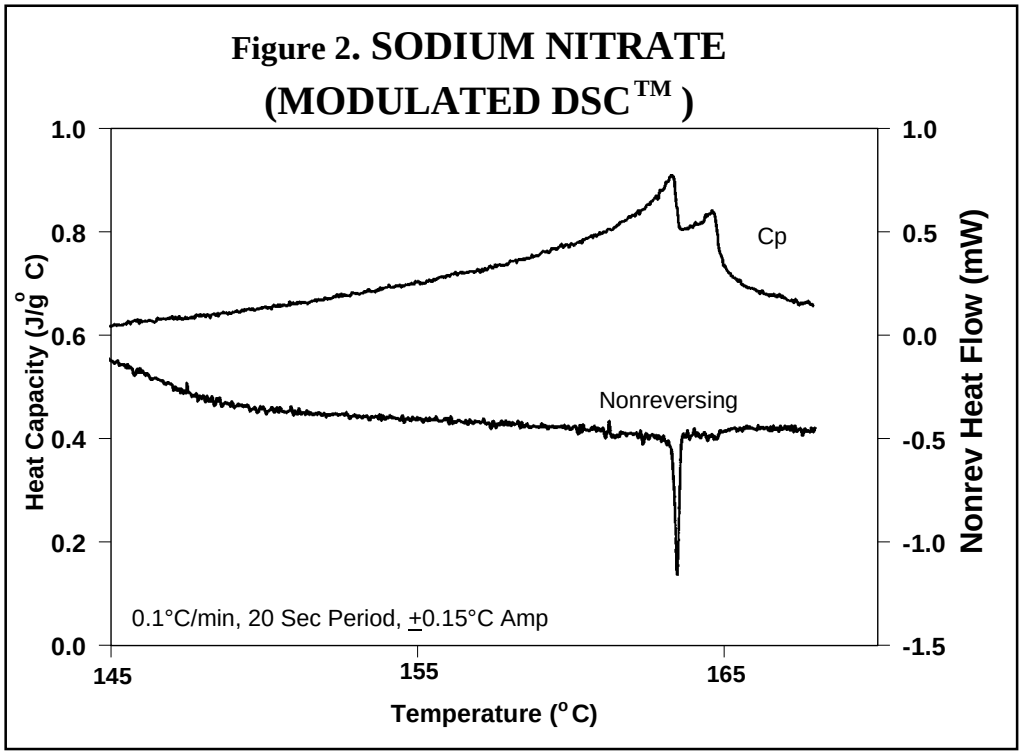
**Figure 1. SODIUM NITRATE (CONVENTIONAL DSC)**



first transition. It should be noted that MDSC™ is able to measure these heat capacity changes even at a very slow average heating rate of 0.1 °C/minute. This is possible because the heat capacity information in MDSC is generated from the instantaneous (modulated) heating rate, which under the conditions used here varies from -2.7 to 2.9°C/minute.

**REFERENCES**


1. Sinku-Riko Inc. Product Brochure AC Calorimeter ACC-1.
2. TA Instruments Modulated DSC Product Brochure TA-074A.





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