Introduction

The selection of appropriate crucibles is decisive in achieving good reproducibility for measurement results. Repeatability tests were carried out using both standard aluminum and Concavus pans which were developed to optimize the repeatability of DSC measurements.

Test Conditions

Two polypropylene samples of the same batch were prepared; the first one with a sample mass of 11 mg in a Concavus pan, the second one with a mass of 10.94 mg in a standard aluminum crucible. Each sample was measured four times with the DSC 214 Polyma. Between each of these four measurements, the sample was removed from the device, rotated 90°, and then returned to the DSC before being measured again. All measurements were carried out from 50°C to 200°C at a heating rate of 10 K/min. Two heating runs each were performed; the second one was evaluated.

Test Results

The second heating run for each of the measurements carried out in the standard aluminum crucible are displayed in figure 1. Two peaks were detected. The main peak, found between 169.7°C and 170.8°C (peak temperature), is due to the melting of polypropylene. The second peak (peak temperature between 119.3°C and 120.8°C) can be attributed to the melting of a small amount of polyethylene. The temperatures of the polypropylene and the polyethylene peaks vary by 0.5°C and 0.6°C, respectively (standard deviation).
The equivalent plot with the measurements employing the Concavus pan is given in figure 2. For comparison purposes, exactly the same scale was used as for figure 1. The better reproducibility is obvious: the curves match better than those for the measurements in standard aluminum crucibles. The standard deviation of the peak temperatures was calculated as well: it amounts to 0.2°C for both peaks.

**Conclusion**

These measurements illustrate the superiority of the Concavus pan with regard to repeatability. Even rotating the crucibles between the measurements does not have much influence on the resulting DSC curve.