PO E 12

DSC BY TGA/SDTA 851^e CONSIDERING MASS CHANGES

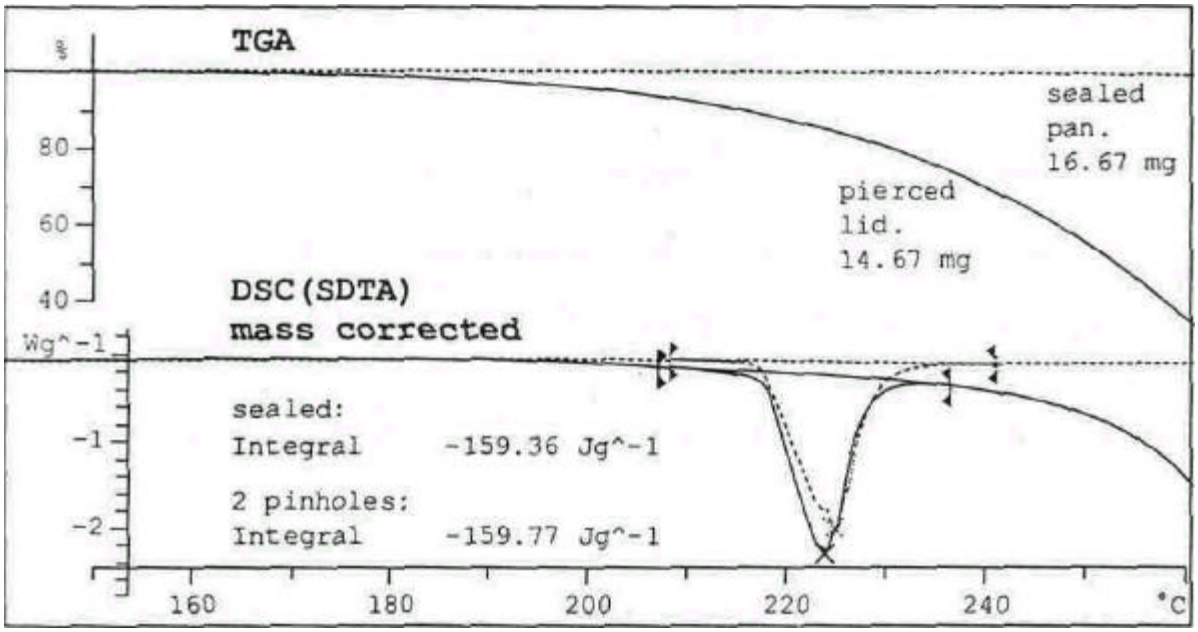
Rudolf Riesen, Kurt Vogel

Mettler-Toledo GmbH, Sonnenbergstrasse 74, CH-8603 Schwerzenbach, Switzerland

Abstract

DSC results (e.g. heats of reaction) are usually normalized to the initial sample mass. But, for the determination of heats of transitions (e.g. melting), a possible mass change during the measurement can not be neglected [1]. Hence, the heat flow has to be normalized to the actual mass. This can easily be accomplished, provided the TGA module delivers simultaneously also a SDTA signal. In this case, a temperature dependent calibration factor is determined which converts the SDTA signal into a heat flow curve (DSC curve). The SDTA signal is the temperature difference between the temperature measured directly at the sample and the model reference temperature [2, 3].

As a result, heats of reactions and transitions can correctly be determined including mass changes before or during physical or chemical transitions. Hence, open systems can be analyzed by DSC despite the disturbing evaporations, see the example below. As an other application example, the condensation reaction of a formaldehyde resin has be analyzed by DSC even under ambient pressure. The influence of the presence or the release of volatile reaction products on the reaction rate and kinetic parameters was shown. In general, the method can be used to correct DSC curves for thermal effects related to weight change.



Example: Heat of melting of anthracene in partially open (with evaporation) and in closed pan measured by TGA/SDTA at 5 K/min, 50 ml/min N2. Neglecting the actual mass correction in case of the pierced lid would give a value of 116 J/g only . Lit. value: 161 J/g.

[1] A.A. van Dooren, B.W. Müller, Thermochimica Acta 49 (1981) 181
[2] R. Truttmann, R. Riesen, G. Widmann, J Thermal Analysis, 47 (1996) 259
[3] M. Kelsey, R. Truttmann, American Laboratory, January 1997, 17