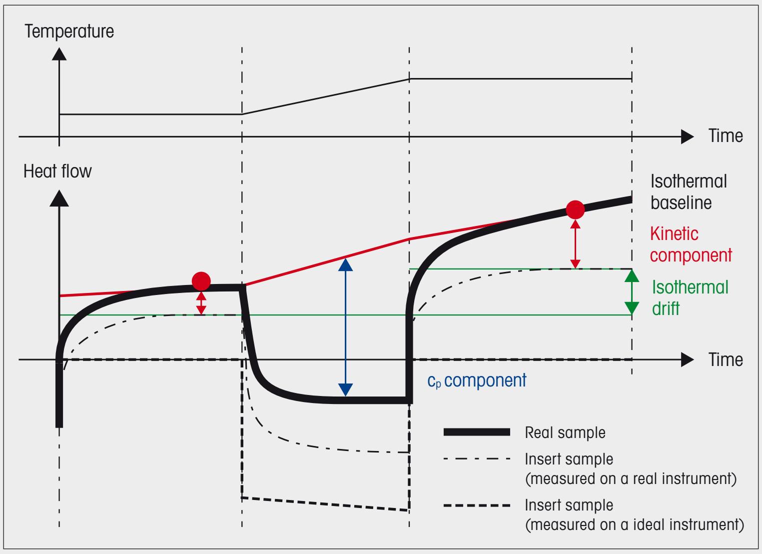
Thermal Analysis Option

IsoStep™

Separation of Simultaneous Effects

Thermal effects can often occur simultaneously, IsoStep™ is a Temperature Modulated DSC (TMDSC) software option that can help evaluation of complicated DSC curves. A typical IsoStep™ temperature program consists of alternating dynamic and isothermal segments. This leads to the separation of the reversing (isothermal segments) and non-reversing (dynamic segments) thermal effects, as well as obtaining highly accurate specific heat capacity (c_p) data.



A typical IsoStep™ experiment consists of alternating heating and isothermal segments. Kinetic phenomena are measured during isothermal segments, whereas the heat flow occurring in the dynamic segments is mainly due to the thermal mass and the heat capacity of the sample.

Isothermal drift caused by heating is corrected using the baseline correction procedure shown in the diagram.

→ Isothermal segments should be at least 1 minute in length and a maximum of 50 K apart.

IsoStep™ is a temperature-modulated DSC method that allows you to determine specific heat capacity and kinetic effects separately in a single DSC run. Overlapping thermal events can therefore be clearly distinguished. IsoStep™ provides a straightforward approach to the determination of heat capacities.

Features and benefits

- **Separation of reversing and non-reversing processes** – separation of overlapping effects permits the very accurate determination of heat capacity
- **C_p calculation based on DIN standard** – sapphire reference material ensures accurate c_p determination

Isothermal Drift Correction
Better C_p Accuracy

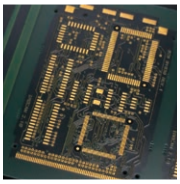
The evaluation method is based on the work of Dr. S. C. Mraw and Dr. H. Dörr. Isothermal segments allow for correction of instrumental drift that occurs during dynamic segments, resulting in higher c_p accuracy (< 2%).

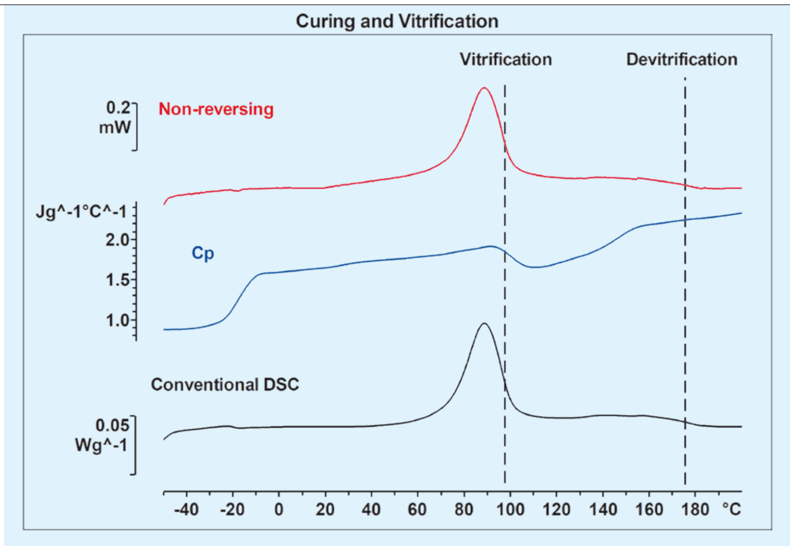
Temperature modulated DSC (TMDSC), such as IsoStep™, enables the separation of the measured DSC output signal into what is called the reversing heat flow (c_p information) and the non-reversing heat flow (excess heat production); this allows you to understand and interpret the different thermal events that occur in the sample and to distinguish between overlapping effects.

IsoStep™ provides useful information that can help evaluation of DSC curves for the following application fields:

Industry	Application
Automobile and aerospace	Curing reactions, influence of moisture, glass transition, vitrification
Chemical	Exothermic reactions (safety investigations), glass transition, kinetics, crystallization behavior, polymorphism, drying, heat capacity
Electronics	Curing reactions, glass transition, vitrification
Paints	Curing reactions, influence of moisture, glass transition, drying, vitrification, evaporation
Rubber (elastomers)	Glass transition, phase behavior, melting, vulcanization
Plastics/Polymers	Curing reactions, influence of moisture, enthalpy relaxation, glass transition, cold crystallization, phase separation, melting, melting and crystallization, vitrification, heat capacity
Foodstuffs	Influence of moisture, gelation, glass transition, stickiness, polymorphism, drying
Pharmaceuticals	Influence of moisture, glass transition, melting (isothermal step melting point), crystallization behavior, polymorphism, drying, heat capacity, decomposition behavior

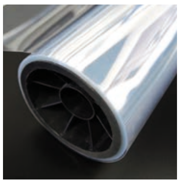
Application Examples

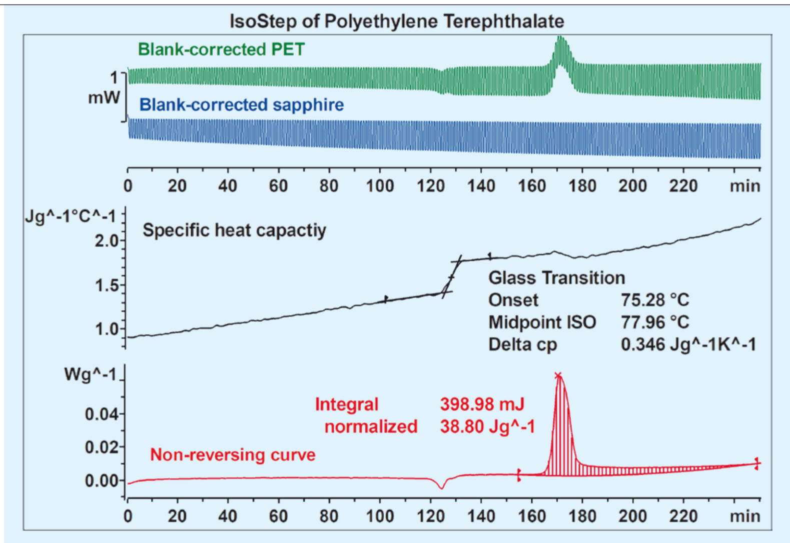




Curing behavior of epoxy resin

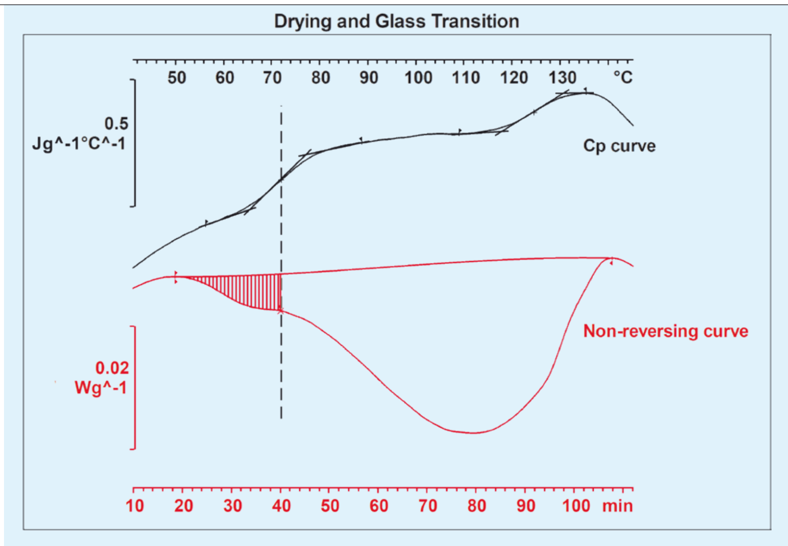
IsoStep™ was used to measure the curing behavior of a two-component epoxy resin system because conventional DSC cannot directly separate the effects of vitrification and devitrification from the curing reaction. The IsoStep™ c_p curve shows a decrease in heat capacity due to vitrification at about 100 °C. The slow heating rate gives the sample sufficient time to form a relatively dense polymer network, therefore the glass transition increases and becomes larger than the actual sample temperature, leading to vitrification of the sample. At this point, the reaction rate of curing slows considerably. On further heating, the new glass transition of the partially cured material is about 150 °C. For comparison, a conventional DSC curve is also presented. It shows the initial glass transition at about –20 °C and the curing reaction at about 90 °C.





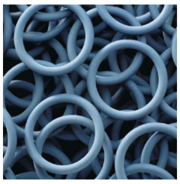
PET Film

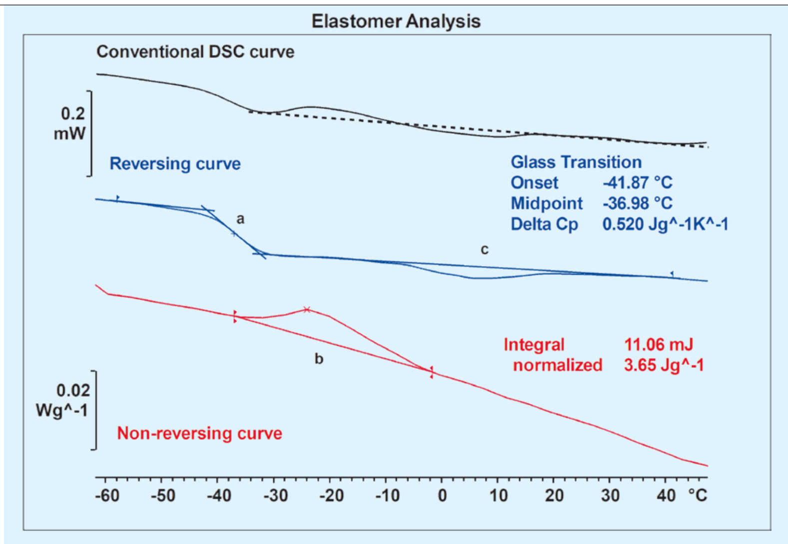
A typical IsoStep™ experiment consists of measuring a blank corrected sample and sapphire curves. Extracting the information from the dynamic segments the c_p curve is constructed, while the information from the isothermal segments creates the non-reversing curve. The c_p curves clearly displays the glass transition at about 78 °C, this is accompanied by the enthalpy relaxation peak visible on the non-reversing curve. The sample then crystallizes at about 120 °C, as indicated by the large peak on the non-reversing curve. Crystallization leads to a slight decrease in c_p.



Drying and T_g of a pharmaceutical substance

The diagram shows the c_p and non-reversing heat flow curves of a pharmaceutical product dried at 80 °C for 20 minutes. The c_p curve shows two glass transitions at about 70 °C and 125 °C. The broad evaporation peak present in the non-reversing curve extends over the range of both glass transitions. The peak is so large that the glass transitions would not resolved with a conventional DSC experiment. The first glass transition is due to the higher water content. Once part of the water has evaporated the T_g shifts to approximately 120 °C.





Interpretation of an elastomer analysis

The interpretation of DSC results from polymeric materials is not always straightforward. The diagram shows the conventional DSC curve of an SBR-based elastomer, as well as the IsoStep™ reversing and non-reversing curves. The conventional DSC curve exhibits several thermal events that are not easily evaluated. IsoStep™ helps to clarify the curves and simplify interpretation. A glass transition at around –35 °C is clearly visible in the reversing curve (a). The non-reversing curve on the other hand, shows an exothermic peak (b) attributed to the crystallization of additives. On further heating, melting occurs at about 5 °C, this endothermic peak is visible in the reversing curve (c).

METTLER TOLEDO Group
Analytical Division
Local contact: www.mt.com/contacts

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