Glass Transition Temperature and Step Height from DSC Curves: All Evaluations Described in the International Standard DIN EN ISO 11357-2

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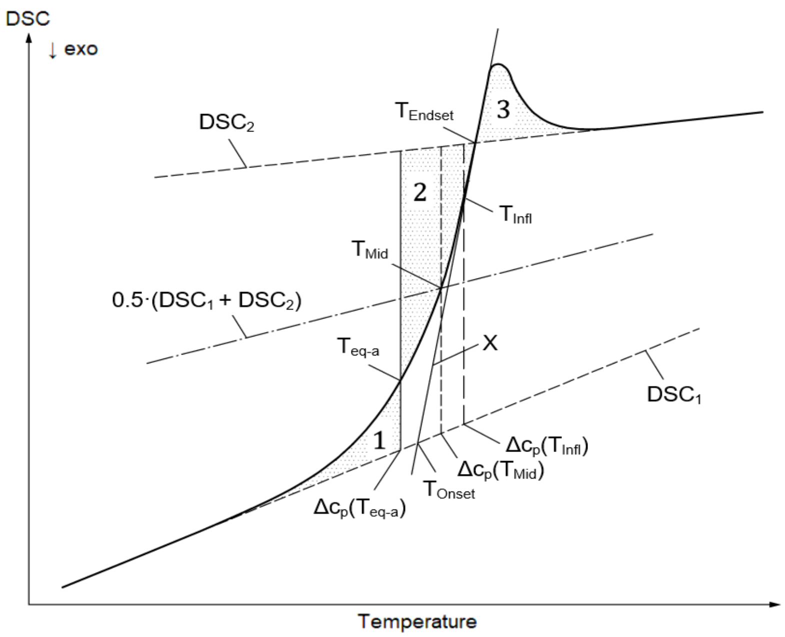
From version 9.6 onward, *Proteus*® analysis allows for the evaluation of all characteristic properties of a glass transition occurring in a DSC curve as described in the international standard DIN EN ISO 11357-2 [1]; the evaluations of the glass transition step height measured by the equal-areas method and by the inflection-point method have been added compared to earlier *Proteus*® versions.

Introduction

There are several national and international standards regarding the evaluation of the glass transition temperature and step height from DSC curves [1-3], of which DIN EN ISO 11357-2 [1] is the most comprehensive one. As illustrated in figure 1, the DSC signal exhibits a step, in some cases superimposed by a relaxation peak upon heating from below to above the glass transition temperature. The calculation of all quantities requires the extrapolated heat-flow rates below and above the glass transition temperature, DSC_1 and DSC_2. The extrapolated onset and endset temperatures, T_{Onset} and T_{Endset}, are at the intersection points between DSC_1 and DSC_2 and the line of the steepest slope, X, respectively. The "equal-areas temperature" according to Richardson*, $T_{eq\text{-}a}$, is defined as the temperature where area 1 is equal to area 2 minus area 3 (see figure 1). The half-step-height or mid temperature, T_{Mid}, is at the intersection point between the equidistant line $0.5{\cdot}(DSC_1 + DSC_2)$ and the DSC measurement curve. The inflection temperature, T_{Infl}, is at the point of the steepest slope of the DSC curve.

*This temperature was denoted as T_f in earlier *Proteus*® versions.



1 Explanation of different characteristic quantities of a glass transition evaluation described in the standard DIN EN ISO 11357-2 [1].

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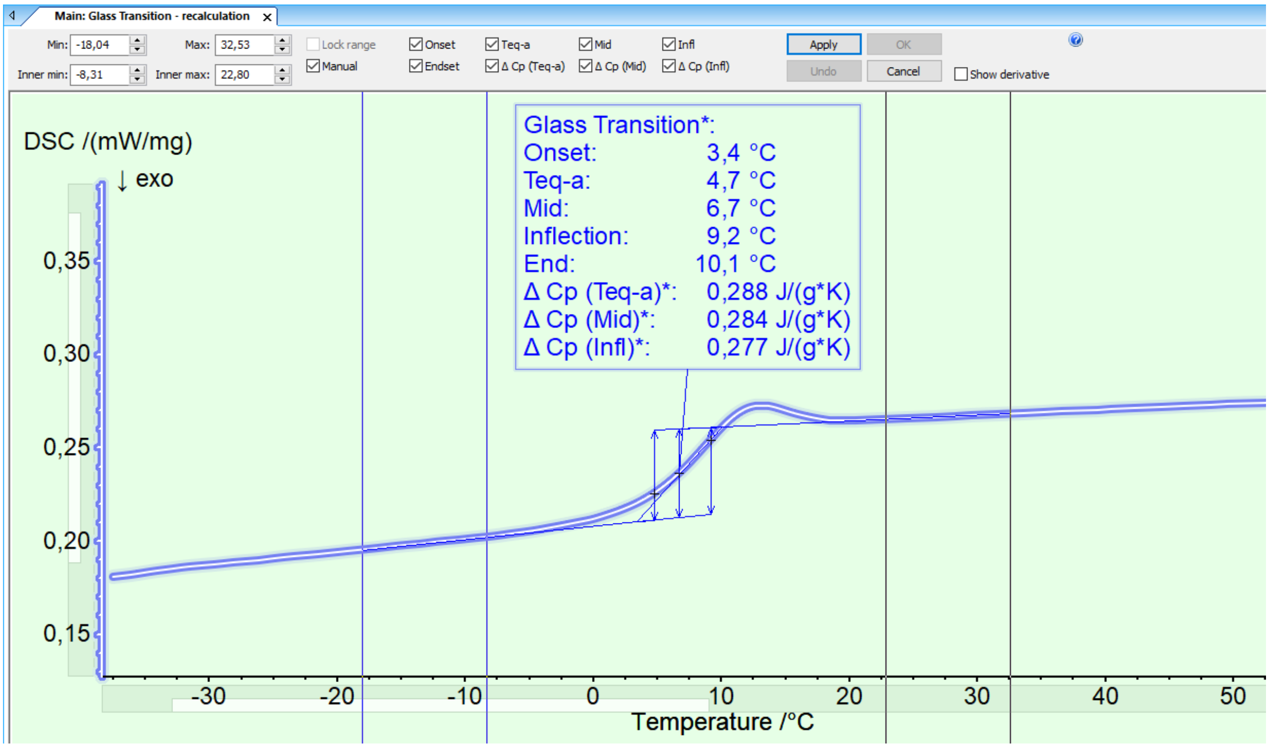
Consequently, three different step heights of the glass transition can be defined, $\Delta c_p(T_{eq\text{-}a})$, $\Delta c_p(T_{Mid})$ and $\Delta c_p(T_{Infl})$, which were calculated from the difference between the extrapolated tangent lines DSC_1 and DSC_2 at the corresponding temperatures $T_{eq\text{-}a}$, T_{Mid} and T_{Infl}. If the slopes of the tanget lines DSC_1 and DSC_2 are identical, then all three definitions of the step height, $\Delta c_p(T_{eq\text{-}a})$, $\Delta c_p(T_{Mid})$ and $\Delta c_p(T_{Infl})$, reveal the same value.

The DIN EN 11357-2 standard states that preferably, the equal-areas method shall be used for determination of the glass transition temperature; the half-step-height method and the inflection point method may result in significant deviations from the glass transition temperature for asymmetric glass transition step profiles, in particular for samples showing enthalpy relaxations such as those reflected by the DSC curve displayed in figure 1.

Implementation in *Proteus*® Analysis

Figure 2a depicts the dialogue of the glass transition evaluation in *Proteus*® analysis version 9.6, where a showcase glass transition that occurs in a DSC measurement was already evaluated before. At the left side of the upper part, the four positions of the evaluation cursors can be set, defining the tangent lines explained above. When "Manual" is not checked, it is sufficient to define only the two outer cursors, and inner cursors will be set automatically. The checkboxes visible in the center of the upper part of figure 2a allow for the choice of which characteristic temperatures and Δc_p values should be evaluated and displayed.



2a Dialogue of the glass transition evaluation in *Proteus*® analysis with a glass transition alreaedy evaluated in a DSC curve.

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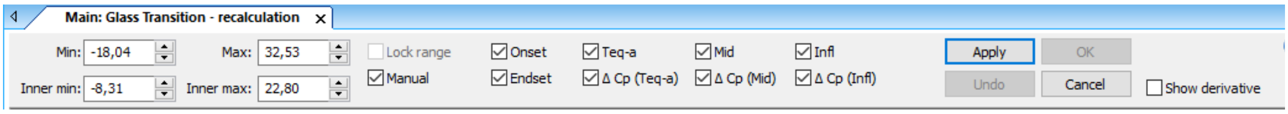
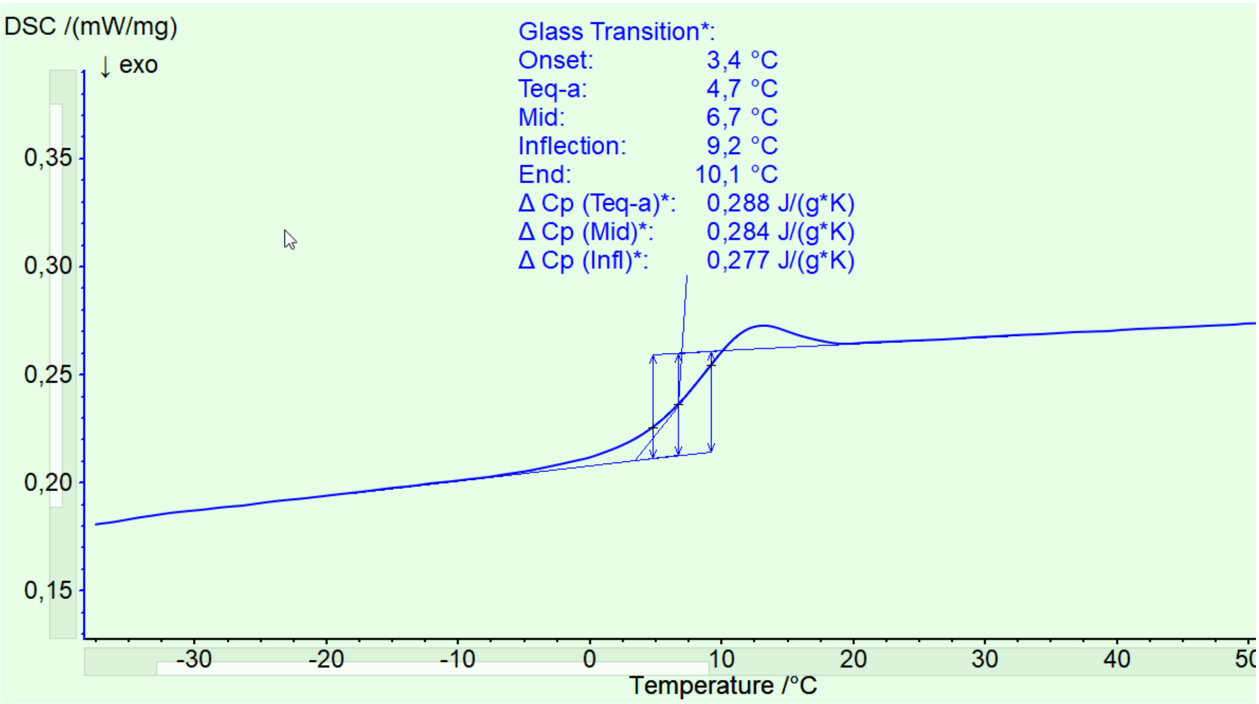
Figures 2b and 2c show the dialogue and the evaluated DSC curve (the result after pressing OK) separately.

Finally, it should be mentioned that the evaluation of a glass transition in a DSC curve can also be the result of *AutoEvaluation*.



2b Dialogue of the glass transition in *Proteus*® analysis.



2c Evaluated glass transition in a DSC curve (*Proteus*® analysis).

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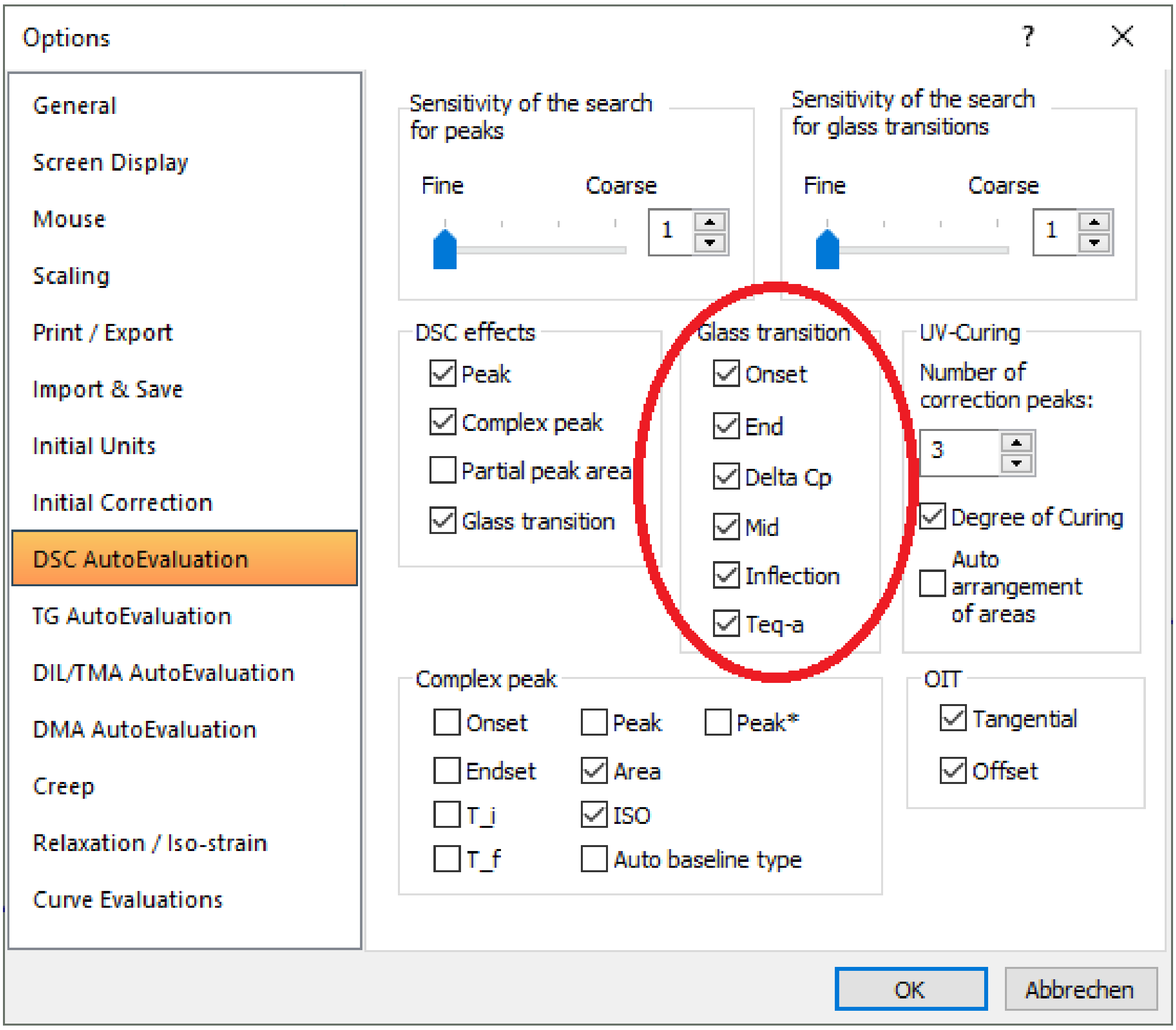

3 Setting dialogue in *Proteus*® analysis

Which characteristic properties of the glass transition should be evaluated and shown can be defined in the Settings of *Proteus*® analysis, as depicted in figure 3.

Summary

Proteus® analysis versions 9.6 and higher allow for evaluation of all characteristic properties of a glass transition occurring in a DSC curve as described in the international standard DIN EN ISO 11357-2 [1]. These properties include the temperatures T_{Onset}, $T_{eq\text{-}a}$, T_{Mid}, T_{Infl} und T_{Endset} as well as the step heights $\Delta c_p(T_{eq\text{-}a})$, $\Delta c_p(T_{Mid})$ and $\Delta c_p(T_{Infl})$.

References

[1] DIN EN ISO 11357-2:2020 (E), Determination of glass transition temperature and step height.
[2] ASTM E1356-23, Standard Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry.
[3] DIN 51007:2019, Differenz-Thermoanalyse (DTA) und Dynamische Differenzkalorimetrie (DSC) – Allgemeine Grundlagen.

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