The New 'Polymer Mixtures NETZSCH' Library for *Identify*

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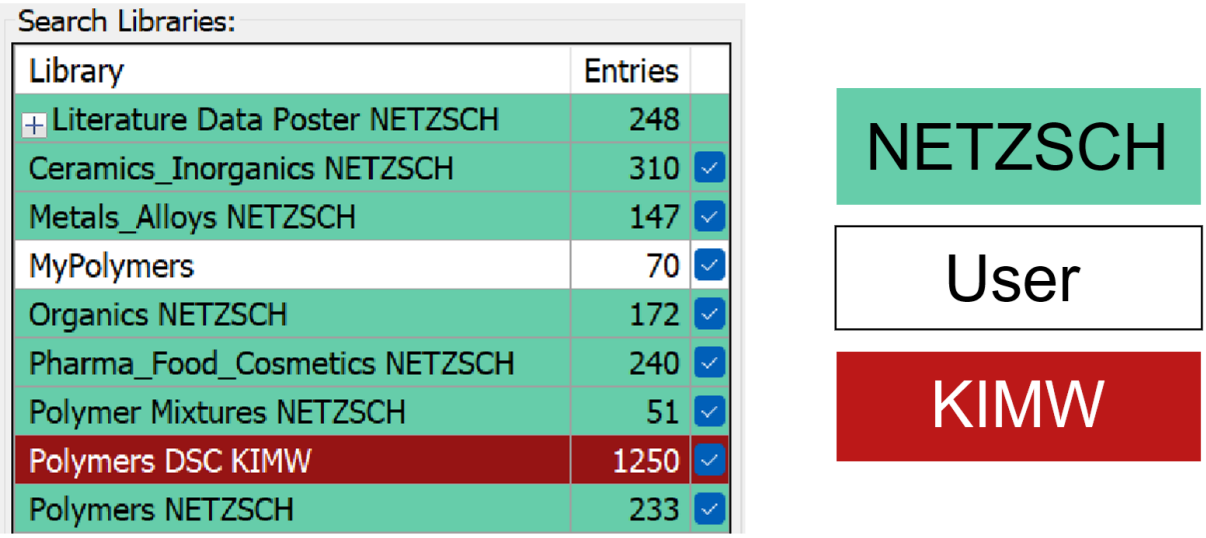
As of *Proteus*® Version 9.8, the new 'Polymer Mixtures NETZSCH' library is available and included as standard in the *Identify* package. *Identify* is part of *Proteus*® analysis and is generally useful for automatic curve recognition, material recognition and thus for interpretation also in terms of quality control. In addition, it can be used as an archive for measurements and measurement conditions. Currently, measurements and signals of DSC, TGA, TGA-c-*DTA*®, STA, c_p, DIL/TMA and DMA types are supported. The new 'Polymer Mixtures NETZSCH' library initially contains DSC measurements and is intended to improve identification of polymer mixtures and also facilitate quantification of polymer components. Applications can be found, for example, in failure analysis or the investigation of recyclates.

Analysis of a Mixture of PE-LD and PP

Figure 1 shows all the *Identify* libraries available as of *Proteus*® 9.8. onwards. The new 'Polymer Mixtures NETZSCH' library initially contains 51 DSC measurements on mixtures of the polymers PE-LD, PE-LLD, PE-HD, PP, EVOH and PET, with plans to expand the library in future *Proteus*® versions. The samples used originate from various sources and, as usual for polymers, were heated at 10 K/min to above the melting temperature, cooled again at 10 K/min and heated a second time at 10 K/min. The second heating curves are used for the analyses with *Identify*.



1 *Identify* libraries (as of 1/2025, *Proteus*® 9.8). The libraries created by users ('User') can be expanded indefinitely and can be shared and used collectively within the network; the 'KIMW library' for polymers is available as an option.

NGB · Softwre Innovation 028 · EN · 1025 · Technical specifications are subject to change.

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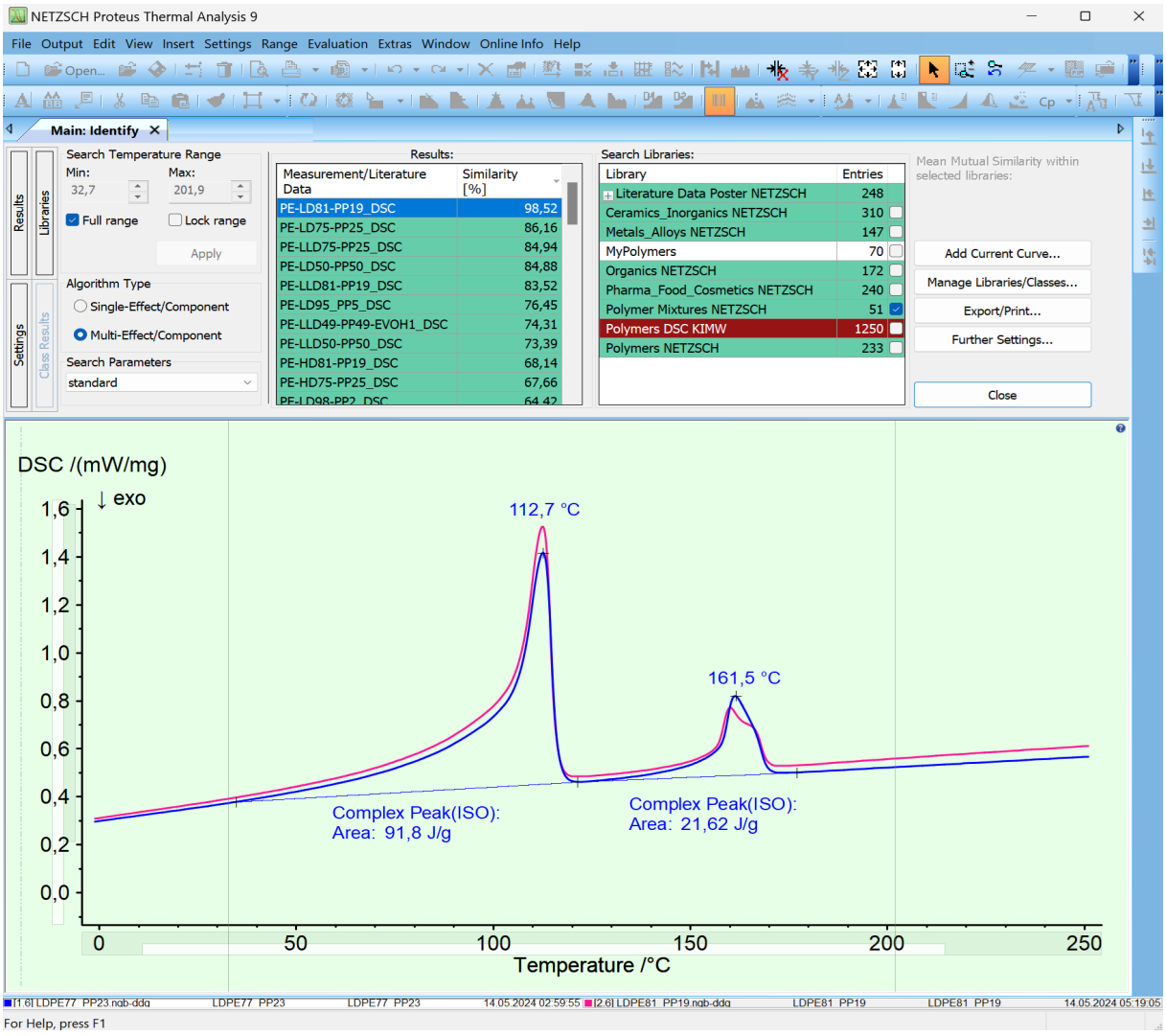
Figure 2a shows the result of an investigation of a DSC curve* on a polymer mixture, nominally consisting of 77% PE-LD and 23% PP. During the analysis across the entire temperature range, *Identify* found the best match with the very similar database curve, 'PE-LD81-PP19_DSC' (pink curve in figure 2a) from the 'Polymer Mixtures NETZSCH' library; the composition of the underlying sample was 81% PE-LD and 19% PP. This means that the polymer mixture was analyzed correctly in terms of both qualitative and quantitative composition within a few percent.



2a *Identify* analysis of a mixture of PE-LD and PP (blue DSC curve) across the entire temperature range.

*Measurement conditions according to the curves in the library.

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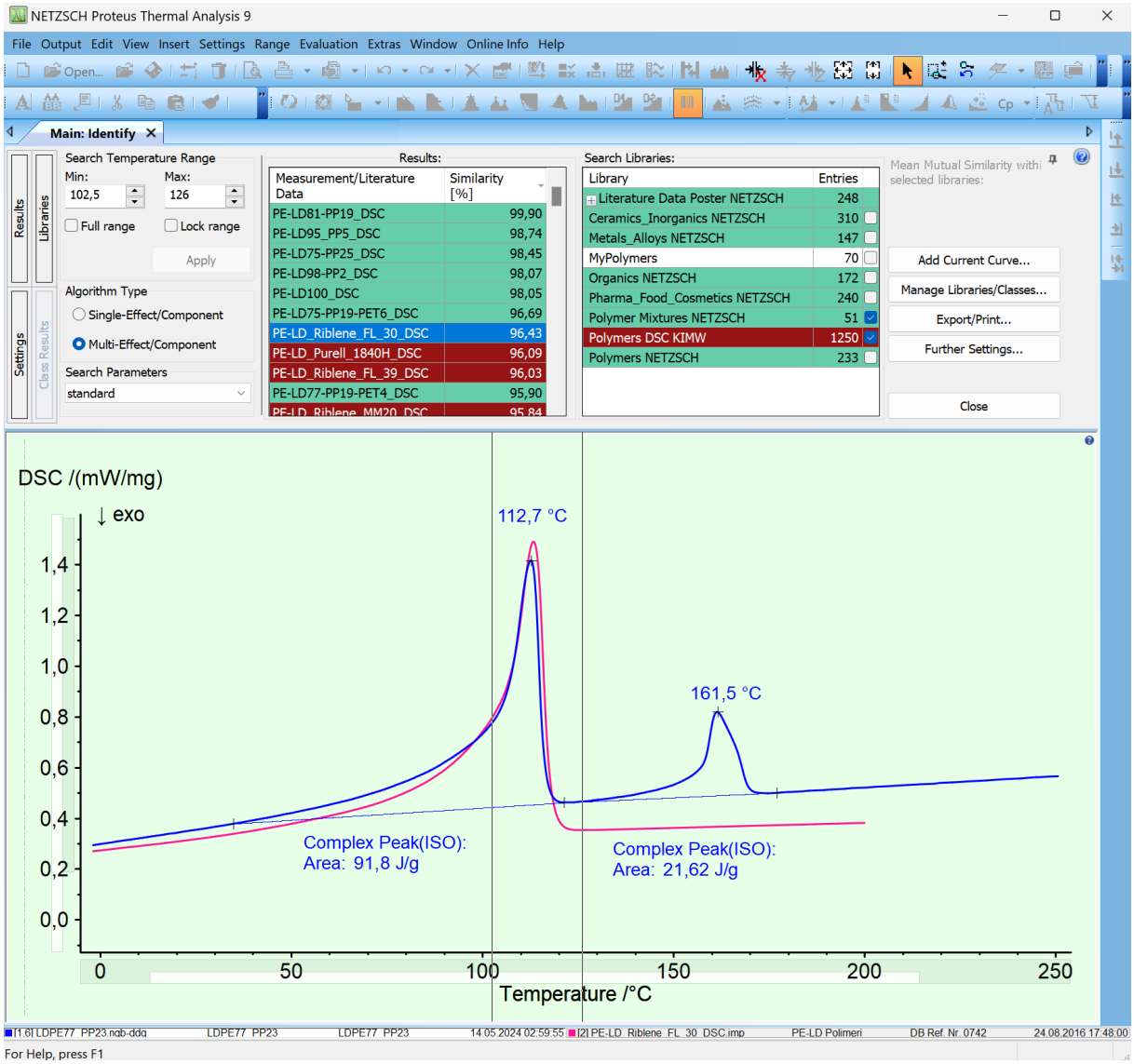
Identify offers the possibility of limiting the temperature range in which the database comparison is to be performed, e.g., to examine melting effects or glass transitions individually. This can be seen in figure 2b, where the selected temperature range covers the melting peak occurring at lower temperatures. In addition to the 'Polymer Mixtures NETZSCH' library, the 'KIMW library' was also selected, which currently contains DSC curves for 1,250 different polymer products. The result shown in figure 2b clearly proves that the melting peak at around 113°C can be attributed to PE-LD: for example, the database curve 'PE-LD_Riblene_FL_30_DSC' displayed in pink is very similar to the input curve. In addition, the hit list in figure 2b shows that PE-LD is virtually the only possibility for the melting peak at around 113°C; the other 173 polymer types contained in the KIMW database can be ruled out, which is an additional valuable piece of information.



2b *Identify* analysis of a mixture of PE-LD and PP (blue DSC curve) over the temperature range 102.5 … 126°C. In comparison to figure 2a, the 'KIMW library' was also selected.

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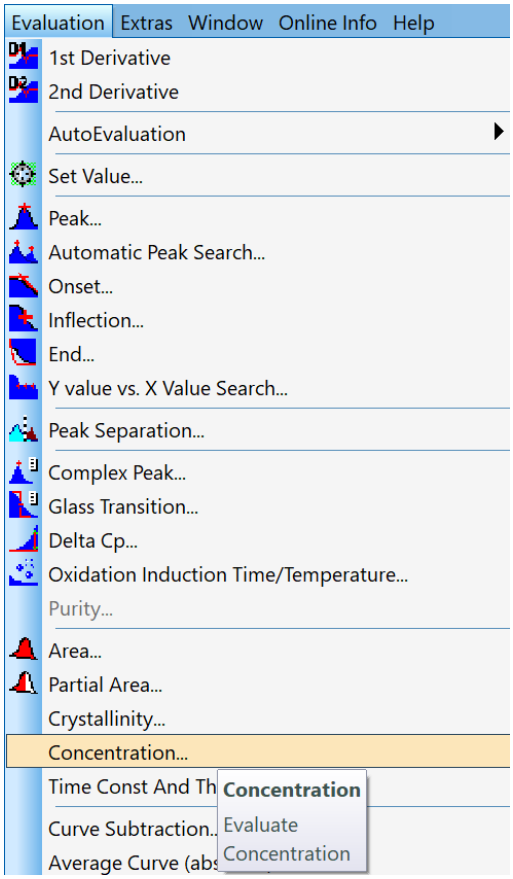
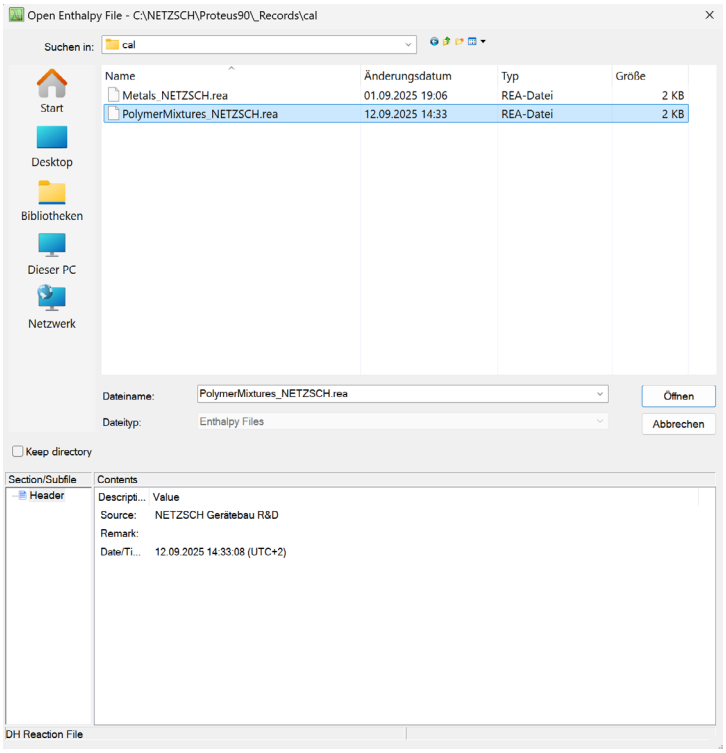
Quantification with 'Concentration'

The 'Concentration' evaluation feature is also available in *Proteus*® analysis for quantifying the amount or concentration of a substance in a DSC sample. This was introduced by Software Innovation 07. When the 'Concentration' function is retrieved (see figure 3a), the user must first select an enthalpy library, as shown in figure 3b. This library contains the melting enthalpies of pure substances, i.e., for a concentration of 100%. *Proteus*® includes enthalpy libraries for metals and polymer mixtures, more specifically for the polymers PE-LD, PE-LLD, PE-HD, PP, EVOH and PET found in the mixtures. Users can also create their own enthalpy libraries. The enthalpy values of the polymers *dHmelt_100%* are to be understood as average values, which in principle were determined from the equation $dHmelt = dHmelt\_100\% \cdot c$ using the melting enthalpies *dHmelt* of the polymers occurring in the mixtures with known concentrations c. An unknown concentration is calculated using $c = dHmelt / dHmelt\_100\%$.

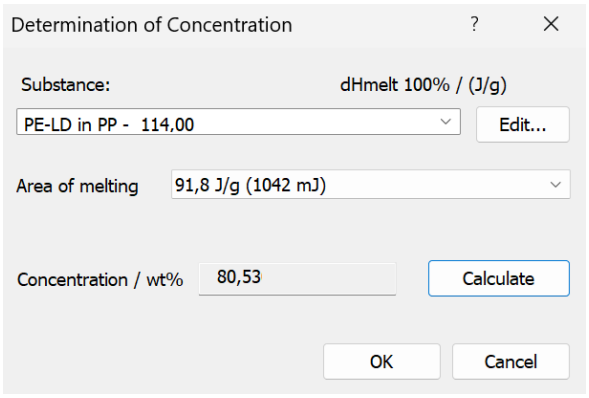
Figure 3c shows the calculation of the concentration of PE-LD from the melting enthalpy of 91.8 J/g of the DSC peak at approximately 113°C. The result is 80.5%, or around 81%, which deviates by approximately 4% from the nominal value of 77%.



3a Access to the 'Concentration' function via the 'Evaluation' menu.



3b Selection of the 'PolymerMixtures_NETZSCH' enthalpy library.



3c Dialog box for determining the concentration after selecting the substance.

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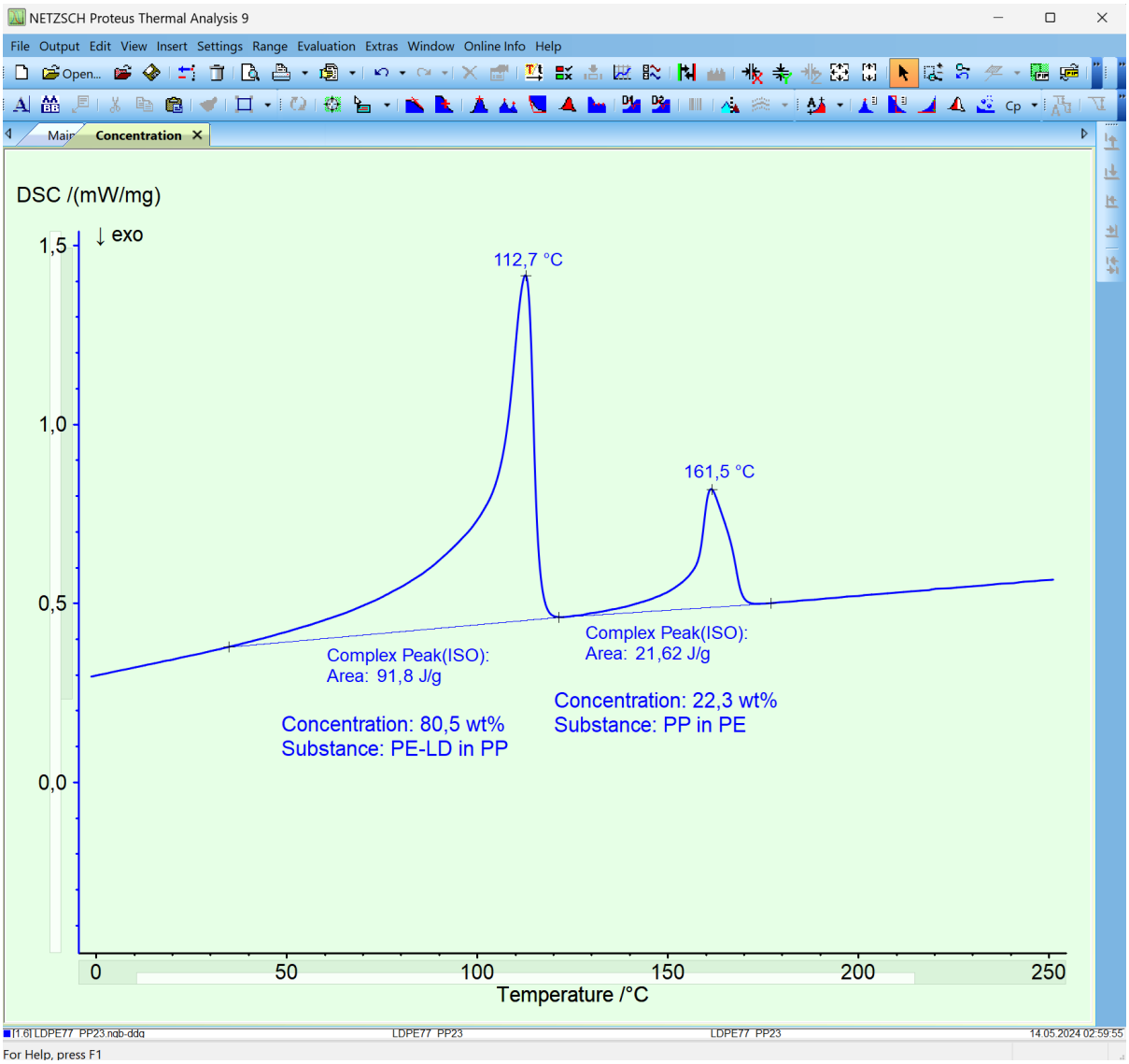
The accuracy of the method is limited by the uncertainty of the library values *dHmelt_100%* and the determination of the DSC peak area to be evaluated. At high concentrations, the accuracy is in the order of ±5%; at low concentrations, the accuracy can also be significantly better if the corresponding DSC peak of the polymer is not superimposed by DSC peaks of another polymer. In the example discussed about the mixture of PE-LD and PP, the 'Concentration' function yielded a PP concentration of around 22%, which agrees very well with the nominal concentration of 23% (see figure 4).



4 Results of the 'Concentration' evaluation feature applied to the evaluated DSC curve of the mixture of PE-LD and PP.

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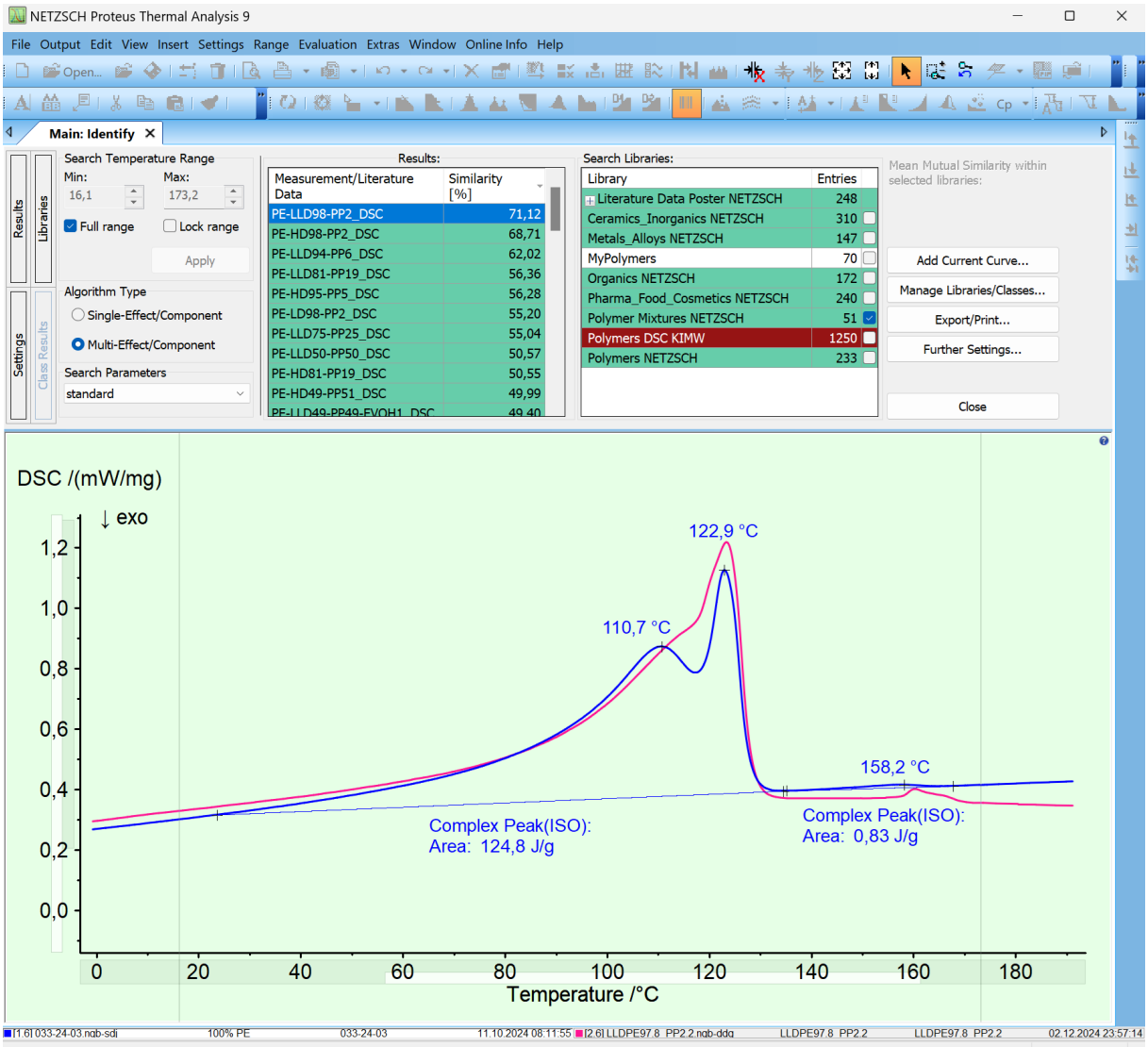
Analysis of Contaminated PE

Figure 5a shows an *Identify* analysis of a DSC curve obtained from a nominally pure PE sample ('100% PE'). The hit list and the curve comparison with the best hit 'PE-LLD98-PP2_DSC' (98% PE-LLD + 2% PP, pink curve) immediately indicate that the sample was of the PE-LLD type, which is reflected in the superimposed melting effects in the DSC curve at peak temperatures of approximately 111°C and 123°C. The second statement is that the small DSC peak at approximately 158°C is due to a PP content of less than 2% in the sample, i.e., the PE-LLD sample was not a pure PE sample but was contaminated with PP.



5a *Identify* analysis of contaminated PE (blue DSC curve) across the entire temperature range.

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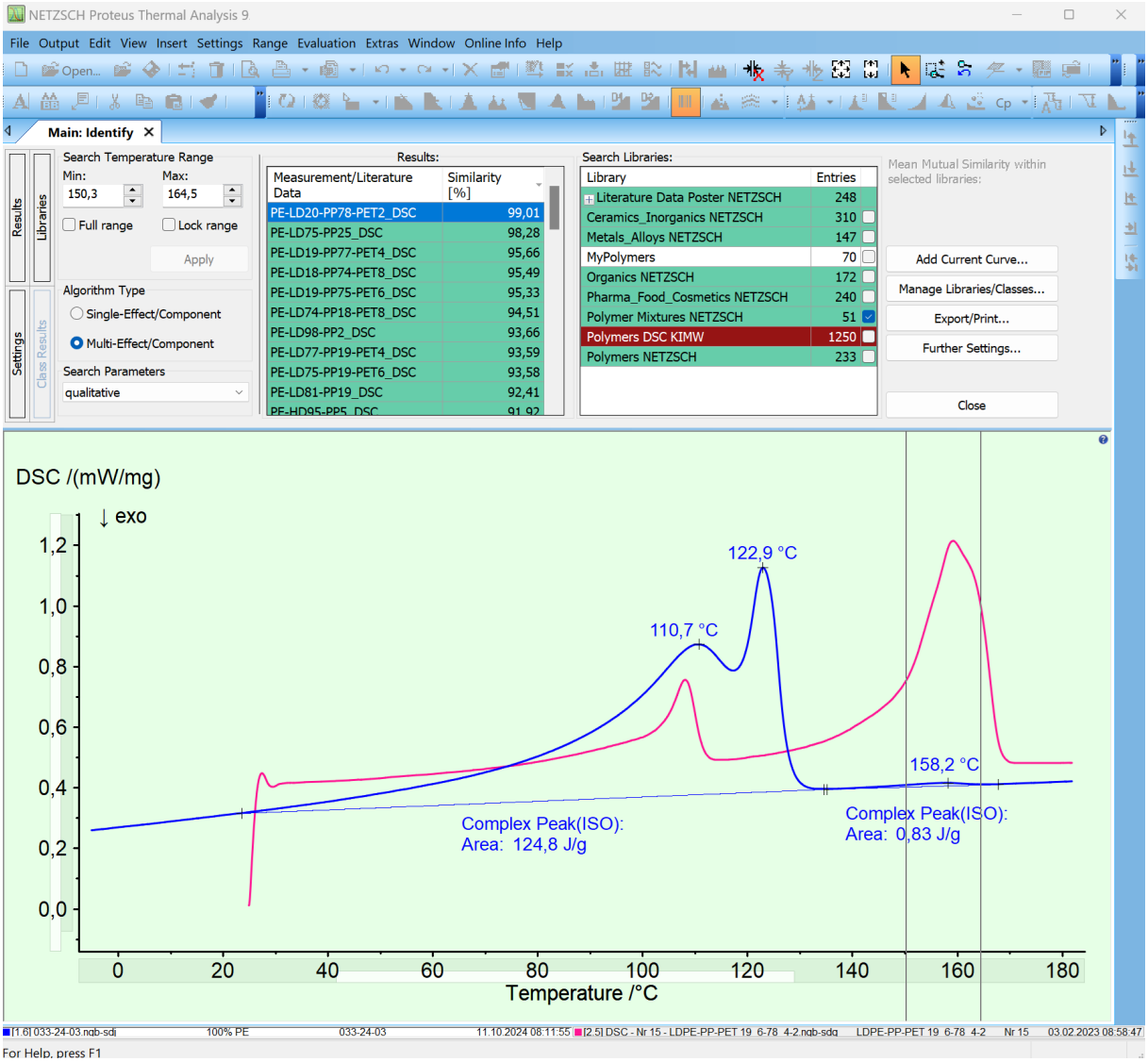
The similarity value of the best hit (pink curve in figure 5a) is only about 71% with the selected settings, which is partly due to the differing temperature ranges of the melting effect of PP. A separate, qualitative detection of this effect with *Identify* shows that there are DSC curves in the database where the position of the DSC peak originating from PP better matches that in the input curve (see figure 5b).



5b *Identify* analysis of contaminated PE (blue DSC curve) over the temperature range 150.3 … 164.5°C. Compared to figure 5a, the search parameters were changed to 'qualitative'.

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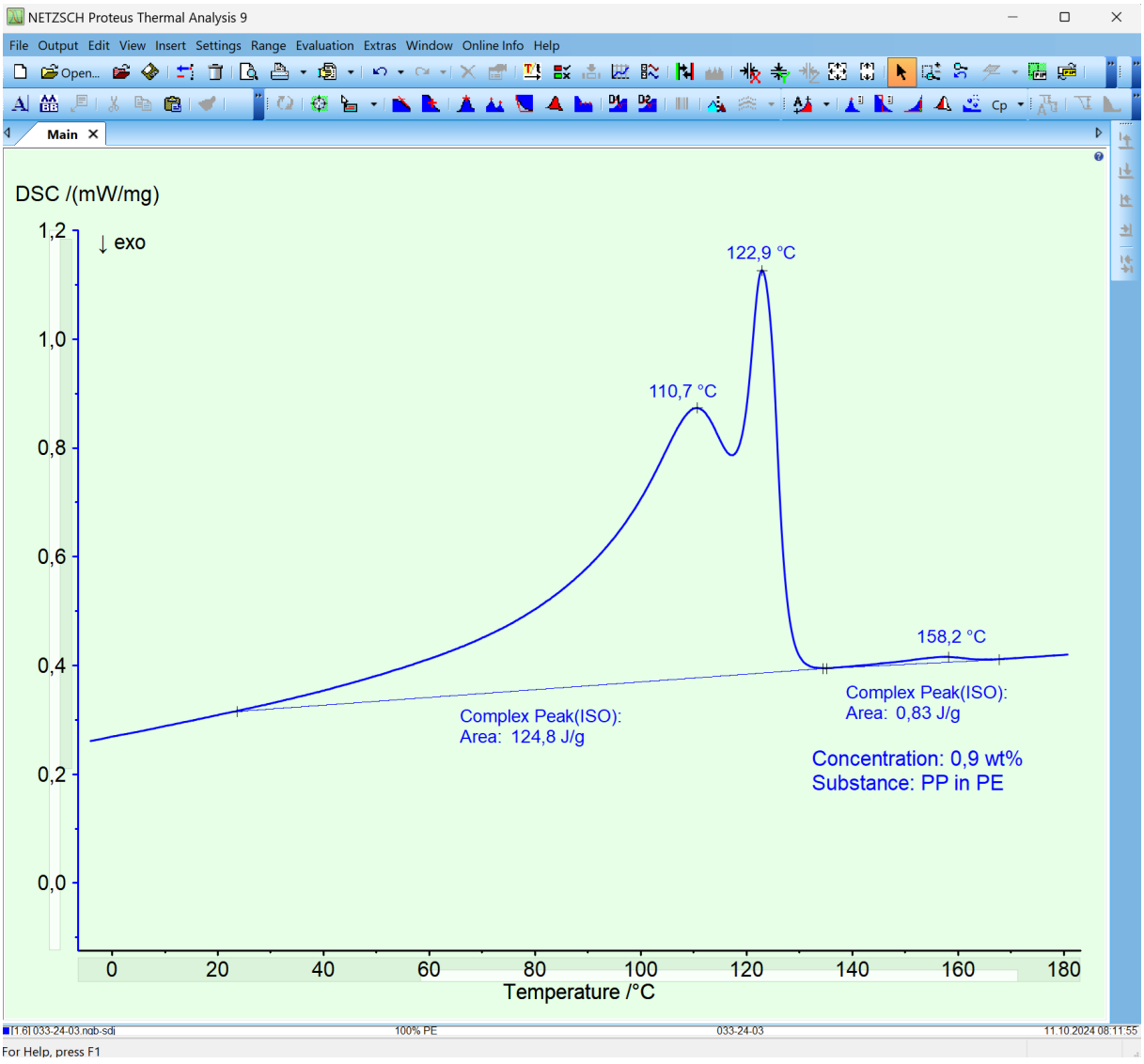
The DSC peak at approximately 158°C shown in figures 5, caused by PP contamination, was also evaluated using the 'Concentration' function; this resulted in a PP concentration of 0.9%, i.e., around 1% (see figure 6).

Finally, it should be mentioned that overlapping DSC effects can be described using the *Peak Separation* function of *Proteus*® analysis by superimposing individually calculated DSC peaks. These calculated DSC curves can be evaluated using both *Identify* and the 'Concentration' function, which can increase the accuracy of the results.

Summary

As of *Proteus*® version 9.8, the new 'Polymer Mixtures NETZSCH' library is available and included as standard in the *Identify* package. This library is designed to improve the identification of polymer mixtures and also facilitate the quantification of polymer components. In addition, the application of the 'Concentration' analysis function was demonstrated, which also enables the quantification of polymers using a DSC curve.



6 Results of the 'Concentration' evaluation feature applied to the evaluated DSC curve of the contaminated PE.

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