TG-Threshold and TG-Stability Detection During TG and STA Measurements

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From *Proteus®* version 9.8 onwards, the two *TG-Threshold Detection* and *TG-Stability Detection* functions are available for TGA and STA instruments*. *TG-Threshold Detection* recognizes when a user-defined mass-change value is exceeded, triggering automatic termination of the measurement.

With *TG-Stability Detection*, fulfilment of a user-defined mass-stability criterion triggers either a jump to the next segment of the temperature program or termination of the measurement, depending on the definition by the user.

Both functions are particularly helpful when the course of a thermogravimetric measurement cannot be predicted in detail. This is the case with unknown samples. In addition, the measured mass changes generally depend not only on temperature, but also on the measurement conditions such as the heating rate, the type of gas and the gas flow, making detailed prediction difficult.

Applications of *TG-Threshold Detection* include the detection of a small mass loss, e.g., due to decomposition or evaporation of the sample, but also identification of the time point of an expected mass loss, e.g., due to complete decomposition. *TG-Stability Detection* can be considered complementary to this. In this application, the magnitude of mass change that occurs during sample drying, for example, is not precisely known. However, it is known that after a certain time, the sample mass will reach a constant state. The function can detect this state in order to switch to the next temperature segment or start a new measurement.

Both functions can thus serve to save measurement time while maximizing the information content of the measurement. The safety of the device is also increased when, for example, unwanted evaporation of the sample is detected and thus minimized.

TG-Threshold Detection

Figure 1a shows how *TG-Threshold Detection* can be activated within a measurement definition: The last line of the temperature program defines the emergency reset temperature, i.e., the temperature above which the measurement is interrupted for safety reasons. In addition, the user can specify that the measurement is also automatically terminated if a certain mass change is exceeded. As shown in the example, this is set to a mass loss of 1%.

This threshold criterion can be combined with an additional delay time between the exceeded mass change and the actual termination of the measurement. No delay time was defined in the example shown in figure 1a; i.e., the measurement should terminate immediately after the defined mass change is exceeded. The measurement result is depicted in figure 1b. A melting peak can be seen in the DSC signal (blue curve) at around 190°C. Above this temperature, a slight mass loss is visible in the TG signal (green curve) due to the onset of decomposition. As expected, this triggered the termination of the measurement by *TG-Threshold Detection*. It should also be noted that the TG signal in the example was corrected in real time during the measurement using the *TGBeFlat®* mathematical buoyancy correction.

*TGA refers to Thermogravimetric Analysis, also called thermogravimetry (TG). STA refers to Simultaneous Thermal Analysis

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The example of *TG-Threshold Detection*, illustrated in figures 1a and 1b, clearly describes the application of automatically terminating a measurement when the mass loss begins due to decomposition or evaporation. As a further, contrasting application, the complete mass loss can also be automatically detected, which can essentially be achieved by the user-defined threshold value for mass change.
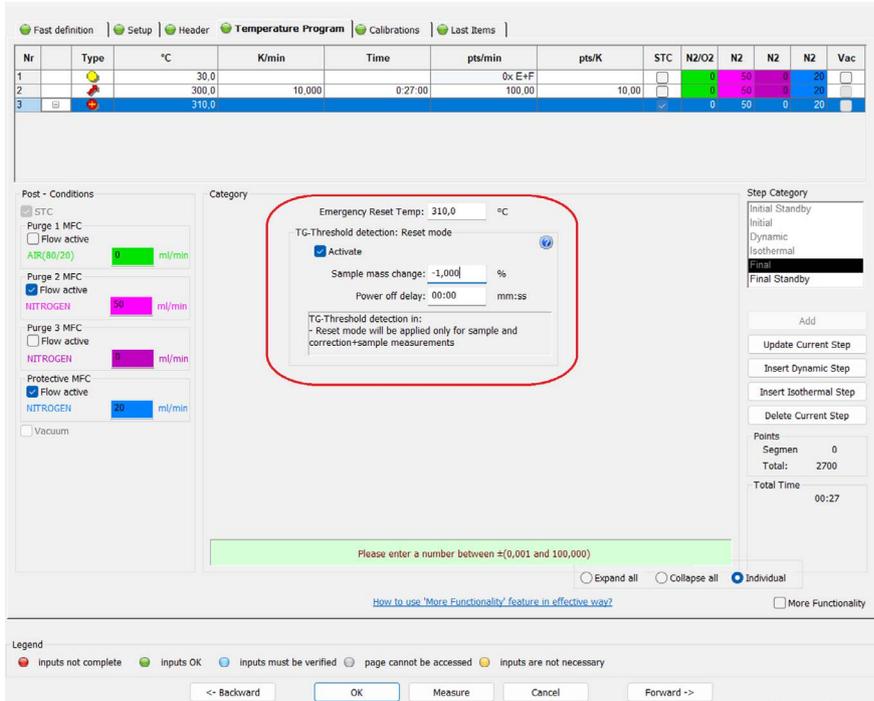
1a Activation and settings for *TG-Threshold Detection* when defining a measurement.
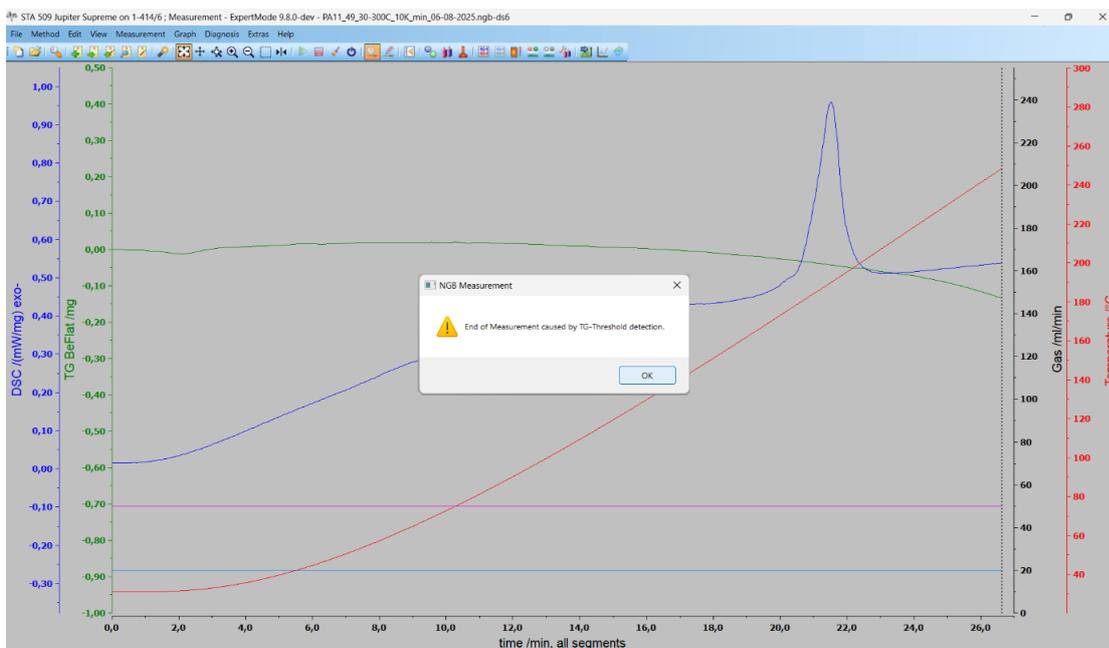
1b Automatic termination of a measurement, carried out with the STA 509 *Jupiter® Supreme* on a polymer sample (polyamid 11), based on the *TG-Threshold Detection* defined in Fig. 1a.

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In the measurement definition shown in figure 2a, a threshold value of -99.5% was set in combination with a delay time of one minute. The result is shown in figure 2b: The measurement was automatically terminated at approximately 520°C after the pyrolytic decomposition of the polymer sample was complete and the TG signal was constant. *TGBeFlat®* was also used in this example.

2a Activation and settings for *TG-Threshold Detection* when defining a measurement.

2b Automatic termination of a measurement based on the *TG-Threshold Detection* defined in Fig. 2a. The measurement on polyethylene was performed using a TG 309 *Libra® Supreme*.

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TG-Stability Detection

As shown in figure 3, *TG-Stability Detection* can be activated in the setup when defining a measurement or method.

Once *TG-Stability Detection* is activated by the user, the corresponding tab appears, within which a few settings must be specified (see figure 4a). In this example, there is a 30-minute isothermal segment at 110°C, which is intended to completely dry the sample. For this segment, T*G-Stability Detection* is activated in 'Jump mode', i.e., as soon as the specified stability criterion for mass is met, a jump to the next segment of the temperature program will take place, in which the temperature is to be increased to 800°C at 20 K/min. The mass stability criterion is a combination of two values (see figure 4a): In the example, a maximum mass change of 0.1%, relative to the initial sample mass, must be achieved within a time window of 3 minutes. The user can also employ the 'Relative value' check box to specify that instead of a relative value, the mass change should fall below an absolute value in milligrams. In addition, a delay time between the stability criterion being met and the jump to the next segment can be defined; in

the example shown in figure 4a, the default value 00:00:00 (hh:mm:ss) is used, i.e., no delay time. In the last segment of the temperature program, *TG-Stability Detection* is activated in 'Reset mode', which may lead to the measurement being terminated when the stability criterion is reached. At the beginning of this segment, i.e., from 800°C, the purge gas in the sample chamber is to be switched from nitrogen to air, which is not visualized in figure 4a.

The result of the measurement according to the definition shown in figure 4a is depicted in figure 4b. It can be seen that *TG-Stability Detection* triggered the jump to the next segment after a measurement time of approximately eight minutes, i.e., after the sample had finished drying at 110°C. At approximately 49 minutes, the measurement was interrupted by *TG-Stability Detection* because the mass-stability criterion was met again – in this case, after the combustion of the so-called pyrolysis soot was complete. Overall, the duration of the measurement was reduced from 78 minutes to only 49 minutes in this example due to *TG-Stability Detection*; in other words, the time saved was approximately half an hour.
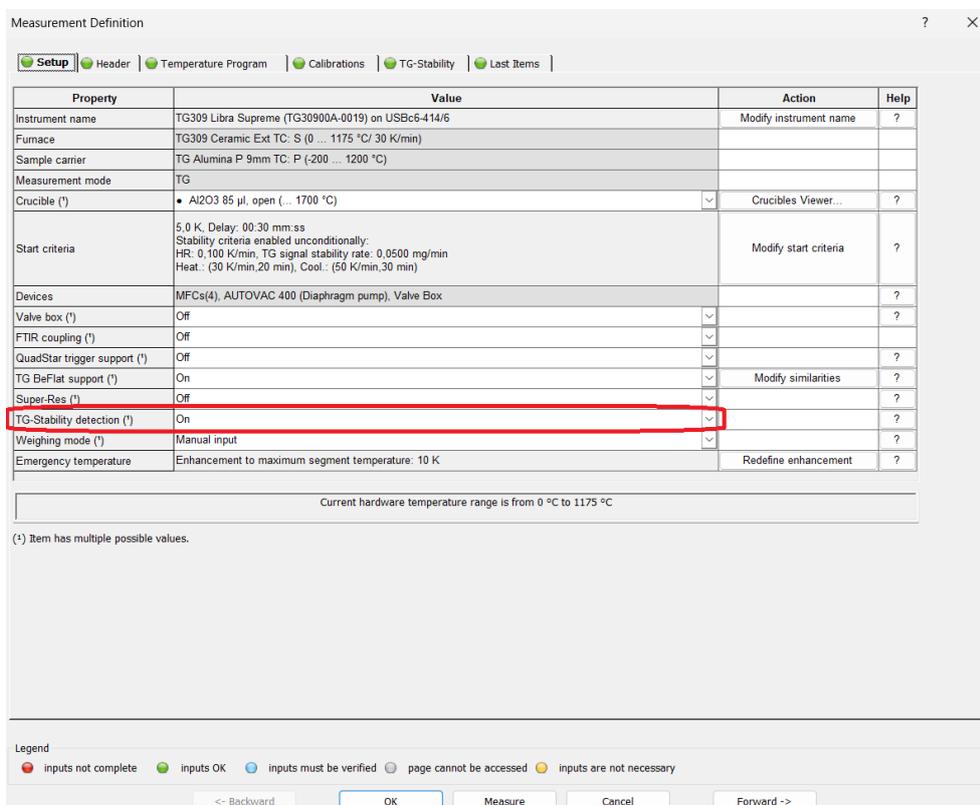

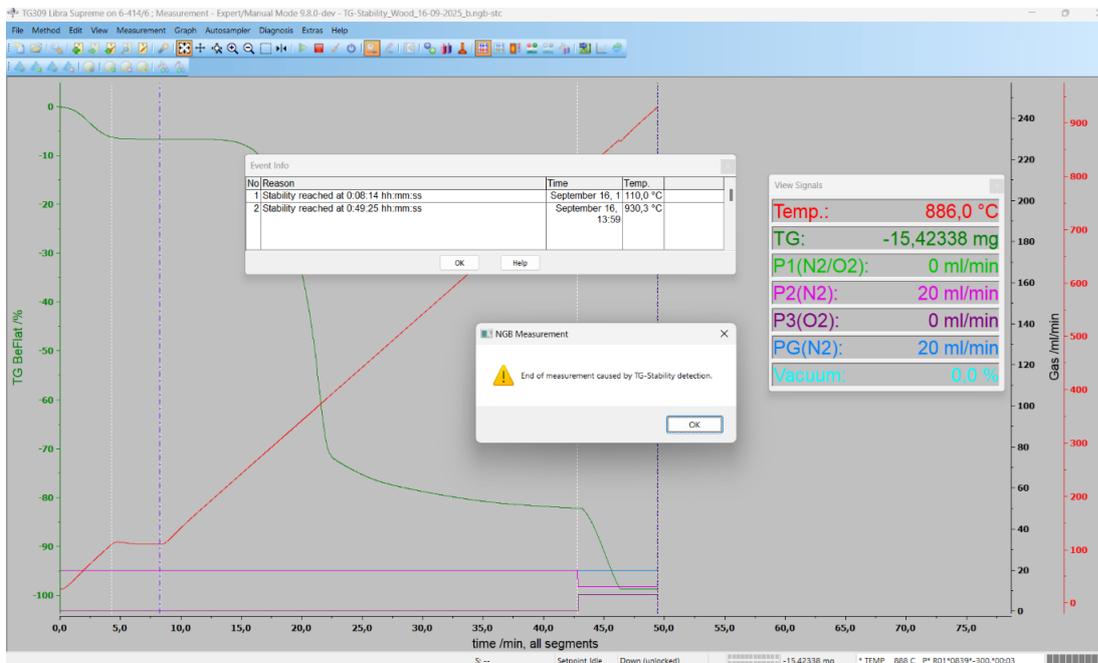**3** Activation of *TG-Stability Detection* when defining a measurement.

The measurement shown in figures 4 was performed on another wood sample of the same type in a modified form. In case of the first wood sample, drying, pyrolysis and combustion were performed in a single measurement. In case of the second sample, drying was performed in a first measurement and pyrolysis and combustion in a second measurement on the same sample. Before the second measurement, the (dried) sample was weighed again using the internal balance of the TG 309 *Libra® Supreme*.
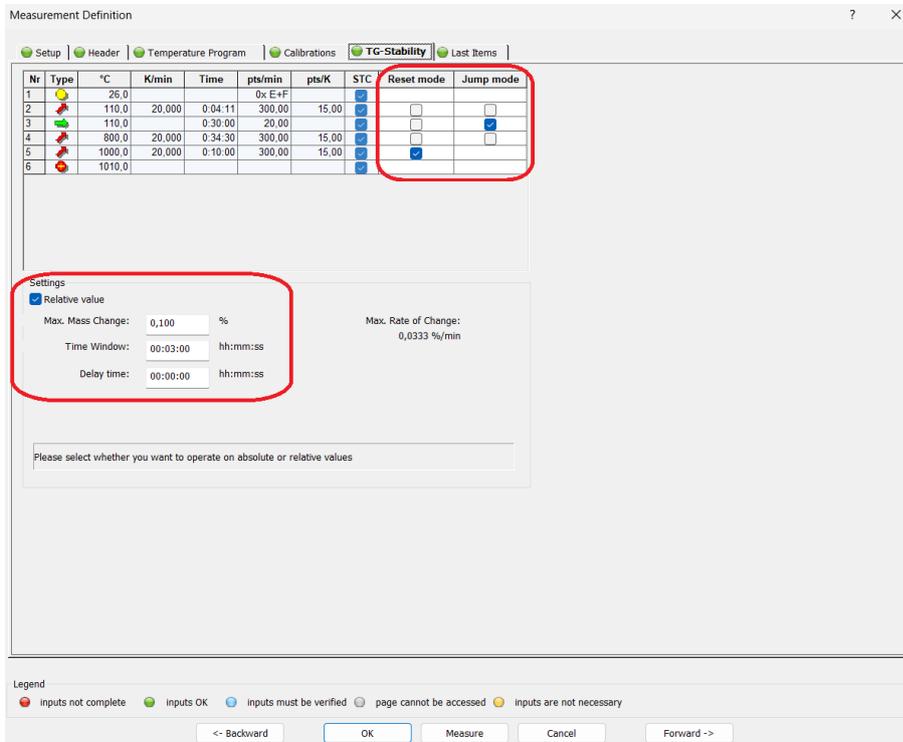
**4a** Settings for *TG-Stability Detection* in a measurement definition.

4b Automatic termination of a measurement based on the *TG-Stability Detection* defined in fig. 4a. The measurement on wood was performed usind a TG 309 *Libra® Supreme*.

The results of both measurements, which were carried out using appropriate measurement methods and the TG 309's automatic sample changer (ASC), can be seen in figures 5: In both, drying (see figure 5a) and pyrolysis and combustion of the wood sample, the measurement was terminated prematurely by the *TG-Stability Detection.*
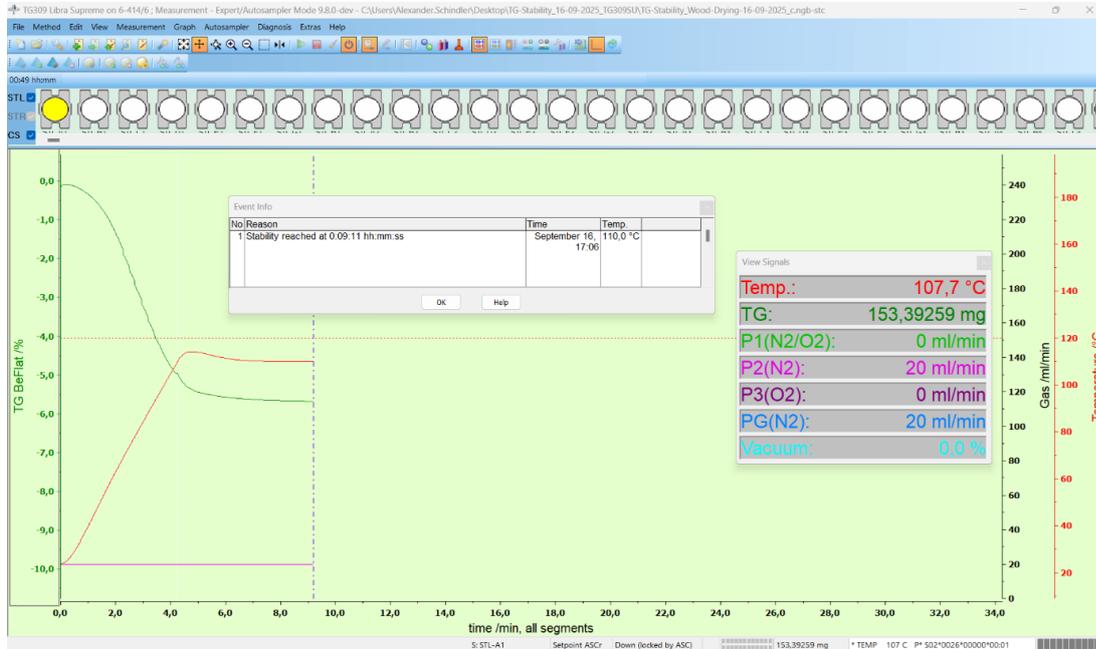
5a Automatic termination of a measurement (drying of a wood sample) caused by *TG-Stability Detection*.
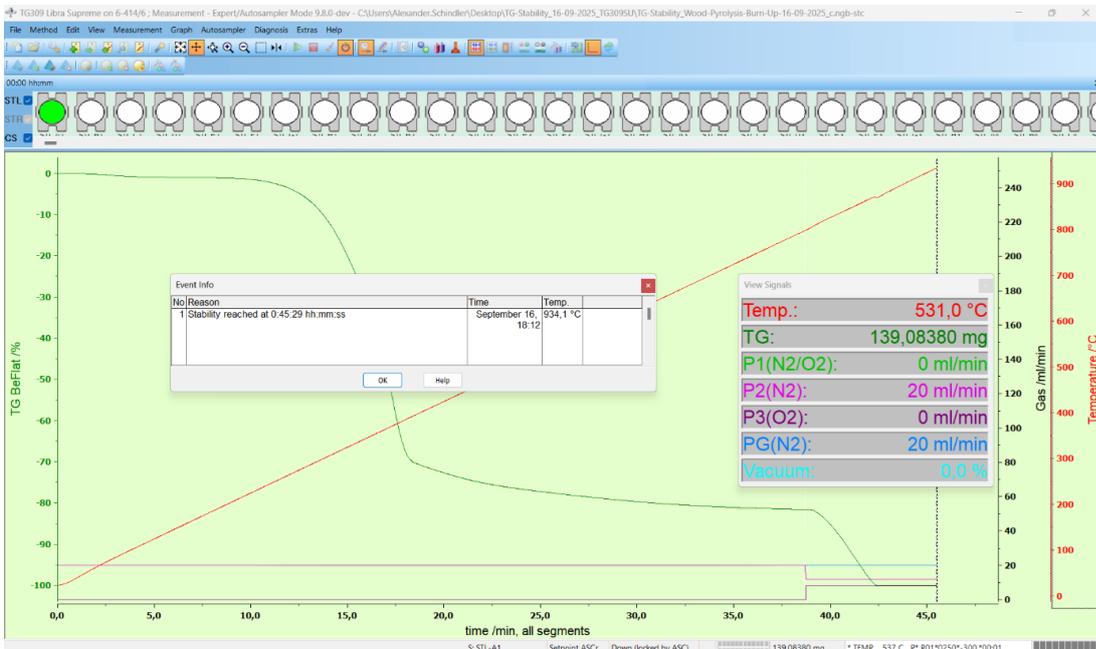
5b Automatic termination of a measurement (pyrolysis and combustion of a dried wood sample) caused by *TG-Stability Detection*.

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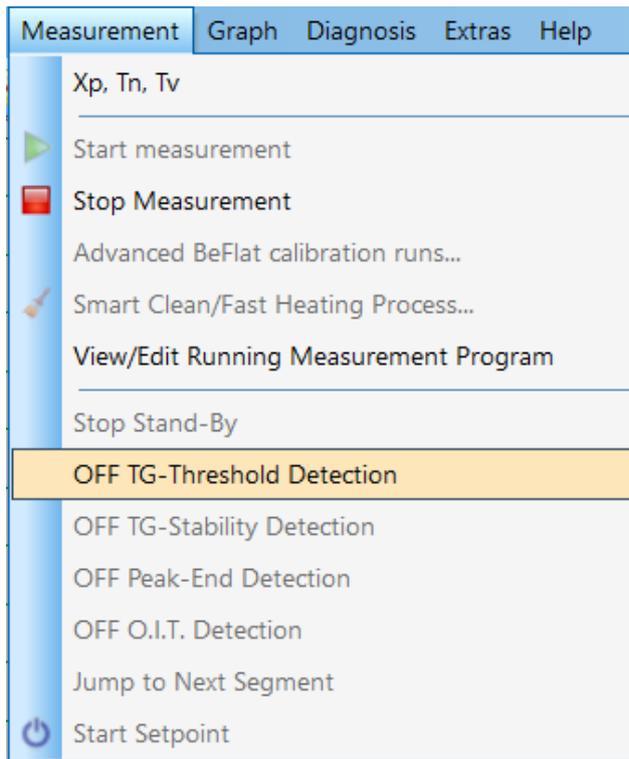
6 Deactivation of the *TG-Threshold* and *TG-Stability Detection* functions during a running measurement.

Finally, it should be noted that both *TG-Stability Detection* and *TG-Threshold Detection* can be deactivated during the measurement if necessary, provided that these functions have been activated in a measurement (see figure 6).

Summary

As of *Proteus*® version 9.8, the two *TG-Threshold Detection* and *TG-Stability Detection* functions are available for TG and STA devices. *TG-Threshold Detection* recognizes when a user-defined value for mass change is exceeded, triggering an automatic termination of the measurement. With *TG-Stability Detection*, the fulfilment of a user-defined stability criterion for the mass triggers either a jump to the next segment of the temperature program or termination of the measurement, depending on the definition. Both functions help save measurement time. In addition, the safety of the device can be increased if, for example, unwanted evaporation of the sample is detected and subsequently minimized.

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