**DSC purity**

You can use this purity determination in the chemical and pharmaceutical industries and also for additives in the food and plastics industries. In research, development and the quality lab, the content of the main component of a sample is of interest. However, with the purity analysis the identity can also be checked, essentially by means of the melting temperature and the heat of fusion.

The DSC purity analysis is based on the Van’t Hoff law of melting point depression of eutectic systems. For such substances, purities between 90 and 100 mol% can be determined with the accuracy of the result being typically 10% of the impurity.

Compared with a conventional melting point determination in which the pure substance also has to be measured, the DSC purity analysis has the advantage of requiring only a single DSC measurement. The possibility to heat the samples very quickly (even up to 10 °C/min) permits decomposition effects of the substances to be separated from the melting effects (however, ASTM E928 stipulates a maximum heating rate of 0.7 °C/min!).

**Theory**

The normal purity analysis («purity») uses the simplified Van’t Hoff law:

\[
T_{\text{ fus}} = T_o - \frac{x_{2.0} \cdot R \cdot T_o^2 \cdot m}{M} \cdot \frac{1}{A_{\text{ tot}} + c}
\]

This can also be used to evaluate partial melting peaks, an important advantage if the sample starts to decompose immediately following melting.

The complete Van’t Hoff law (menu: Purity Plus) is particularly recommended for higher concentrations of impurities (> 5 mol%):

\[
T_{\text{ fus}} = T_o + \frac{R \cdot T_o \cdot T_{\text{ fus}}}{\Delta H_{\text{ fus}}} \cdot \frac{1}{x_{2.0}} \cdot \ln \left( \frac{A_{\text{ tot}} + c}{A_{\text{ part}} + c} \right)
\]

- \( T_{\text{ fus}} \): temperature of the sample during melting
- \( T_o \): melting point of the pure substance (= T Fusion Pure)
- \( x_{2.0} \): mole fraction of the impurity in the sample
- \( A_{\text{ fus}} \): measured peak area in mJ
- \( A_{\text{ part}} \): measured partial peak area in mJ
- \( m \): sample mass
- \( M \): molar mass of the main component (pure substance)
- \( \Delta H_{\text{ fus}} \): enthalpy of fusion of the pure substance in J/mol
- \( c \): linearization correction in mJ
- \( R \): gas constant, 8.134 J/mol K
- \( F \): liquid fraction = \( \frac{A_{\text{ part}} + c}{A_{\text{ tot}} + c} \)

The 1/F plot allows a visual assessment of the raw data and the linearized function.
**Application examples**

**Purity analysis of acetylsalicylic acid**

As the example of acetylsalicylic acid shows, in addition to the purity and the melting temperature the following results can also be calculated if desired:

- **T Fusion 10%** Melting temperature at 10% liquid fraction ("start of melting")
- **Depression** Melting point depression ($T_o - T_{fus}$)
- **Correction** Linearization correction in % of $A_{tot}$
- **Delta H + Corr** Correct heat of fusion in J/g and kJ/mol
- **Suggested Rate** Suggested maximum heating rate; determined from peak shape
- **Cryos Constant** Cryoscopic constant in % per K depression

**Purity determination of dimethyl terephthalate**

The example opposite shows the purity analysis of different samples of highly impure dimethyl terephthalate (DMT). The impurity makes the peak broader and lowers the peak temperature.

Pure DMT was weighed into an aluminum crucible and increasing amounts (0–11 mol%) of salicylic acid added as an impurity. These crucibles were hermetically sealed and measured at a heating rate of 2 K/min. Evaluation was then performed with “Purity Plus”. The results show that even with high levels of impurity there is a good agreement between the value calculated from the initial weight (calculated value) and the measured purity.

**Purity analysis of a metal**

With zinc as an example, the purity analysis of a highly pure metal showed that at a heating rate of 0.5°C/min melting occurred. The freely selectable result block contains all desired information. The purity of 99.98% calculated using the simplified law of Van't Hoff agrees well with the producer’s specification of 99.9%.

The 1/F plot shows the melting temperature $T_{fus}$ as a function of the reciprocal of the liquid fraction 1/F both before and after the required linearization.