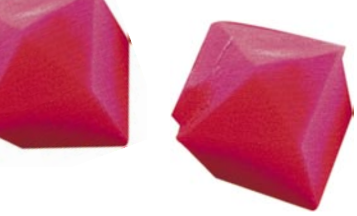
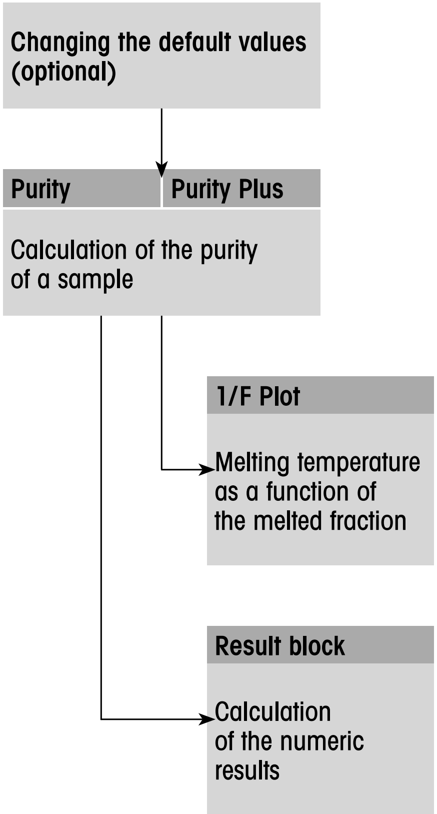


DSC purity

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:

$$T_{fus} = T_o - \frac{x_{2.0} \cdot R \cdot T_o^2 \cdot m}{M} \cdot \frac{1}{(A_{part} + c)}$$

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%):

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

T_{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_{tot}	measured peak area in mJ
A_{part}	measured partial peak area in mJ
m	sample mass
M	molar mass of the main component (pure substance)
ΔH_{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 = $(A_{part} + c) / (A_{tot} + c)$

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

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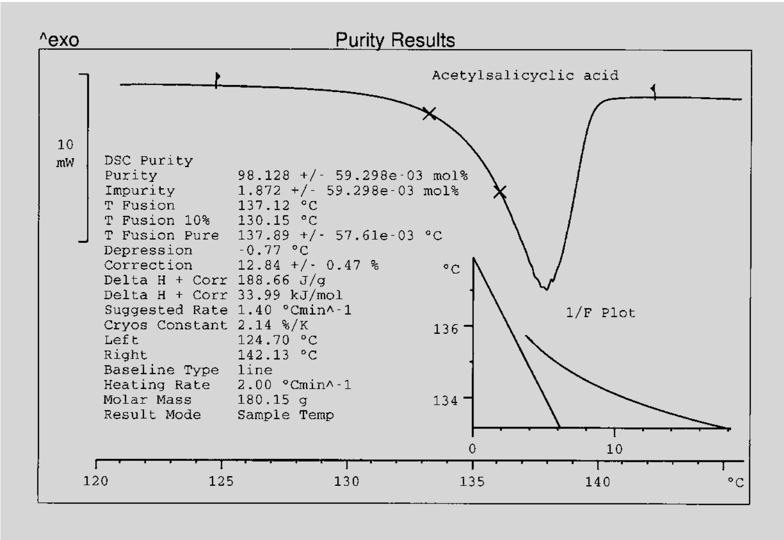
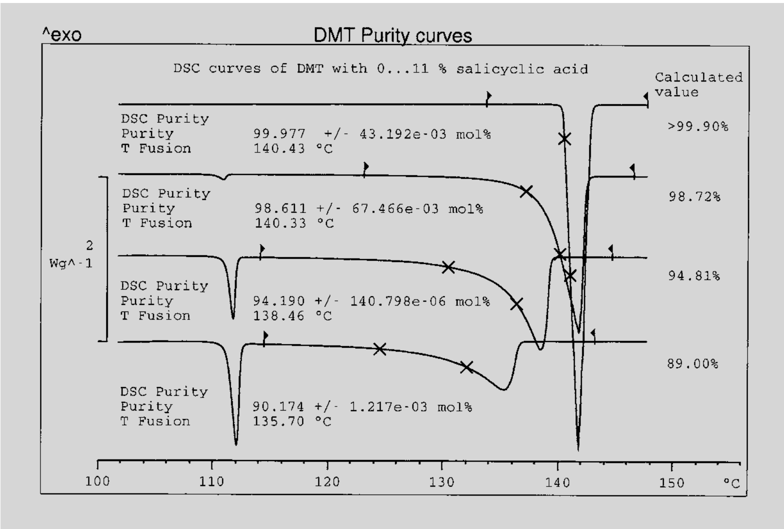
Application examples



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:

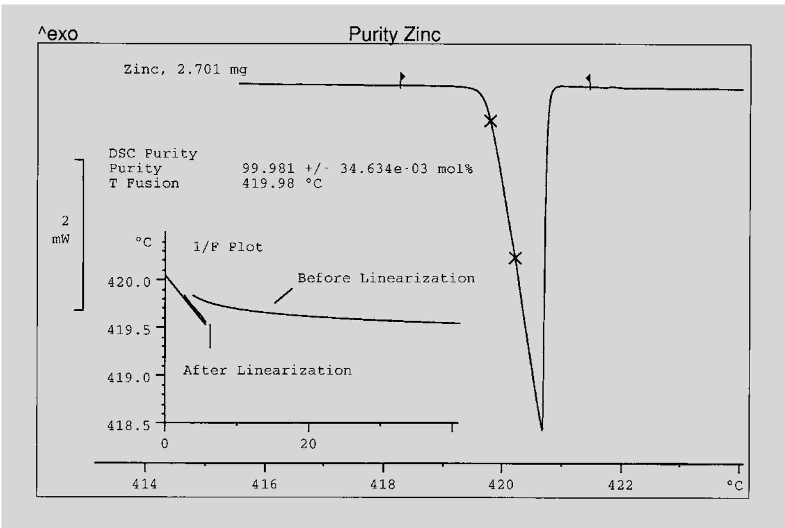
T Fusion 10%	Melting temperature at 10% liquid fraction ("start of melting")
Depression	Melting point depression ($T_o - T_{fus}$)
Correction	Linearization correction in % of A_{tot}
Delta H + Corr	Correct heat of fusion in J/g and kJ/mol
Suggested Rate	Suggested maximum heating rate; determined from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

Mettler-Toledo GmbH, Analytical
Postfach, CH-8603 Schwerzenbach
Phone (01) 806 77 11, Fax (01) 806 73 50
Internet: http://www.mt.com/ta

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