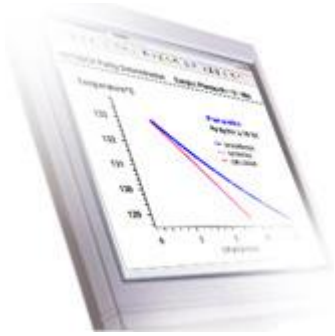


Purity determination

Purity Determination is a program for determining the purity of a material through analysis of the DSC melting peak.

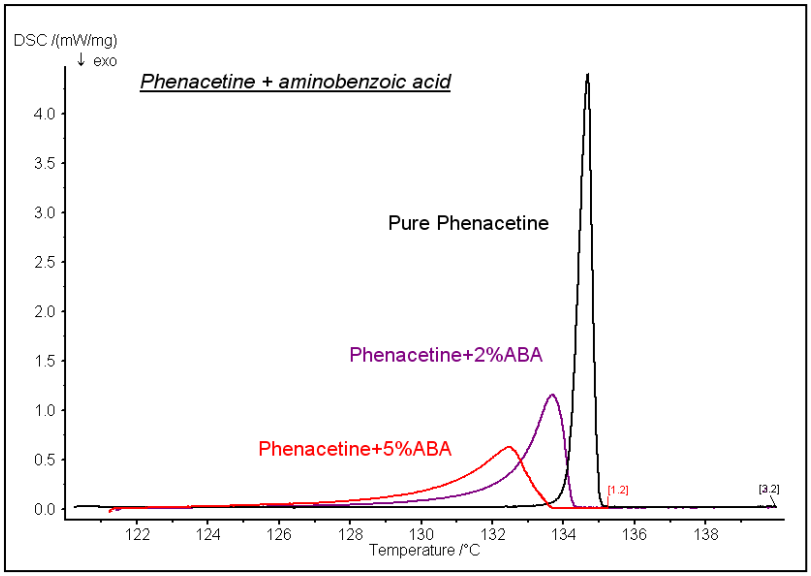
The calculation is carried out without any simplification of Van't Hoffr's equation. The adjustment of thermal resistance on the basis of purity determination of a pure metal itself increases the reliability of values of high purity.

Applications :

Purity Determination software is widely applied in organic chemisry, pharmaceutical and chemical industry.

With increasing contents of impurity, the melting process is observed at lower temperatures as well as broadening of the DSC melting peak. These facts form the basis of purity determinaion according to the VAN'T HOFF theory.

The figure presents the dependence of DSC peak shape on the content of amino-benzoic acid in phenacetin (purity standard set of NBS). The using of ASCII files as common interface allows the analysis of data, measured with different instruments



The NETZSCH purity software uses the VAN'T HOFF equation in the non-linearized format. The melting temperature of the pure main component *T0/°C,* the content of impurity *x2/mol%* and the correction parameter *Corr* are determined without any mathematical approximation by means of non-linear regression. The partial peak area *F* and sample temperature *Tm* are taken from measured curve.

$$T = \frac{T_0}{1 + \frac{RT_0}{\Delta H_f} \cdot \ln\left(1 - x_2 \cdot \frac{1 + Corr}{F + corr}\right)}$$

In order to achive high accuracy of results, especially in the range of small impurities, the measured DSC signal is corrected for the thermal resistance of the DSC sensor and crucible arrangement. The thermal resistance is precisely determined from measurements with materials of exactly known high purity.

The following features set this program apart from the known solutions as well as from ASTM E 928:

- determination of purity using the VAN'T HOFF equation without a mathematical approximation. Thus, the range of application can be extended beyond the threshold of impurity of 5 mol%. The estimated upper boundary could be 10 mol%
- adjustment of the thermal resistance in such a way that the nominal value is calculated for a substance with a known degree of purity. This yields not only a high degree of precision in the high purity range, but substances of a lower purity can be used to determine the thermal resistance

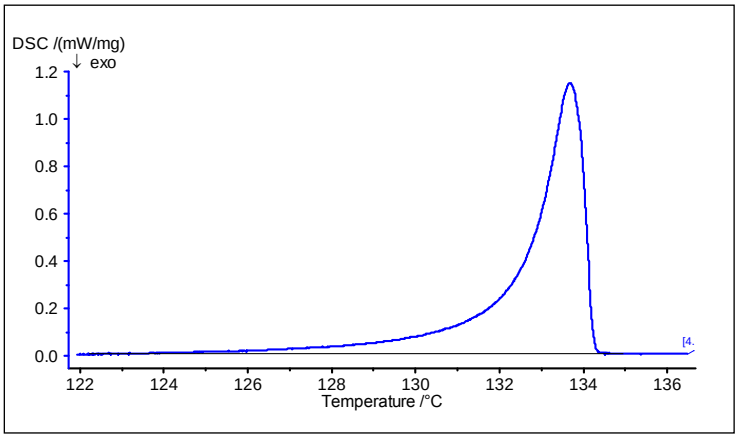
The names and the molar masses of the substances used are stored in a table and are available after every start. ASCII-Files serve as the general interface for the transfer of data.

DSC Measurement

The DSC data can be corrected using three different baselines:

- linear,
- horizontal area-proportional and
- tangential area-proportional.

In application example the DSC melting peak of 2% aminobenzoic acid in phenacetin (purity standard set of NBS) is used as the data for determination of purity with linear baseline type.



Report of Data

For analysis according to good laboratory practice (GLP), all parameters of analysis are shown in the heading of result list.

NETZSCH Purity Determination

Date/Time: 04/02/2008 at 14:52

Data identity:	041-3-04-03 21.07.2004 16:25:51		
Original file:	041-3-04-03.sd3		
ASCII file:	041-3-04-03.txt		
Sample:	Phenacetin + 2% ABA		
Substance:	Phenacetine	Heating rate/(K/min):	0.64
Molecular mass/(g/mol):	179.22	Sample mass/mg):	1.02
Base line:	linear	LeftPts: 1	RightPts: 1
Crucible:	Aluminum, pierced	Thermal resistance/(K/mW):	0.0875
Start at partial area:	0.10	TempMin/°C:	121.44
End at partial area:	0.50	TempMax/°C:	135.72
End at rel. peak height:	0.90		

RESULTS

Parameter	Value	t*Stand.Dev.
Impurity/mol%	1.8555	0.0071
Purity/mol%	98.1445	0.0071
To/°C	134.6177	0.0065
TempClear/°C	133.8037	constant
TempBegin/°C	128.8632	constant
Correction/%	5.7219	0.0483
Enthalpy/(kJ/mol)	31.7466	constant

STATISTICS

Corr.Coeff.:	1.00000	t-Val(0.95,118):	1.972

The following results are listed in the report:

Name of Parameter	***Meaning***
Impurity/mol%	*concentration of the impurity(ies)*
Purity/mol%	*purity,*
To/°C	*melting temperature of the pure substance, extrapolated temperature for 1/Partial Area => 0.*
TempClear/°C	*temperature is obtained through extrapolation of 1/Partial Area ->1.*
TempBegin/°C	*temperature for which the Partial Area = 0.1,*
Correction/%	*correction of the peak area*
Enthalpy/(kJ/mol)	*melting enthalpy of the substance (measured from the DSC peak).*

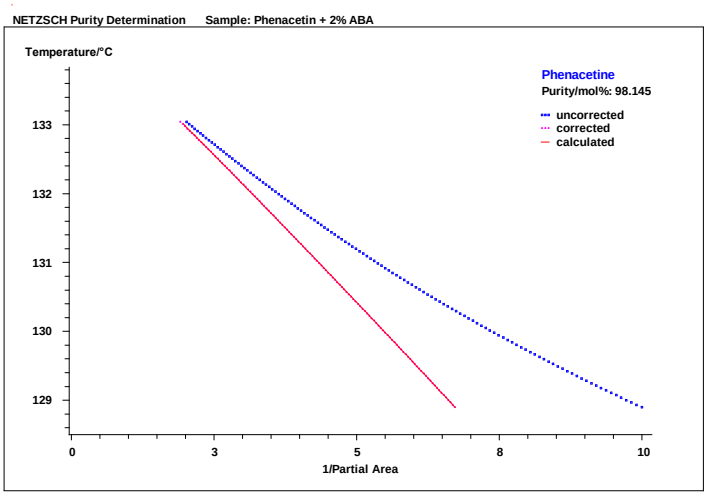
The results can be printed out with the graph.

Van’T Hoff plot

The software determines the partial area F which is proportional to the molar fraction melted at temperature *Tm*.
Here the range *F* from 0.1 up to 0.5 is selected to determine the purity. The plot of *Tm* versus inverse partial area at that temperature, normally gives a curved line. Therefore, a correction of partial areas is necessary to transfer VAN’T HOFF curve into a straight line.

The slope of this line is proportional to the total contents of impurity; the intercept is the melting temperature of the pure material.

Impurity value 1.85 mol% is in the good agreement with certified level of ABA 1.91±0.12.



Additional information can be found on www.therm-soft.com