Preface

The application of Thermic Analysis (TA) to food technology is relatively new. In the past, thermic analysis was used, above all, as a standard method for the investigation of polymers. The processes of conversion in complex foods and even in pure food components (e.g. the denaturation of proteins) often exhibit only very low energy changes. This places very high demands on the measurement system and the evaluation software. In the meantime, these performance standards have been attained, with the result that DSC is now being used more and more in the food industry for routine process analysis and quality control and not just for research and development.

This booklet presents an overview of the main areas of application of thermic analysis: storage lifetimes or process parameters can be determined as well as interactions between food components. Every method, of course, requires a specific evaluation period before it can be routinely employed, during which time Mettler Toledo application engineers will be pleased to be of assistance.

Most of the applications described in this booklet were performed in the Fraunhofer Institute of Food Technology and Packaging, Freising, Germany where DSC is applied to widely different development problems in industry. Some additional measurements were carried out in the market support laboratory of Mettler Toledo in Schwerzenbach, Switzerland and the layout is done by Helga Judex.

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Georg Widmann, Schwerzenbach

This application booklet presents selected application examples. These have been tested with the utmost care using the analytical instruments mentioned in the booklet. The experiments were conducted and the resulting data evaluated according to the current state of our knowledge.

The application booklet does not however absolve you from personally testing the suitability of the examples for your own methods, instruments and purposes. As the use and transfer of an application example are beyond our control, we cannot accept responsibility.

When chemicals, solvents and gases are used, the general safety rules and the directions of the manufacturer must be observed.

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Introduction to Thermal Analysis

Thermic Analysis (TA) is the term used to describe the analytical techniques that measure the physical and chemical properties of a sample as a function of temperature or time. The sample is subjected to a temperature program, which consists of a series of preselected segments in which the sample is heated or cooled at a constant rate or held at a constant temperature. In many experiments the atmosphere is also of importance. In particular, one distinguishes between the use of inert and oxidizing gases.

Differential Scanning Calorimetry, DSC

A differential scanning calorimeter measures the difference between the heat flows to a sample and a reference pan that are subjected to the same temperature program. Heat flow corresponds to transmitted power and is measured in watts (W) or milliwatts (mW). If the heat flow or power is integrated with respect to time then a quantity of energy is obtained which is expressed in units of mWs = mJ. If the sample absorbs energy then the enthalpy change is called endothermic. If the sample liberates energy then the enthalpy change is said to be exothermic.

DSC measurements provide information on thermal effects which are characterized by an enthalpy change and by the temperature range, such as melting behavior, crystallization, solid-solid transitions and chemical reactions. Since the specific heat capacity is also measured, a change in heat capacity such as that which occurs at the glass transition can also be determined.

Typical DSC curve of an edible fat:

1. initial deflection proportional to the heat capacity of the sample
2. part of the DSC curve with no thermic effects, i.e. baseline
3. melting peak
4. onset of oxidation in air

Thermogravimetric Analysis, TGA

Thermogravimetric analysis measures the mass of a sample which is subjected to a temperature program. The measurement is performed in a defined atmosphere, usually in inert conditions (nitrogen) or in an oxidative environment (air or possibly oxygen). The mass is measured with a highly sensitive electronic balance. Interfering buoyancy or gas flow effects are compensated by a blank curve correction. Volatile components evolved from the sample are also sometimes analyzed (EGA, Evolved Gas Analysis), e.g. in a mass spectrometer coupled to TGA module.
Thermogravimetric analysis provides information on the content of volatile components such as solvents or water, on decomposition behavior and on the ash or filler content.

Thermomechanical Analysis, TMA

Thermomechanical analysis measures dimensional changes of a sample. In a TMA experiment the deformation of a sample under a constant load is measured while the sample is subjected to a temperature program. If instead of a constant load, a periodically changing load is applied, then the technique is known as DLTMA, dynamic load TMA. This method provides information on the viscoelastic behavior of the sample. In dilatometric measurements the load on the sample is low, almost negligible. The TMA signal i.e. the change in length is measured with a high resolution displacement sensor.

TMA provides information on softening temperatures, on dimensional stability on heating, and on viscoelastic behavior. Dilatometric measurements supply information on the coefficient of linear expansion, on the glass transition, on possible polymorphic transformations that cause volume changes, and shrinkage or expansion of any solid material including packaging films.
**Application Overview Food**

The table shows the effects and properties of food products that can be investigated by thermal analysis. The more important applications are marked with a large spot.

<table>
<thead>
<tr>
<th>Application</th>
<th>DSC</th>
<th>TGA</th>
<th>TMA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting and crystallization</td>
<td>●</td>
<td></td>
<td>●</td>
</tr>
<tr>
<td>Drying, evaporation, sublimation, desorption</td>
<td>●</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>Polymorphism</td>
<td>●</td>
<td></td>
<td>●</td>
</tr>
<tr>
<td>Glass transition</td>
<td>●</td>
<td></td>
<td>●</td>
</tr>
<tr>
<td>Specific heat capacity</td>
<td>●</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oxidation stability</td>
<td>●</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>Thermal stability</td>
<td>●</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>Enthalpy changes, reaction enthalpies</td>
<td>●</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemical reaction, denaturation</td>
<td>●</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>Compositional analysis, purity</td>
<td>●</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>Characterization of plastic packaging materials</td>
<td>●</td>
<td>●</td>
<td>●</td>
</tr>
</tbody>
</table>
1 Denaturation of Vegetable Proteins

Samples

Commercial seeds from health food store.

Conditions

Measuring cell: DSC20
Pan: Aluminum standard 40 µl, hermetically sealed
Reference: Approximately the same amount of water in a sealed pan
Sample preparation: Mill seeds, aqueous extraction at pH 4.5, centrifuge, discard supernatant, re-extract residue at pH 8.5, supernatant evaporated to desired dry substance content under vacuum.

DSC measurement: Heating from 30 °C to 110 °C at 5 K/min

Interpretation

Four different untreated vegetable proteins were investigated. Each curve shows an endothermic peak in the range between 72 °C and 100 °C that is characteristic for protein denaturation. The proteins differ in their thermic stability and enthalpy of denaturation. With comparable sample weights and protein concentrations, the integrals of the peaks are a direct measure of the specific reaction enthalpies.

Spelt contains the proteins that are most temperature sensitive and that have the highest enthalpy of denaturation. Whereas the wheat proteins are already extensively denatured at 86 °C, the main fraction of the soybean protein is still native. In this case, denaturation does not set in until 88 °C.

The enthalpies of denaturation for grain and oil seed proteins usually lie in the range 3 – 10 J/g protein. The enthalpy changes depend on the phase of the proteins, so that the enthalpy of denaturation of seed proteins in the original condition is less than in the dissolved state in aqueous solutions.
The higher the enthalpy of denaturation of the native protein, the better the measurement of the fractions of denatured protein in processed protein samples by DSC.

The free selection of the heating rate with DSC can be of use to help improve the measurement and detection of reactions. The higher the heating rate, the larger the detected signal, i.e. the larger the measured peak. The calculated peak area should of course be the same if the reaction process is the same. However, it should be noted that at high heating rates the reaction is shifted to higher temperatures.

Identification of vegetable proteins by DSC is an application that is less likely to be used in practice and would also be complicated since the reaction enthalpy can vary depending on the variety and growth conditions. A native protein must, therefore, always be measured as a reference when estimating the degree of denaturation.

The DSC curve is a ‘fingerprint’ of the measured sample. Such fingerprints of protein fractions in foods are characteristic of the condition of the protein.

<table>
<thead>
<tr>
<th>Sample</th>
<th>DS content</th>
<th>Sample weight</th>
<th>Reaction enthalpy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wheat</td>
<td>12.5</td>
<td>3.44</td>
<td>3.5</td>
</tr>
<tr>
<td>Spelt</td>
<td>20.0</td>
<td>5.40</td>
<td>17.2</td>
</tr>
<tr>
<td>Lupines</td>
<td>23.0</td>
<td>6.80</td>
<td>4.2</td>
</tr>
<tr>
<td>Soybean</td>
<td>13.0</td>
<td>4.78</td>
<td>5.2</td>
</tr>
</tbody>
</table>

DS = dry substance

**Conclusion**
The measurements discussed show that, in principle, any product which contains protein can be investigated by DSC. This applies all the more to animal proteins (see also egg, blood and muscle protein) owing to the generally higher original protein concentration in animal products (muscle), the greater measurement sensitivity and their occurrence in a aqueous environment.
2 Egg Protein Denaturation

Sample
Egg white of a hen's egg

Conditions
Measuring cell: DSC20
Pan: Aluminum standard 40 µl, hermetically closed
Reference: Approximately the same amount of water
Sample preparation: Egg white was separated and stirred for 2 min. Content of dry substance: DS = 11.6%. Sample mass 32.9 mg
DSC measurement: Heating from 30 °C to 110 °C at 10 K/min

Interpretation
Heating up the egg white of a fresh hen’s egg shows two main endothermic peaks at 70 °C and at 87 °C (double peak). The first peak relates to the denaturation of the conalbumin fraction (13.8% of the total protein, heat of denaturation 19.8 J/g[1]), the second peak represents the denaturation of the ovalbumin fraction (65% of the total protein, heat of denaturation 28.9 J/g[1]). Since the lysozyme fraction (3.4% of the total protein [1]) denatures between the two main peaks, the first peak is not completely separated from the second peak.

Evaluation

<table>
<thead>
<tr>
<th></th>
<th>First peak</th>
<th>Second peak</th>
</tr>
</thead>
<tbody>
<tr>
<td>Endothermic area, mJ</td>
<td>19.3</td>
<td>47.9</td>
</tr>
<tr>
<td>Temperature of peak, °C</td>
<td>69.9</td>
<td>87.4</td>
</tr>
</tbody>
</table>

Conclusion
The typical denaturation peaks can be used for identification purposes.

3 Influence of Thermal Treatment of Egg White

Sample: Egg white

Conditions:
- Measuring cell: DSC20
- Pan: Aluminum standard 40 µl, hermetically sealed
- Reference: Approximately the same amount of water
- Sample preparation: After weighing the egg white, the sealed pan is immersed in a thermostatted water bath (80 °C) for the specified exposure times.
- DSC measurement: Heating from 30 °C to 110 °C at 10 K/min

Interpretation:
Denaturation occurs during the pretreatment so that the remaining reaction enthalpy decreases with increasing exposure. The conalbumin fraction is no longer detected in the sample treated for 30 seconds, which shows that the sample has been heated to at least 80°C. The intact ovalbumin peak with a double maximum at 83°C and 88°C shows that the temperature did not exceed 80°C. With increasing treatment time, the peak shifts to an absolute maximum at 90°C. This thermically induced rearrangement is the transition from ovalbumin to the thermically more stable form S-ovalbumin (see also the next application example). The fact that the reaction enthalpy remains constant shows that neither the ovalbumin nor the S-ovalbumin fraction are significantly damaged by the heat treatment. The rearrangement is irreversible and takes place via intermediate products. The increased thermic resistance is a consequence of a change in the covalent structure. The pH value increases from 8.1 to 9.0. The transition to the more heat resistant structure amounts only to about 10% of the usual enthalpy of denaturation.
### Evaluation

<table>
<thead>
<tr>
<th>Exposure time minutes</th>
<th>Sample weight mg</th>
<th>Reaction enthalpy J/g egg white</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0 (fresh)</td>
<td>25.03</td>
<td>1.8</td>
</tr>
<tr>
<td>0.5</td>
<td>25.14</td>
<td>1.0</td>
</tr>
<tr>
<td>2.0</td>
<td>29.32</td>
<td>0.8</td>
</tr>
<tr>
<td>5.0</td>
<td>36.64</td>
<td>0.7</td>
</tr>
</tbody>
</table>

### Conclusion

The measurements show that DSC can both verify the extent of thermal treatment (for proof of the treatment) and determine the influence of the process parameters temperature/time on the protein quality for process optimization purposes.
4 Influence of Egg Storage Time

Sample
Commercially available eggs, class A
Packaging date: June 6
Purchase date: June 18

Conditions
Measuring cell: DSC20
Pan: Aluminum standard 40 µl, hermetically sealed
Reference: Approximately the same amount of water
Sample preparation: Storage of the eggs in a physiological saline solution at 30 °C.
At the end of the storage time, the egg white was separated and stirred for two minutes.
DSC measurement: Heating from 30 °C to 110 °C at 5 K/min

Interpretation
In addition to the spontaneous transitions due to the influence of heat or pH, slower conformational changes also occur in food and can be measured by DSC if the reaction behavior of the stored products changes. For instance, the storage time (including also the post-mortem phase of meat) can be deduced if required.

The above-mentioned reactions include the protein rearrangement of ovalbumin in egg white to S-ovalbumin (or retrogradation in the case of carbohydrates). Fresh egg white shows an ovalbumin denaturation peak at 86 °C (first day). After a storage time of just two days, two maxima appear at 88 °C and 91 °C, a sign that the rearrangement to S-ovalbumin has started. After five days the main maximum has shifted to 91 °C. After twelve days the entire protein fraction has changed to the S-conformation with a peak maximum at 91 °C. The shift in the peak temperature correlates directly with the storage time.
## Evaluation

<table>
<thead>
<tr>
<th>Date</th>
<th>Sample weight</th>
<th>Sample</th>
<th>Peak 1</th>
<th>Peak 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>June 19</td>
<td>27.38 mg</td>
<td>1st day</td>
<td>70.4 °C</td>
<td>86.2 °C</td>
</tr>
<tr>
<td>June 21</td>
<td>27.38 mg</td>
<td>3rd day</td>
<td>72.1 °C</td>
<td>88.4 °C</td>
</tr>
<tr>
<td>June 24</td>
<td>33.66 mg</td>
<td>6th day</td>
<td>72.9 °C</td>
<td>90.0 °C</td>
</tr>
<tr>
<td>July 02</td>
<td>30.47 mg</td>
<td>14th day</td>
<td>74.0 °C</td>
<td>92.0 °C</td>
</tr>
</tbody>
</table>

## Conclusion

Even though the shift of the peak maximum between the fresh sample and the sample stored for twelve days is only 5 K, the measurement is so reproducible, that within the first ten days of storage the storage time can be readily estimated to the nearest two days. After complete rearrangement of the ovalbumin fraction to S-ovalbumin, it is no longer possible to follow the storage time.
5 Influence of pH on Bovine Hemoglobin

Samples  Bovine whole blood, stabilized with citrate

Conditions  Measuring cell: DSC20
             Pan: Aluminum standard 40 µl, hermetically sealed
             Reference: Approximately the same amount of water
             Sample preparation: Destroying the cells by freezing. The native pH is 7.06, the adjustment has been done with 0.5 mol/l HCl.
             DSC measurement: Heating from 30 °C to 110 °C at 10 K/min

Interpretation  The actual pH value and whether it is kept constant or changes has a large influence on the course of the process and the product quality.

In the present measurement, the main protein component of the red blood corpuscles, hemoglobin, is exposed to different pH values. The globin chains, which are a protein fraction of the hemoglobin molecule, are denatured on heating.

The physiological pH value of the blood is 7.35. In this state, the hemoglobin can not be measured because it immediately coagulates. A value of 18 J/g is assumed to be the native denaturation enthalpy measured with addition of a small amount of citrate at pH 7.06.

When the pH is lowered to a slightly acidic value (pH 5.8), the enthalpy shows no significant decrease, but the peak maximum is shifted to lower temperatures. In other words the pH value influences the thermic stability of the protein.
Heme stabilizes the structure of hemoglobin. In an acidic denaturation, the molecule becomes unstable and hence more sensitive to heat. A significant feature is the lowering of the temperature of the peak maximum. Moreover the peaks become broader. At pH 4.28 the molecule unfolds, but the heme is still attached to the globin chains. Only a very small enthalpy change can be measured and the reaction takes place at much lower temperatures (60°C to 70°C).

In the acidic pH range (pH 3.3), the sample is already fully denatured, i.e. uncoiled as a result of the high hydrogen ion concentration, so that the thermic treatment of the DSC measurement induces no further reaction. Only a straight baseline is recorded.

<table>
<thead>
<tr>
<th>Evaluation</th>
<th>Sample</th>
<th>Sample weight</th>
<th>Peak temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>mg</td>
<td>°C</td>
<td></td>
</tr>
<tr>
<td>7.06</td>
<td>29.51</td>
<td>85.4</td>
<td></td>
</tr>
<tr>
<td>5.80</td>
<td>29.07</td>
<td>81.1</td>
<td></td>
</tr>
<tr>
<td>4.08</td>
<td>29.12</td>
<td>65.9</td>
<td></td>
</tr>
<tr>
<td>3.32</td>
<td>22.28</td>
<td>no peak</td>
<td></td>
</tr>
</tbody>
</table>

**Conclusion**

The stability of the proteins and the effect of the process conditions are readily shown by DSC.
6 DSC of Meat

Samples
Chicken, turkey and veal

Conditions
Measuring cell: DSC12E with cryostat cooling
Pan: Aluminum standard 40 µl, hermetically sealed
Reference: Approximately 70 mg alumina to compensate the heat capacity of the sample.
Sample preparation: Small pieces were cut with a sharp knife
DSC measurement: Heating from 30 °C to 110 °C at 10 K/min

Interpretation
The meat proteins undergo several conformational changes. There are the transitions of myosin and sub-units at 55 °C to 62 °C, of sarcoplasmic proteins and collagens around 67 °C and of actin at 78 °C to 83 °C [1]. The DSC curves are characteristic ‘fingerprints’ of the various muscle proteins.

Conclusion
The DSC curves show the denaturation temperatures. They are the key parameters for processing and serve for identification purposes.

7 Gelatinization of Starch

Samples
Corn starch, rice starch, wheat starch, potato starch

Conditions
Measuring cell: DSC30
Pan: Aluminum standard 40 µl, hermetically sealed
Sample preparation: Preparation of a suspension of starch in water (20 weight %) by stirring, homogenized suspension weighed into the pan.
DSC measurement: Heating from 30 °C to 110 °C at 10 K/min

Interpretation
The relatively high fluctuations in the DSC curve in this measurement can originate from the inhomogeneity of the sample, i.e. various starch clusters react stochastically. However, the peak of the starch swelling is easily recognized.

Evaluation
<table>
<thead>
<tr>
<th>Sample</th>
<th>Sample weight in mg starch</th>
<th>Reaction enthalpy in J/g starch</th>
<th>Peak °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corn</td>
<td>4.83</td>
<td>7.5</td>
<td>69.6</td>
</tr>
<tr>
<td>Rice</td>
<td>5.18</td>
<td>8.8</td>
<td>66.3</td>
</tr>
<tr>
<td>Potato</td>
<td>5.33</td>
<td>11.0</td>
<td>64.4</td>
</tr>
<tr>
<td>Wheat</td>
<td>5.46</td>
<td>8.1</td>
<td>59.2</td>
</tr>
</tbody>
</table>

Conclusion
The DSC curves show the gelatinization temperatures. They are key parameters for processing and serve to identify the different starches.
8 Influence of the Starch Content on Swelling in Water

Sample
Potato starch

Conditions
Measuring cell: DSC30
Pan: Aluminum standard 40 µl, hermetically sealed
Sample preparation: Preparation of the starch suspensions by the addition of varying amounts of distilled water; stirring (Ultra-Turrax for 1 min); homogenized suspension weighed into the pan.
DSC measurement: Heating from 20 °C to 110 °C at 5 K/min

Interpretation
One of the most decisive factors in the swelling of starch is the starch/water ratio. With an excess of water (more than 4 molecules of water per anhydroglucose unit), the endothermic peak is only about 10 K wide, (see also previous example with 20% starch in water). If the starch/water ratio rises above a value of 40%, the reaction range broadens or a second endothermic peak is formed. Whereas the first peak (between 60 °C and 80 °C) appears at constant temperature, a reaction at higher temperatures is evident, which initially leads only to the formation of a shoulder on the first peak and the blurring of the temperature range of the reaction but then, at higher ratios, to the appearance second peak. The area of the first peak decreases, but the total enthalpy remains the same.
Whereas in the limiting case of excess water (40% starch) in the above measurement the endothermic peak lies between 60 °C and 70 °C, the overall reaction at 70% starch extends over a range of 30 K.
At high water contents, the system is destabilized through hydration (water uptake). Increased motion of the polymer chains (heat uptake) and melting of the starch granules begins (first endotherm). In more concentrated starch solutions, this effect is reduced as a result of the limited water content. Direct thermic melting of the starch granules occurs at higher temperatures.

### Evaluation

<table>
<thead>
<tr>
<th>Starch content</th>
<th>Sample weight</th>
<th>Reaction enthalpy</th>
<th>Peak</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>mg starch</td>
<td>J/g starch</td>
<td>°C</td>
</tr>
<tr>
<td>49</td>
<td>26.12</td>
<td>7.0</td>
<td>62.4</td>
</tr>
<tr>
<td>58</td>
<td>26.37</td>
<td>7.4</td>
<td>62.2</td>
</tr>
<tr>
<td>70</td>
<td>22.58</td>
<td>7.8</td>
<td>62.7</td>
</tr>
</tbody>
</table>

### Conclusion

These curves show that the water/starch ratio is important for reproducible results. We recommend weighing starch and water directly into the pan (no mixing required, diffusion adjusts local gradients).
9 ADSC of Amorphous Sugar

Sample  Sugar (Saccharose)

Conditions  Measuring cell: DSC821° with IntraCooler
Pan: Aluminum standard 40 µl, with pierced lid
Sample preparation: The sugar is weighed in the pan and heated in the DSC cell at 5 K/min. Immediately after the endothermic fusion peak the pan is removed and shock-cooled to ambient temperature on an aluminum plate in order to obtain the amorphous phase.
DSC measurement: Heating from 50 °C to 170 °C at a mean heating rate of 2 K/min. Modulation amplitude 1 °C, period one minute. The empty cell and an aluminum sample are measured under the same conditions for calibration purposes.
Atmosphere: Nitrogen, 50 cm³/min

Interpretation  In the upper coordinate system the DSC curve obtained using the alternating temperature program is displayed. It shows a number of changes.
Fast Fourier Analysis allows the recording of the mean signal (= total heat flow signal, corresponding to a classical DSC curve measured at 2 K/min), the amplitude curve (reversing curve) and the so called non-reversing curve.
The changes of the specific heat capacity due to the glass transition, crystallization and other phenomena can be observed in the reversing curve. The reversing curve allows the calculation of the temperature function of the specific heat capacity (lower diagram) again showing the c_p changes but in this case directly in J/gK.
The non-reversing effects include for example chemical reactions, cold crystallization on heating amorphous samples, relaxation effects during the glass transition and the evaporation of volatiles (drying).
There is a broad range of substances that are usually crystalline but, when shock-cooled from the molten phase, form a glassy or amorphous state of aggregation. Examples of such substances are sulfur, most organic compounds, some semicrystalline polymers, certain alloys and quartz. On heating they undergo a glass transition in which they change from a solid and brittle state and become soft and rubbery. Due to the mobility of the molecules, they are then able to form crystals (devitrification). This crystallization process leads to an exothermic peak on the DSC curve (total heat flow and non-reversing curve).

**Evaluation**

The glass transition midpoint of 75.6 °C is typical for anhydrous sugar; moisture would act as a plasticizer and lower the temperature range of glass transition.

The heat of crystallization is obtained by integrating the crystallization peak on the non-reversing curve. The value of 94.6 J/g does not correspond to complete crystallization since the heat of fusion of completely crystalline saccharose is approximately 130 J/g. The corresponding change in $c_p$ is 0.31 J/gK.

**Conclusion**

In food technology the glass transition temperature is becoming increasingly important in connection with stability considerations of sweets, with other amorphous dried foods and with deep-freeze processes.

The most attractive feature of ADSC is its ability to separate $c_p$ changes from certain enthalpy changes as shown in the case of crystallization.
10 TGA of Sugar and Starch

**Sample**  
Sugar (saccharose), corn starch

**Conditions**  
Measuring cell: TGA/SDTA851°  
Pan: Alumina 70 µl, no lid  
Sample preparation: If sugar is heated at a rate of 10 K/min it foams up during decomposition and may leave the pan. The addition of approximately 20 mg dry alumina powder helps to keep the sample in the pan.  
Sample weights: 5.26 mg sugar, 4.99 mg starch  
TGA measurement: Heating from 30 °C to 600 °C at 10 K/min  
Atmosphere: Nitrogen, 50 cm³/min

**Interpretation**  
The flat part of the TGA curve up to 200 °C proves that there is no moisture in the sugar (< 0.1%). The first process that occurs is melting at 190 °C, which is only visible in the SDTA curve. In the liquid phase the carbohydrate loses water and caramelizes. Stoichiometrically, from the formula \( C_n(H_2O)_m \) one expects the formation of 60% water and 40% carbon black. But, there is no distinct dehydration step because of concurrent other reactions.

**Evaluation**  
The DTG minimum is normally used as the evaluation limit to separate overlapping steps. The dehydration step of 67.3% is close to the above mentioned value of 60%. The carbon black formed burns exothermically up to 540 °C. The shape of the SDTA curve is called the ‘burning profile’ and gives an indication of the reactivity of the carbon black. The residue of 0.40% at 590 °C is the mineral ash content.
**Interpretation**

Starch contains several percent moisture depending on the relative humidity of the surrounding air. The moisture is eliminated up to 200°C. Stoichiometrically from the formula \( C_n(H_2O)_n \) one expects 60% water and 40% carbon black. Again, there is no distinct dehydration step because of concurrent other reactions.

**Evaluation**

The DTG minimum is used as the evaluation limit to separate overlapping steps. There is 9.2% of moisture detected. The next step of 67.9% is higher than the value expected of 60%. The carbon black formed burns up to 540°C. The residue of 0.19% at 590°C corresponds to the mineral ash content.

**Conclusion**

TGA/SDTA allows the determination of the moisture content, the content of active ingredients and the ash content. In addition, the melting point and the TGA inflection temperatures are used to identify the different carbohydrates.