

# **FOOD** CHARACTERIZATION

## THERMAL ANALYSIS , CALORIMETRY & GAS SORPTION SOLUTIONS

• OILS, FATS, LIPIDS • • CARBOHYDRATES• • PROTEINS • • FOOD PROCESSING •



#### **YOUR CHALLENGES**

Food primarily consists of carbohydrates, lipids, proteins, and water, alongside trace amounts of minerals and diverse organic compounds. The composition and arrangement of these elements significantly influence various attributes of food, ranging from its texture to its shelf life.

Furthermore, factors such as the source, origin, and processing methods of these constituents impact their structure. Given that food undergoes processes like heating, cooling, or pressure application during preparation, employing techniques like thermal analysis and calorimetry becomes essential for characterizing them effectively.

#### **COMMON STUDIES & SOLUTIONS FOR FOOD PRODUCTS**

This brochure presents some of our solutions in this field and we encourage you to contact us for more information.

Lipids

provide food

products with specific

texture, flavor release, or ageing

resistance. But an uncontrolled

change of lipid structure may lead

to an undesired change of product

quality. DSC is used to detect and

understand these structure changes in lipids.

Oils, Fats, Lipids

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Heating of carbohydrates like sugars, starches or gel forming substances means triggering reactions. For instance: water release, melting, decomposition, gelatinization, retrogradation or gel formation. Our TGA and DSC solutions provide data to study and maintain control over these reactions.

Carbohydrates

In the food industry, it is important to test proteins with DSC for their temperature of unfolding, aggregation and of glass transition. Indeed, when transforming, food products may lose their properties and change structure.

Proteins

You may use many food processing methods with thermal or pressure treatments. With DSC, calorimetry and microcalorimetry you can measure interesting data for process engineering. It includes heat capacity, heat of freezing, or the influence of pressure on ingredient properties.

Food processing

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"DSC measurements helped to choose two different heat treatments of whey protein isolate (WPI) dispersion in comparison with native WPI."

**E.** Ruffin et al. / Food Chemistry 151 (2014) 324–332. http://dx.doi.org/10.1016/j.foodchem.2013.11.071

*"Microcalorimetry showed that the ternary model systems successfully explained the fat melting endotherms [...]".* 

N. Hesso et al. / Food Hydrocolloids 51 (2015) 7-15 http://dx.doi.org/10.1016/j.foodhyd.2015.04.013

"DSC was shown to be an invaluable tool for elucidating differences between the thermal behaviour of sodium stearoyllactylate in aqueous dispersion with that in O/W emulsion [...]."

D. Kurukji et al, Journal of Colloid and Interface Science 409 (2013) 88–97 http://dx.doi.org/10.1016/j.foodhyd.2015.04.013

#### THE KEP TECHNOLOGIES ADVANTAGE

KEP Technologies is addressing it's offerings to the food market by making available the widest and most versatile choice of solutions. Now you can consult with one company, KEP Technologies, to address your challenges across the broadest number of food studies on the market.

**EXPERIMENTAL** Each solution embodies our "Reimagine CONTROL Material Characterization" value proposition by delivering the three core customer benefits of Experimental Control, **Instrument Versatility and Quality Results.** We believe solutions that provide these benefits will deliver the highest value to our customers. In addition to our core customer benefits, we are able to provide **customized** OUALITY INSTRUMENT solutions by harnessing the engineering RESULTS VERSATILITY and project management of our highly skilled organization.

#### CUSTOMIZED SOLUTIONS

Modular design allows for upgraded and tailored functionality Access to all previous non-proprietary custom requests Open access to our engineering development team

### **OILS, FATS, LIPIDS**



dfor intuitive and easy data handling

#### SPECIFICATIONS

	SETLINE <sup>®</sup> DSC	SETLINE <sup>®</sup> DSC+	
Temperature range (°C)	-170 to 700	-170** to 700	
Programmable heating rate (°C/min)	0.01 to 100	0.01 to 100	
Cooling time	12 min from 500°C to 100°C (air)       12 min from 500°C to 1         12 min from 25°C to -100°C (LN2)       12 min from 25°C to -1         5 min from 100°C to 0°C       5 min from 100°C to 0°C         (cryothermostat)       (cryothermostat)		
Enthalpy accuracy / precision *** (%)	+/- 0.8 / 2.5		
Temperature accuracy / precision *** (°C)	+/- 0.30 / 0.50		
DSC measurement range (mW)	+/- 6 000		
Atmosphere	Inert gas, air (possible gas switch between 2 gases)		
Gas flow range (ml/min)	10 to 100		
Autosampler	-	59 positions (samples or references)	

\*Lower temperatures can be achieved. The time to reach these minimum temperatures can be over two hours; \*\*When subambient cooling devices are used, the autosampler cannot operate; \*\*\*Based on indium melting tests

#### Determination of the Solid Fat Index (SFI) of Chocolate by DSC

#### **INTRODUCTION**

The melting curve of a fat is generally complex: for a given fat, there is not a melting point, but more a melting range. In processing fat, it is also interesting to know, for a given temperature, what is the amount of fat melted. The DSC technique is now widely used to determine solid-liquid ratios in fats, called the Solid Fat Index (SFI). This method is based on measuring the heat of fusion successively at different temperatures. By reference to the total melting heat, the fraction offat melted is determined. This technique is faster than dilatometry, and gives results comparable with NMR. DSC gives the possibility of tempering the fat at different temperatures prior to index determining.



#### **EXPERIMENT**

#### Sample:

Chocolate, 70% cocoa

#### **Experimental conditions:**

• Atmosphere: Nitrogen, atmospheric pressure

• Sample mass: About 25 mg in a Aluminum 100µL crucible closed with a standard lid.

#### **Experimental procedure:**

The temperature is programmed from -15°C up to 50°C at 5°C/min<sup>-1</sup>.

#### **RESULTS AND CONCLUSION**

During the heating, an large endotherm corresponding to the melting of the chocolate is observed. The amount of chocolate that is already melted at a given temperature is equal to the ratio of the partial heat of melting at this temperature and the total heat of melting.

By difference, the percentage of solid phase or Solid Fat Index (SFI), can be obtained. Calisto Data Processing enables the automatic calculation of the SFI curve versus temperature of the chocolate sample. For a given temperature, this curve enables the determination of the melted amount or of the amount of fat remaining solid. At room temperature (20°C) the tested chocolate is 50% melted.

Then, the chocolate is soft but solid enough to be easily eatable.

### **OILS, FATS, LIPIDS**



### **SPECIFICATIONS**

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Temperature range (°C)	-170 to 700	-170** to 700	
Programmable heating rate (°C/min)	0.01 to 100	0.01 to 100	
Cooling time	12 min from 500°C to 100°C (air) 12 min from 25°C to -100°C (LN2) 5 min from 100°C to 0°C (cryothermostat)	12 min from 500°C to 100°C (air) 12 min from 25°C to -100°C (LN <sub>2</sub> ) 5 min from 100°C to 0°C (cryothermostat)	
Enthalpy accuracy / precision *** (%)	+/- 0.8 / 2.5		
Temperature accuracy / precision *** (°C)	+/- 0.30 / 0.50		
DSC measurement range (mW)	+/- 6 000		
Atmosphere	Inert gas, air (possible gas switch between 2 gases)		
Gas flow range (ml/min)	10 to 100		
Autosampler	-	59 positions (samples or references)	

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REIMAGINE MATERIAL CHARACTERIZATION

### Melting of different types of chocolates by DSC

#### **INTRODUCTION**

Chocolate is mainly composed of cocoa butter, cocoa paste and sugars. Moreover, the composition could differ from one type of chocolate to another. The dark chocolate contains solid cocoa, cocoa butter and sugar. In the milk chocolate, powder milk is added. In the white chocolate, there is no solid cocoa.

Then, the thermal profile is an essential information for the industry to improve the quality and properties of their products, like for example the ability to be solid at room temperature and soft in the mouth.



#### **EXPERIMENT**

#### Samples:

- Dark chocolate 70% of cocoa
- Milk chocolate
- White chocolate

#### **Experimental conditions:**

Instrument : SETLINE DSC Atmosphere: air, atmospheric pressure Sample mass: about 25 mg in a 100µl sealed aluminum crucible **Experimental procedure:** Heating from -50°C up to 45°C at 5°C/min.

#### **RESULTS AND CONCLUSION**

For the dark chocolate, an endothermic effect is recorded with a maximum at about 20°C, corresponding to the melting of the cocoa butter that contains different fatty acids.

The white type presents two maxima attributed to the two main compounds : cocoa butter and milk fats. From the melting peak area, it is noticed that the white chocolate contains less cocoa butter.

For the milk chocolate the observation of the melting peak area shows that it contains less cocoa butter.

### **OILS, FATS, LIPIDS**



manometry, BET instrumentation, gas analyzers, humidity controllers and gas panels

#### **SPECIFICATIONS**

TEMPERATURE	MICROCALVET	
Temperature range (°C)	-45 to 120 Cooling under 0°C requires the use of an auxiliary thermostat	
Temperature accuracy (°C)	+/- 0.07*	
Temperature precision (°C)	+/- 0.15*	
Programmable temperature scanning rate (°C/min)	0.001 to 2	
HEAT & HEAT FLOW		
Enthalpy accuracy (%)	+/- 0.4*	
Calorimetric precision (%)	+/- 0.7*	
GENERAL		
Cells volume (ml)	Up to 1 (standard cell)	
Pressure measured and controlled (bar [psi])	400 [5,800]; 1000 [14,600 ]	

#### REIMAGINE MATERIAL CHARACTERIZATION

### Characterization of Palm Oils at different temperature scanning rates by MICROCALVET

#### INTRODUCTION

Palm oil is mainly constituted of esters of glycerol and fatty acids named glycerides. The composition of glycerides in oil is an important characteristic to control its quality. This composition can be determined following the specific phase transitions between polymorphic forms and solid-liquid phases of the constituents. It is shown that MICROCALVET is an equipment perfectly designed to characterize palm oil.

#### **EXPERIMENT**

#### Samples:

Refined Palm Oil (PO) Refined Palm Kernel Oil (PKO) **MICROCALVET experimental conditions:** Atmosphere: air, atmospheric pressure Sample mass: about 500 mg in a standard cell **Experimental procedure:** 

- +80 to -40°C at different temperature scanning rates
- Isotherm at -40°C during 30 minutes
- -40 to +80°C at the same scanning rate



Palm tree fruits constituted by the flesh (1) and the kernel (2) from which are extracted PO and PKO



#### Figure 1 : Cooling thermograms of PO

Figure 2 : Heating thermograms of PO

PO is constituted of different types of glycerides (saturated and unsaturated) which may have different crystal forms (polymorphism). That is the reason why on the corresponding thermograms, several peaks are observed. Figures 1 and 2 present the cooling and heating curves obtained using MICROCALVET at different temperature scanning rates. When the rate decreases, the resolution of peaks increases and allows for a better separation of the different glycerides. Despite the very slow rates (down to 0.05°C/min), the heat flow remains very easily measurable and allows for a better identification of the different fractions.

#### **RESULTS AND CONCLUSION**

### **OILS, FATS, LIPIDS**



manometry, BET instrumentation, gas analyzers, humidity controllers and gas panels

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HEAT & HEAT FLOW		
Enthalpy accuracy (%)	+/- 0.4*	
Calorimetric precision (%)	+/- 0.7*	
GENERAL		
Cells volume (ml)	Up to 1 (standard cell)	
Pressure measured and controlled (bar [psi])	400 [5,800]; 1000 [14,600 ]	

#### REIMAGINE MATERIAL CHARACTERIZATION

### Characterization of Palm Oils at different temperature scanning rates by MICROCALVET

#### **RESULTS AND CONCLUSION**



Figure 1 : Cooling thermograms of PO

Figure 2 : Heating thermograms of PO

On the contrary, PKO thermograms have very few peaks even when applying a slow rate. One main peak is observable since PKO is particularly concentrated in saturated triglycerides. (Figures 3 and 4).

As the melting and cooling are very complex to analyze due to the several peaks, the Calisto software with its deconvolution function is used to extract from the thermogram the different peaks associated to each glycerides and to enable a better identification of the spectrum (Figures 5 and 6).







Figure 6 : Deconvolution of PO heating thermogram at 0.05°C/min

The MICROCALVET with its large sample volume combined with a high sensitivity, is ideal to characterize very accurately the different transitions in palm oil and access to the glycerides' composition and polymorphic forms of their crystals. Associated with the Calisto software a separation of the different peaks is made available for a betteridentification of the composition.

### CARBOHYDRATES



### **SPECIFICATIONS**

	SETLINE <sup>®</sup> DSC	SETLINE <sup>®</sup> DSC+	
Temperature range (°C)	-170 to 700	-170** to 700	
Programmable heating rate (°C/min)	0.01 to 100	0.01 to 100	
Cooling time	12 min from 500°C to 100°C (air)       12 min from 500°C to 1         12 min from 25°C to -100°C (LN2)       12 min from 25°C to -10         5 min from 100°C to 0°C       5 min from 100°C to 0°C         (cryothermostat)       (cryothermostat)		
Enthalpy accuracy / precision *** (%)	+/- 0.8 / 2.5		
Temperature accuracy / precision *** (°C)	+/- 0.30 / 0.50		
DSC measurement range (mW)	+/- 6 000		
Atmosphere	Inert gas, air (possible gas switch between 2 gases)		
Gas flow range (ml/min)	10 to 100		
Autosampler	-	59 positions (samples or references)	

\*Lower temperatures can be achieved. The time to reach these minimum temperatures can be over two hours; \*\*When subambient cooling devices are used, the autosampler cannot operate; \*\*\*Based on indium melting tests

#### Melting of different sugars by DSC with SETLINE DSC

#### **INTRODUCTION**

Different types of sugars are present in food products (e.g. sucrose, fructose, maltose, saccharose). Many are used in the food industry, as powders or liquids. During food processing, they are heated, cooled, quenched according to different heat treatments. The DSC test is useful in this case to characterize the thermal behavior of the sugar when heated or cooled, to know if the sugar is in a crystalline or amorphous form according to the preparation. In the example the melting of four different sugars and a sugar substitute (Sorbitol) were investigated.



#### **EXPERIMENT**

#### Sample:

Analytical grade samples of Sorbitol, Maltose, Mannose, Glucose, Saccharose

#### Experimental conditions:

- Atmosphere: nitrogen, atmospheric pressure
- Sample mass: about 5 mg in a 100µl aluminum crucible

#### **Experimental procedure:**

Heating at 5°C/min from room temperature until the end of melting for each sample

	SORBITOL	MALTOSE	MANNOSE	GLUCOSE	SACCHAROSE
Melting temperature (°C)	99.0	121.2	130.9	151.8	182.2
Melting heat (J/g)	195.67	181.35	158.93	190.66	116.39

#### **RESULTS AND CONCLUSION**

Sorbitol has the lowest melting point and saccharose the highest.

### CARBOHYDRATES

#### **INSTRUMENT**

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### MICROCALVET

#### -45°C to 120°C

#### HIGHEST HEAT MEASUREMENT ACCURACY

Calvet 3D sensor based on Peltier elements with Joule effect calibration.

#### **MODIFIABLE TEMPERATURE CONDITIONS** for increased flexibility and replication of real life conditions.

### CONVENIENT INTERCHANGEABLE CRUCIBLES AND CELLS

to perform even the most demanding experiments using one instrument :

- high pressure (1000bar) and high vacuum
- pressure measurement and control
- mixing experimentin one instrument

#### **EXTERNAL COUPLING CAPABILITY**

designed to increase your research options including manometry, BET instrumentation, gas analyzers, humidity controllers and gas panels

#### **SPECIFICATIONS**

TEMPERATURE	MICROCALVET	
Temperature range (°C)	-45 to 120 Cooling under 0°C requires the use of an auxiliary thermostat	
Temperature accuracy (°C)	+/- 0.07*	
Temperature precision (°C)	+/- 0.15*	
Programmable temperature scanning rate (°C/min)	0.001 to 2	
HEAT & HEAT FLOW		
Enthalpy accuracy (%)	+/- 0.4*	
Calorimetric precision (%)	+/- 0.7*	
GENERAL		
Cells volume (ml)	Up to 1 (standard cell)	
Pressure measured and controlled (bar [psi])	400 [5,800]; 1000 [14,600 ]	

#### Gelatinization of different starches in water by MICROCALVET

#### **INTRODUCTION**

Starch is a base product in food materials. It is especially true for rapid cooking, where it is used for its gelatinization properties. When starch grains are placed in contact with water and heated, an effect of gelatinization occurs at a well-defined temperature. This temperature depends on starche's botanic origin. In the present note, 4 starches (rice, corn, potato, wheat) are compared.



#### **EXPERIMENT**

Samples: Mixtures of different starches in water (25% starch by weight) :

- Atmosphere: air , atmospheric pressure
- Samples masses: about 325 mg of mixture in standard cells
- Heating profile
- 23°C to 90°C at 0.5 K/min
- Instrument: MICROCALVET

#### **RESULTS AND CONCLUSION**

An endothermic effect, corresponding to the formation of the gel network occurs around  $60^{\circ}$ C. The different samples have a range in the peak of the gelatinization effect of about  $10^{\circ}$ C. Wheat starch has the lowest temperature of gelatinization (59.4°C) and rice starch has the highest (71.1°C).

Rice and corn starches have similar heats of gelatinization. The maximum heat that is observed, corresponds to potato starch (3.31 J/g of mixture).

### CARBOHYDRATES

#### **INSTRUMENT**

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### **MICROCALVET ULTRA**

#### -20°C to 170°C

#### HIGHEST HEAT MEASUREMENT ACCURACY

3D sensor based on Peltier elements with Joule effect calibration.

**MODIFIABLE TEMPERATURE CONDITIONS** for increased flexibility and replication of real life conditions.

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#### **SPECIFICATIONS**

TEMPERATURE	MICROCALVET ULTRA	
Temperature range (°C)	-20 to 170	
Temperature accuracy (°C)	+/- 0.07*	
Temperature precision (°C)	+/- 0.15*	
Programmable temperature scanning rate (°C/min)	0.001 to 2	
HEAT & HEAT FLOW		
Enthalpy accuracy (%)	+/- 0.5*	
Calorimetric precision (%)	+/- 0.8*	
GENERAL		
Cells volume (ml)	Up to 1 (standard cell)	
Pressure measured and controlled (bar [psi])	400 [5,800]	

#### **APPLICATION**

#### Starch retrogradation and bread staling

#### **INTRODUCTION**

Gelatinized starch goes through retrogradation, which involves recrystallization of amylose and amylopectin. Retrogradation characteristics depend on parameters like botanic specie or water content. It is the main source of bread staling effect.

The ageing of maize, of wheat starch dough and of bread crumb, was studied with the MICROCALVET ULTRA 4C microcalorimeter.



Figure 1 – Thermogram of the analysis of wheat starch dough at t = 0



Figure 3 – Thermogram recorded during the analysis of bread crumb



with wheat starch storage time.

#### **EXPERIMENT**

• Dough are composed of maize and wheat starch (Merck) with deionized water (34% w/w), heated at 110°C for 1 hour. Bread crumb is freshly baked, wheat flour based.

• Test procedure: dough and bread were stored at ambient temperature in closed containers and sampled every day to be heated in a calorimetric vessel. Mass~ 400 mg, heating 10°C->180°C at 0.7 K/min

#### **RESULTS AND CONCLUSION**

Starches: Each starch exhibit three main thermal events (Figure 1):

- Melting of the recrystallized amylopectin (endotherm between 30 and 80°C). Heat of melting increases with the ageing of the dough and stabilizes with time (Figure 2)

- Melting of amylose-lipid complexes (endotherm at around 120°C). Heat of melting increases with the ageing of the dough and stabilizes with time .

- Melting of the recrystallized amylose (endotherm at around 160°C).

Bread crumb: Heat of melting of recrystallized amylopectin increases with ageing on the bread crumb thermogram (Figure 4). At higher temperatures a large exothermic effect occurs and could be linked with Maillard reaction occuring between the protein and polysaccharide contents of the crumb (Figure 3).

Starch retrogradation and to some extend bread staling can be monitored by microcalorimetric systems that allow a combination of a large sample mass, a high sensitivity and slow scanning rates that keep the tested samples close to thermal equilibrium.

### CARBOHYDRATES

#### **INSTRUMENT**

### MICROCALVET

#### -45°C to 120°C

#### HIGHEST HEAT MEASUREMENT ACCURACY

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Programmable temperature scanning rate (°C/min)	0.001 to 2	
HEAT & HEAT FLOW		
Enthalpy accuracy (%)	+/- 0.4*	
Calorimetric precision (%)	+/- 0.7*	
GENERAL		
Cells volume (ml)	Up to 1 (standard cell)	
Pressure measured and controlled (bar [psi])	400 [5,800]; 1000 [14,600 ]	

#### Melting and gelation of kappa-Carrageenan by MICROCALVET

#### **INTRODUCTION**

Carrageenans are extracted from red algae and used in food processing for their gelation properties. This polysaccharide shows an order-disorder transition when heat-treated.

The kappa-carrageenan, obtained under aggregating conditions in the presence of K+ ion, shows a single molecular process when heated and cooled. The kappa-carrageenan forms rigid, thermally reversible, high strength gels e.g. dessert gels, petfood gels, air freshener gels.

The microcalorimetry technique is well adapted to investigate the formation and the melting of such gels.



#### **EXPERIMENT**

k-carrageenan (0.5 %) in KCl 0.1 M solution pH 5.

#### **MICROCALVET** experimental conditions:

Sample mass: about 620 mg Cells : Standard cells **Experimental procedure:** Heating from 20°C up to 80°C at 0.5°C/min.

#### **RESULTS AND CONCLUSION**

During the heating phase, an endothermic effect corresponding to the melting of the carrageenan is observed with a maximum at 68.8°C.

When cooling, an exothermic effect is detected at 50.2°C, with a significant hysteresis, corresponding to the gelation process.

### PROTEINS

#### **INSTRUMENT**

### **MICROCALVET ULTRA** -20°C to 170°C HIGHEST HEAT MEASUREMENT ACCURACY 3D sensor based on Peltier elements with Joule 👸 setaram effect calibration. **MODIFIABLE TEMPERATURE CONDITIONS** for increased flexibility and replication of real Vetuites life conditions. **CONVENIENT INTERCHANGEABLE CRUCIBLES AND CELLS** to perform even the most demanding experiments using one instrument : • high pressure (1000bar) and high vacuum • pressure measurement and control • mixing experimentin one instrument

#### EXTERNAL COUPLING CAPABILITY

designed to increase your research options including manometry, BET instrumentation, gas analyzers, humidity controllers and gas panels

#### **SPECIFICATIONS**

TEMPERATURE	MICROCALVET ULTRA	
Temperature range (°C)	-20 to 170	
Temperature accuracy (°C)	+/- 0.07*	
Temperature precision (°C)	+/- 0.15*	
Programmable temperature scanning rate (°C/min)	0.001 to 2	
HEAT & HEAT FLOW		
Enthalpy accuracy (%)	+/- 0.5*	
Calorimetric precision (%)	+/- 0.8*	
GENERAL		
Cells volume (ml)	Up to 1 (standard cell)	
Pressure measured and controlled (bar [psi])	400 [5,800]	

#### **APPLICATION**

#### **Denaturation of Albumin by Microcalorimetry**

#### **INTRODUCTION**

Albumins are major proteins which are found in many food products such as egg white, milk or meat. These proteins are able to coagulate under the influence of the temperature. This thermal property is commonly used in food processes, such as in sugar refining to clarify the solutions or as emulsifying and gelling agents.



#### **EXPERIMENT**

- Sample: Albumin from egg
- Sample mass: 550 mg of 10% albumin in a 0.1M NaCl solution pH 5.
- Reference: 550 mg of 0.1M NaCl solution
- Type of cell: Batch cell

Experimental procedure:

The temperature was programmed from 20 °C up to 95 °C at 1 °C/min.

#### **RESULTS AND CONCLUSION**

The denaturation of albumin corresponds to an endothermic event. It occurs at 68.1°C with a heat of 0.73 J/g of solution or 7.3 J/g of albumin.

The denaturation temperature (corresponding to the peak maximum) is measured at 76.4°C.

At the beginning of the denaturation, an overlaying exothermic event is detected. It is attributed to the partial aggregation of the protein.

### **FOOD PROCESSING**



#### **SPECIFICATIONS**

TEMPERATURE	CALVET PRO	
Tomporaturo rango (°C)	Ambient to 830°C	
lemperature range ( C)	-120 to 200 °C (with cooling accessory)	
Temperature accuracy (°C)	+/- 0.05*	
Temperature precision (°C)	+/- 0.15*	
Programmable temperature	0.01 to 30	
scanning rate (°C/min)	0.01 to 50	
HEAT & HEAT FLOW		
Enthalpy accuracy (%)	+/- 0.8*	
Calorimetric precision (%)	+/- 0.4*	
MASS VARIATION***		
Weighing accuracy (%)	+/- 0.1**	
Weighing precision (%)	+/- 0.05**	
Weight Range (mg)	+/- 200	
GENERAL		
Crucible or cells volume (ml)	Up to 0.32 depending on the chosen design and material (aluminium, incoloy, graphite, alumina, platinum, etc)	
Pressure (bar [psi])	400 [5,800] (measured and controlled); 500 [7,250] (resistant)	

\* Based on indium melting tests \*\* Based on CuSO4. 5H2O dehydration \*\*\* With TG option

REIMAGINE MATERIAL CHARACTERIZATION

#### Freezing of an extract of liquid coffee

#### **INTRODUCTION**

For the frozen products, the DSC method can provide four different characteristics : the temperature at the beginning of freezing (especially for the solutions), the amount of ice vs temperature, the temperature at the beginning of melting, the detection of recrystallization. The temperature at the beginning of freezing is especially interesting for industrial operations. Moreover, it defines the temperature range for freeze drying operations.

In the present example, the temperature of freezing of an extract of liquid coffee is measured.



#### **EXPERIMENT**

Sample : Extract of liquid coffee Mass : 150.6 mg Crucible : Stainless steel Cooling : 2 K/min The sample was cooled from room temperature down to – 50°C.

#### **RESULTS AND CONCLUSION**

Freezing starts at around – 23°C. The integration of the peak provides the heat of freezing (66.88 J/g). It allows the amount of ice to be determined by comparison with the heat of freezing of water.

Some supercooling effects can appear when cooling the sample. Before starting the freeze-drying of a new product, determining the temperature at the beginning of melting is of interest for knowing the temperature range over which the water is fully frozen.

### **OILS, FATS, LIPIDS**



#### **SPECIFICATIONS**

	SETLINE <sup>®</sup> DSC	SETLINE <sup>®</sup> DSC+	
Temperature range (°C)	-170 to 700	-170** to 700	
Programmable heating rate (°C/min)	0.01 to 100	0.01 to 100	
Cooling time	12 min from 500°C to 100°C (air)       12 min from 500°C to 10         12 min from 25°C to -100°C (LN2)       12 min from 25°C to -10         5 min from 100°C to 0°C       5 min from 100°C to 0°C         (cryothermostat)       (cryothermostat)		
Enthalpy accuracy / precision *** (%)	+/- 0.8 / 2.5		
Temperature accuracy / precision *** (°C)	+/- 0.30 / 0.50		
DSC measurement range (mW)	+/- 6 000		
Atmosphere	Inert gas, air (possible gas switch between 2 gases)		
Gas flow range (ml/min)	10 to 100		
Autosampler	-	59 positions (samples or references)	

\*Lower temperatures can be achieved. The time to reach these minimum temperatures can be over two hours; \*\*When subambient cooling devices are used, the autosampler cannot operate; \*\*\*Based on indium melting tests

#### Palm Oil refining process studied by DSC

**Atmosphere:** nitrogen N<sub>2</sub>, atmospheric pressure

**Sample mass:** about 20 mg in a 100 µl aluminum

• +60 -60°C at different temperature scanning rates

#### **INTRODUCTION**

**EXPERIMENT** 

**Experimental procedure:** 

isotherm at -60°C during 30 min
-60 +60°C at the same rate

crucible

Classically, crude palm oil has to undergo processes to improve its qualities before being proposed on the market. In a first step, an absorption bleaching is performed to produce refined palm oil. This latter is constituted by a liquid phase and a solid phase which could be separated by a fractionation process.

This process is based on the crystallization temperature of each phase, that is why, the thermal profile of palm oil should be clearly established.

#### Cooling Cooling Cooling Cooling Science Cooling Coo



#### **RESULTS AND CONCLUSION**

On the PO thermogram (figure 1), the two main peaks represent the low and high melting points fractions. These fractions are Olein (peak 1) (liquid at room temperature) and Stearin (peak 2) (solid at room temperature). The temperature range between these two peaks is an essential information to control palm oil refining processes. The PKO thermograms (figure 2) present only one peak even when using a slow temperature ramp.



Figure 2 : Cooling thermograms of PKO

This can be explained because it is particularly concentrated in saturated triglycerides.

It shows that SETLINE DSC is an equipment perfectly adapted to characterize palm oil in terms of crystallizationand quality control.



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