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# Development of a Test Standard for PCM and TCM Characterization

## Part 1: Characterization of Phase Change Materials

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Report A2.1 part 1, of the working group A2 Test and Characterization

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## Compact Thermal Energy Storage: Material Development for System Integration



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## Short description Working group A2 Test and Characterization

### INTRODUCTION

As the knowledge of the behavior of heat storage materials is very essential to design an application it is important to characterize these materials. The important characteristics of heat storage materials are their heat capacity, the thermal conductivity, the density and viscosity all in dependency on temperature. All these parameters are necessary to size a thermal storage or to develop heat exchanger to charge and discharge such storages. Simulations are also very often used to analyze applications or components of it and their interaction with the storage material. Such simulations will not be valid if the used material data is not describing its behavior in a correct way, so also for this purpose good and reliable results from the characterization are needed.

Talking about PCMs means to have a very strong relationship between temperature and the mentioned characteristics. In principle it seems to be very easy to characterize materials since there is equipment available to determine all the needed parameter, but unfortunately the way in which the materials will be measured influences the results. Figure 1 gives an example of a DSC measurement using different heating rates, as well the first measurement using 0.5 K/min and the second one. Every rate is leading to a different shape of the curve. The question is: Which is the correct measurement? This is just one example but in principal all parameters which can be determined for a PCM show in some way a more or less dependency on the used measurement procedure and there is always the question which is the right one.

So there are already differences caused by the measurement method itself but we get even bigger differences if we use for example DSC from different manufacturers and/or located at different laboratories.

The activities of working group A2 aims to develop measurement procedures for thermal storage materials. The goal is to introduce standard procedures to make it possible to compare measurement results obtained using different measurement equipment at different laboratories. Therefore the following targets for the working-group A2 "Test and characterization" were defined at the beginning of the Task 42 / Annex 24:

- To determine the differences in characterization of heat storage materials for heat capacity and melting behavior, viscosity, thermal conductivity, density and cycle stability, water uptake (TCM).
- To develop measurement standards to obtain the "real" materials characteristic.
- To develop a standards that lead to the "same" results also when they are determined at different laboratories.

The following deliverables are set during the 2<sup>nd</sup> meeting in Lleida, Spain:

<i>Nr.</i>	<i>Deliverable</i>	<i>Month</i>
A2.1	List of existing standards	15
A2.2	Definition of draft standard for RRT	18
A2.3	RRT results and final standard	36
A2.4	Database	42

This final report includes all listed deliverables excluding the database, which is already set up but not online.

The work in the group started with a comparison of DSC-results. The most common equipment used to determine the most important parameter namely the melting and solidification enthalpy of PCMs as well as its melting and crystallization behavior. The work was started using the same PCM which was sent to different laboratories and it was expected to get not the same results but to get comparable results. But the results showed really big differences and so it was realized that it will not be possible to establish a measurement procedure for all above mentioned parameters. In the case of the PCMs the working group is still focused on the DSC measurement and just started on rheometry in September 2011.

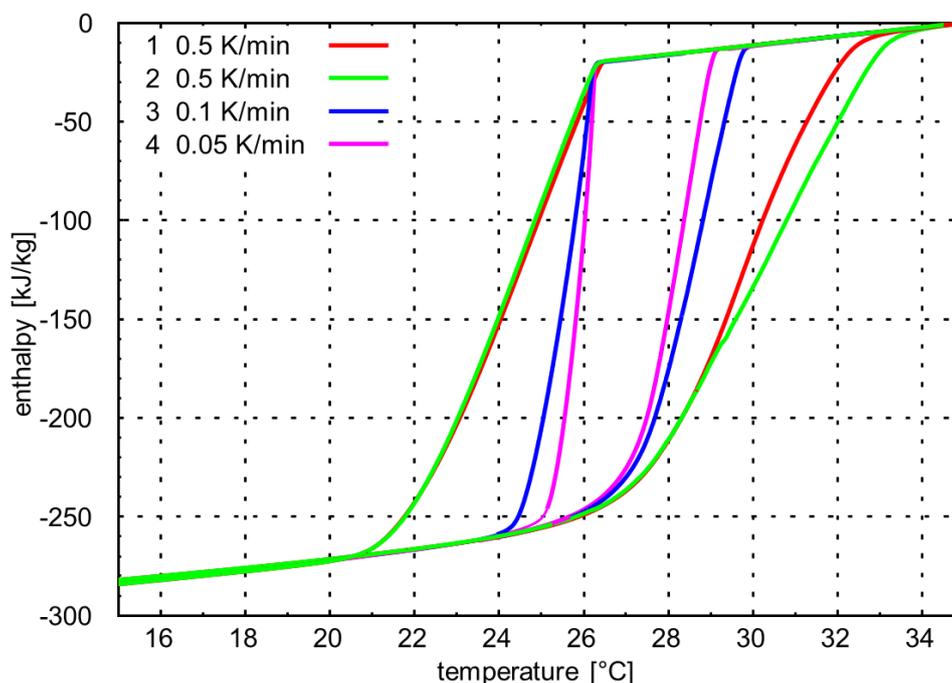


Figure 1 : Example of a DSC measurement using different heating rates.

## 1 EXISTING STANDARDS FOR MATERIAL CHARACTERIZATION

The list below summarizes the standards defined to measured parameters of materials that are also important when characterizing PCMs. Almost all of these standards are defined for materials that primarily will not be used as heat storage material. As it can be seen further down in this report, the characterizations of PCMs especially the determination of melting and crystallization behavior requires standards that are really applied to the phase transition of these materials. So these standards which were

developed to characterize materials for another kind of usage might not be appropriate to get the needed knowledge about PCM necessary to apply them into storage applications. The only standard existing so far for PCM was established in Germany. It is a standard which was defined in the frame of the RAL with the target to ensure the quality of PCM-Products. "RAL is an independent institution that is responsible for the creation of RAL Quality Marks (Gütezeichen). The RAL Quality Assurance Associations are responsible for awarding the Quality Marks (Gütezeichen) to companies. These Quality Assurance Associations recognised by RAL represent particularly quality-conscious service providers and manufacturers. Only those companies who voluntarily submit themselves to the strict RAL Quality Assurance and Test Specifications receive the right to carry the RAL Quality Mark (Gütezeichen). Compliance with these regulations is ensured through regular self-monitoring and by the regular monitoring carried out by independent bodies." Taken out of the RAL web-page [1]. To get the RAL-Gütezeichen for a PCM means that the PCM-Product has to fulfill several conditions which are defined within the quality criteria [4]. These criteria describe also what has to be measured and how it has to be measured as well as the limits that has to be reached to get the RAL-Gütezeichen for a PCM-product. The standard procedures to measure PCM defined there can be taken as a method to apply a measurement standard for PCMs within the IEA Task 42 and Annex 24.

The base for the inquiry, in addition to the RAL standard, were web search engines [1][2].

## 1.1 Melting points

### 1.1.1 ASTM D87-07a

#### **Standard Test Method for Melting Point of Petroleum Wax (Cooling Curve)**

1.1 This test method covers the determination of the melting point (cooling curve) of petroleum wax. It is unsuitable for waxes of the petrolatum group, microcrystalline waxes, or blends of such waxes with paraffin wax or scale wax. Note 1: For additional methods used for testing petroleum waxes, see Test Method D 127 and Test Method D 938. Results may differ, depending on the method used. For pharmaceutical petrolatum, Test Method D 127 usually is used.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

### 1.1.2 ISO 2207:1980

#### **Petroleum waxes -- Determination of congealing point**

The procedure specified allows the determination of the temperature at which a sample being cooled develops a set or resistance to flow. At that temperature the product may be at or close to the solid phase. In the case of petrolatums, congealing is associated with the formation of a gel structure as the sample cools.

### 1.1.3 ASTM D4419-90(2005)

#### **Standard Test Method for Measurement of Transition Temperatures of Petroleum Waxes by Differential Scanning Calorimetry (DSC)**

Scope:

1.1 This test method covers the transition temperatures of petroleum waxes, including microcrystalline waxes, by differential scanning calorimetry (DSC). These transitions may occur as a solid-solid transition or as a solid-liquid transition.

1.2 The normal operating temperature range extends from 15C to 150C (Note 1).

1.3 The values stated in SI units are to be regarded as the standard.

### **1.1.4 Heat capacity**

#### **1.1.5 ISO 11357-4:2005**

Plastics -- Differential scanning calorimetry (DSC) -- Part 4: Determination of specific heat capacity

#### **1.1.6 ISO 11357-4:2005**

specifies methods for determining the specific heat capacity of plastics by differential scanning calorimetry.

## **1.2 Enthalpy**

### **1.2.1 ISO 11357-3:1999**

#### **Plastics -- Differential scanning calorimetry (DSC)**

Part 3: Determination of temperature and enthalpy of melting and crystallization

#### **1.2.2 ASTM E793-06**

#### **Standard Test Method for Enthalpies of Fusion and Crystallization by Differential Scanning Calorimetry Scope**

1.1 This test method describes the determination of the enthalpy (heat) of fusion (melting) and crystallization by differential scanning calorimetry (DSC).

1.2 This test method is applicable to solid samples in granular form or in any fabricated shape from which an appropriate specimen can be cut, or to liquid samples that crystallize within the range of the instrument. Note, however, that the results may be affected by the form and mass of the specimen, as well as by other experimental conditions.

1.3 The normal operating temperature range is from -120 to 600°C. The temperature range can be extended depending upon the instrumentation used.

1.4 This test method is generally applicable to thermally stable materials with well-defined endothermic or exothermic behavior.

1.5 Computer or electronic based instruments, techniques, or data treatment equivalent to those in this test method may also be used.

1.6 SI units are the standard.

1.7 The enthalpy of melting and crystallization portion of ISO 11357-3 is equivalent to this standard.

#### **1.2.3 ASTM E794-06**

#### **Standard Test Method for Melting And Crystallization Temperatures By Thermal Analysis**

Scope:

1.1 This test method describes the determination of melting (and crystallization) temperatures of pure materials by differential scanning calorimetry (DSC) and differential thermal analysis (DTA).

1.2 This test method is generally applicable to thermally stable materials with well-defined melting temperatures.

1.3 The normal operating range is from 120 to 600°C for DSC and 25 to 1500°C for DTA. The temperature range can be extended depending upon the instrumentation used.

1.4 Computer or electronic based instruments, techniques, or data treatment equivalent to those in this test method may be used.

1.5 SI units are the standard.

### 1.3 Thermal conductivity

#### 1.3.1 ISO 22007-2:2008

Plastics -- Determination of thermal conductivity and thermal diffusivity -- Part 2: Transient plane heat source (hot disc) method

Specifies a method for the determination of the thermal conductivity and thermal diffusivity, and hence the specific heat capacity per unit volume, of plastics. The experimental arrangement can be designed to match different specimen sizes. Measurements can be made in gaseous and vacuum environments at a range of temperatures and pressures.

This method is suitable for testing homogeneous and isotropic materials, as well as anisotropic materials with a uniaxial structure. In general, the method is suitable for materials having values of thermal conductivity,  $\lambda$ , in the approximate range  $0.01 \text{ Wm}^{-1}\text{K}^{-1} < \lambda < 500 \text{ Wm}^{-1}\text{K}^{-1}$  and values of thermal diffusivity,  $\alpha$ , in the range  $5 \times 10^{-8} \text{ m}^2\text{s}^{-1} \leq \alpha \leq 10^{-4} \text{ m}^2\text{s}^{-1}$ , and for temperatures,  $T$ , in the approximate range  $50 \text{ K} < T < 1 \text{ 000 K}$ .

The thermal-transport properties of liquids can also be determined, provided care is taken to minimize thermal convection.

#### 1.3.2 ISO 22007-4:2008

Plastics -- Determination of thermal conductivity and thermal diffusivity -- Part 4: Laser flash method.

Specifies a method for the determination of the thermal diffusivity of a thin solid disc of plastics in the thickness direction by the laser flash method. This method is based upon the measurement of the temperature rise at the rear face of the thin-disc specimen produced by a short energy pulse on the front face.

The method can be used for homogeneous solid plastics as well as composites having an isotropic or orthotropic structure. In general, it covers materials having a thermal diffusivity,  $\alpha$ , in the range  $1 \times 10^{-7} \text{ m}^2\text{s}^{-1} < \alpha < 1 \times 10^{-4} \text{ m}^2\text{s}^{-1}$ . Measurements can be carried out in gaseous and vacuum environments over a temperature range from  $-100 \text{ }^\circ\text{C}$  to  $+400 \text{ }^\circ\text{C}$ .

For inhomogeneous specimens, the measured values may be specimen thickness dependent.

#### 1.3.3 ISO 13787:2003

Thermal insulation products for building equipment and industrial installations -- Determination of declared thermal conductivity

ISO 13787:2003 establishes a procedure for the determination and verification of declared thermal conductivity, as a function of temperature, of thermal insulating materials and products used for the insulation of building equipment and industrial installations.

Informative annex B gives an optional method for establishing the thermal conductivity curve or table from measured values.

It is not applicable to thermal insulating products used in building envelopes. For the procedures which are used for these products, see ISO 10456, "Building materials and products - Procedures for determining declared and design thermal values".

## 1.4 Density

### 1.4.1 ASTM D1481-02(2007)

#### **Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer**

Scope:

1.1 This test method covers the determination of the density of oils more viscous than 15 cSt at 20°C ( $\text{mm}^2/\text{s}$ ), and of viscous oils and melted waxes at elevated temperatures, but not at temperatures at which the sample would have a vapor pressure of 100 mm Hg (13 kPa) or above.

Note 1—To determine the densities of less viscous liquids at 20 or 25°C use Test Method D 1217.

1.2 This test method provides a calculation procedure for converting density to relative density (specific gravity).

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

### 1.4.2 ISO 3675:1998

#### **Crude petroleum and liquid petroleum products -- Laboratory determination of density -- Hydrometer method**

### 1.4.3 ISO 12185:1996

#### **Crude petroleum and petroleum products -- Determination of density -- Oscillating U-tube method**

Abstract:

This standard gives a method for the determination, using an oscillation U-tube densitometer, of the density of crude petroleum and related products within the range  $600 \text{ kg/m}^3$  to  $1\ 100 \text{ kg/m}^3$  which can be handled as single-phase liquids at the test temperature and pressure.

## 1.5 Viscosity

### 1.5.1 ASTM D2534-88(2007)

#### **Standard Test Method for Coefficient of Kinetic Friction for Wax Coatings**

Scope:

1.1 This test method covers the determination of the coefficient of kinetic friction for a petroleum wax coating or wax-based hot melt coating when sliding over itself.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

### 1.5.2 ASTM D2669-06

#### **Standard Test Method for Apparent Viscosity of Petroleum Waxes Compounded with Additives (Hot Melts)**

Scope:

1.1 This test method covers the determination of the apparent viscosity of petroleum waxes compounded with additives (hot melts). It applies to fluid hot melts having apparent viscosities up to about 20 Pas at temperatures up to 175°C (347°F).

Note 1

For petroleum waxes and their blends having low apparent viscosities, below about 15 mPas, Test Method D 445, is especially applicable.

1.2 The values stated in SI units shall be regarded as the standard. One pascal second (Pas) = 1000 centipoises (cP). One millipascal second (mPas) = 1 centipoise (cgs units).

### 1.5.3 ASTM D1986-91(2007)

Title: Standard Test Method for Determining the Apparent Viscosity of Polyethylene Wax  
Scope:

1.1 This test method covers the determination of the viscosity in centipoise of polyethylene wax using the Brookfield LVT or LVF viscometer and thermosel system.

### 1.5.4 ASTM F766-82(2005)

Title: Standard Test Method for Melting Point of Waxes

Scope:

1.1 This test method covers the determination of the initial and final melting points and recovery point of waxes using a hot stage and microscope.

## 1.6 Thermal stability

### 1.6.1 ASTM E537-07

#### **Standard Test Method for The Thermal Stability Of Chemicals By Differential Scanning Calorimetry**

Scope:

1.1 This test method describes the ascertainment of the presence of enthalpic changes in a test specimen, using minimum quantities of material, approximates the temperature at which these enthalpic changes occur and determines their enthalpies (heats) using differential scanning calorimetry or pressure differential scanning calorimetry.

1.2 This test method may be performed on solids, liquids, or slurries.

1.3 This test method may be performed in an inert or a reactive atmosphere with an absolute pressure range from 100 Pa through 7 MPa and over a temperature range from 300 to 800 K (27 to 527°C ).

1.4 SI values are the standard.

1.5 There is no ISO standard equivalent to this test method.

## 1.7 Compatibility

### 1.7.1 ISO 175:1999

Plastics. Methods of test determination of the effects of immersion in liquid chemicals.

## 1.7.2 ISO 62:2008

Plastics -- Determination of water absorption

ISO 62:2008 describes a procedure for determining the moisture absorption properties in the "through-the-thickness" direction of flat or curved-form solid plastics. It also describes procedures for determining the amount of water absorbed by plastic specimens of defined dimensions, when immersed in water or when subjected to humid air under controlled conditions. The "through-the-thickness" moisture diffusion coefficient can be determined for single-phase material by assuming Fickian diffusion behavior with constant moisture absorption properties through the thickness of the test specimen. This model is valid for homogeneous materials and for reinforced polymer-matrix composites tested below their glass transition temperature. However, some two-phase matrices such as hardened epoxies may require a multi-phase absorption model which is not covered by this International Standard.

The results obtained using the methods described in this International Standard are applicable to most plastics but are not applicable to cellular plastics, granulates or powders, which can show additional absorption and capillary effects. Plastics exposed to moisture under controlled conditions for defined periods of time provide relative comparisons between them.

## 1.7.3 ISO 4611:2008

Plastics -Determination of the effects of exposure to damp heat, water spray and salt mist

It specifies the conditions of exposure of plastics to

- damp heat;
- water spray;
- salt mist;

and the methods for the evaluation of the change in some significant characteristics after given exposure stages.

It is, in general, suitable for all plastics in the form of standard test specimens, and finished articles or parts thereof.

It considers separately methods for the determination of

- change in mass;
- change in dimensions and appearance;
- change in physical properties.

## 1.8 German RAL-Standard for PCM

### 1.8.1 Phase transition temperature and stored heat

#### ***1.8.1.1 Determining the stored heat as a function of temperature***

The stored heat is determined according to one of the following measurement methods:

- hf-DSC dynamic measurement with constant rates of heating and cooling,
- hf-DSC quasi-stationary measurement with a step profile for heating,
- m-DSC,
- T-history method,
- CALVET calorimeter,
- multilayer calorimeter procedure.

### **1.8.1.2 Performance of the measurement**

When the measurements are made, the requirements of the Quality Association PCM e.V. must be observed, particularly with regard to

- number of samples and measurements,
- procedures to ensure thermal equilibrium within the sample with sufficient accuracy,
- regulations for measuring the phase transition temperature and stored heat.

### **1.8.1.3 Contents of the test results and the test report**

The stored heat as a function of temperature must be specified separately for the cases of heating and cooling, as there will be differences in the temperature profile in most cases. It is tabulated in a predetermined temperature range. This must be done in each case with respect to

- the sample mass in J/g,
- the sample volume in J/ml.

The values with respect to the sample volume will be calculated from the values with respect to the sample mass by multiplication with the minimum sample density in the operating temperature range.

## **1.8.2 Cycle stability**

The examination of the cycle stability is carried out to award the quality mark for PCM and PCM composites. In practice, usually thermal cycling is carried out using equipment that has been designed especially for the relevant product, which can go through many cycles in the shortest possible testing time. The suitability of the apparatus used needs to be confirmed by the Quality Association PCM e.V

### **1.8.2.1 Testing the quality criteria**

At regular intervals, tests need to be carried out on various quality criteria. Apart from the criteria phase transition, temperature and stored heat that are always necessary, depending on the sample being examined, a test may need to be carried out on mass or thermal transition changes for example. The Quality Association PCM e.V. sets the quality criteria to be tested and the tolerances to be complied with.

Stored heat:

Determining the stored heat as a central quality criterion must be carried out analogously to the measurement regulations in accordance with section 3.6.1 Phase transition temperature and stored heat.

Enthalpy–temperature profile:

Deviations in the enthalpy-temperature profile for each of the onset, offset and peak temperatures are permissible providing the tolerances set by the Quality Association PCM e.V. are complied with. Greater deviations in at least one of these three temperatures are to be regarded as a case of damage. The measurement regulations can be found in the section 'Phase transition temperature and stored heat' and must be applied.

Density:

Checking the density of encapsulated PCM is to be carried out using suitable measurement procedures which are approved by the Quality Association PCM e.V.

### 1.8.2.2 Cycle category

Depending on the number of cycles tested, categories A – F are attainable.

Depending on the intended category, apart from the start measurement at the beginning of cycling, control measurements at various frequencies need to be carried out on the damage quality criteria listed in 3.6.2.1. The categories and the resulting control frequency are listed here.

Sensitive samples (e.g. hygroscopic, encapsulated, etc.), for which the sample composition can be affected by removal of part of the sample for a control measurement, must be removed as a complete unit (e.g. bag) and may not be used thereafter for further cycling. For this reason, sufficient individual samples must be included from the beginning of the cycling process. For the control measurements, samples are removed from the cycling apparatus and are measured. The samples removed may not be cycled any further after the control measurement.

If the intended category is not attained, then the last category proven to be passed can be awarded.

Category name	Number of cycles	Check the quality criteria every
A	≥ 10,000 cycles	1,000 cycles
B	≥ 5,000 cycles	500 cycles
C	≥ 1,000 cycles	250 cycles
D	≥ 500 cycles	100 cycles
E	≥ 100 cycles	25 cycles
F	≥ 50 cycles	10 cycles

### 1.8.2.3 Performance of the measurement

The temperature ramp that the cycling is based on is defined on the basis of a measurement of the whole enthalpy and the enthalpy temperature profile in accordance with section 3.6.1 for every material to be tested. The switching temperatures and upper and lower driving temperatures are determined on the basis of this measurement result. The duration of the pauses and the permissible lowest and highest temperatures should be taken from the manufacturer's specifications. To determine the temperature ramp, the specifications set out by the Quality Association PCM e.V. need to be complied with, in particular on defining the measurement cycle based on:

- temperature limits,
- cycling width,
- permissible heating rates,
- on-/offset temperatures used.

The measured values must be recorded using suitable measurement technology and must be kept to document the cycles completed.

Further specifications of the Quality Association PCM e.V. on measurement apparatus, positioning the temperature sensors and measurement technology used are to be complied with.

#### **1.8.2.4 Contents of the test results and the test report**

The test report must provide information on all measurements carried out during cycling. This includes in particular:

- Documentation of the initial measurements
- Parameters of temperature ramp
- Documentation of the control measurements and results
- Proof of cycles completed (e.g. through attached measurement data)
- Where applicable, information on cases of damage
- Information on the cycle categories successfully tested

### **1.8.3 Thermal conductivity**

#### **1.8.3.1 Thermal conductivity as a function of temperature**

Thermal conductivity is determined above and below the melting point with an accuracy of at least:

- $\pm 5\%$  in the thermal conductivity,
- $\pm 0.5\text{ °C}$  in the temperature.

This accuracy currently corresponds to the known state of technology. More stringent demands on accuracy can be made in future by the Quality Association PCM e.V. if state of the art technology develops further.

#### **1.8.3.2 Determining the thermal conductivity above and below the melting point**

According to the current state of knowledge, suitable measurement methods are:

See Table 3.6.3.2: Suitable measurement methods

Due to their individual characteristics, the measurement methods are not equally well suited for all PCM. The most suitable measurement method for the relevant sample should be selected. The Quality Committee can specify testing for only one state for certain products.

Other measurement methods can be categorized as being suitable by the Quality Association PCM e.V. on the basis of new findings. In all of the listed measurement methods, the temperature measurement points and the measurement parameters must be chosen such that melting processes in the sample do not affect determining the thermal conductivity. This selection must be based on an enthalpy graph that must be available for the material to be tested.

At the start of every individual measurement, the thermal equilibrium within the sample must be warranted. To ensure the reliability of test results, the functionality and accuracy of the measurement system used must be checked by test measurements on standard materials and must be documented immediately before an initial test or external monitoring.

## 2 Development of a Measurement Standard for DSC measurements

This section describes the steps which were done so far to come to a measurement standard for the thermal characterization of PCMs. This means to be focused on the characterization of PCMs using DSCs.

### 2.1 Reference PCM

The starting point was to get the state of art of this type of PCM characterization. So the first step was to learn how big the actual deviations of measurement results are determined by different laboratories. Therefore octadecane was chosen as reference PCM, which is a synthetic octadecane synthesized by Sasol Wax using the Fischer-Tropsch process. The chemical data are plotted in the table below (Table 1).

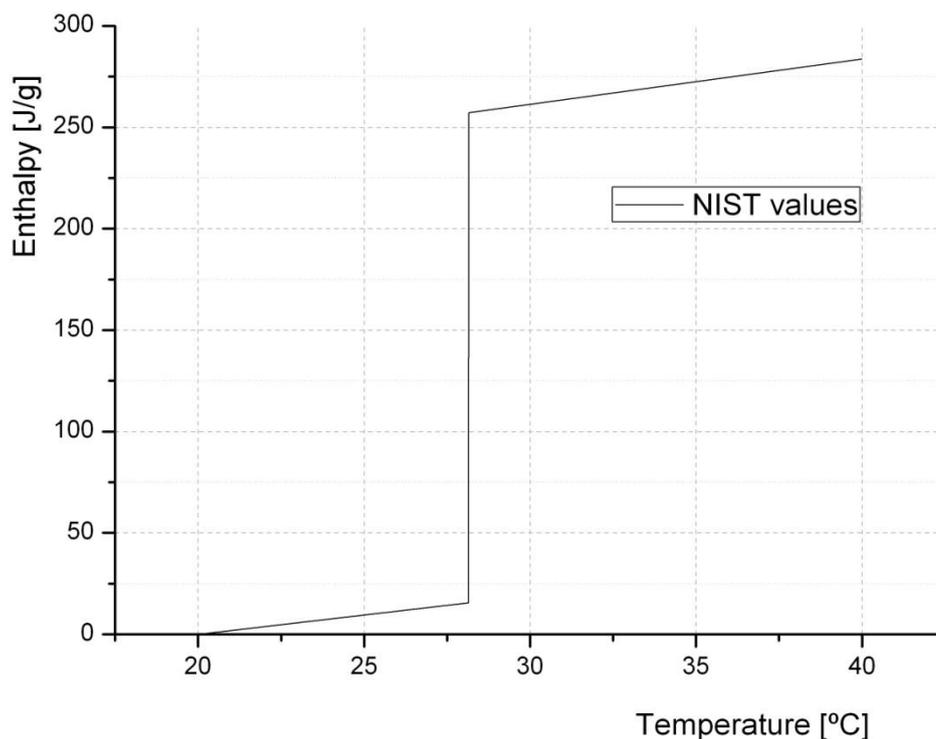
*Table 1: Compounding of the reference octadecane*

Name	Parafol 18-97
C16	0.07 wt.-%
C18	98.11 wt.-%
iC18	0.4 wt.-%
C20	0 wt.-%
Bromine	0.01 mg/100mg

The thermal properties for pure octadecane given by the NIST are shown in table 2. Figure 2 shows the enthalpy of octadecane in function of temperature built with the reference values.

*Table 2: Reference values of the thermal properties of pure octadecane*

Property	Value	Value (SI)	Reference
Constant pressure heat capacity of solid	485.64 J/mol K	1.9083 J/gK	K Messerly, Guthrie, et al., 1967
Constant pressure heat capacity of liquid	568.325 J/mol K	2.23315 J/gK	K Hoehne, 1981
Enthalpy of fusion	61500 J/mol	241.66 J/g	K Domalski and Hearing, 1996
Temperature of fusion	301.3 K	28.15 °C	Domalski and Hearing, 1996



## 2.2 Partners for the Round Robin Test

The reference material was sent to several labs. The target was to get an overview on the results of different types of equipment and also over a range of different laboratories doing PCM characterization. Table 2 shows a list of institution having equipment to obtain the melting and solidification curves.

Figure 2 shows the countries involved into the working group and the interest on topic. Those having interest in PCM characterization using heat flux DSCs and/or calvet DSCs. DSCs from all common manufacturers are available within the group Figure 3.

Table 2: List partners doing PCM characterization by DSC and other devices.

Institute / Company	Country	Type of Measurement	Manufacturer
AIDICO	Spain	heat flux DSC	Mettler Toledo
AIDICO	Spain	TG-DSC	Mettler Toledo
Universitat de Barcelona	Spain	heat flux DSC	Mettler-Toledo
Fraunhofer ISE	Germany	calvet DSC	Setaram
Fraunhofer ISE	Germany	heat flux DSC	TA-Instruments
Fraunhofer ISE	Germany	T-history	self built
Fraunhofer ISE	Germany	heat flux	self built
National Institute of Chemistry (NIC)	Slovenia	TG-DSC	TA Instruments
University of Zaragoza	Spain	T-history	selfe built
University of Zaragoza	Spain	Energy Balance	self built
University of Zaragoza	Spain	heat flux DSC	Netzsch
TU Wildau	Germany	calvet DSC	Setaram
University of Lleida	Spain	heat flux DSC	Mettler Toledo
Polymer Institute of Slovak Academy of Science	Slovakia	heat flux DSC	Metler Toledo
ERTES	France	??	Perkin Elmer
University of Basque Country	Spain	heat flux DSC	Metler Toledo
University of Basque Country	Spain	T-history	self built
Warsaw University of Technology	Poland	heat-flux	Perkin-Elmer
Warsaw University of Technology	Poland	Heating with constant power,	WUT
Université de Pau	France	heat flux DSC	Perkin Elmer
Institut für Luft und Kältetechnik	Germany	calvet DSC	Setaram
Institut für Luft und Kältetechnik	Germany	heat flux DSC	TA Instruments

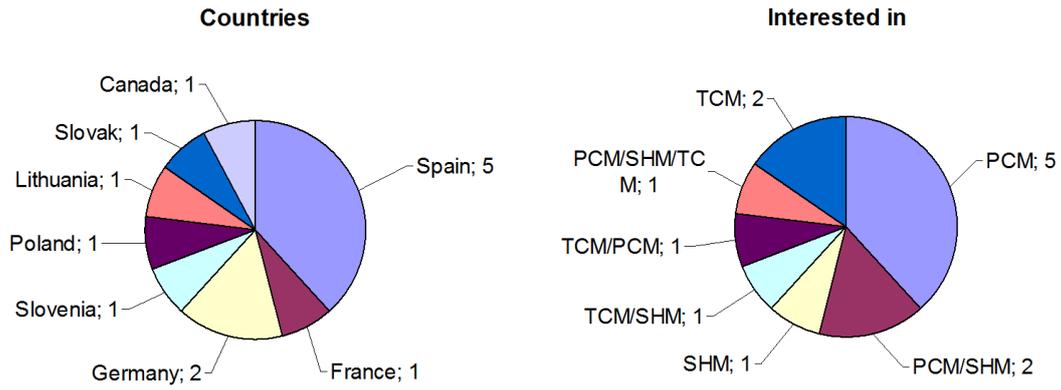


Figure 2: Countries and interest of partner. TCM: thermo-chemical material, PCM: phase change material, SHM: sensible heat storage material

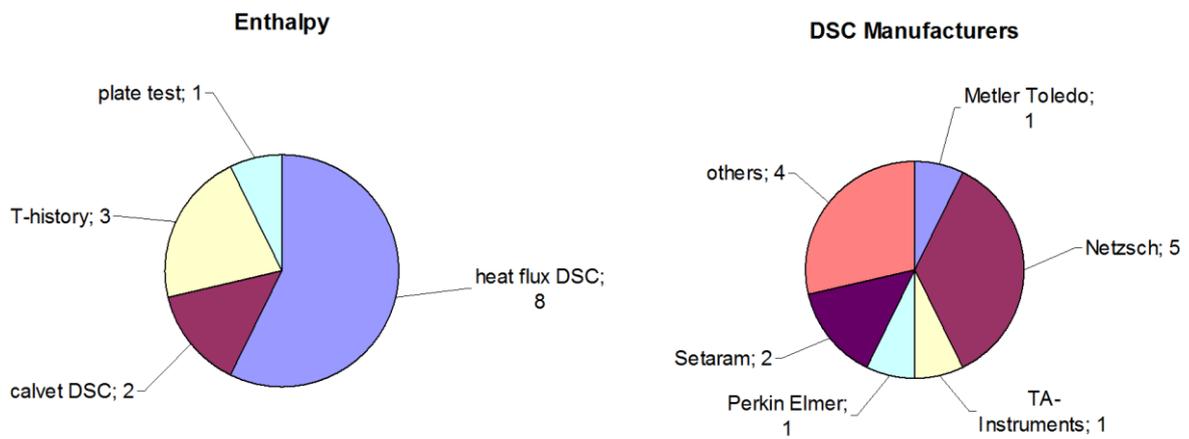


Figure 3: Type of DSC equipment and manufacturers.

### 2.3 Result of first RRT

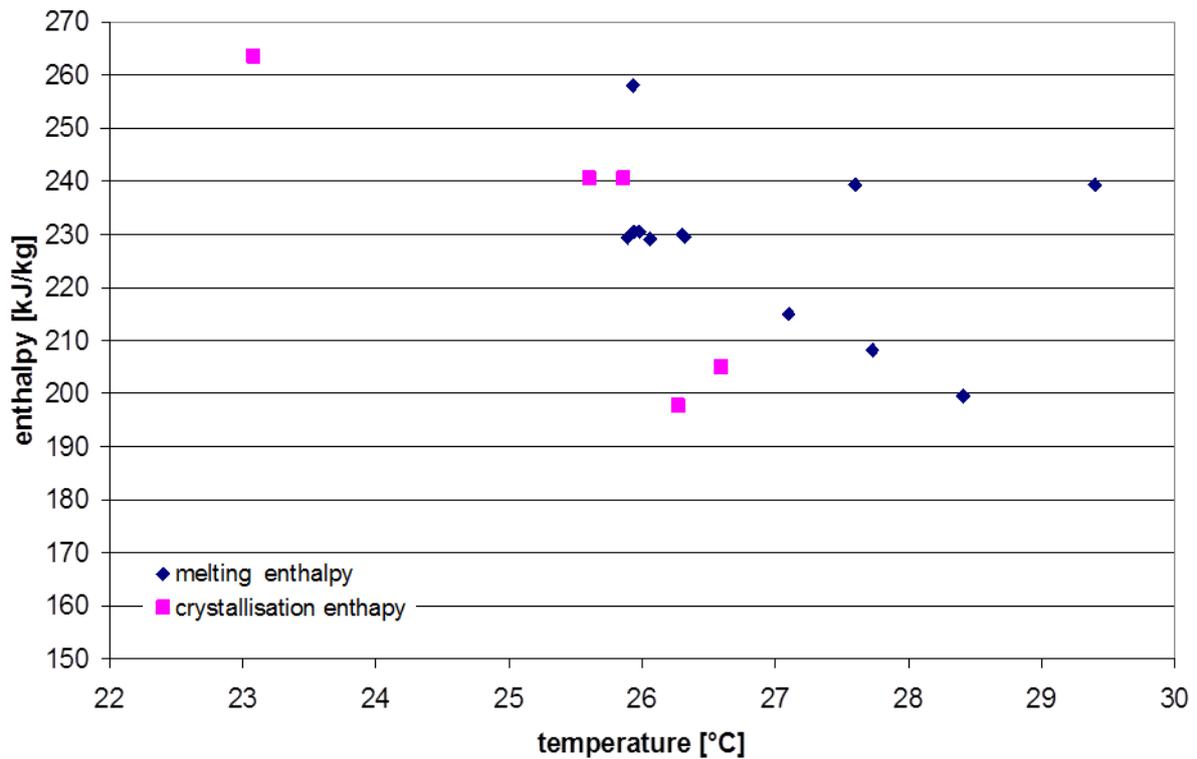


Figure 4 shows the result of the First Round Robin Test (RRT). There was no procedure defined for the measurements. So every partner measured according to its own definition. The obtained result shows a wide range in melting as well as in solidification temperatures. Also the measured melting enthalpies deviate in a large range. The enthalpy curves displays the differences between the single results more detailed (Figure 5). These results give a good impression how comparable values are which are published by different labs today and it underlines the importance of a common standard for the characterization of PCMs.

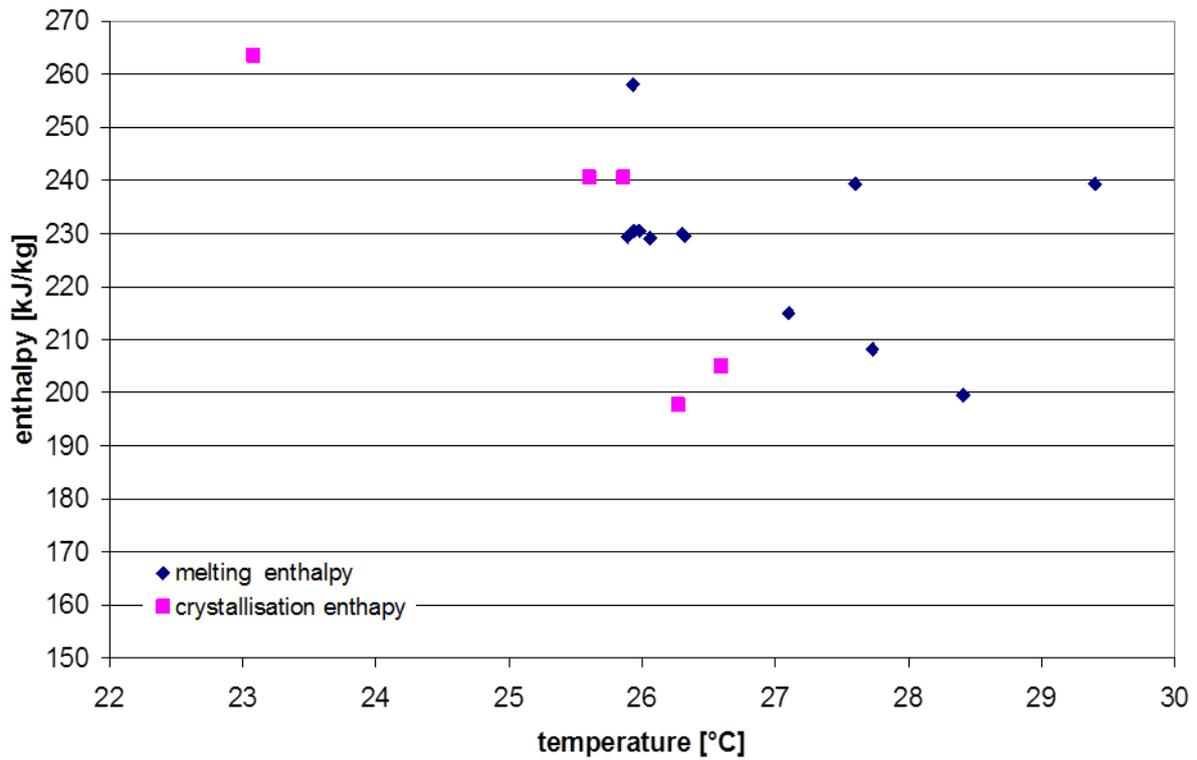


Figure 4: State of the art in DSC-measurement. Comparison of results from different laboratories.

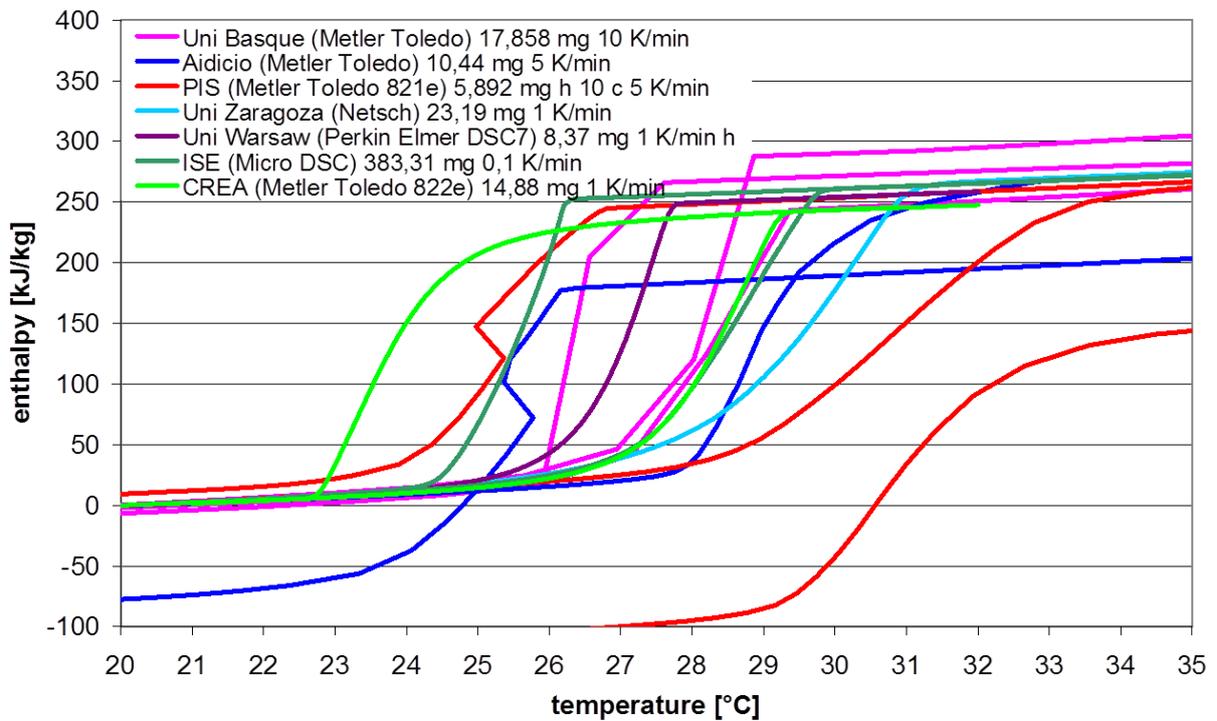


Figure 5: Results of first RRT as function of temperature

## 2.4 Pre-definition of a DSC measuring standard according to RAL

After the first result (2.3) was obtained the first definition for the measurement procedure was defined according to the definition of the German RAL standard. This definition was sent to the partner to do a second RRT. The procedure is:

1. Three samples per material (**For the test in T4224, Please just measure 1 sample!**)
  - a. At least one measurement per sample which consist of three cycles (heating and cooling ramps) which includes the whole melting and cooling peak,
2. Procedure to insure the thermal equilibrium
  - a. The sample has to be in a isothermal stage during the measurement
  - b. The method to insure the thermal equilibrium
    - i. Dynamic measurement with constant heating and cooling rate
      1. Slow down the heating rate by "halve" the heating rate from one full cycle to the next (e.g 1.6; 0.8; 0.4; 0.2; 0.1; 0.05; 0.025; 0.0125)
      2. Plot the results of all cycles as Enthalpy vs. temperature
      3. The feasible min. heat rate is reached when the inflexion point of the heating and the cooling ramp differ not more than 0.2 K to the next slower heat/cooling ramp (see Figure 6)
      4. Or the temperature from the inflexion points of the heating ramp and the cooling ramp differ not more than 0.5 K (see Figure 7)
      5. The slower heating/cooling ramp with which the criteria above was first reached has to be applied to all samples of this material
      6. Are there more than one phase transitions the criteria defined above have to be true for all phase transitions
    - ii. Dynamic measurement with the T-history method
      1. It is like in (i), a proper end-temperature has to be determined. This has to be done by variation of the end-temperature
      2. Divide by two the temperatur difference between melting/crystallisation temperature and the end-temperature, the difference of the curves have to be less than 0.2 K
      3. Or less than 0.5K between the results of melting and crystallisation measurement
    - iii. Isothermal measurement (stepwise with the DSC)
      1. The time at temperature has to be long enough to make sure that the sample is in isothermal state. This can be checked by a decline of the measurement signal to the base line

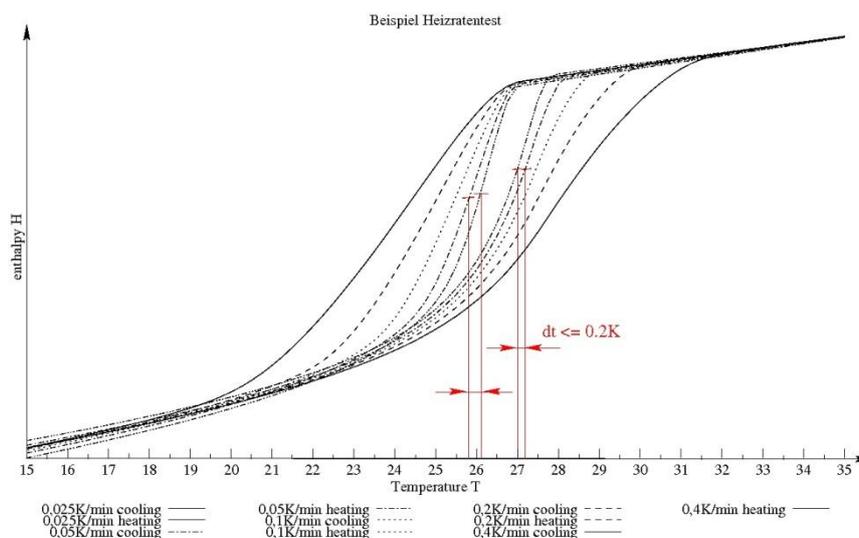


Figure 6: To ensure the thermal equilibrium (case 1) (source: RAL Guetegemeinschaft)

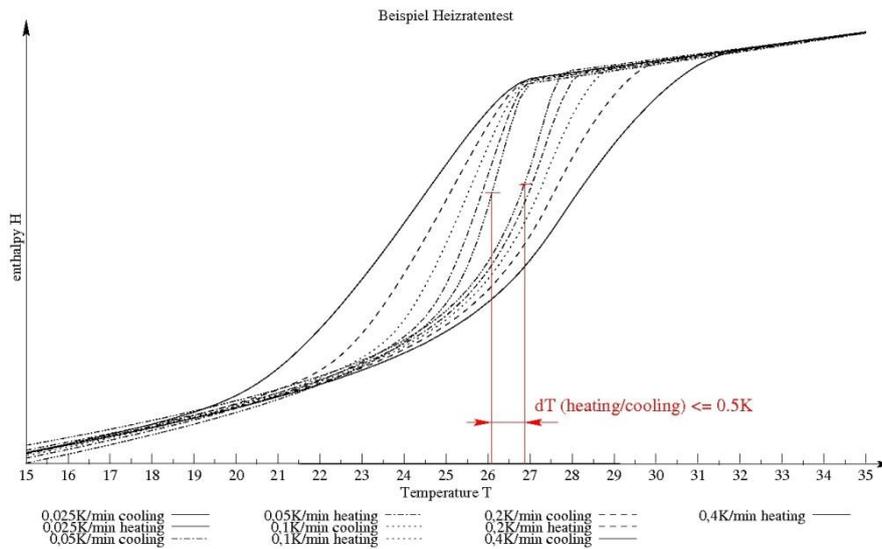


Figure 7: To ensure the thermal equilibrium (case 2) (source: RAL Guetegemeinschaft)

The basis for this procedure is the knowledge that the sample mass and the chosen heating rate have an influence on the result (shape of the curves). This can be seen in Figure 6 as well as in Figure 7. This influence was also investigated for the reference PCM by the Warsaw University of Technology and University of Leida (Figure 8 to Figure 10).

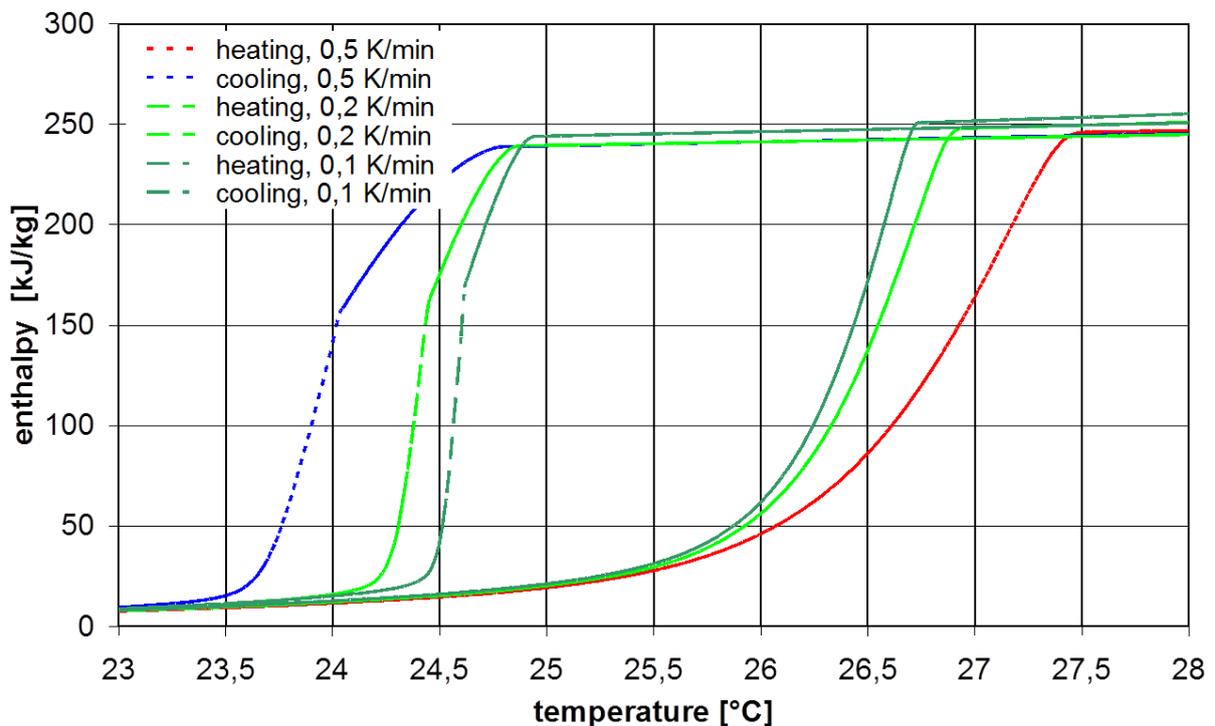


Figure 8: Influence of heating and cooling rate on the results for the reference PCM using a Perking & Elmer DSC, sample mass 15.56 mg (Warsaw University of Technology)

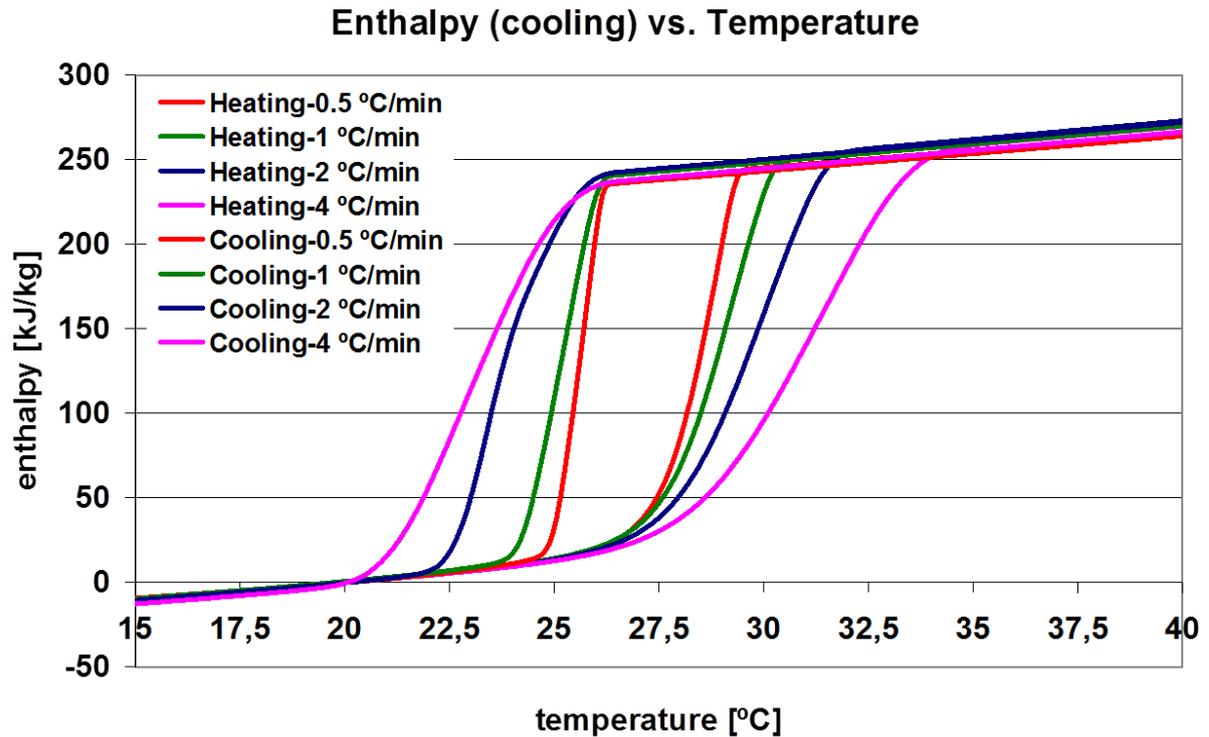


Figure 9: Influence of heating and cooling rate on the results for the reference PCM using a Mettler Toledo DSC (University of Lleida)

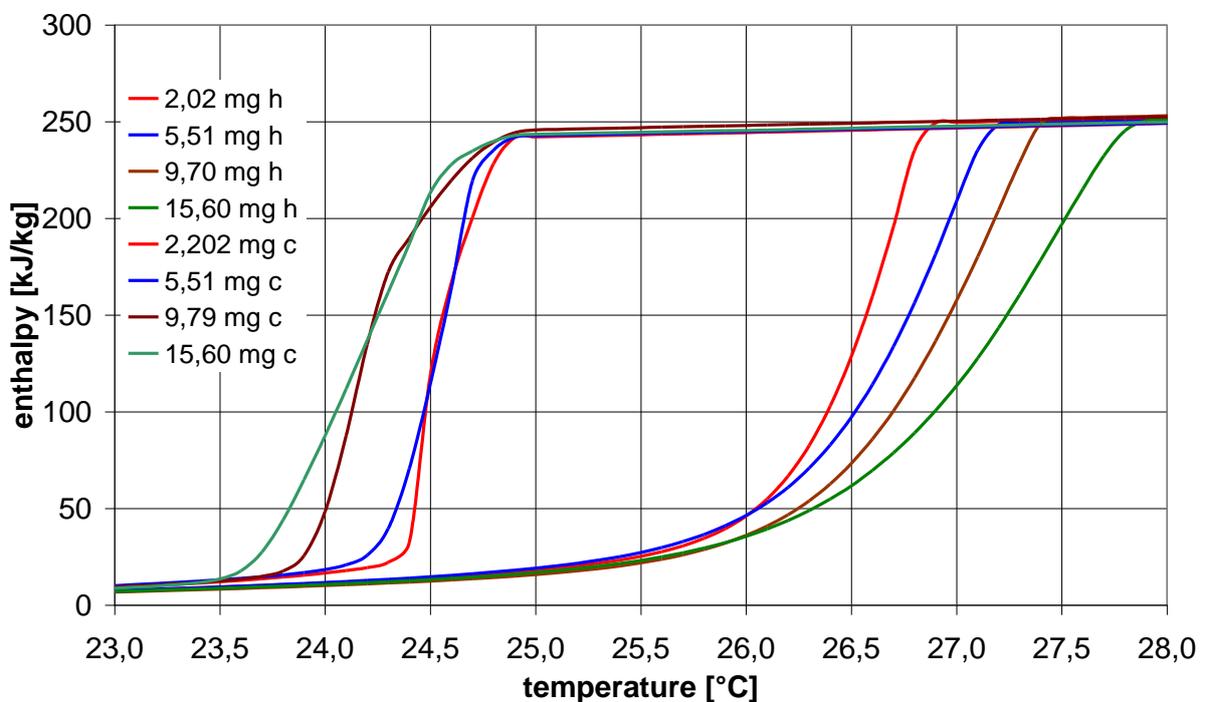


Figure 10: Influence of the results by different sample masses (heating/cooling rate 0.5 K/min) using the Perkin & Elmer DSC, heating rate 0.5 K/min (Warsaw University of Technology)

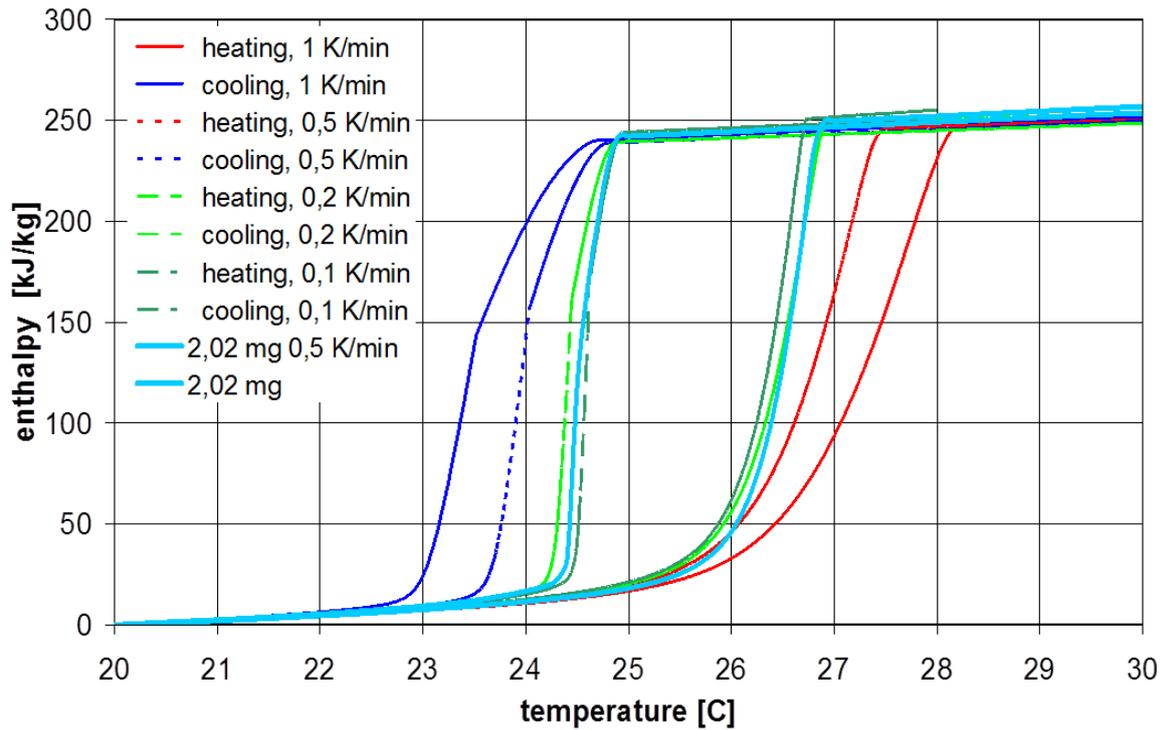


Figure 11: Comparison of different heating rates (15.56 mg) and reduced sample mass (Warsaw University of Technology)

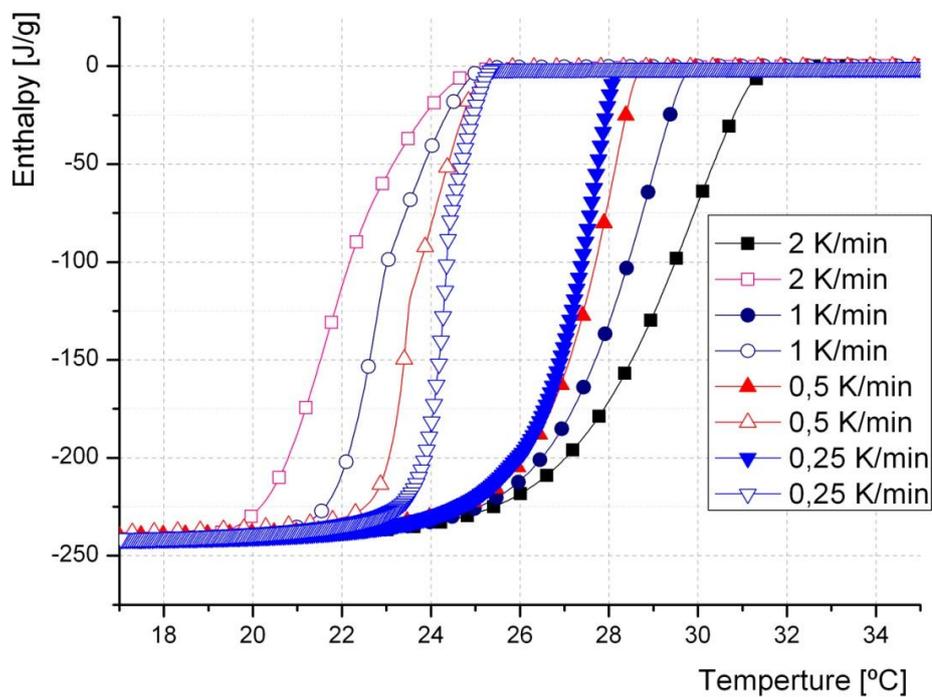


Figure 12: Comparison of different heating rates (15.45 mg) for the reference PCM using a NETZSCH DSC (University of Zaragoza)

## 2.5 Results of second RRT according to RAL

Figure 13 shows the comparison of results of those measured according to the defined procedure. Almost all measurement lead approximately to the same enthalpy except the measurement from the University of Lleida (UDL), which show lower enthalpies. But the measurement reveals also a wider range of melting and solidification curves. The measurement from ISE lies in-between the curves of UDL while the results of ILK and WUT are more shifted to the left side.

So for the enthalpies the result is much better than the one got before (see 2.3). But the differences in temperatures are still too big to compare the results.

As the resulting temperatures are much related to the DSC and the whole procedure from the calibration of the device to the preparation of samples and its measurement it was decided to make a workshop on DSC measurement to have the chance to learn more about the differences of DSC and to establish a common basis for the measurement procedure within the test and characterization group.

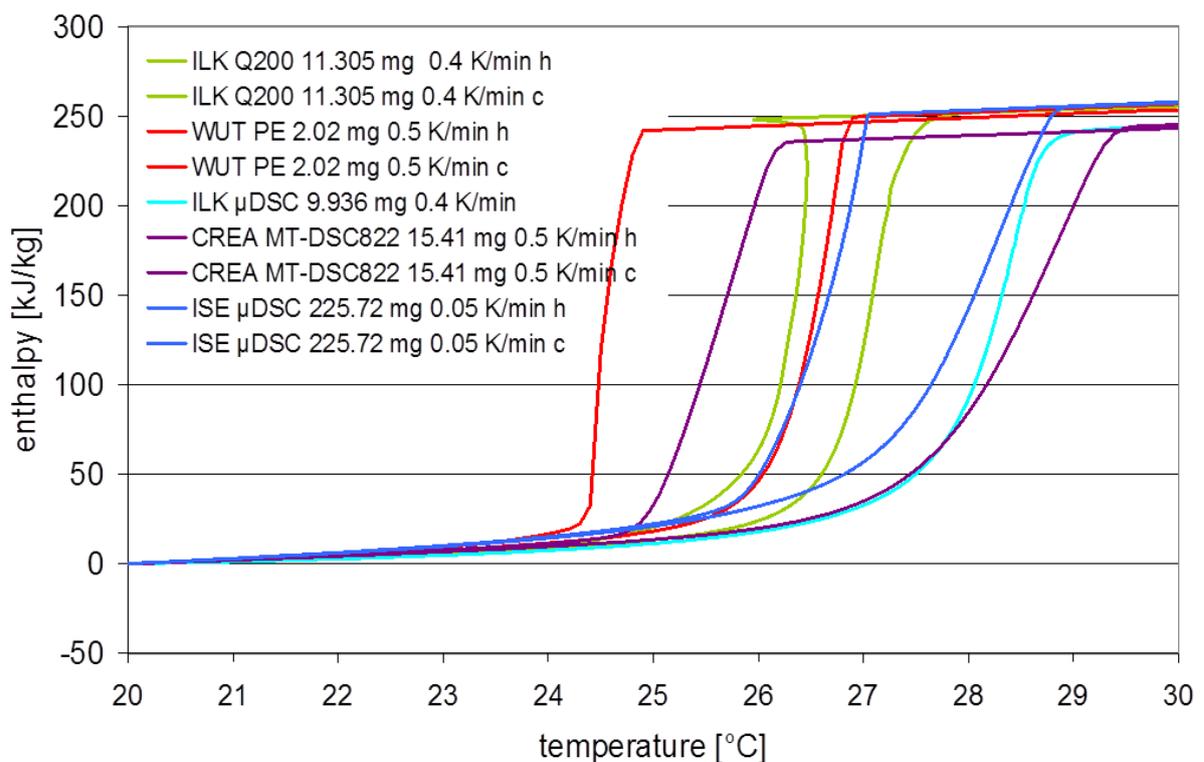


Figure 13: Comparison of DSC results measured according to the RAL pre-definition

## 2.6 DSC Workshop on PCM Characterization

As seen above although the measurement were done according to the RAL standard for DSC measurement of PCM there is still a wide variation of melting and solidification temperatures visible. These deviations can be related to:

- the DSC itself,
- the preparation of the samples,
- The calibration of the DSC

To get answers of this questions and to get a better impression on the differences of the DSCs as well as to bring the participations of the RRT to a common basis a workshop was suggested at the Task 42/Annex 24 meeting in Belfast in February 2011. The workshop was held in Freiburg in May 23<sup>rd</sup> until 27<sup>th</sup> 2011. It was connected to a COST-Action TU0802 meeting which was hosted at the same time also at the Fraunhofer Institute in Freiburg.

For the workshop DSCs from, Netzsch (DSC 204 F1), Perkin & Elmer (DSC 4000), Mettler Toledo (DSC 1) and TA-Instruments (Q200) were provided from the manufacturers, so that it was possible to do the measurement on the DSCs in parallel and to compare the results right after the measurements. Figure 14 shows the different DSCs.

Harald Mehling and Eva Günther from the ZAE Bayern gave presentations in the frame of the workshop. Harald Mehling's presentation was about DSC-measurement in general and the RAL procedure as well as about data evaluation and Eva Günther's presentation was about DSC calibration.

Seven people participated at the workshop: three from Spain, one from France and three from Germany. All participants stood for the whole week. In addition also instructors for the DSC coming from the manufacturers supported us during the workshop.



Figure 14: The DSCs used in the workshop, clockwise from top: Mettler Toledo, Netzsch, TA-Instruments and Perkin & Elmer

In the workshop a measurement was conducted according the RAL procedure using the reference PCM. Here it was observed, that all DSC have to go below 0.5 K/min to get a difference from peak to peak of less than 0.2 K/min. After this experiment a calibration was done for all DSC using ice and indium with a heating rate of 0.5 K/min. This calibration was used to measure the reference PCM again. Figure 16 shows the result of this measurement. As it can be seen in Figure 14, the Netzsch device as well as the DSC from Mettler Toledo show unstable baselines. For the Netzsch DSC the measurement was repeated at the University of Zaragoza and for the Mettler Toledo DSC at the University

of Basque Country. There a much more stable baselines were obtained (Figure 17). Figure 18 compares the heating curves of these measurements. The curve measured by the Mettler Toledo DSC (MT) shows a much too high specific heat but the measured shape of the curve within melting range fits quite well to those measured with the other DSCs. Also the measured melting temperatures determined by the Netzsch DSC at the workshop fits well to the other results. The comparison shows a clear improvement compared to the result of the RRT done before (Figure 13). Here the deviation of melting temperature was more than 3 K now it's less than 0.5 K.

The experiment looks like:

1. Measuring of Baseline: Reference crucible closed, sample crucible without sample and open lid.
2. Applying sample to the sample crucible and close the lid.
3. Measurement of the sample.

This means that the sample crucible looks different from the baseline-measurement to the sample-measurement. Figure 19 shows a comparison of baseline measurements: In the first measurement both crucible were open (lid not closed). During the second measurement the lid of the reference crucible was closed and the sample crucible was still open. So there is an influence on the baseline but so far the influence on the sample measurement is not determined.

Several problems were observed during the workshop:

- Only calibration with Indium, i.e. at  $T=156\text{ °C}$  -> no calibration in the temperature range of the ringtest sample (solution in the workshop: calibration using water/ice at  $0\text{ °C}$ ).
- Unstable baselines make it difficult to determine enthalpy versus temperature.
- Crucibles that loose material when the lid is closed (P&E, Figure 15).

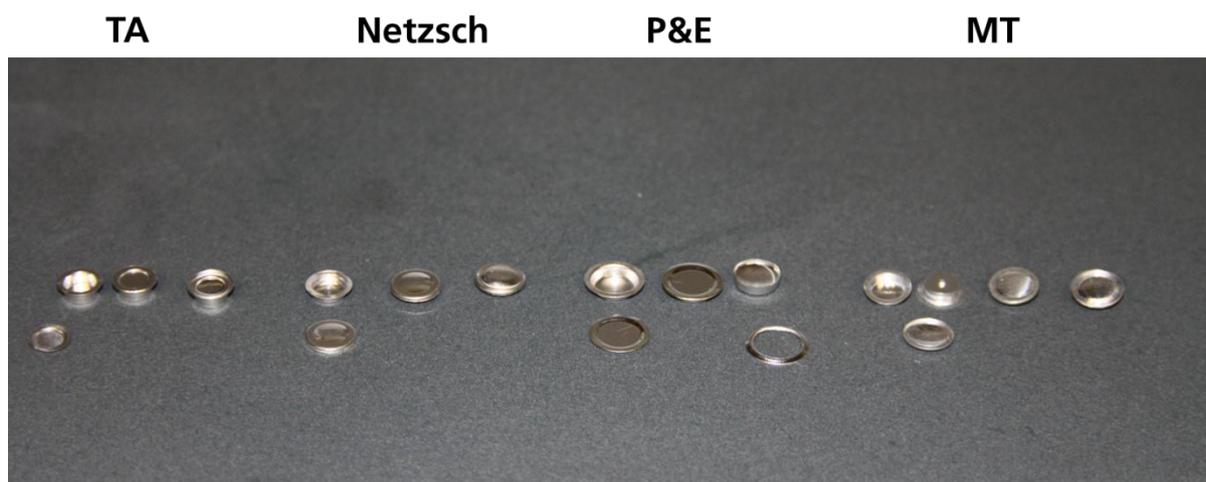


Figure 15: The crucibles (pan and lids), open pan and lid, lid laid on pan and pan closed

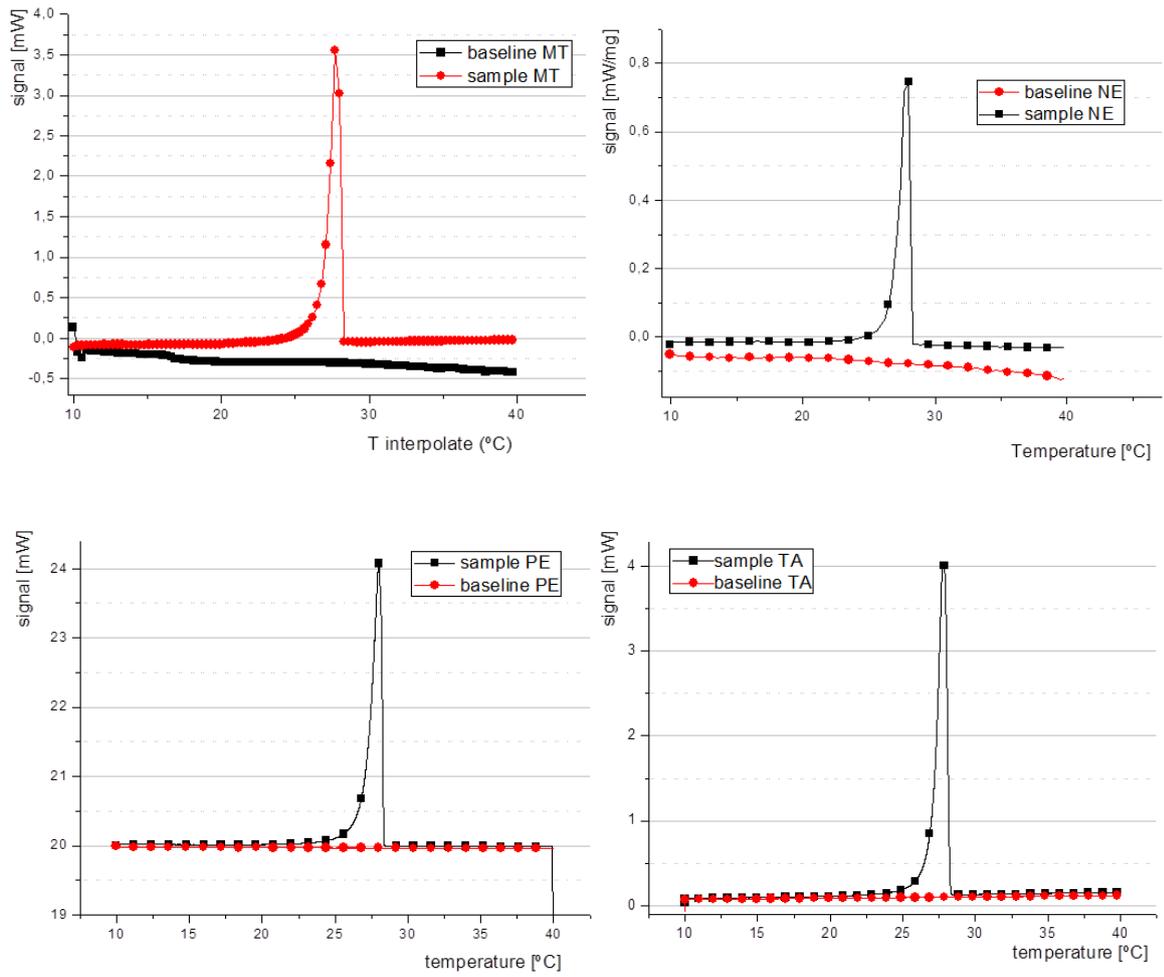


Figure 16: Results of the PCM measurements after calibration with ice. Clockwise from the top: Mettler Toledo, Netzsch, Perkin & Elmer and Ta-Instruments

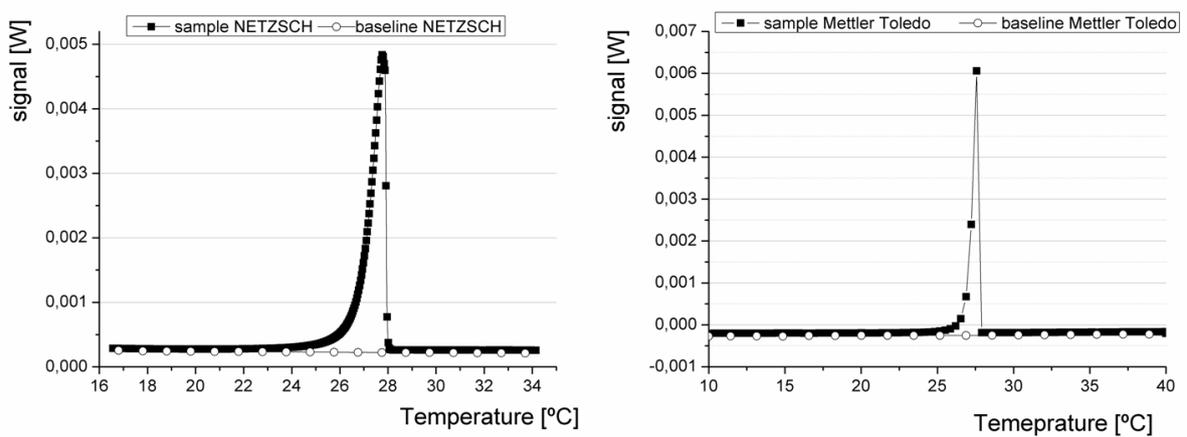


Figure 17: Measurement for the Netzsch device which was repeated at the University of Zaragoza and for the Mettler Toledo device which was repeated at the University of Basque Country.

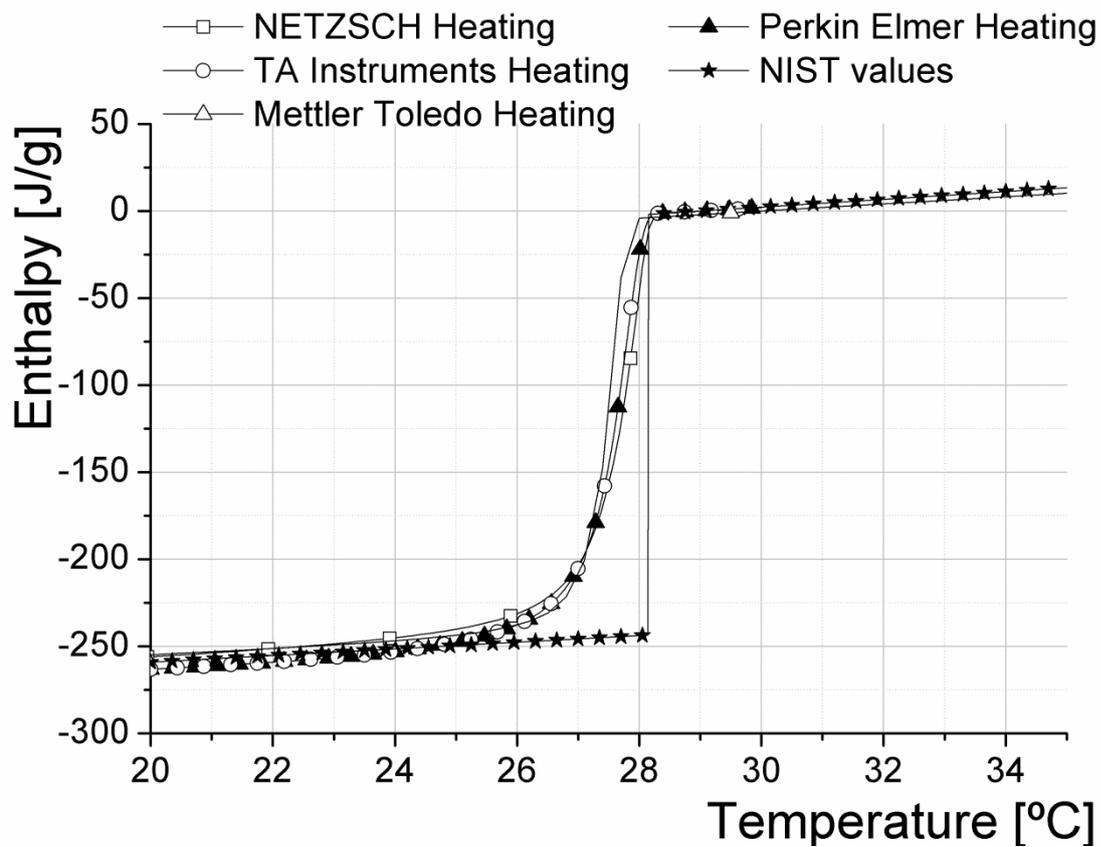


Figure 18: Comparison of the DSC results.

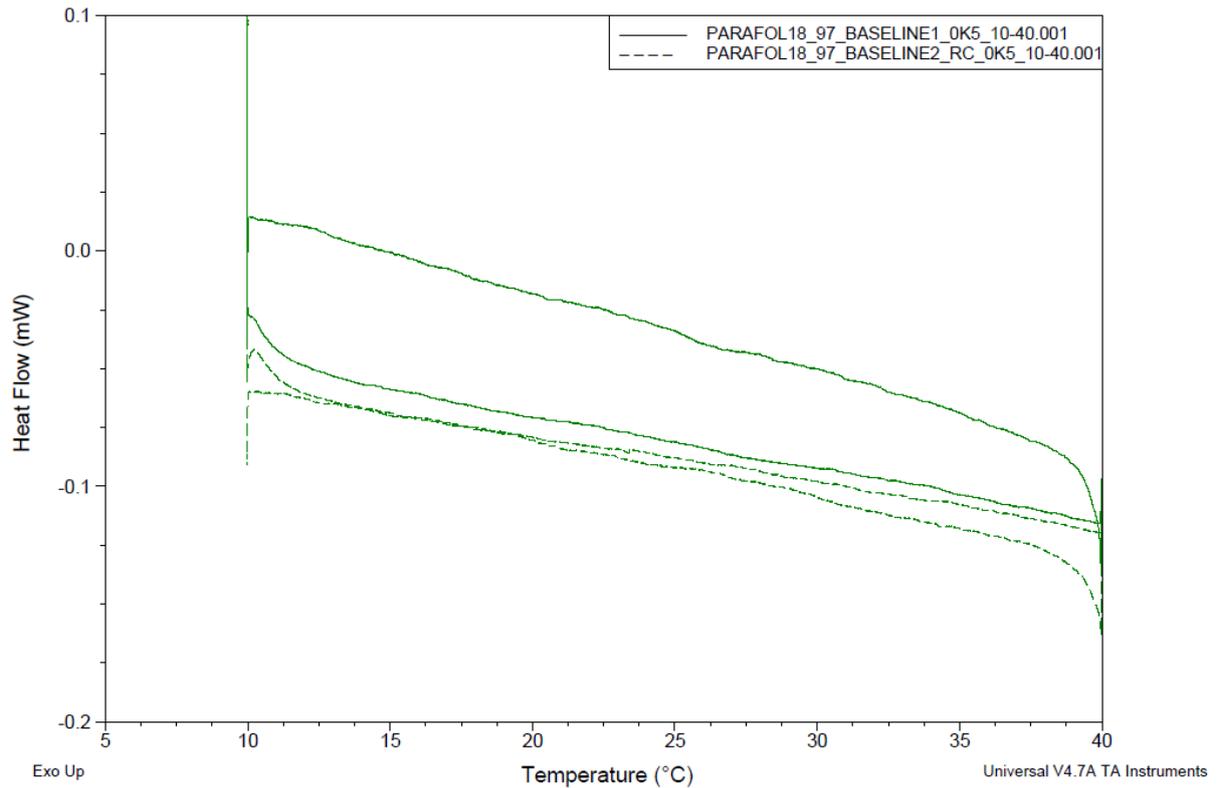


Figure 19: Experiment baseline-measurement, solid line: reference and sample pan open (lid only laid on pan), dashed line: lid of reference closed sample still open, heating rate 0.5 K/min

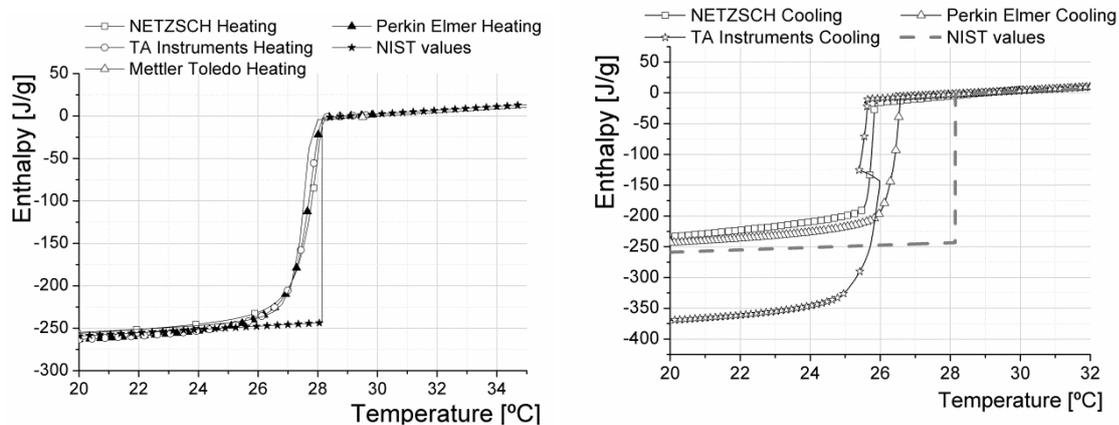


Figure 20: Comparison of the result obtained for heating (left) and for cooling (right)

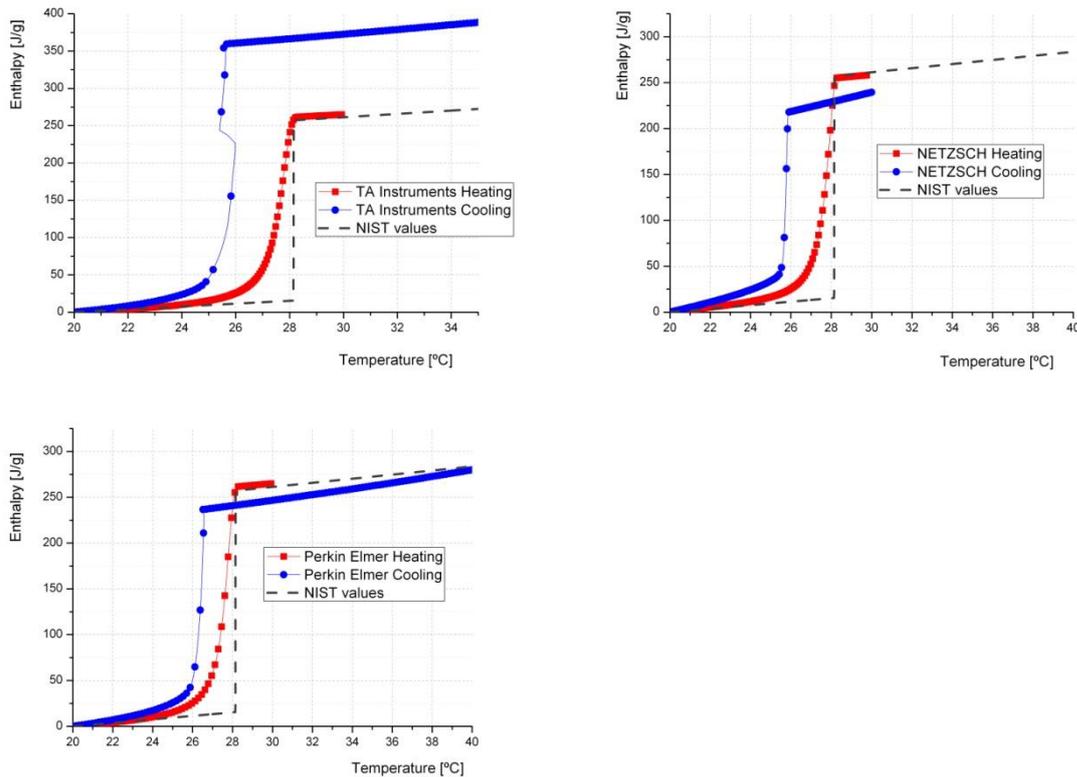


Figure 21: Comparison of the DSC results from the different equipments

The results obtained from the cooling measurements show more problems related to the agreement, the specific heat values and the total enthalpy change than the results from heating measurements.

In order to study the reproducibility of the measurements, three different samples were measured using the same procedure. Also the results obtained by two different measuring modes available for the NETZSCH DSC were compared. With STC means that the temperature program is followed by the sample crucible and without STC means that the temperature program is followed by the furnace temperature. Results show that the reproducibility is ensured for heating measurements (figure 21).

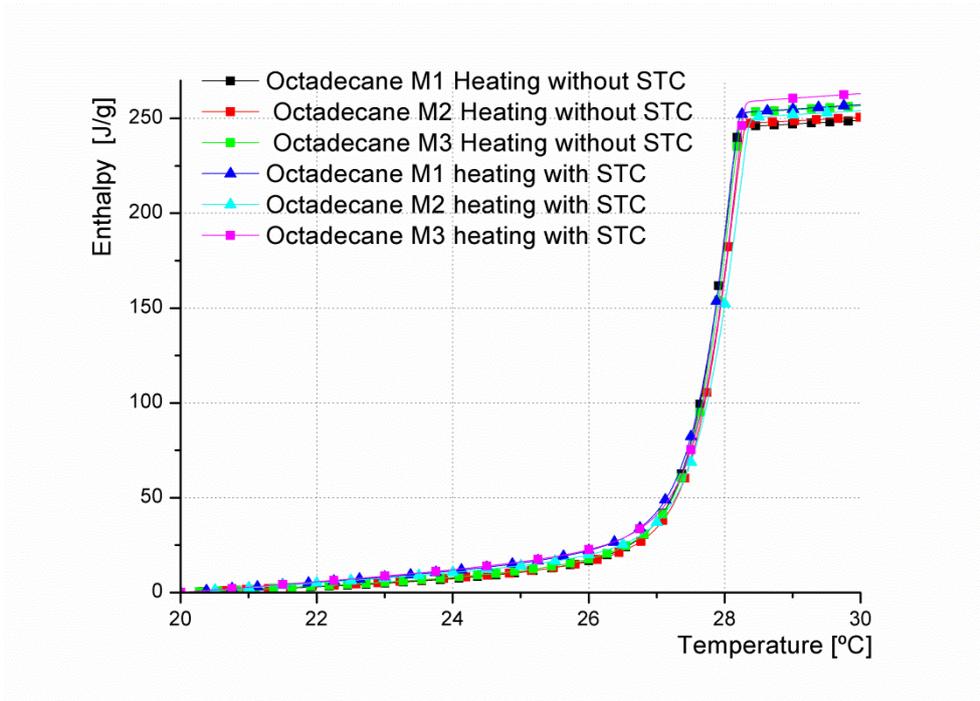


Figure 22: Comparison of the heating results from the different samples for the NETZSCH DSC at the University of Zaragoza, Spain.

As shown in figure 22, cooling mode based on liquid Nitrogen is not valid for the cooling measurements since results are no reproducible.

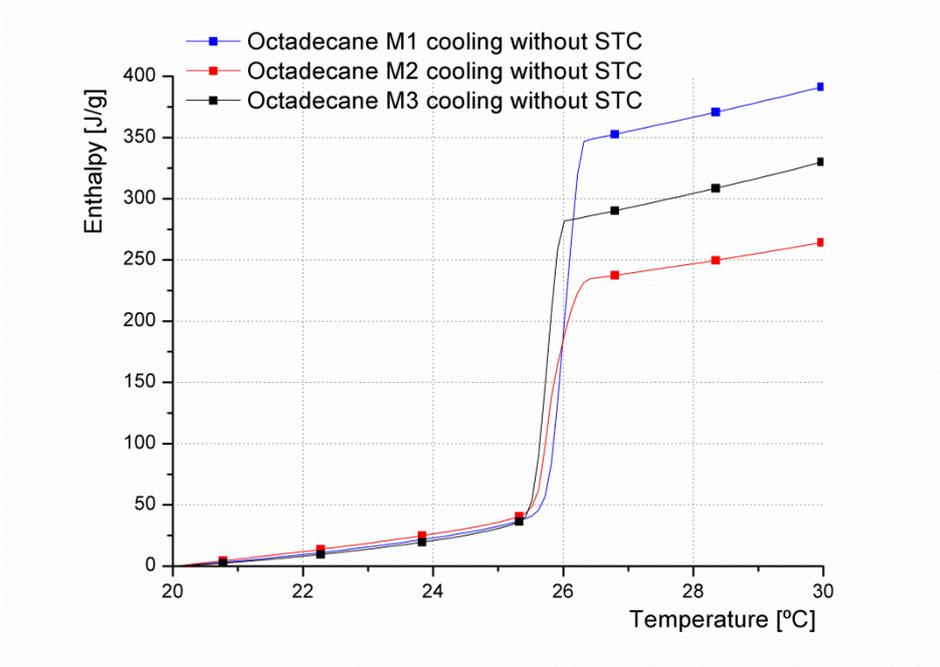


Figure 23: Comparison of the cooling results from the different samples for the NETZSCH DSC at the University of Zaragoza, Spain.

### 3 T-History Measurement

#### 3.1 Experimental Setup and Measuring Procedure

Investigation on T-History method was undertaken by KTH Sweden. The KTH in-house built T-history setup consists of three identical sample holders insulated in 15 mm thick polyurethane foam. The sample holders are 20 mm diameter test tubes measuring 250 mm in height. Temperature of the samples and temperature of the climate chamber were measured with 4-wire PT100 RTD sensors. The complete setup was placed inside a humidity and temperature controlled climate chamber and the data were recorded with 24 bit Keithely 2701 data logger to a computer. The T-History setup and the schematic are shown in Figure 24.

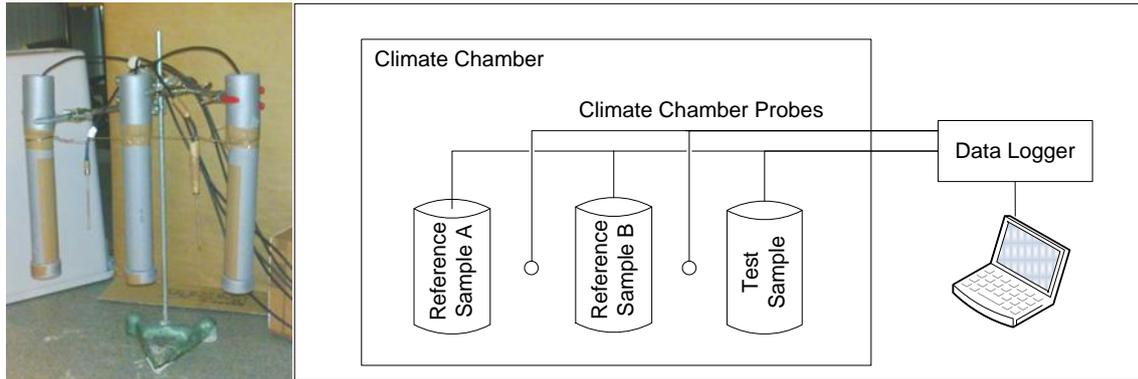


Figure 24: T-history setup (left) and Schematic (right)

The obtained data were temperature measurements over the testing period. Data processing methods leading to enthalpy and specific heat capacity have been proposed by many prominent researchers [5][6][7]. The approach utilized in concatenating input heat flux to that of temperature variation between the environment (climate chamber) and the ref/test samples is shown in Eq. 1 and Eq. 2.

$$\dot{Q}_{\text{ref}} = k_1 \cdot \text{lmtd}_{\text{ref}}^2 + k_2 \cdot \text{lmtd}_{\text{ref}} \quad \text{Eq. 1}$$

$$\dot{Q}_{\text{pcm}} = k_1 \cdot \text{lmtd}_{\text{pcm}}^2 + k_2 \cdot \text{lmtd}_{\text{pcm}} \quad \text{Eq. 2}$$

where  $k_1$  and  $k_2$  are constants

The log mean temperature differences are shown in Eq. 3 and Eq. 4.

$$\text{lmtd}_{\text{ref}} = \frac{(T_{\text{ref}}^{n-1} - T_{\text{chamber}}^{n-1}) - (T_{\text{ref}}^n - T_{\text{chamber}}^n)}{\ln\left[\frac{(T_{\text{ref}}^{n-1} - T_{\text{chamber}}^{n-1})}{(T_{\text{ref}}^n - T_{\text{chamber}}^n)}\right]} \quad \text{Eq. 3}$$

$$\text{lmtd}_{\text{pcm}} = \frac{(T_{\text{pcm}}^{n-1} - T_{\text{chamber}}^{n-1}) - (T_{\text{pcm}}^n - T_{\text{chamber}}^n)}{\ln\left[\frac{(T_{\text{pcm}}^{n-1} - T_{\text{chamber}}^{n-1})}{(T_{\text{pcm}}^n - T_{\text{chamber}}^n)}\right]} \quad \text{Eq. 4}$$

where  $T_{n-1}$  and  $T_n$  depict the sampled temperatures at instant  $n-1$  and  $n$ .

Detailed calculation procedure is available in [5].

The material used was 98% pure Octadecane received from IEA ECES Annex 24/Task 42 Working Group A2. The measurements were performed over three freezing/melting cycles. The duration of each cycle was set to 24 hours in both heating and cooling mode.

An intermediate temperature stage was introduced at 32°C in heating and 23°C in freezing (4.5°C above and below the phase change temperature) so as to create a dynamic step mode test which reduces the heat flux to the samples during the phase change process and hence improves the accuracy of the measurement. The tested Octadecane sample mass was  $37.5 \pm 0.1$  g.

The RTD sensors used were calibrated class B PT100. The error analysis performed follows the procedure as proposed by Moffat [10]. Error analysis shows that the obtained enthalpy reading is with an error of  $\pm 2.4\%$ .

### 3.2 Results and Discussion

The average Biot number in this experiment is below 0.1. The enthalpy obtained with T-history method for one complete cycle is shown in Figure 2. Both enthalpies obtained in freezing and in melting cycles are set at reference temperature of 20°C. The enthalpy change from 20°C to 30°C is  $225 \pm 5$  kJ/kg for melting and  $240 \pm 6$  kJ/kg for freezing. A possible explanation for the difference in melting and in freezing enthalpies is shown later in section **Fout! Verwijzingsbron niet gevonden..** With the T-history method, the phase change temperature range measured for melting and freezing is from 27.2°C to 27.7°C. A subcooling of 1.1°C marked the sudden increase in temperature from 26.6°C to 27.7°C in the cooling cycle.

The enthalpy change from 20°C to 30°C obtained with DSC measurements (240-260 kJ/kg), Figure 3 [11], lies slightly above that measured with T-history method (225-240 kJ/kg). Different degrees of hysteresis from 1°C to 3°C were obtained with various DSCs at different heat fluxes, while no significant hysteresis was seen with T-history.

The majority of the DSC measurements did not show noticeable subcooling with the tested sample. Similar subcooling phenomenon was obtained at ILK with DSC-Q200 with 0.4 K/min cooling rate from 25.8°C to 26.2°C, Figure 3.

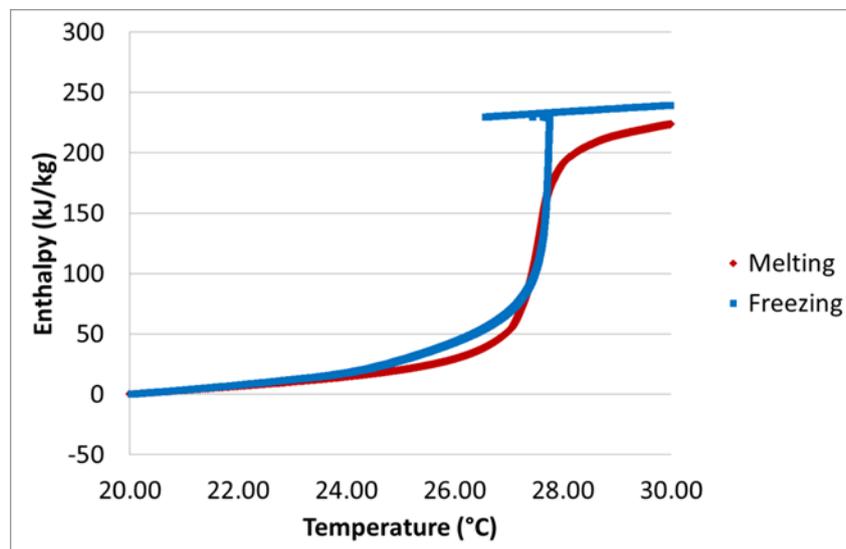


Figure 25: Enthalpy of Octadecane Characterized with T-History Method performed at KTH.

### 3.3 Gravitational effect on T-History Method

From the study performed in section **Fout! Verwijzingsbron niet gevonden.**, it was shown that enthalpy change may differ depending on the heat transfer direction (freezing or melting) of the measurement performed. A hypothesis is proposed for this observation and preliminary study will be carried out.

T-history method is based on Lumped Capacitance model where the temperature gradient inside the test material is considered small. This condition is valid when Biot number is smaller than 1, eq. (1).

$$Bi = \frac{\text{Heat Transfer Coefficient} \times \text{Characteristic Length}}{\text{Thermal Conductivity}} = \frac{U \cdot L}{k} \ll 1 \quad (1)$$

The validity of the criterion is however an approximation as Biot number leans towards zero but never reaches zero. While Biot number is non-zero, a temperature gradient will always exist in the sample. The temperature gradient is reflected by separation of solid phase to liquid phase, e.g. a non-insulated melting cycle is shown in Figure 4. Here the solid phase with higher density deposits at the bottom and the liquid phase with lower density stays afloat due to buoyancy.

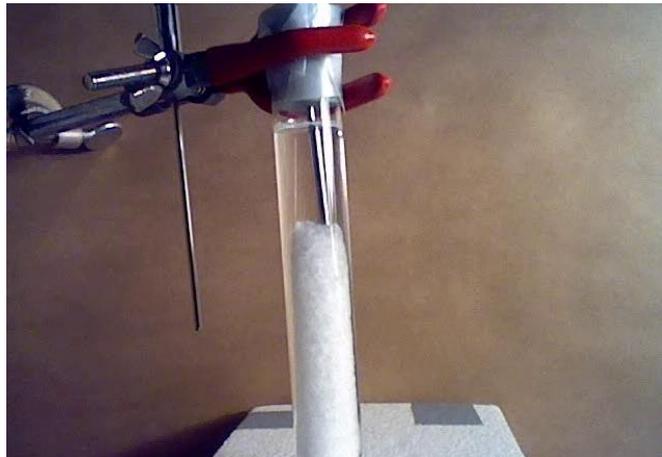


Figure 26: Phase Separation during Melting of Octadecane

It is suspected by the authors that the separation of phases due to buoyancy effect generates non-uniformity in temperature distribution where the upper part is at a higher temperature and the lower part at a lower temperature. Depending on the height at which the temperature probe is placed, the retrieved temperature data will either be too low or too high as compared to the average sample temperature. The objective of this part of work is to compare the T-History method with a vertically placed setup to that of a horizontally placed disposition.

### 3.4 Experimental Setup and Measuring Procedure

Two in-house built T-history setups were manufactured, Figure 5 and Figure 6. Both setups consist of a 30 mm thick polyurethane box that holds the sample containers in place and reduces convective heat transfer from the climate chamber to the samples. The temperature probes are 4-wire RTD PT100, the measuring chain is identical to the one described in section 1.1. Two sensors are placed inside the box for air temperature measurement and three are placed inside the samples. The mass of Octadecane sample placed in the vertical setup amounts to  $30.9 \pm 0.1$  g, and the mass of Octadecane sample placed in the horizontal setup amounts to  $31.9 \pm 0.1$  g.

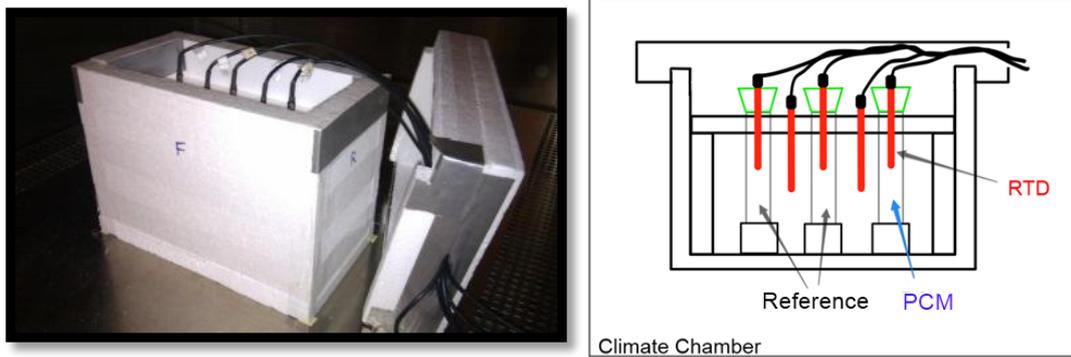


Figure 27: Vertical T-History Setup

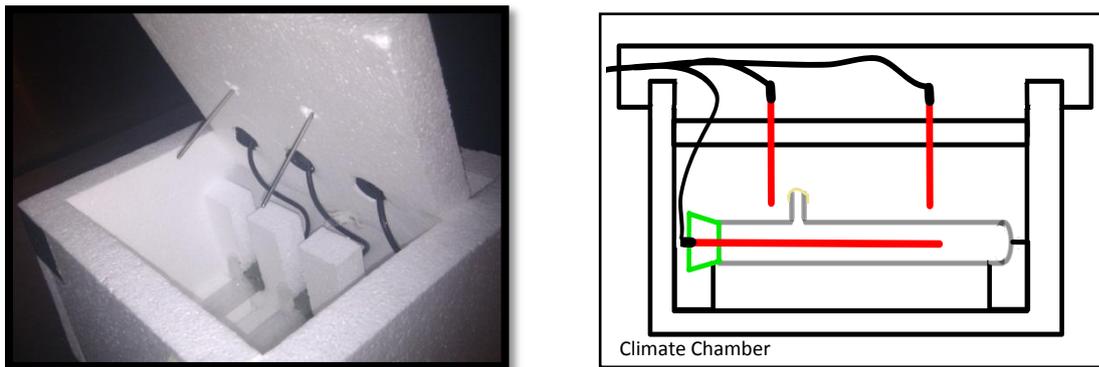


Figure 28: Horizontal T-History Setup

### 3.5 Results and Discussion

The obtained enthalpy graphs for the 98% pure Octadecane over the range of 20°C to 30°C are shown in Figure 29. The results shown are averaged melting and freezing enthalpies.

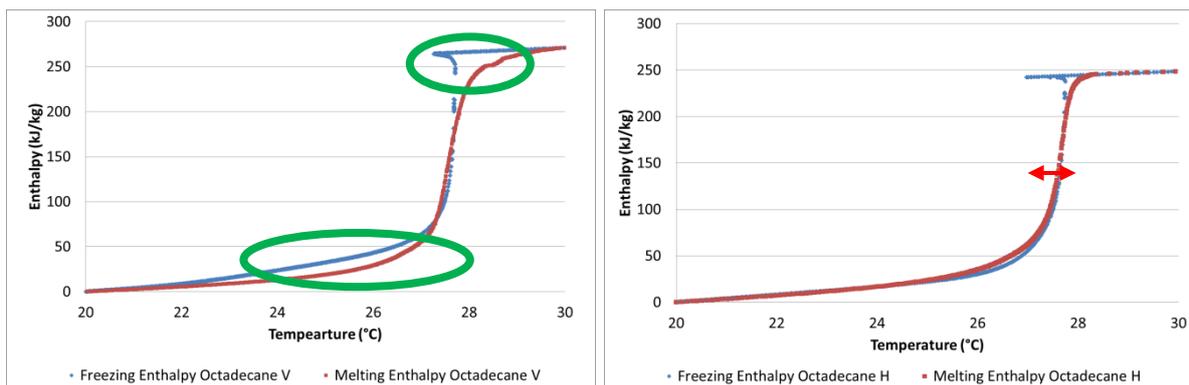


Figure 29: Enthalpy Measurement with Vertical Setup (left) and Horizontal Setup (right)

It is seen from the vertical setup that the freezing and melting enthalpy trend show discrepancies in the solid and in the liquid phases (encircled in green), while the horizontal setup shows more matching results, Figure 29 right. In comparison to the large hysteresis obtained with DSC in the Round Robin tests, c.f. **Fout!**

**Verwijzingsbron niet gevonden.**; the melting and freezing enthalpy during phase change for both vertical and horizontal T-History setups are within 0.1°C difference.

The total enthalpy changes between the vertical and the horizontal setups in the range of 20°C to 30°C are however different. The vertical setup here shows a 9% higher enthalpy (270 kJ/kg) than that of the horizontal setup (248 kJ/kg) and reaches 20% higher value than that obtained with the first vertical setup presented in section 1 (225-240 KJ/kg). The discrepancy between two vertical setups further supports the effect of Buoyancy on T-History measurement.

### 3.6 Conclusion

T-history method is shown to be an alternative method in giving enthalpy measurement of a PCM. The phase change temperature is sharper than that obtained with DSC. No significant hysteresis is observed. Subcooling is well characterized. However, enthalpy change obtained in melting and freezing cycles reached 10% difference. An explanation and an improvement to the T-History method is proposed in the following section.

It is shown through this study that T-history method provides an alternative means for characterizing enthalpy of a PCM. The hysteresis observed with DSC based measurements is minimized with T-history characterization. The phase change temperature for the tested 98% pure Octadecane lies between 27.2°C and 27.7°C, this temperature range is much sharper than that obtained with DSC possibly due to larger sample volume. The enthalpy change from 20°C to 30°C amounts to 248 kJ/kg based on the horizontal T-History setup. It is also shown from this work that horizontally placed samples are recommended over vertical setup for the reason of Buoyancy reduction.

#### Acknowledgement

The authors would like to acknowledge Swedish Energy Agency for funding this project.

## 4 TCM-Stability

A principal problem encountered in case of the commercially available samples is the hydrothermal stability of these materials. Unfortunately, all evaluated materials show a strong degradation within only 20 cycles. As shown in the figure below, the samples were treated between 140°C and 40°C under humid conditions (red curve). Already after the first full cycle, which means one adsorption and one desorption the initial weight and therefore the initial loading cannot be reached anymore (green curve). The progressive degradation is clearly visible within the next full cycles.

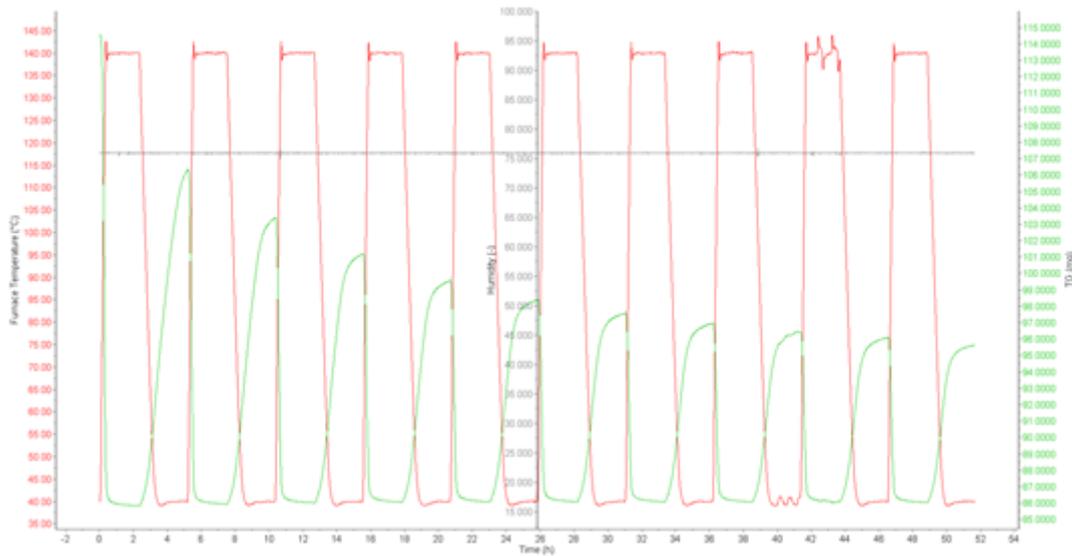


Figure 30: Progressive degradation of a commercial available MOF material under hydrothermal conditions. (Source : [12])

In addition to the novel class of metal organic frameworks, in a close collaboration with the KIC (National Institute of Chemistry, Ljubljana), a small-pore microporous aluminophosphate was evaluated for solar energy storage with promising results. [14]

#### 4.1 Comparative Thermo-Gravimetric measurements

Comparative measurements on the two selected reference materials (zeolite 4 ABF and 13XBF by CWK) were performed by Jochen Jänchen TH Wildau and Fabian Fischer ZAE Bayern.

First results are promising; however there are still differences between the various measurement apparatus and methods visible.

The transformation of the data into a proper thermodynamic model, in this case according to Dubinin, shows a good correlation between thermogravimetric measurements, but a strong deviation compared to volumetric measurements (see figure below). Further investigations are necessary.

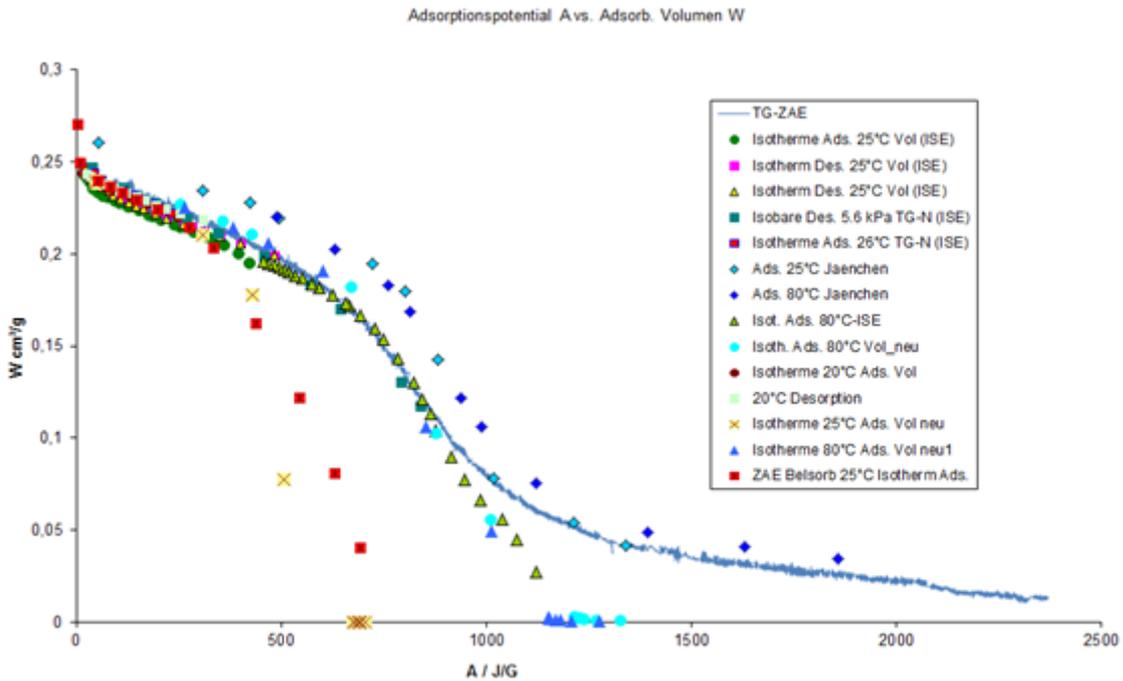


Figure 31: Overall comparison of RRT measurements performed at different labs (TH Wildau, ZAE Bayern, ISE Freiburg) with different apparatus.

However, the direct comparison between volumetric measurement at ISE and ZAE with two different apparatus (Hydrosorb vs. Belsorb) shows an excellent agreement (see figure below).

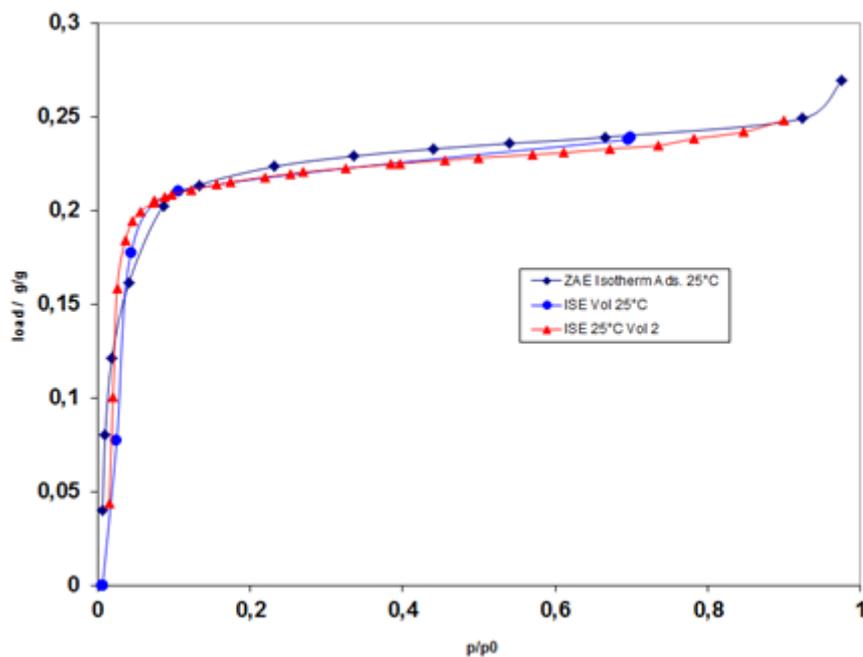


Figure 32: Direct comparison of volumetric measurements at ISE and ZAE on the reference material.

## 5 Literature

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## IEA Solar Heating and Cooling Programme

The *International Energy Agency* (IEA) is an autonomous body within the framework of the Organization for Economic Co-operation and Development (OECD) based in Paris. Established in 1974 after the first “oil shock,” the IEA is committed to carrying out a comprehensive program of energy cooperation among its members and the Commission of the European Communities.

The IEA provides a legal framework, through IEA Implementing Agreements such as the *Solar Heating and Cooling Agreement*, for international collaboration in energy technology research and development (R&D) and deployment. This IEA experience has proved that such collaboration contributes significantly to faster technological progress, while reducing costs; to eliminating technological risks and duplication of efforts; and to creating numerous other benefits, such as swifter expansion of the knowledge base and easier harmonization of standards.

The *Solar Heating and Cooling Programme* was one of the first IEA Implementing Agreements to be established. Since 1977, its members have been collaborating to advance active solar and passive solar and their application in buildings and other areas, such as agriculture and industry. Current members are:

Australia	Finland	Singapore
Austria	France	South Africa
Belgium	Italy	Spain
Canada	Mexico	Sweden
Denmark	Netherlands	Switzerland
European Commission	Norway	United States
Germany	Portugal	

A total of 49 Tasks have been initiated, 35 of which have been completed. Each Task is managed by an Operating Agent from one of the participating countries. Overall control of the program rests with an Executive Committee comprised of one representative from each contracting party to the Implementing Agreement. In addition to the Task work, a number of special activities—Memorandum of Understanding with solar thermal trade organizations, statistics collection and analysis, conferences and workshops—have been undertaken.

Visit the Solar Heating and Cooling Programme website - [www.iea-shc.org](http://www.iea-shc.org) - to find more publications and to learn about the SHC Programme.

**Current Tasks & Working Group:**

- Task 36 *Solar Resource Knowledge Management*
- Task 39 *Polymeric Materials for Solar Thermal Applications*
- Task 40 *Towards Net Zero Energy Solar Buildings*
- Task 41 *Solar Energy and Architecture*
- Task 42 *Compact Thermal Energy Storage*
- Task 43 *Solar Rating and Certification Procedures*
- Task 44 *Solar and Heat Pump Systems*
- Task 45 *Large Systems: Solar Heating/Cooling Systems, Seasonal Storages, Heat Pumps*
- Task 46 *Solar Resource Assessment and Forecasting*
- Task 47 *Renovation of Non-Residential Buildings Towards Sustainable Standards*
- Task 48 *Quality Assurance and Support Measures for Solar Cooling*
- Task 49 *Solar Process Heat for Production and Advanced Applications*

**Completed Tasks:**

- Task 1 *Investigation of the Performance of Solar Heating and Cooling Systems*
- Task 2 *Coordination of Solar Heating and Cooling R&D*
- Task 3 *Performance Testing of Solar Collectors*
- Task 4 *Development of an Insolation Handbook and Instrument Package*
- Task 5 *Use of Existing Meteorological Information for Solar Energy Application*
- Task 6 *Performance of Solar Systems Using Evacuated Collectors*
- Task 7 *Central Solar Heating Plants with Seasonal Storage*
- Task 8 *Passive and Hybrid Solar Low Energy Buildings*
- Task 9 *Solar Radiation and Pyranometry Studies*
- Task 10 *Solar Materials R&D*
- Task 11 *Passive and Hybrid Solar Commercial Buildings*
- Task 12 *Building Energy Analysis and Design Tools for Solar Applications*
- Task 13 *Advanced Solar Low Energy Buildings*
- Task 14 *Advanced Active Solar Energy Systems*
- Task 16 *Photovoltaics in Buildings*
- Task 17 *Measuring and Modeling Spectral Radiation*
- Task 18 *Advanced Glazing and Associated Materials for Solar and Building Applications*
- Task 19 *Solar Air Systems*
- Task 20 *Solar Energy in Building Renovation*
- Task 21 *Daylight in Buildings*
- Task 22 *Building Energy Analysis Tools*
- Task 23 *Optimization of Solar Energy Use in Large Buildings*
- Task 24 *Solar Procurement*
- Task 25 *Solar Assisted Air Conditioning of Buildings*
- Task 26 *Solar Combisystems*
- Task 27 *Performance of Solar Facade Components*
- Task 28 *Solar Sustainable Housing*
- Task 29 *Solar Crop Drying*
- Task 31 *Daylighting Buildings in the 21<sup>st</sup> Century*
- Task 32 *Advanced Storage Concepts for Solar and Low Energy Buildings*
- Task 33 *Solar Heat for Industrial Processes*
- Task 34 *Testing and Validation of Building Energy Simulation Tools*
- Task 35 *PV/Thermal Solar Systems*
- Task 37 *Advanced Housing Renovation with Solar & Conservation*
- Task 38 *Solar Thermal Cooling and Air Conditioning*

**Completed Working Groups:**

*CSHPSS; ISOLDE; Materials in Solar Thermal Collectors; Evaluation of Task 13 Houses; Daylight Research*



## IEA Energy Conservation Through Energy Storage Programme

The *International Energy Agency* (IEA), based in Paris, is an autonomous agency linked with the *Organisation for Economic Co-operation and Development* (OECD). The IEA is the energy forum for 26 Member countries. IEA Member governments are committed to taking joint measures to meet oil supply emergencies. They have also agreed to share energy information, to co-ordinate their energy policies and to co-operate in the development of rational energy programmes.

The R&D programme *Efficient Energy End-Use Technologies* contains 14 different *Implementing Agreements* (IAs) of which one is the IA on energy storage. The full name of this IA is *Energy Conservation through Energy Storage* (ECES IA). This IA was founded in 2004 and intends to promote co-operative research, development, demonstrations and exchanges of information regarding energy conservation through energy storage.

The continued development, application and deployment of energy efficient end-use technologies has the potential to significantly reduce energy consumption and greenhouse gases in the buildings, electricity generation, industry, and transport sectors. Energy storage technologies can overcome the temporal mismatch between energy supply and demand, especially regarding renewable energy technologies, the use of waste energy and energy from ambient sources such as cold from the natural environment.

Active participants in ECES IA are at present:

Belgium	Germany	Sweden
Canada	Italy	USA
China	Japan	Turkey
Finland	Korea	
France	Norway	

Three sponsors are participating in the ECES at the moment:

IF Technology B.V. (Netherlands)  
 Institute of Heat Engineering, University of Technology of Warsaw (Poland)  
 Energesis Ingeniería, S.L. (Spain).

Other countries have signed the agreement but are not presently engaged in the activities. Further countries have recently shown interest in participating in the near future, including countries from Eastern Europe.

The work within the ECES IA is lead by an *Executive Committee* (XC) and the work undertaken by this XC is done by mutual agreements defined in Annexes as listed on the next page. The work is led by Chairman Halime Paksoy (Turkey) and Secretary Hunay Evliya (Turkey).

To find ECES publications and learn more about the Programme visit <http://www.iea-eces.org> or contact the ECES Secretariat, Hunay Evliya, e-mail: hevliya@cu.edu.tr

## Ongoing Annexes:

- Annex 20 *Sustainable Cooling with Thermal Energy Storage*
- Annex 21 *Thermal Response Test for Underground Thermal Energy Storages*
- Annex 22 *Thermal Energy Storage Applications in Closed Greenhouses*
- Annex 23 *Applying Energy Storage in Ultra-low Energy Buildings*
- Annex 24 *Material Development for Improved Thermal Energy Storage Systems*
- Annex 25 *Surplus Heat Management using Advanced TES for CO<sub>2</sub> mitigation*
- Annex 26 *Electric Energy Storage: Future Energy Storage Demand*

## Completed Annexes:

- Annex 1 *Large Scale Thermal Storage Systems Evaluation*
- Annex 2 *Lake Storage Demonstration Plant in Mannheim*
- Annex 3 *Aquifer Storage Demonstration Plant in Lausanne Dorigny*
- Annex 4 *Short Term Water Heat Storage Systems*
- Annex 5 *Full Scale Latent Heat Storage Installations*
- Annex 6 *Environmental and Chemical aspects of Thermal Energy Storage in Aquifers and Research and Development of Water Treatment Methods*
- Annex 7 *Innovative and Cost Effective Seasonal Cold Storage Applications*
- Annex 8 *Implementing Underground Thermal Energy Storage Systems*
- Annex 9 *Electrical Energy Storage Technologies for Utility Network Optimization*
- Annex 10 *Phase Change Materials and Chemical Reactions for Thermal Energy Storage*
- Annex 12 *High-Temperature Underground Thermal Energy Storage (HT UTES)*
- Annex 13 *Design, Construction and Maintenance of UTES Wells and Boreholes*
- Annex 14 *Cooling with TES in all Climates*
- Annex 17 *Advanced Thermal Energy Storage Techniques – Feasibility Studies and Demonstration Projects*
- Annex 18 *Transportation of Thermal Energy Utilizing Thermal Energy Storage Technology*
- Annex 19 *Optimised Industrial Process Heat and Power Generation with Thermal Energy Storage*

