# Events Calendar

## Main Training courses

<table>
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<tr>
<th>Subject of the training</th>
<th>Dates for session in English</th>
<th>Dates for session in French</th>
</tr>
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<tbody>
<tr>
<td>Thermogravimetry applied to Setsys TG</td>
<td>June 1-3, 2005</td>
<td>March 16-18, 2005</td>
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<tr>
<td>DSC (DSC131)</td>
<td>October 1-21, 2005</td>
<td>September 14-16, 2005</td>
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<tr>
<td>MicroDSC (models II and VII)</td>
<td>December 8-9, 2005</td>
<td>November 17-18, 2005</td>
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<tr>
<td>Colorimetry applied to C80</td>
<td>March 9-1, 2005</td>
<td>January 19-21, 2005</td>
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Other sessions are scheduled. Do not hesitate to contact us: sales@setaram.com

## Exhibitions Conferences 2005

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<tr>
<th>FEBRUARY</th>
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<tr>
<td>Magyaregula</td>
<td>Feb 23 - 25</td>
<td>Budapest, Hungary</td>
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<tr>
<td>PITCON</td>
<td>Feb 27 - March 3</td>
<td>Orlando, FL - USA</td>
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<tr>
<td>ACS</td>
<td>March 13 - 17</td>
<td>San-Diego, CA - USA</td>
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<tr>
<td>Les Journées Annuelles du GFC</td>
<td>March 15 - 17</td>
<td>Paris, France</td>
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<tr>
<td>Ulf-Freiburger Kalorimetriege</td>
<td>March 16 - 18</td>
<td>Freiberg, Deutschland</td>
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<tr>
<td>CPE Stabilité des principes actifs et produits formulés</td>
<td>March 16 - 18</td>
<td>Lyon, France</td>
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<tr>
<td>XXXI JEEP Journées d’Etude des Équilibres entre Phases</td>
<td>March 31 - April 01</td>
<td>Barcelona, Spain</td>
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<tr>
<th>MARCH</th>
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<tr>
<td>TAC 2005</td>
<td>April 6 - 7</td>
<td>Norwich, UK</td>
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<td>ACERES</td>
<td>April 10 - 14</td>
<td>Baltimore, MD - USA</td>
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<td>RENACES</td>
<td>April 27 - 29</td>
<td>Meknes, Morocco</td>
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<td>European Symposium</td>
<td>April 30 - May 04</td>
<td>Barcelona, Spain</td>
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<td>of the Protein Society</td>
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<th>APRIL</th>
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<tr>
<td>Offshore Technology</td>
<td>May 1 - 5</td>
<td>Houston, TX - USA</td>
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<tr>
<td>JCAT 2005</td>
<td>May 17 - 19</td>
<td>Rouen, France</td>
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<td>WASTEENG 05</td>
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<td>Albi, France</td>
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<td>ENMAC</td>
<td>May 24 - 27</td>
<td>Basel, Switzerland</td>
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<td>North Amer. Catalysis Society Meeting</td>
<td>May 22 - 27</td>
<td>Philadelphia, PA - USA</td>
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<th>MAY</th>
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<tr>
<td>IWPCPS</td>
<td>June 5 - 10</td>
<td>Kona, Hawaii - USA</td>
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<tr>
<td>GAS HYDRATES 2005</td>
<td>June 13 - 16</td>
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<td>CALCON</td>
<td>June 27 - 30</td>
<td>Gothenburg, MD - USA</td>
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<th>JUNE</th>
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<tr>
<td>MEDICTA</td>
<td>July 2 - 6</td>
<td>Tsaldomi, Greece</td>
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<td>IFT</td>
<td>July 16 - 20</td>
<td>New Orleans, IA - USA</td>
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<tr>
<th>JULY</th>
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<tr>
<td>SETARAM SA</td>
<td></td>
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<td>7, rue de l’Oraïre</td>
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<td>69300 Colomiers - France</td>
<td>Phone +33(0)4 72 10 25 25</td>
<td>Fax +33(0)4 78 26 63 55</td>
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<td>SETARAM Inc.</td>
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<td>Fax +1 856 778 7377</td>
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Setaram is the best solution designed to your needs, providing a high level expertise in thermal analysis.
* Equipment, Parts and Accessories
* Technical Support
* Test Applications
* Training (see planning)
* Application and Publications Library

Sales offices in UK, Germany, Italy
sales@setaram.com / www.setaram.com
Dear Readers,

Day after day, you in the research laboratories invent the world of tomorrow. Whether by understanding phenomena, improving the properties of materials, or developing new processes that are safer and more environmentally-friendly, your research work helps fit together the pieces of the never-ending puzzle of science. Setaram Instrumentation has been taking part in this adventure for more than 50 years, proposing innovative standard or tailored solutions in the field of thermal analysis and calorimetry.

Thanks to you the world is changing, and we have to adapt.

Today we have decided to better anticipate your needs by increasing our Marketing action. The aim is to offer you solutions that are adapted to your field of application, be it pharmaceutical, biology, oil, nuclear power, metallurgy, ceramics, etc. We also want to better serve you by reinforcing our Alter-Sales Service and its worldwide network. Our mission is to accompany you throughout the lifetime of the instrument you have acquired.

We have decided to mark this new phase by revamping our magazine. The ‘Setaram News’ you knew in the past is now changing face and name to become ‘Thermal Analysis SOLUTIONS by SETARAM Instrumentation’. We have endeavored to ensure that it is just as informative but more stimulating reading. In short, a true magazine!

Another important piece of news is that Setaram Instrumentation has entered into a partnership with the Swiss company AKTS Software, leader in the field of kinetic analysis software and process safety. Thanks to this partnership we can now offer a global solution for material stability studies.

I will close this editorial by wishing you enjoyable reading. If you would like to receive our magazine regularly (and free of charge), please contact us at the following e-mail address: sales@setaram.com or via our web site: www.setaram.com.

Gilles WIDAWSKI
Managing Director

SETARAM Instrumentation has been developing and selling "large volume" calorimeters for the characterization of nuclear materials, and more particularly the analysis of radioactive waste and quantification of radioactive elements (e.g. curium, plutonium, tritium). These instruments are also used to measure the energy of hard gamma and neutron emitting sources in shielded containers. At the end of 2003, the Military Application Department ordered two large-volume calorimeters capable of accommodating 60- and 90-liter containers, for the CEA (Atomic Energy Commission) center of Valduc, France. These calorimeters, developed and supplied by SETARAM Instrumentation in 2004, are to be used to measure a mass of radioactive gas contained in sealed metal containers.

Thanks to their excellent sensitivity, these calorimeters can accurately measure very low thermal effects (less than 10 µW/litre, that is to say 10 nW/ml), which makes them powerful working tools for nuclear applications. The calorimeters, which conform to EC standards, are placed in glove boxes under controlled atmosphere. The construction materials are chosen for their good fire resistance and the fact that they can be easily decontaminated. All the electronic unit ensuring temperature control, heating power and data acquisition, can be remotely positioned outside the "contaminating zone".

1. General description of the calorimeters:

The two calorimeters are of identical design, differing only in their dimensions and performance.

### CALORIMETERS SPECIFICATIONS

**390 Calorimeter**

- **Reference measuring cells**: 390, 680
- **Internal volume**: 60 liters, 90 liters
- **Closed calorimeter**: 960 mm, 1260 mm
- **Height**: 1500 mm, 1500 mm
- **Depth**: 1000 mm, 1000 mm
- **Approximate weight**: 1000 kg, 1200 kg

**Performance**

- **Detection limit**: 45 µW, 97 µW
- **Measurement range**: 0.5 - 13000 mW, 1 - 26000 mW
- **Sensitivity**: 0.60 µW/mW, 1.55 µW/mW
- **Signal stabilization time**: 30 hours
- **Working temperature**: 20°C

**490 Calorimeter**

- **Reference measuring cells**: 490, 780
- **Internal volume**: 90 liters, 120 liters
- **Closed calorimeter**: 1020 mm, 1320 mm
- **Height**: 1500 mm, 1500 mm
- **Depth**: 1000 mm, 1000 mm
- **Approximate weight**: 1500 kg, 1800 kg

**Performance**

- **Detection limit**: 45 µW, 97 µW
- **Measurement range**: 0.5 - 13000 mW, 1 - 26000 mW
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- **Signal stabilization time**: 30 hours
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The six sides of each cavity are covered with fluxometric plates that can pick up an electric signal (emf) proportional to the energy evolved inside the measuring chamber. The arrangement of the Peltier modules on all the surfaces enables virtually all the heat flux produced inside the cell and passing to the exterior to be integrated. The calorimeters are made up of three chambers fitted inside one another.
The inner chamber comprises the two cells (measurement and reference) on which the fluxmeters are placed. The temperature of this chamber is maintained by laminar heaters that are regulated by several platinum probes.

The intermediate chamber consists solely of aluminum plates and insulating plates providing protection against external disturbances and homogenized the temperature gradients in the center of the calorimeters.

The external chamber, the technical principle of which is identical to that of the intermediate chamber, carries a water circulation circuit consisting of copper tubes on its external surface. This circuit constitutes a stable controlled-temperature thermal potential to minimize any external thermal disturbances.

Access to the measurement and reference chambers is via a lid consisting of an alternation of metal plates, insulating plates and a water circuit. The lid is opened by two low-voltage electric actuators each developing a force of 6000 N. Opening is by a dual drive mechanism that affects a vertical translation movement followed by rotation.

3. Experimental results:

Our calorimeters have been validated by the dissipation of different thermal powers using special metallic cells called JE (Joule Effect) cells. The difference between the power actually measured and the setpoint power is less than 0.3%.

The following performance levels were verified:
- Signal stabilization time
- Signal stability over a Joule effect
- Long-term stability (48h) of the signal on a blank test
- Reproducibility of blank tests and JE measurements
- Detection limit

Signal stability is based on the static notion of the relative standard deviation of the signal with respect to its mean value (calculated from a minimum of 600 points over a period of 6 hours). A Joule effect produced at a power of 200 mW in the 390 calorimeter was used to calculate a sensitivity of about 160 µV/mW, confirms that the base line before the Joule effect is stable with a relative standard deviation of less than 0.23% and that the return to the base line after the Joule effect is highly satisfactory (difference of 0.14% with respect to the average).

The background noise (peak-to-peak) observed on this same calorimeter over a period of 7 hours (about twice the time constant of the calorimeter) is 30 µV, that is to say a background noise value of less than 200 µW for a cell volume of 60 liters.

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Calorimeter opening mechanism

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The study of the active principle (AP):
The study of the AP associated with a compatible excipient. Each of these forms must be studied in detail in order to determine the forms to use in the composition of the drug and in its production and production process. Thanks to SETARAM’s products, the following properties and phenomena can be studied:

- Hydratation, water content (with a SETARAM Evolution thermobalance coupled with a Wetsys humidity generator).
- Crystalinity, polymorphism (with micro DSC, C80 calorimeter, TSC II).
- Amorphy, amorphous form quantification (with the micro DSC, C80 microcalsorimeter, TSC, etc.).
- Purity, stoichiometry, solubility (with the micro DSC, the C80, etc.).
- Stability (with the micro DSC, the C80).

Other analysis techniques are used, but we will focus on detailing the use of thermal analysis:

- The choice of a compatible excipient with the AP.
- The number and the nature of the analyses to perform are similar to those for the AP.

The study of the AP associated with a compatible excipient, or the study of the drug in its definitive form.

Dissolution characteristics, stability according to temperature or relative humidity, AP-excipient compatibility: all these aspects can be studied using Setaram instruments such as the micro DSC, the C80, the Wetsys, or the TSC II.

Furthermore, most of these instruments allow the drug to be analyzed in its final form, without any kind of preparation (for example in pill, capsule or powder form, etc.).

In the following chapter you will find the main applications of thermal analysis for pharmaceutical products.

**For which applications is thermal analysis used?**

**Influence of humidity**
The water content of a pharmaceutical compound is an important property to characterize and quantify. This can be carried out quite easily with a thermobalance (Setsys Evolution). This technique enables the weight loss of the sample and the speed of weight loss to be measured as a function of time or temperature.

**Fig.1. The different forms of AP**

All drugs have to be stored during their life cycle. The stability of the product must therefore be studied under a controlled-humidity atmosphere. Given that, wherever you are, the ambient air always contains some moisture – even if only a very small amount – the phenomenon of hydration must always be considered.

To do this, Setaram proposes a complete system comprising a SETSYS Evolution thermobalance coupled with a WETSYS relative humidity generator that is capable of controlling levels of humidity of between 5 and 95% within the thermobalance analysis chamber. Likewise, the Wetsys can be coupled to any measuring instrument in which a controlled humidity atmosphere has to be generated.

**Compatibility between an active principle (AP) and its excipient (E)**

When a pill containing an active principle is prepared, it is vital to have full compatibility between the active principle and its excipient in the months preceding its ingestion by the patient. AP/E compatibility is often studied using a micro DSC III instrument. Figure 2 shows the thermograms of a single active principle associated with two different excipients, thus simulating their behavior during storage. The red curve is characteristic of poor AP/E compatibility as an interaction is observed between the active principle and the excipient. Conversely, the black curve is characteristic of good compatibility as no effect is observed. It is to be noted that this type of application requires the use of a highly sensitive instrument (signals of just a few microvolts are measured capable of analyzing relatively voluminous samples (1ml, for example).

**Phase diagram**

Lorenz et al. states that "the detailed knowledge of fundamental solid-liquid equilibrium (SLE) data expressed in phase diagrams is essential for designing and optimizing crystallization processes."

A particular advantage of the SETARAM DSC 111 device with regard to solubility measurements is the comparatively large sample amount usable, which is up to 150 mg in closed stainless steel crucibles and 250 ml in aluminum ones. This is important, because the low heating rates required to achieve equilibrium measurements generally lead to poor signal-to-noise ratios, when the heat loss / consumption rate is low, as is the case in non-isothermal dissolution experiments."

**Polymorphism**

When a pharmaceutical compound is capable of existing in several crystalline forms that share the same primary structure, it is said to display polymorphism. This phenomenon is often studied by DSC, which enables the different polymers to be distinguished by their fusion or by phase transitions. It is nevertheless sometimes impossible to distinguish polymorphs using this method. An alternative is to use their heat of dissolution using a C80 calorimeter, for example.

Figure 4 shows the curves corresponding to the dissolution of two crystalline forms of a given pharmaceutical compound. The exothermic effects associated with the dissolution of each crystalline form have a heat of 49.37 J.g⁻¹ and 73.36 J.g⁻¹.

This shows that calorimetry can provide a solution when DSC is ineffective.

**Crystalline phase/amorphous phase**

Another way of studying crystalline and amorphous substances is to use the still too little-known technique of Thermo Stimulated Currents (TSC). This principle of this technique is to submit the sample to an electric field that reveals any molecular mobility within it. At specific temperatures, peaks associated with molecular mobility are observed and subsequently associated with a chemical sequence. Figure 5 thus shows the spectra obtained for two crystalline forms in a study carried out by the pharmaceutical group Sanofi-Aventis. These crystalline forms, which could not be distinguished by DSC, were detectable by TSC which revealed specific molecular mobility of a single crystalline lattice at low temperature.

Moreover, with any pharmaceutical compound synthesized in crystalline form one must ask whether an amorphous fraction might be present in this compound. Any such fraction must be quantified because it can influence the therapeutic role of the medication in terms of bioavailability. Owing to its unstable nature, the amorphous phase displays high molecular mobility and is therefore ideally suited for detection and quantification using TSC.

**Denaturation of proteins**

When a protein is heated it becomes denatured. Denaturation is an irreversible transformation of the molecule, characterized by an endothermic effect, which is sometimes followed by aggregation. This latter phenomenon, which is characterized by an exothermic reaction, results in precipitation.

Studying the denaturation of a protein solution is a very useful exercise because it provides information on the protein structure and thermal stability of the solution.

In biochemical and pharmaceutical applications, the protein solutions studied are often highly diluted, for reasons of cost. The associated endothermic reaction in such cases is extremely weak and generally cannot be observed by conventional DSC. The micro DSC III instrument is particularly well suited to studies of this nature because it is far more sensitive. Furthermore, it allows the study of samples in the form of diluted solutions, solids, powders or viscous liquids. The most appropriate instrument for studying very highly diluted solutions (less than 0.1% solutions) is the Nano DSC II, which has the highest sensitivity.

**Fig. 2. Study of the compatibility of two active principle + excipient formulations with micro DSC III**

**Fig. 3. DSC curves of different mixtures of mandelic acid enantiomers (2)**

**Fig. 4. Dissolution of two crystalline forms by C80**

**Fig. 5. Superposition of the complex TSC spectra of the hexagonal form (-) and the triclinic form (····) of Interfin®.**

**Fig. 6. Denaturation of a solution of Interlukin-2 by micro DSC IV**

**Fig. 7. Denaturation of a solution of Interlukin-2 by Nano DSC IV**
SETARAM Solutions for the pharmaceutical industry

Below is a brief description of each SETARAM instrument mentioned:

**Micro DSC III**
- Very high sensitivity DSC (20/120°C)
- For solids, liquids and dilute solutions
- Capable of handling in situ mixing, circulation of gases/liquids and batch samples
- Featuring the exclusive SETARAM three-dimensional sensor with Joule effect calibration for the most sensitive and precise calorimetric measurements
- With the Micro DSC III you can detect and measure precisely and accurately the subtlest transitions, which the standard DSC cannot.

**Calvet C80**
- Mixing and reaction calorimeter, ambient temperature/300°C
- Large sample volume, excellent sensitivity, ability to monitor/central pressure, etc.
- Featuring the exclusive SETARAM three-dimensional Calvet sensor with Joule effect calibration for the most precise and accurate calorimetric measurements
- You will also appreciate its titration and in situ mixing reaction capabilities.

**Calvet TG-DSC 111**
- 120/830°C, three-dimensional Calvet sensor, simultaneous TGA-DSC, Joule effect calibration
- With Calvet TG-DSC111 you benefit from both the exclusive SETARAM three-dimensional DSC sensor and the high-sensitivity thermobalance. You can characterize your material’s transitions and decomposition processes in just one experiment.

**SETSYS Evolution**
- DTA, DSC, TGA, TGA/DTA/DSC, TMA, TGA-EGA
- State-of-the-art modular thermal analyzer designed for the most demanding research applications
- Hang down design for best baseline performance and reproducibility, coupling systems for FTIR, MS, GC.

**Applications of Thermal Analysis at Eli Lilly**

**Indianapolis, IN - USA**

**Tera L. Deal & Bradley M. Campbell**

“Approximately two years ago, we purchased a Setaram micro DSC III and micro DSC VII, which quickly became two important assets to our laboratory. These microcalorimeters had several advantages over our competitor’s instrumentation. One advantage of the micro DSC III and VII were the removable cells, which allow for relatively easy sample preparation and cleaning, providing more time for laboratory experiments rather than housekeeping. Another advantage of these instruments is the excellent hardware and software. Because of its high sensitivity, stable baseline and low background noise, analysis only requires a small amount of costly sample, which keeps expenses at a minimum. Also, user-friendly software allows for quick turnaround of accurate results, which makes this instrumentation an essential productive laboratory tool.

Lastly, the universality of the micro DSC III and VII was another advantage. These instruments are capable of operating in isothermal mode, scanning mode or a combination of both in order to perform an experiment. Setaram has a variety of vessels available for more flexibility in the types of samples analyzed and the studies carried out.

In our laboratory, we currently have three different types of vessels: closed “batch”, heat capacity and mixing “batch” vessels. We primarily use the closed “batch” vessel because experiments containing either solid and/or liquid samples can be performed. Not only do we use different vessels, but we also use all of the modes to monitor samples (i.e., isothermal, scanning or a combination of both) depending on the project.

Because these instruments have enormous experimental flexibility, our laboratory is able to perform a wide assortment of experiments including: investigations of protein denaturing and aggregation, amorphous content determination, polymorph conversions, drug substance and product stability assessment, and drug-exipient compatibility studies.

Because of the various methods and devices available for this instrument, innovative applications in the pharmaceutical industry are being discovered and development. We expect the micro DSC II and VII to continue to be an essential piece of equipment in our laboratory.

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**Customer Testimony**

**Protein – Ligand interactions**

When it comes to studying interactions between a drug and a receptor or between a protein and a ligand, the technique of Isothermal Titration Calorimetry (ITC) is particularly suitable owing to its great sensitivity. The ITC II instrument, with its removable cells (making for easy cleaning) is therefore ideal for studying systems in which gels or viscous products are formed. The more sensitive Nano ITC III instrument can be used to study the interactions with more dilute solutions. Figure 8 shows the curve obtained with the Nano ITC III for the titration of 1ml of solution.

**The applications of thermal analysis to pharmaceutical products**

The applications of thermal analysis to pharmaceutical products are numerous and are vital for product analysis and characterization. Thermal analyzers must be extremely sensitive and offer various possibilities in terms of sample capacity and coupling with other instruments. SETARAM offers a full range of high-sensitivity original technology products for the thermal analysis of pharmaceutical products.

**Conclusion**

The applications of thermal analysis to pharmaceutical products are numerous and are vital for product analysis and characterization. Thermal analyzers must be extremely sensitive and offer various possibilities in terms of sample capacity and coupling with other instruments. SETARAM offers a full range of high-sensitivity original technology products for the thermal analysis of pharmaceutical products.


SETARAM Instruments and AKTS Software:

A global solution for solid state kinetic analysis and determination of the thermal stability of materials

A synergy is found between accurate thermal analysers and precise kinetic evaluation and prediction. Advanced Kinetics and Technology Solutions builds on a 10 years experience in thermal analysis, kinetics, advanced mathematics and software programming.

AKTS user-friendly Software is available from SETARAM Instrumentation.

Don’t hesitate in asking us for a free demo CD! Or download it directly from our Website: http://www.setaram.com/kinetics/software.html

### Problems in kinetic evaluation

Very often, raw thermoanalytical data are not sufficient to explain the thermal behaviour of a substance. Further description requires information on the kinetics of temperature induced reactions. But many of those reactions are assumed to obey a simple rate law, even when they actually have a complex multi-step nature. The use of too simplified or conservative kinetic models for the assessment of such reactions leads to improper prediction of their thermal behaviour. What is in turn the case of, e.g., energetic materials leads additionally to economic drawbacks. It often results in unnecessary large safety margins.

Moreover most of the available techniques focus on extremely long, time consuming isothermal experiments.

**AKTS solution**

Advanced Kinetics and Technology Solutions has developed the software based on advanced numerical techniques for an accurate kinetic evaluation of complex reactions [1-4].

Due to the accuracy of determined kinetic parameters it enables fine prediction of the reaction progress of substances in a broad range of temperature profiles.

Applying the results obtained by a few (three to five) thermoanalytical or calorimetry non-isothermal runs using DTA (Differential Thermal Analysis), DSC (Differential Scanning Calorimetry), Calvet Calorimeters or TG (Thermo Gravimetric Analysis) or EGA (Evolved Gas Analysis: TGAMS, TGA-FITR), it is possible to calculate and compare:

- Model free kinetics
- Model fitting kinetics selecting in a provided list of commonly applied equations
- Model fitting kinetics based on user defined equations

The calculated kinetic parameters are subsequently employed to predict the reaction progress of the investigated samples under temperature modes such as:

- Isothermal, stepwise
- Modulated or periodic temperature variations [example 20 ± 10°C, period 24 h]
- Rapid temperature increase (temperature shock)
- Real atmospheric temperature profiles for global investigation [selection from a list of 7000 worldwide climates]

Providing accurate input data thanks to SETARAM Instruments a supplementary process safety oriented module allows the prediction of:

- Adiabatic conditions for the determination of TMR, at any temperature and thus build a unique T=f (TMR=24hrs) thermal safety diagram
- The effect of scale, geometry, heat transfer [insulation], thermal conductivity

Upstream the kinetic evaluation is the accurate measurement of thermal effects. State of the art thermal analysers and calorimeters are required to provide valuable input data.

Such data can be easily measured and imported from our SETSYS Evolution coupled with MS, Standard DSC, MicroDSC, Calvet DSC, Calvet Calorimeters.

Among those instruments, C80 is certainly the best fitted for the study of thermal decompositions followed by the kinetic evaluation and prediction of the reaction progress. Heat measurements in a C80 Tian-Calvet type microcalorimeter is done by two fluxmeters, each of which measures the thermal power exchanged constantly between the experimental cell and the calorimetric unit. The main difference in comparison to the DSC plate transducer technique is that the fluxmetric transducer envelopes the sample and is therefore capable of measuring almost all the exchanges between the cell and the unit, a characteristic that gives this device a clear metrological advantage in terms of both the quantification of the measurements and the capacity to measure very weak effects.

Also, because large C80 cells enable the set up of experiments with 1-10 ml of products, the upscale predictions are more accurate than those obtained with a usual DSC 100 ml crucible.

Two types of cells (localised pressure measurement cells and safety cells) allow simultaneous measurement of the internal pressure. In case of gas releasing decomposition study, gas evolution can be quantified.

AKTS software applied to the derivative of the pressure signals can also be used for vent sizing applications [4].

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* Nano DSC III and Nano ITC III are manufactured by CSC (Calorimetry Sciences Corp., USA) and are distributed in Europe by SETARAM Instrumentation.

For countries in or outside Europe please contact SETARAM Instrumentation or sales@setaram.com.
Typical applications:
- Stability of substances (explosives, pharmaceutical materials, polymers, etc.)
- Complex temperature profile exposure simulation (process)
- Gas releasing reactions
- Thermal aging of materials
- Process safety
- Fire exposure simulation (insulation efficiency)
- Container design
- etc.


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Swiss Federal Institute of Technology (EPFL), Institute of Process Sciences, 1015 Lausanne, Switzerland.

Download the following examples directly from our website and watch the corresponding live videos to see how to proceed with AKTS-Thermokinetics and AKTS-Thermal Safety Software:

Download this article directly from:
http://www.setaram.com/kinetics/newsletter.html
Our laboratory is specialized in research into polymers. We bought a SETARAM SETSYS Evolution TGA (Thermo-Graavimetric Analysis) instrument with humidity generator and controller module. This TGA instrument is coupled with an AGILENT Technologies 6890 N’ gas chromatograph (GC) which in turn is coupled with an AGILENT 5973 Network mass spectrometer (MS). We added a series of loops (ILS module) at the TGA-GC interface to collect gas fractions. The GC is the module driver.

Below are some examples of the work we have carried out using these coupled techniques:
- Collection of gases from the thermal degradation of a polymer additive,
- Quantification of the collected gases,
- Study of a chemical hydration reaction (e.g. Alcoxisilane+H₂O → Alcohol).

In this paper we present an example of polymer additive degradation with the collection and analysis of degradation gases. The analyzed product is a polymer modified by an organic additive. Some TGA graphs are shown below:

The weight loss between 70°C and 180°C represents 0.73% of the total weight, and corresponds to the evaporation of water (adsorbed or not). Between 200°C and 400°C, the weight loss represents 29.57% of the total mass and comes from the organic additive.

To identify the additive components, the TGA gas emissions are collected in loops then injected into the GC. The chromatograms are produced in total ions for each peak with their corresponding mass spectra. The result comprises qualitative and quantitative analyses. We illustrate this with two examples of GC total ions and the corresponding mass spectra for each ion: We were able to characterize the following ions using the different spectra (Spectrum 1 and Spectrum 2):

- CH₃– (m/z 252)
- (CH₃)₂– N – CH₂ (m/z 58)

Conclusion

Coupling TGA with GC and MS enabled us to determine the additive in the polymer and its percentage content. Thanks to the GC, we loaded fractions of emission gases at regular time intervals and analyzed them. With the Area versus Time curves, the chemical kinetics of degradation of the additive compound can be determined. This kind of analysis is limited by many factors, such as polymer degradation, secondary effects, or maximum temperature and the separating power of the GC column.
Optimized designs for our future instruments

1. Introduction:
In 2002, SETARAM Instrumentation decided to invest in a thermomechanical modeling and simulation program in order to optimize the design of its future instruments.

Directly integrated into our CAD software, CosmosWorks is a design aid whose principle of operation is based on the finite elements method. This method consists in dividing the 3D digital models produced by the CAD system into elementary entities interlinked with one another by common points, and solving the differential equations relative to a true physical phenomenon for each of these points.

Alumina sleeve for Labsys thermobalance oven

Thanks to this state-of-the-art tool, it is possible to compare several different designs very rapidly and determine which one is optimal. This considerably reduces the number of prototypes, optimizes the performance of the appliance and shortens development times.

Practical example: Sensitivity calculation

In order to optimize the number of fluxmeters to be placed on each of the two cells, we decided to calculate the calorimetric sensitivity according to the number of fluxmeters. The calculations performed with our software enabled us to determine the temperature gradients on each fluxmeter and therefore a resultant signal. We thus knew that with 16 fluxmeters per cell surface, the sensitivity of the calorimeter would be about 153 µV/mW. The actual value turns out to be exactly 159 µV/mW (4% error).

2. Design of large-volume calorimeters for the CEA:
In 2003 the CEA (Atomic Energy Commission) placed a special order with out for two large-volume differential calorimeters called 390 (Wall = 60 liter) and 680 (Wall = 90 liter). These two instruments were designed entirely using the modeling and simulation software, enabling us to bypass a prototyping phase.

The design calculations have enabled us to optimize the sensitivity of our calorimeters, the thermal insulation, the uniformity of heating, as well as the mechanical strength of the various components of the robot-controlled opening mechanism.

Practical example: Sensitivity calculation

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Optimization of the number of fluxmeters

Since 1963, SETARAM was active in China with CALVET microcalorimeters, thermobalances and DTA, massflowmeters,… with the “COMEF Group” in BEIJING, SHANGHAI & XI’AN.

In 1990 , SETARAM appointed a specialist of Thermal Analysis equipments : Mr Jason S. Yang , to promote the different equipment.

In 1996, Mr Jason S. Yang started his own company “AWS Go” to distribute, install, demonstrate, service, … the analysers of SETARAM and other Europeans companies.

Now, “AWS” has 11 persons, highly qualified with an University degree and speak fluently English .

Several universities or Research Institutes are used as “Demo Lab” for customers willing to have practical tests on their own samples .

Due to the recent development of researches in High Temperatures (up to 2400 °C) for ceramics and composites materials, a new “Demo Lab” will be finished in SHENYANG with several TG/DTA/DSC/ DILATOMETERS analysers with Gas Analysis.

In future, other “Demo Laboratories “with full equipment to be used by potential customers, and as proof of the quality of the SETARAM INSTRUMENTATION equipment, will be installed in the South part of China (SHANGHAI area), and in XI’AN for applications in Bio-Technology and Pharmacy.
Our partner laboratory in China

The High-Performance Ceramics Division headed by Prof. Zhou Yanchun belongs to the Shenyang National Laboratory for Materials Science (SYNL). It is located in Shenyang, a heavy industry city in north-eastern China.

The main research activities of the High-Performance Ceramics Division are focused on:
1. Multi-scale (electronic, crystal and microstructural) design and processing of high-temperature ceramics and composites;
2. Developing low-cost techniques for the processing of bulk and small-dimension materials (powders and thin films);
3. Investigating the mechanical behavior under static, dynamic and cyclic loads;
4. Investigating the thermal/chemical stability of ceramics and composites in ultrahigh environments.

The researchers at the SYNL High-Performance Ceramics Division have recently succeeded in developing new methods for processing layered machinable ceramics including Ti$_3$SiC$_2$, Ti$_3$AlC$_2$, Ti$_7$SnC, Cr$_2$AlC. Basing their work on the investigation of electronic structure-property relations, they have designed a number of solid solutions with superior properties. In the past 5 years they have published more than 100 papers in high-impact international journals such as Appl. Phys. Lett., Acta Mater., Chem. Mater., J. Mater. Rec., J. Mater. Chem., Phys. Rev. B, J. Appl. Phys., Carbon, Mater. Res. Innovat. et al.


At the end of 2004, the SYNL High-Performance Ceramics Division and the Setsys 16/18 thermal analyzer (TG to 1750°C, TG/DSC and TG/DTA to 2400°C) were purchased for $500,000. The main facilities used in this laboratory include the Setsys 16/18 thermal analyzer (TG to 1750°C, TG/DSC and TG/DTA to 1600°C), the Setsys 24 thermal analyzer with MS (TG and TG/DTA to 2400°C), and the Setsys 24 thermal mechanical analyzer.

ISO 9001 certification to improve customer satisfaction

On April 23rd, 2004, SETARAM received ISO 9001: 2000 certification from the BVQI for its 2 activities, namely scientific equipment for laboratories (SETARAM instrumentation) and industrial equipment for baking furnaces (SETARAM Engineering).

This certification is the result of a process initiated in 2001 in the framework of a quality policy highlighting:
- The improvement in the quality of our products and services,
- Customer attentiveness to our customers needs,
- The implementation of a quality management system.

The operation is placed under the responsibility of Planète Urgence’s management. The objective is to support various types of technical, educational or material development actions in third-world countries or in France.

Professor Hiroshi Suga

Professor Hiroshi Suga, a member of SETARAM’s Industrialization Department, has invested himself in these actions of solidarity by volunteering his services to Planète Urgence for a mission. His assignment forms part of the ‘congé solidaire’ (Solidarity Leave), which changed its name to “Planète Urgence” (Planet Emergency) in 2004.

He purchased other Calvet calorimeters for low-temperature (BT2.15) and high-temperature (HT1000) applications. After receiving the Award, Professor Hiroshi Suga gave a lecture entitled “A calorimetric study of transition phenomena in molecular solids”.

SETARAM sponsors an ecological monitoring mission

Created in 1999 by the association “Congé Solidaire” (Solidarity Leave), which changed its name to “Planète Urgence” (Planet Emergency) in 2004, the “congé solidaire” enables employees to become involved in humanitarian missions during their free time. The objective is to support various types of technical, educational or material development actions in third-world countries or in France.

Philippe Joly, a member of SETARAM’s Industrialization Department, has invested himself in these actions of solidarity by volunteering his services to Planète Urgence for a mission. His assignment forms part of the permanent research activities in the biophysical reserve of the National Park of Pendjari in the Republic of Benin, West Africa.

These activities include monitoring vegetation growth, the development of bush fires, the development of wet zones, large mammal, carnivore and bird populations, and establishing an ecological monitoring database.

Encouraged by this success, SETARAM now wants to go further and use quality management as a lever to make improvements in the following key areas:
- The development of its personnel skills management,
- The watch over its market sectors,
- The customer satisfaction through permanent and continuous communication.

And of course we continue to seek the best solutions for your thermal analysis measurement needs, and endeavor to constantly improve the performance of our products.