micro DSC III
specifications

Temperature range	Nota : cooling to 0°C and down requires using auxiliary thermostat	-20 / 120°C
Scanning rate		0,001 to 1.2°C.min^{-1}
Vessel		"Batch" or circulating
volume		850 µl
Resolution		0.03 µW
Noise RMS		0.03 µW
Specific noise RMS		4.10^{-5} µW/µl
Cooling rate from 120°C to room temperature		1h30

The micro DSC III is equipped with SETSOFT 2000, the thermal analysis software from SETARAM.

SETARAM Scientific & Industrial Equipment
KEP TECHNOLOGIES
Excellence in thermal analisys and calorimetry.

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micro DSC III
high sensitivity DSC and calorimetry

SETARAM Scientific & Industrial Equipment
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micro DSC III
the ultra-high sensitivity response

-20°C +120°C

For more than ten years SETARAM has been marketing micro DSC type calorimeters throughout the world. The combination of isothermal and DSC-mode calorimetry, the choice of experimental vessels on offer and the ultra-high sensitivity of the micro DSC have all attracted great numbers of laboratories.

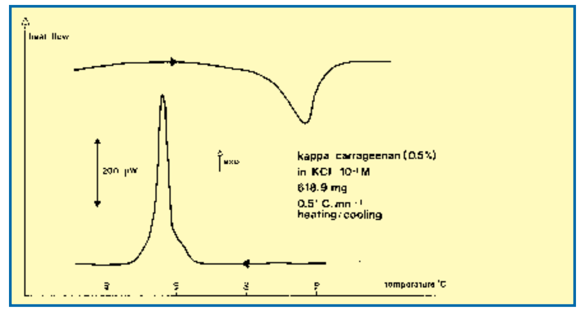
With the micro DSC III an even greater temperature range, from -20°C to 120°C, is given thereby extending the applications available.

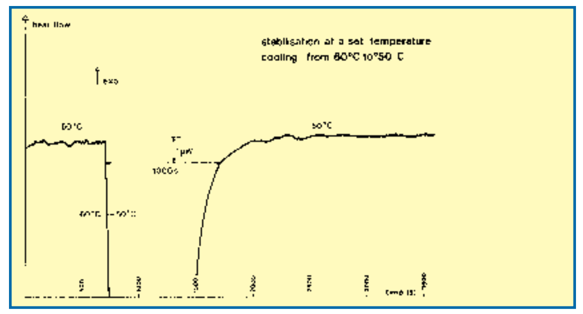
New experimental vessels have been developed, especially for studying reactions.
With its facilities for rising and falling temperature scanning and its integrated cooling system the micro DSC III offers research laboratories specifications and measuring levels unattainable by isothermal calorimetry alone or adiabatic-type calorimetry.

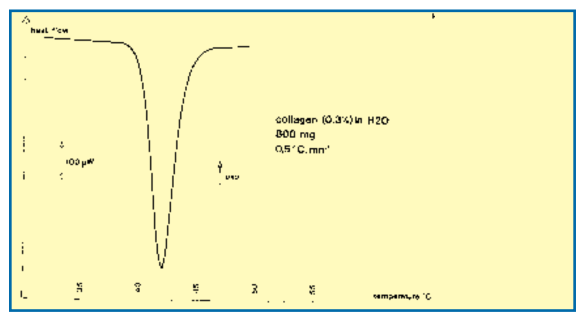
micro DSC III
high-level performance and wide-ranging facilities with a single instrument

• High detection limit.
The detection limit on the **micro DSC III** is very much better (by a factor of ten) than on conventional DSC (power compensated, thermocouple-detector). Variations in the calorimetric signal below one microwatt can be detected.

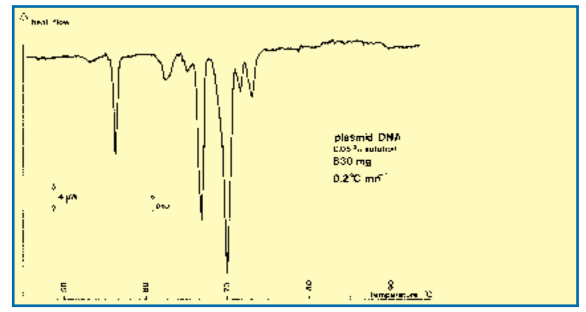
• Very good stability on the signal and low background noise.
Use of a liquid thermostat and piloting via the CS 32 controller import the **micro DSC III** with very good base-line stability, especially when running isothermally, over very long periods. Background noise is thus less than 0.2 µW.

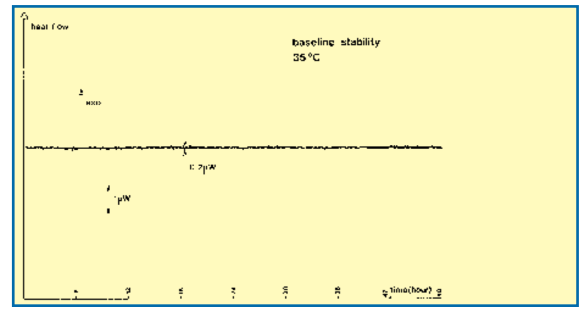






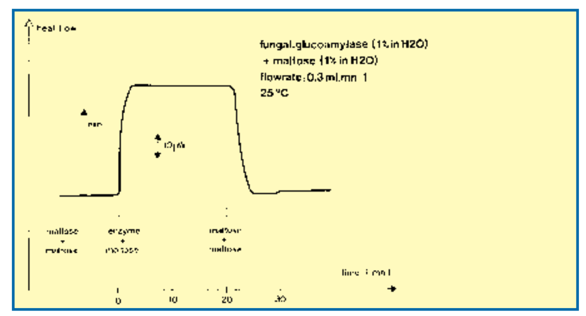
• Analyzing solids or solutions.
The **micro DSC III** can carry out equally well analyses on solid samples or diluted solutions. There is a large volume available for the sample (of the order of 1 cm³).





• Operation with rising and falling temperatures.
The **micro DSC III** has scanning over the temperature range from -20°C to 120°C, with rise and fall being identical and no outside cooling being needed. Scanning rates as low as 0.001°C.min-1 can be accurately achieved.

• Short response time and rapid thermal equilibrium.
The **micro DSC III** has a short response time providing major scanning flexibility and rapid thermal equilibrium after thermal disturb, such as one due to a change in temperature or introduction of an experimental vessel.



• Adapted for varied experimental vessels.
With removable experimental vessels the **micro DSC III** offers a varied choice for applications : closed vessel, for introducing gases or liquids, mixing, for measuring heat capacity.

micro DSC III

• micro DSC III... the calorimetric block.

The **micro DSC III**'s calorimetric block is made up of a gold- plated metal cylinder with high thermal conductivity. This assembly provides for excellent temperature homogeneity with accurate control, which in turn determines the very good base-line stability in the calorimeter.

Two hollows are machined into the block to take the experimental vessels. The "measurement" vessel takes the solid or liquid sample to be analyzed. The "reference" vessel contains an inert material to compensate for the thermal effect linked to the sample's heating up.

Various types of experimental vessels can be used, depending on the applications to be carried out.

• micro DSC III... the heat flow transducer.

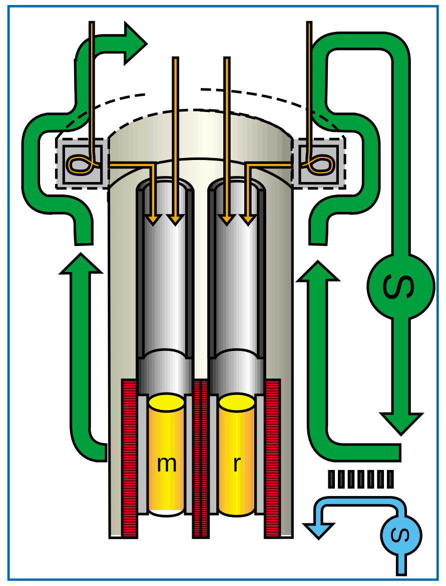
Each vessel is surrounded by a plane-surfaced detector with very high sensitivity providing the thermal link with the calorimetric block.

These transducers are good thermal conductors which keep the temperature in the vessels identical to that in the calorimetric block.

Their electrical signal is proportional to the heat transfer between the vessel containing the sample and the calorimetric block.

Setting the two transducers in opposition on the "measurement" and "reference" vessels removes the perturbations common to both vessels.

The heat-flow transducer as defined imports the **micro DSC III** with ultra-high sensitivity (detection limit : 0.2 µW) and very good measuring accuracy.



• micro DSC III... the temperature.

The calorimeter's temperature is set by thermostatic control of circulating liquid which is heated or cooled via piloting from the CS 32 controller from -20°C to 120°C.

Cooling the calorimeter does not require any outside source.

External water circulation removes the heat.

The transducer zone is protected by thermal buffers preventing outside interference, but providing direct access to the vessels.

Where circulation vessels are used the reactive agents are under thermostatic control via a built-in pre-stabiliser.

Use of a liquid thermostat and the accuracy of temperature control reduce the effect of outside-temperature fluctuations, as well as enabling very small thermal effects to be measured with accuracy both in isothermal and DSC modes.

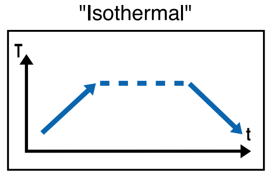
• micro DSC III... temperature control.

With the flexibility of scanning and temperature control offered by the CS 32 controller, the **micro DSC III**'s specifications provide for various modes of operation depending on the applications to be carried out :

"Isothermal"

• Constant temperature (isothermal calorimetry mode)

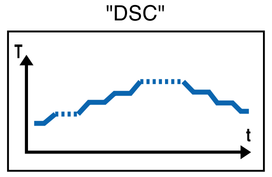
The isothermal mode is especially appropriate for studying metabolisms, bacterial growth, for checking stability, compatibility between materials and for simulating reactions (mixing, wetting, enzymatic-reaction,...)



"DSC"

• Scanning temperature (DSC mode)

The DSC mode enables a sample's thermal behaviour to be explored as a function of the temperature, especially for studying the changes in phase or structure (fusion, gelification, transition, denaturation...) and for checking how the phenomenon is reversible by temperature cycling.



"By step"

• Step scanning temperature

Scanning by temperature increment is especially attractive for determining the heat capacity of solids and liquids. The measurement has greater accuracy with this method.

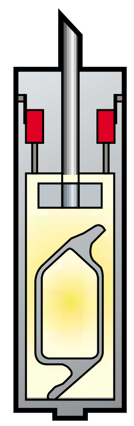
micro DSC III
vessels and uses

The micro DSC III's flexibility consists in the choice of experimental vessels available to fit in with the various requirements of laboratories. Based on the applications the vessels are used either as closed ("batch" type) or are combined with fittings for introducing reactive agents ("flow" type). All the vessels can be used in isothermal and DSC mode. They are made of Hastelloy C, have a volume of 1 cm³ and can easily be cleaned.

• Closed, "batch" vessels.

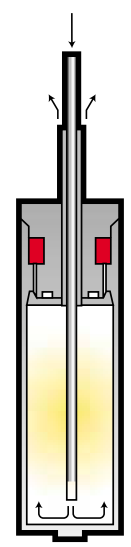
The closed, "batch", vessel is designed for analyzing solid or liquid samples, isolated from the outside environment.

The vessel's fluid-tightness is provided by an elastomer seal, able to stand a maximum internal pressure of 20 bars. This vessel is especially designed for studying denaturation, phase transitions and gelification.



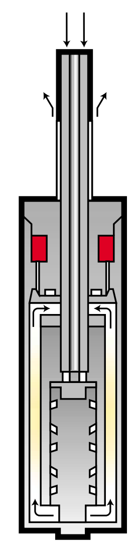
• Ampoule vessel.

The ampoule vessel is specifically designed for studying wetting or hydration of powders. Powder is first degassed in the ampoule under vacuum, before the sealing operation. Then the sealed ampoule is immersed in the liquid. Using a push rod the ampoule breaks easily ensuring the instantaneous wetting of the powder.



• Fluid-circulation vessel.

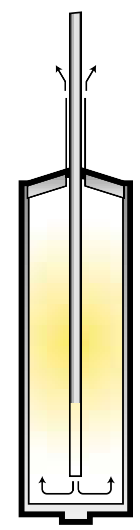
The circulation vessel contains a tube for introducing a fluid (liquid or gas) and a concentric, exhaust tube. The fluid has previously been thermostatically controlled in the pre-stabiliser on the calorimeter's inlet so as to provide thermal balance on the inlet and outlet fluids.This vessel is designed for studying interactions between a fluid and a solid sample (adsorption, substrate on immobilized enzyme). This vessel is also appropriate for measuring the activity of a circulating liquid taken for sampling from the heart of a reactor (fermentation, decomposition, reaction).



• Vessel for mixing circulating liquids.

The circulation-mixing vessel is designed for studying mixing of two liquids "in situ". The liquids enter the vessel via two separate tubes which come together in a mixer.

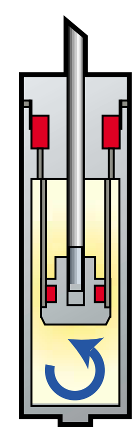
The resulting mixture is removed from the vessel via a third, concentric tube. Before mixing the liquids are thermostatically controlled in the pre-stabiliser on the calorimeter's inlet. Studying mixing, dilution, neutralization and enzymatic reaction are the main applications of the circulation-mixing vessel.



• Heat capacity vessel (liquid).

The accuracy of measuring a liquid's heat capacity via the calorimetric method depends on the correction due to the vapour phase above the liquid. To overcome this difficulty the **micro DSC III**'s heat capacity vessel is fitted with two tubes welded to the vessel's body.

The vessel is filled via one of the tubes so as to avoid any vapour phase in the vessel. Determining the heat capacity for the corresponding volume of liquid is done via the step scanning method. For measuring the heat capacity of solids the closed "batch" vessel is used.



• Mixing "batch" vessel.

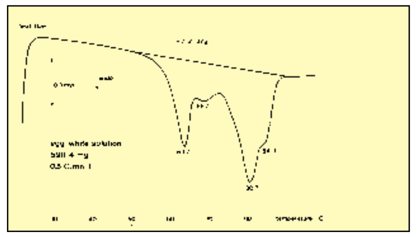
To study the reaction between a powder and a liquid, the mixing "batch" vessel is fitted with two separate chambers to envelope the samples.

These are brought into contact and mixed using a push rod. As a single part of the first compartment the latter ensures the mixture is well stirred. This vessel is especially appropriate for studying enzymatic reactions.

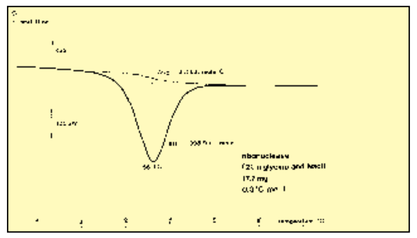
micro DSC III
applications in scanning mode

-20°C +120°C

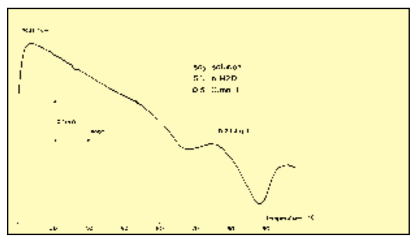
Using the micro DSC III in the DSC mode opens up a great number of applications, such as denaturing effects on proteins and enzymes, the effects of aggregation, transitions in lipids and membranes, protein-lipid interactions, fusion and gelification of polysaccharides and gelatins, but also phase transitions in liquid crystals or crystallizing effects in paraffin as well. These are only a few examples of how it can be used in dealing with biological, food or pharmaceutical products, but many other fields are still to be explored, especially those with polymers or organic products.



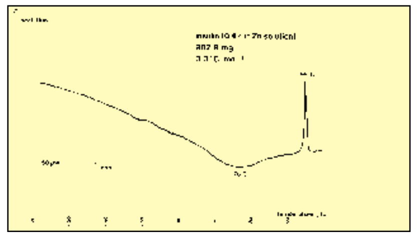
• Denaturation of animal protein.
A solution of the analyzed white of a hen's egg has been stabilised in relation to thermal denaturation by adding Al^{3+} ions. Analysis by the **micro DSC III** enables this stabilisation's efficiency to be distinguished up to 50°C, before highlighting how the various proteins present in the solution denature.



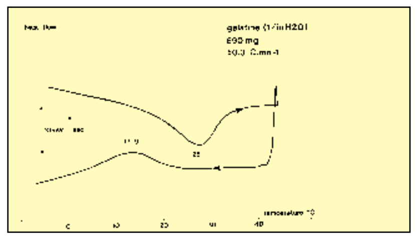
• Enzyme denaturation
Enzymes are catalysts specific to biochemical reactions, but denature during heating as they loose their properties. The **micro DSC III** enables the enzyme's thermal stability and its operating conditions to be accurately distinguished, as with ribonuclease A, an extract of the bovine pancreas (Buffer : 0.2 M glycine; 0.2 M NaCl; pH 4). It is worth noting the variation in heat capacity which accompanies the enzyme's change of structure.



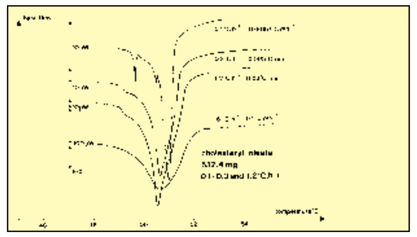
• Denaturation of vegetable protein.
Isolated vegetable proteins are part of the food ingredients used more and more in not only animal feedstuffs but also human feedstuffs. The **micro DSC III** is especially appropriate for studying these proteins in powder-form or dissolved solution, particularly for how they denature thermally during heating. This is the case with a solution of soja protein (5% in water) which exhibits two distinct features of denaturing.



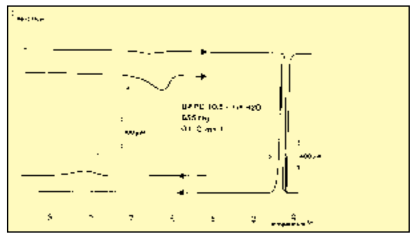
• Protein denaturation-aggregation.
Insulin is a hormone produced by the pancreas, which reduces sugar concentration in the blood. Nowadays insulin can be synthesized by genetic engineering. Stored in an injection-ready solution, insulin tends to denature and form aggregates. Using the **micro DSC III** enables these two phenomena to be checked (endothermic denaturation, exothermic aggregation) as these must be ascertained to prevent them in practice.



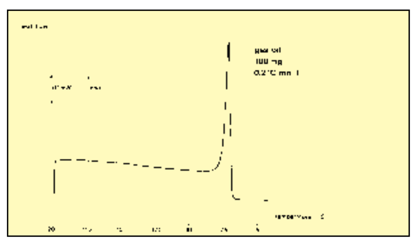
• Fusion-gelification.
Hydrocolloids (pectin, carrhagenane, alginate, gelatin, xanthane) exhibit the special properties of the thickeners or gelifiers widely used in the food industry.
Working with a gelatin solution (1% in water), cooling the solution initially taken to 40°C highlights the gel formation.
On heating the gel can be observed melting.
The **micro DSC III** makes studying the reversibility of these phenomena very easy.



• Phase transitions (liquid crystal).
Liquid crystals exhibit intermediate phases between the solid and the liquid structure. Using a conventional DSC analysis sometimes does not highlight these perfectly.
The **micro DSC III**, with its ultra-high sensitivity, offers the facility of analyzing these transitions very accurately, especially by using very slow temperature scanning rates (up to $0.1°C.h^{-1}$). The isotropic, liquid -> liquid crystal transition is analyzed in this way for a crystal of cholesteryl oleate.



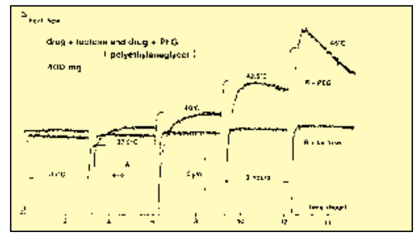
• Phase transitions (lipids).
Lipids are one of the main constituents in cellular membranes. When dispersed in water two thermal transitions can be observed : at low temperature a gel/gel transition due to a molecular rearrangement, followed by a gel/liquid crystalline phase linked to the fusion of hydrocarbon chains. With the **micro DSC** and working with DPPC (0,5% in water the temperatures of these transitions are accurately measured, and their reversibility highlit.



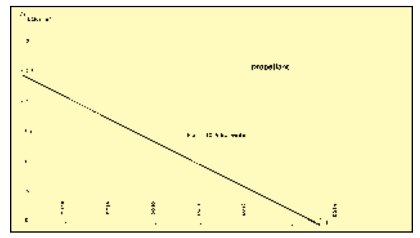
• Crystallization.
The facilities for slow-rate cooling offered by the **micro DSC III** are ideal for studying the phenomenon of paraffin crystallization in gas oils. Rates of $0.001°C.min^{-1}$ can be scanned, providing for simulation of the real temperature variations during the cycle of a day.

micro DSC III
applications in isothermal mode

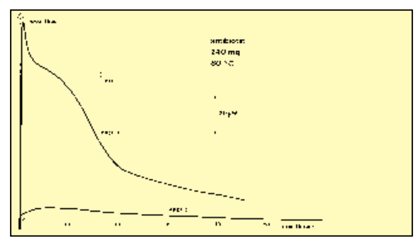
Using the micro DSC III as an isothermal calorimeter provides items of quantitative information in real time over a very wide range of applications : thermal stability of unstable materials (powders, explosives), of foodstuff ingredients (aromas), of pharmaceutical products (antibiotics), compatibility between drug and excipient, metabolic activity in living organisms, bacterial growth and fermentation, enzymatic reaction and any mixing reaction of a general nature.



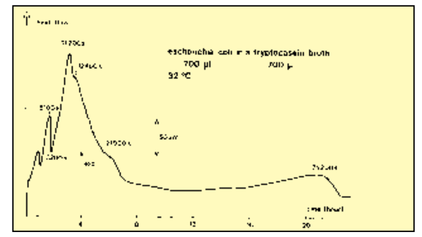
• Drug-excipient compatibility
Choosing an excipient, combined with a drug, is a vital stage in developing a medicine.There must be compatibility between the two products, with no reaction affecting the drug's capacities. With the **micro DSC III** a rapid method is available for checking this compatibility. By successively studying the mixture at various temperatures it can be noted, for example, that a drug is compatible with lactose, but reacts with polyethylene glycol. The compatibility test is carried out in less than 24 hours.



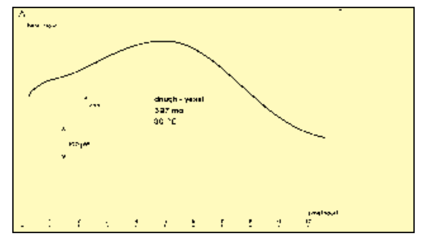
• Stability of unstable materials.
An explosive is generally a mixture of substances which violently decompose after firing. However, depending on the storage conditions, the mixture can self-decompose, and this phenomenon must be accurately monitored. By studying this decomposition at various temperatures it is possible to predict, using kinetic models, the hazards connected with the storage temperature, as well as the powder's life time.
The same type of procedure can be applied to the self-discharge of electrolytic batteries.



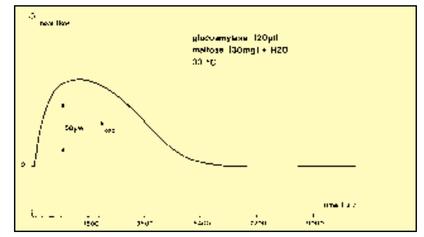
• Stability of pharmaceutical products.
The stability test is primordial for certifying pharmaceutical products and the **micro DSC III** provides a rapid and accurate method for this evaluation.
An analysis at 80°C on two different preparations containing the same antibiotic highlights a very different thermal behaviour. This is because preparation 1 exibits an exothermic phenomenon linked with a process of decomposition.



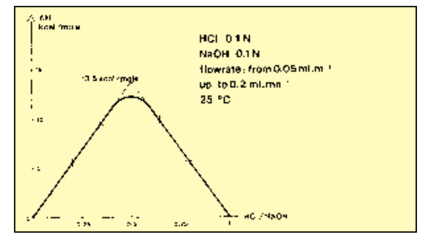
• Bacterial growth.
Studying bacterial growth in aerobic or anaerobic conditions is undertaken very easily with the **micro DSC III** as it enables bacterial development to be followed in real time.
An example is provided by the growth of a colony of Escherichia Coli in a Trypticase Soja culture medium. The various exothermic effects read correspond with the various stages of growth in the bacteria.



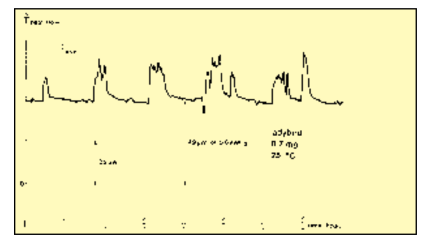
• Fermentation.
Fermentation produced by types of yeast is a reaction which can easily be simulated in isothermal calorimetry. The mixture of flour, yeast and water can be prepared initially and rapidly introduced into the calorimeter so as to follow the fermentation. It is possible to use the mixing "batch" vessel providing for mixing "in situ" and for producing more information on the start of the reaction.



• Enzymatic reaction.
The **micro DSC III** is used for distinguishing the temperature operating of the enzymes, as well as, for simulating the enzymatic reactions.
The reaction can be followed up continuously by using the circulation-mixing vessel, or in the "batch" mode with the twin-compartment, mixing vessel. The second technique is used to measure the maltose being transformed by the glucoamylase dissolved in the water.



• Mixing diagram.
Various types of mixing reactions can be simulated with the **micro DSC III**. A particular example is the calorimeter using the circulation-mixing vessel which is especially appropriate for determining the mixing diagram between two liquids. This is done by measuring the mixing heat at various concentrations, as it is done with the acid-base mixture : HCl/NaOH.



• Thermogenesis.
For many years now isothermal calorimetry has been considered as the ideal technique for following the metabolic activity in living organisms. The items of information may be produced in an aerobic or anaerobic condition.
The fluid-circulation vessel provides for modifying the gas' properties around the organism under study. When studying the lady-bird the cycle of periods of activity and rest was clearly highlited.