TA Instruments
MICROCALORIMETRY


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MULTI CELL DIFFERENTIAL SCANNING **CALORIMETRY**

The MCDSC is a versatile, multipurpose microcalorimeter that provides continuous temperature-scanning, step-scanning or isothermal measurements on three samples and one reference simultaneously. The one milliliter sample capacity ampoules and microwatt sensitivity ensures maximum flexibility for identifying phase transitions in chemicals and biologicals, metabolic activity in cellular organisms and thermal events occurring in complex reactions of liquids and/or solids during admixing. The superior baseline stability and short equilibration time permits faster measurements and more rapid temperature changes than is possible with other isothermal calorimeters. The availability of reusable, probe-accessible and high pressure removable ampoules allows the study of materials with experimental designs that are not compatible with other microcalorimeters.

MC DSC **SPECIFICATIONS**

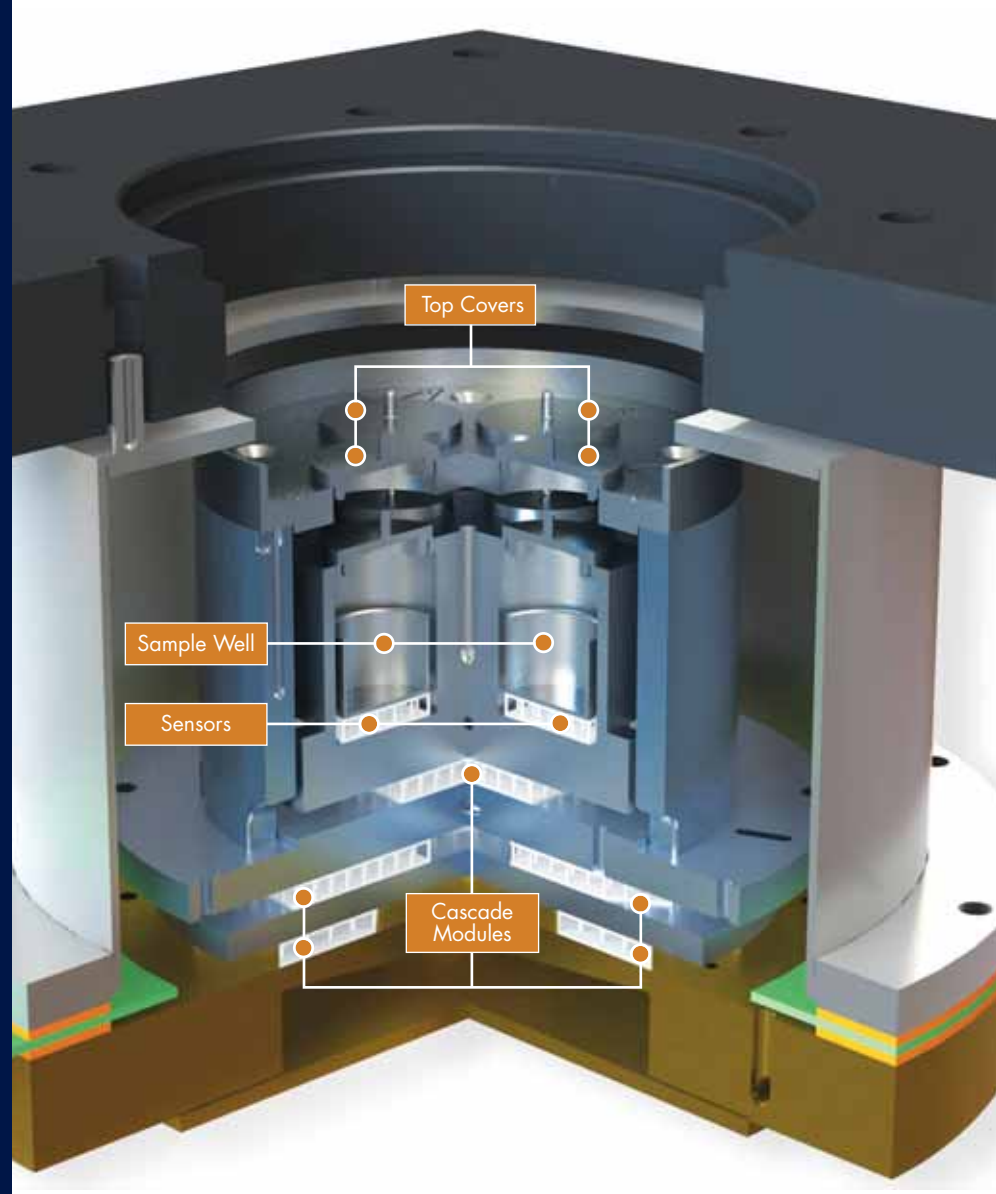
Sample Cells	3
Reference Cells	1
Temperature Range	-40 to 150 °C
Detection Limit	0.2 µW
Cell Volume	1 ml
Sample Volume	up to 1 ml
Short Term Noise Level	0.2 µW
Baseline Repeatability	2 µW
Scan Rate	0 (isothermal) to 2 °C/minute
Response Time	90 second time constant or 9 minutes for 99% response
Heat Measurement Method	Heat flux

MC DSC **TECHNOLOGY**

No other DSC on the market offers more flexibility for studying thermal stability of almost any sample as a function of temperature or time. The calorimeter exploits ultra-sensitive Peltier technology for both temperature control and as sensors to detect heat effects. Peltier cascades allow precise temperature control for isothermal operation and reproducible scan rates up to 2 °C per minute. The Peltier sensors give true microwatt detection independent of scan rate. Equipped with four removable cells, this is truly a workhorse instrument.

The MC DSC runs one reference and three samples simultaneously in removable, Hastelloy ampoules sealed with O-rings to prevent loss of volatiles. With an extra set of ampoules there is no downtime for cleaning and separate ampoule sets are convenient for different user groups. The wide-mouth ampoule design allows easy cleaning and accommodates large pieces of solids and viscous liquids as well as suspensions and solutions. The O-ring seal provides a reliable, truly hermetic seal for pressures up to 15 atmospheres, sufficient to retain the vapor pressure of liquid water up to 200 °C. Hastelloy ampoules are resistant to corrosion by aggressive solvents including concentrated bases, H_2SO_4, HCl and HNO_3 and are inert to biological materials such as proteins and lipids.

The top covers are designed for easy access to the calorimeter cells which allows operation with high pressure, batch reaction, and probe accessible ampoules for a wide range of non-traditional applications.



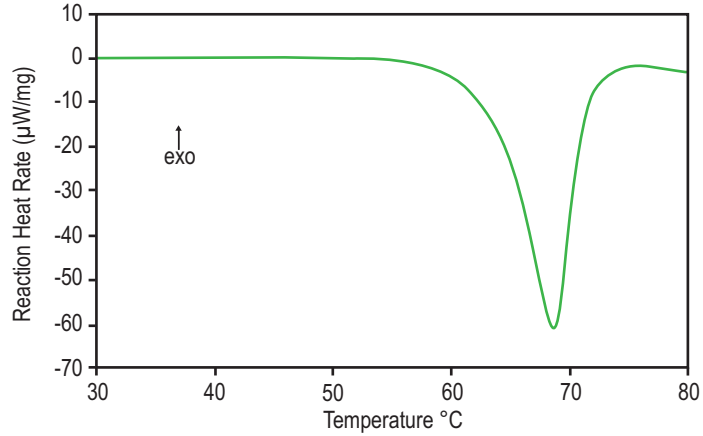
MC DSC **AMPOULES**

Standard Ampoules	Hastelloy®, 1.0 ml volume, 11mm diameter, 5mm depth
High Pressure Ampoules	Hastelloy®, 0.5ml volume, Kalrez® O-rings, max pressure 6000psi
Probe Accessible Ampoules	Standard Ampoule with special connections for pressure measurements, and connecting to spectrophotometers, mass specs, and gas chromatographs.
Batch Reaction Ampoules	Special Hastelloy® cells with two separate, sealed compartments for studying the reaction (and heat of reaction) between solids, liquids, and vapors.

MC DSC **Applications**

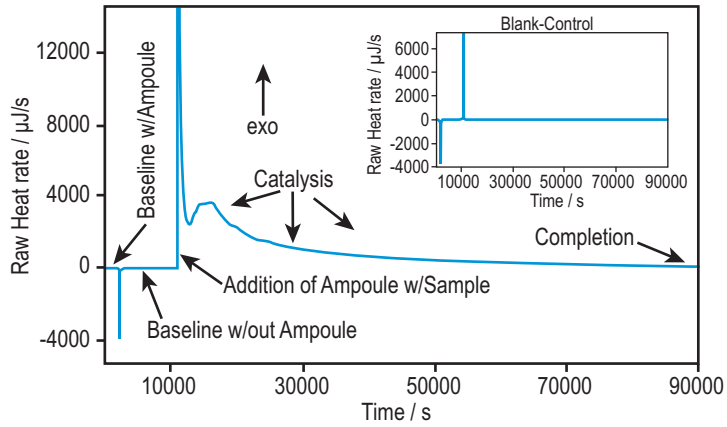
Drug-Excipient Compatibility

Because of the large sample capacity and multiple cell design, the MC DSC can be used to rapidly determine the temperature dependence of rates of drug-excipient reactions. This figure shows the results from a continuous temperature scan of 155mg of a 1:1 mixture of aspirin and magnesium stearate. The rate of the endothermic reaction indicating incompatibility becomes measurable at about 50 °C. Because of the wide-mouth (11mm) and depth (5mm) of the ampoules, whole tablets and capsules can be studied. The continuous temperature scan method is also useful for rapidly establishing useful temperatures for isothermal calorimetric measurements of stability of pure drugs and mixtures.



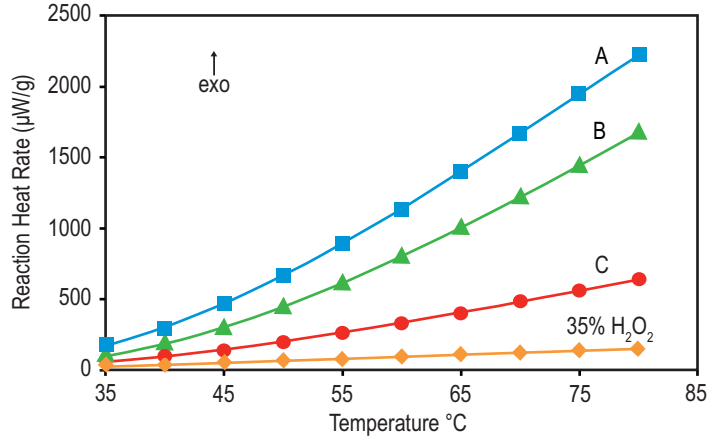
Biofuel Production

Determining reaction rates is a necessary prerequisite for understanding the mechanism of a chemical process. The MCDSC is a precision isothermal analysis tool, capable of analyzing both short- and long- term reaction kinetics. In the presence of a 3% w/v sodium hydroxide (NaOH) in ethanol solution, soybean oil undergoes transethylesterifcation to form a biofuel (biodiesel). Following the initial baseline periods (with- and without- ampoule), a sealed 1 mL ampoule containing the premixed reaction components was positioned into one of the three channels of the MCDSC equilibrated to 25 °C. The initial deflection at ~12000 s is due to the insertion of the ampoule into the MCDSC. The maximum peak for the catalysis in the reaction rate is shown at ~16000 s. Following the reaction maximum, the decreasing rate of the reaction is shown until completion. The inset shows the control experiment, whereby ethanol without NaOH was mixed with soybean oil and measured simultaneously in a second ampoule.



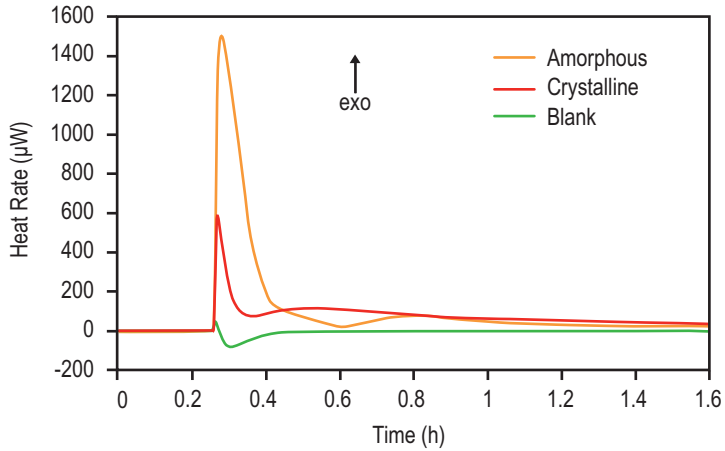
Shelf-Life Prediction by Step-Scanning

In the step-scan method, the calorimeter temperature is rapidly scanned to a programmed temperature and then held isothermal for sufficient time (30-45 min) to allow for equilibration to accurately measure the steady-state heat rate, then stepped to the next temperature for another measurement of the isothermal heat rate. Step-scan measurements are particularly useful for accurate determination of activation energies and temperatures at which reaction mechanisms change. The figure shows data collected by the step-scan method on 100 mg aliquots of three different brands of peroxide bleaches used for whitening teeth. 35% H_2O_2 is shown for comparison. The higher the decomposition rate, the faster the product whitens teeth, but the shorter the working time. The method is applicable to reactions in a wide range of materials; for example, cleaning agents, drug-excipient mixtures, adhesives, cell cultures, and small organisms.



Polymorph & Amorphous Crystallization

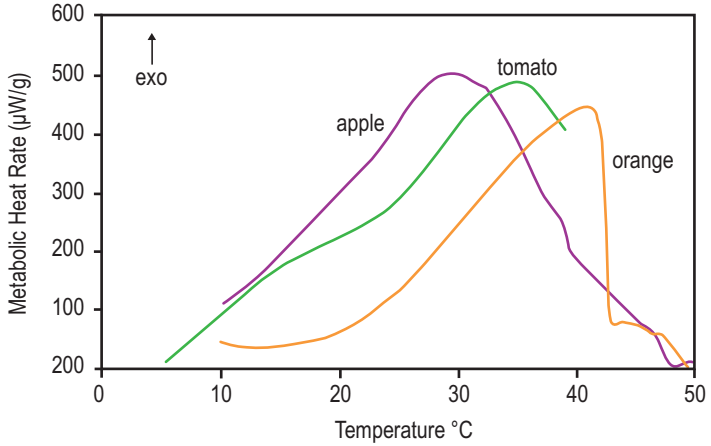
The batch reaction ampoules make it particularly useful for measurement of the heat and kinetics of reactions between volatile liquids and solids. The figure shows the immediate crystallization of 30 mg of a partially amorphous lactose sample on exposure to 100% humidity. Water sorption by 30 mg of crystalline lactose is shown for comparison. The blank in dry N_2 shows the exothermic heat of opening followed by the endothermic evaporation of water. The thermodynamics and kinetics of reactions such as oxidation, decomposition, hydrolysis, and curing can be determined by similar isothermal measurements in standard ampoules. Evolution of gases and changes in solution composition can be followed by adding pressure tranducers, spectral probes or mass spectrometer inlets to the probe accessible ampoules.



MC DSC **Applications**

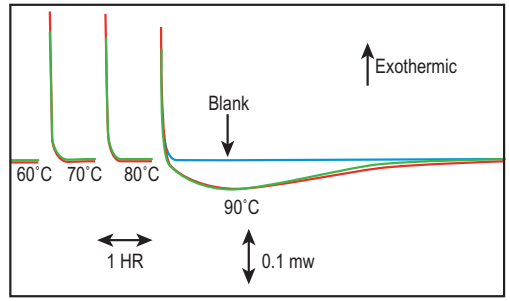
Temperature Dependence of Metabolic Rate

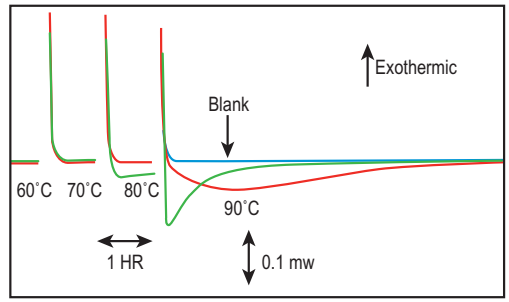
In many organisms, metabolic heat rate is equivalent to the oxygen uptake rate, but heat rate is much easier to measure in cell cultures, tissues, and small organisms. Data on respiration rate as a continuous function of temperature is necessary to optimize conditions for cell cultures, for prediction of the effects of climate change on organisms and ecosystems, for selection of cultivars of crop plants for optimum productivity, and for prediction of the invasive potential of exotics. The data shows a comparison of the metabolic heat rates obtained with continuous temperature scans of 50 mg samples of leaf tissue from apple and orange trees and of a tomato cell culture. Near simultaneous measurements of metabolic heat rates and CO^2 rates as functions of temperature can be done by the step-scan method.



Characterisation of solid state drugs by calorimetry

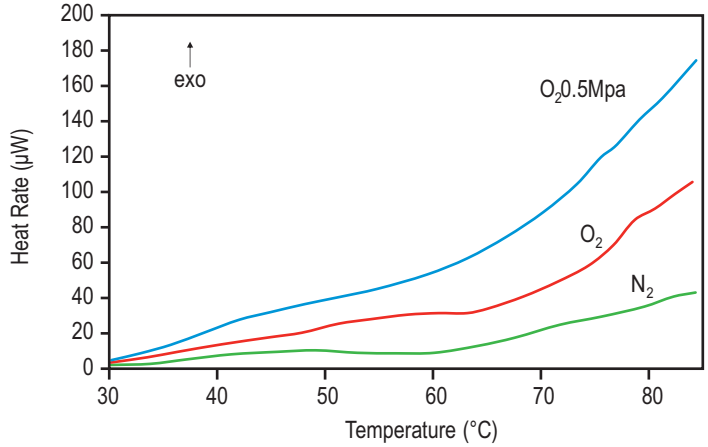
This is an example of a drug where the enantiotropic form A spontaneously converts to form B. This experiment was conducted in the step isothermal mode of the MCDSC. Samples were loaded at 60 °C and measured for 1.5 hrs before the temperature was changed 10°C. The endothermic effect at 90 °C (Graph 1) is the conversion of form A to B, with an excellent reproducibility between duplicate samples. The shape of the curve indicates an autocatalytic mechanism consistent with nucleation and growth. The conversion went to completion in 10 hours. The second graph (Graph 2) shows the same reaction, but in this case a small amount of B was added as an impurity to A, and with this seeding the conversion started at a lower temperature, 80 °C, and at 90 °C the time to completion of the conversion was significantly reduced.





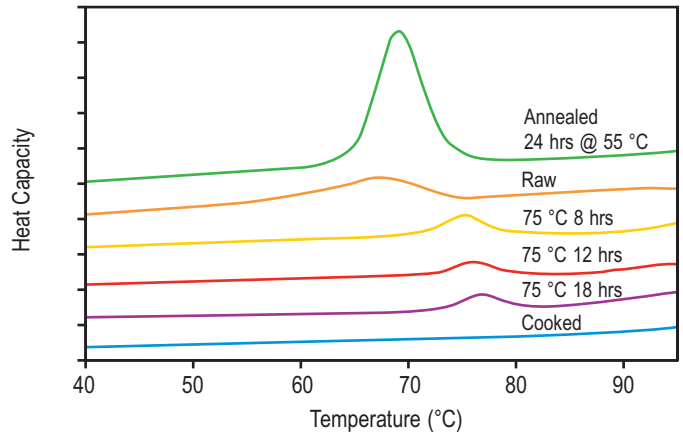
Food Processing

Determination of the temperature dependence of reaction rates is necessary for establishing proper storage, manufacturing, and compounding conditions for foods, drugs, cleaners, and industrial chemicals and products. The rates of reactions in diverse materials can be rapidly determined as a continuous function of temperature with the MC DSC. Continuous scanning is particularly useful for rapidly establishing the optimal temperatures for more definitive, but much slower, determinations of the identity of reactions and of rate laws by isothermal calorimetry and analytical methods. This figure shows data from continuous temperature scans of 0.5 g aliquots of pineapple juice concentrate under various conditions. Hydrolysis of sucrose in the juice produces glucose, a reducing sugar, which is then oxidized by oxygen and also undergoes Maillard reactions with amines in the juice to produce off-flavors and discoloration. Such data can be used to rapidly optimize process conditions.



Phase Transitions in Foods

Phase transitions occurring during cooking, freezing, drying, mixing, and storage of food products are important determinants of texture, taste, and quality. The large, wide-mouth ampoules, wide temperature range, high pressure capability, and multiple sample cells of the MC DSC make it a versatile calorimeter for both temperature scanning and isothermal studies of food products. The data shows temperature scans of whole grain rice after various treatments. The peak is due to gelatinization of crystalline starch remaining after partial cooking.



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