

NETZSCH

Proven Excellence.



Multiple Module Calorimeter MMC 315 *Nexus*®

ARC, Scanning and Coin Cell Modules

Analyzing & Testing

Multiple Module Calorimeter

Synergy of Proven Methods

Scientists and engineers generally recognize that more product and process information can be obtained by multiple analytical methods rather than using a single technique. Different signals can be recorded, and superimposed sample effects can often be much better explained. The technique has to be reliable, fast and easy to use in everyday operations.

The NETZSCH Multiple Module Calorimeter MMC 315 Nexus® is an instrument which can be operated with different modules for the investigation of energetic materials and batteries (coin cells). It can be used in commercial R&D, universities and research centers and in QC/QA for various industries. The MMC is capable of the following on gram-size samples:

- Measurement of chemical reactions
- Measurement of phase changes
- Analysis of process safety
- Characterization of coin cells

This allows for organic/inorganic multiphase mixtures testing. For these tests, the MMC 315 Nexus® can be equipped with four different calorimeter modules: ARC, Scanning and two Coin Cell modules.

The MMC 315 Nexus® consists of two parts: the base unit with electronics and the interchangeable calorimeter modules for maximum flexibility.



MMC 315 Nexus® base unit with status indicator (left) for four interchangeable measuring modules

ARC Module – Process Safety

Knowing the amount of energy released by a given chemical reaction is essential for ensuring safe and reliable processes. The ARC (Accelerating Rate Calorimetry) module allows you to study the worst-case scenarios at elevated temperatures without losing heat to the environment.

ARC systems in accordance with ASTM E1981 have been widely used for decades to simulate the behavior of actual large-scale reactors. However, these instruments have a large footprint. In contrast, the MMC 315 *Nexus*[®] with ARC module has a space-saving table-top design.

Typical ARC Applications

- Chemical process safety
- Thermal runaway (Heat-Wait-Search)
- Storage and transport studies
- Energetic material testing
- Effect of autocatalysis and inhibitors under exaggerated conditions

Scanning Module – Screening of High Reaction Enthalpies

The Scanning module is the choice for more sophisticated applications. It can be used to analyze exo- and endothermal reactions with high energies. The module can be used for both solids and liquids.

Typical Scanning Applications

- Sample screening
- Isothermal tests
- Constant temperature ramp tests



Coin Cell Modules – Testing Battery Coin Cells, Pharmaceuticals or Polymers

The Coin Cell modules are equipped with various sensors – one type optimized for high-temperature applications and another designed for high-sensitivity experiments. These modules are specifically developed for coin cell battery studies. Data obtained from MMC tests can be integrated with results from battery testing systems, enabling comprehensive analysis and visualization of both battery performance and thermal behavior. When filled with pharmaceuticals or polymers, the coin cell container can also serve as a high-volume, DSC-like crucible.

Typical Coin Cell Applications

- Comprehensive characterization of full- and half-stack coin cells to replicate real-world cell performance
- Amount of heat released or absorbed during chemical changes
- Charging/discharging and electrochemical impedance spectroscopy (using external potentiostat)
- Rate of energy change
- Efficiency
- Melting temperature and enthalpy
- Crystallization temperature and enthalpy
- Polymorphism
- Glass transition
- Determination of impurities

Advantages and Key Features of the MMC 315 *Nexus*[®]

- Multiple testing modes in one instrument to cover a wide application range:
 - Scanning mode (constant power, constant heating rate)
 - Isothermal mode (including isothermal charging/discharging)
 - Adiabatic mode with Heat-Wait-Search for process safety tests
- Wide temperature range
- Wide pressure range
- Various sample containers of different materials and volumes
- *Proteus*[®] software for complete evaluation of thermoanalytical data in one plot



Thermal runaway scenarios can be understood by investigating the runaway reaction. Questions arise which can be answered by means of measurements under adiabatic conditions. For this purpose, the NETZSCH MMC 315 *Nexus*[®] can be configured with the ARC module, which can also be equipped with *VariPhi*[®].

The ARC Module

Process Safety

- What is the thermal behavior of the material?
- What is the thermal hazard potential of the material?
- At what temperature does the reaction occur (onset)?
- What is the enthalpy of reaction?
- What is the Time-to-Maximum Rate (TMR)?
- What is the rate of temperature and pressure change?
- How does the material behave within a given time frame; e.g., 24 h?
- What maximum temperature can be expected under adiabatic conditions?
- What is the temperature of no return?
- What are the kinetic parameters?

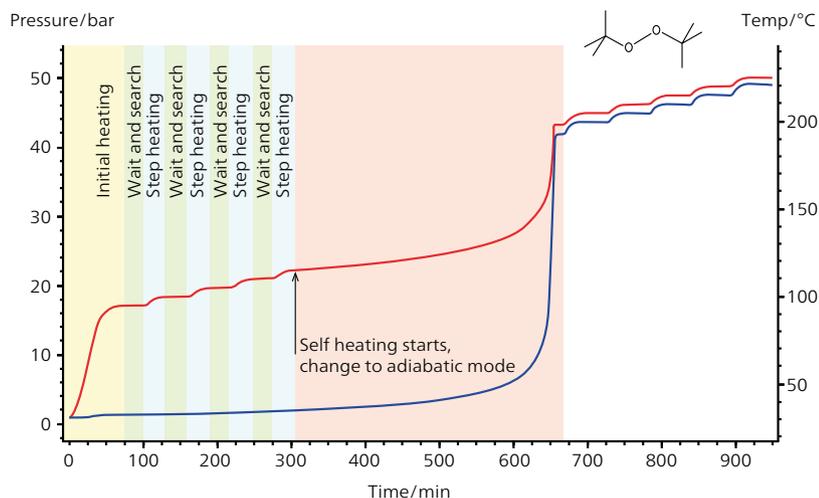
A chemical reaction is said to be 'runaway' when its own heat production rate is higher than heat losses.

Heat-Wait-Search Test

During a Heat-Wait-Search (HWS) test, the sample is heated to a defined temperature (HEAT) and the system is allowed to settle at this temperature (WAIT). The system then checks whether any temperature rise has occurred that stems from the sample itself (SEARCH). If no rise is detected, the procedure starts again by heating to a higher temperature.

In cases where a thermally induced exothermal reaction starts and the self-induced temperature increase of the sample exceeds a predefined value (threshold), the system continues to track the temperature change of the sample.

After the exothermal reaction is finished, the system returns to the HWS operation until either the next exothermal effect is detected or the measurement has completed by means of having reached the maximum temperature (predefined in the software).



HWS test on 20% di-tert-butyl-peroxide (DTBP) in toluene. For the investigation of self-heating behavior, the measurement can include isothermal segments or temperature ramps.

Key Technical Data for the ARC Module

Temperature range	RT to 500°C
Temperature readability	0.01 K
Heating rate	0 to 5 K/min 0 to 2 K/min with <i>VariPhi</i> ®
Pressure limit	150 bar
Pressure readability	0.01 bar
Sample Container volume	0.5 to 8.5 ml
Container	Stainless steel, Inconel, Hastelloy, glass inlet, Titanium
Modes of Operation	Heat-Wait-Search (HWS), constant rate, iso-aging, with <i>VariPhi</i> ®: Heat-Wait-Search (HWS), constant rate, constant power, isothermal
Tracking rate	Up to 50 K/min



The ARC Module – Simulation of the Worst Case Scenario

The best way to understand worst case scenarios is to investigate runaway reactions under adiabatic conditions, which means no heat exchange occurs with the sample's environment.

The ARC module is able to minimize heat loss from the sample container by maintaining the temperature of the surroundings equal to that of the sample temperature.

A defined volume of a sample (ml scale) is placed in a tube-shaped or spherical container which is surrounded by a sophisticated heating system.

If there is no temperature difference between the surrounding heaters and the sample, then all the heat generated by the sample stays inside the sample.

Usually, the sample container has a volume of between 0.1 ml and 8.5 ml.

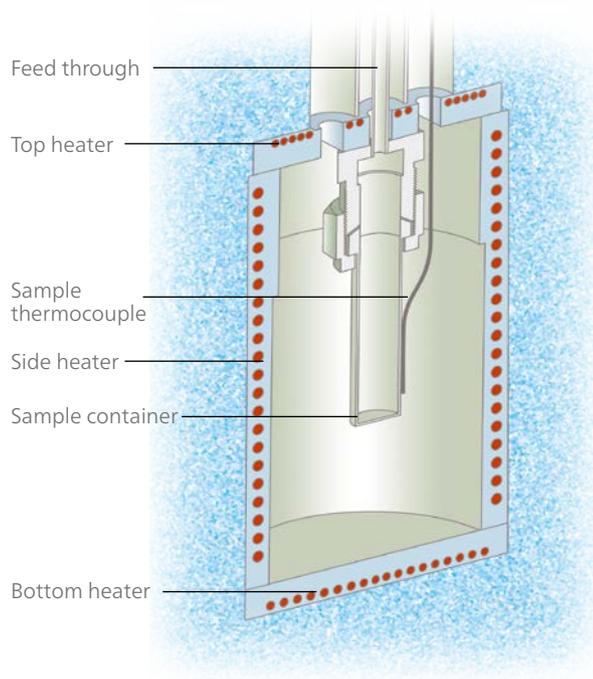
The tracking rate can be allowed up to 50 K/min. Pressures of up to 150 bar can be detected.

Isothermal Aging

It is possible to investigate the sample behavior at a constant temperature using the Iso-Fixed or Iso-Track Mode.

ARC Module – Key Features

- Heat-Wait-Search tests for thermal runaway reactions
- Pressure measurement
- *VariPhi*® (optionally upgradable, see page 7)
- Solid and liquid samples
- Isothermal aging



ARC module

ARC Module with VariPhi® – Determination of Reaction Enthalpies

VariPhi® is an additional controlled variable DC heater which is in contact with the sample material (internal heater). It allows for the definition of thermal inertia for a real-world thermal environment by compensating for heat loss from the sample to the container.

Since both the sample and the container are under adiabatic conditions, the heat generated by the sample causes not only an increase in its own temperature but also in that of the container.

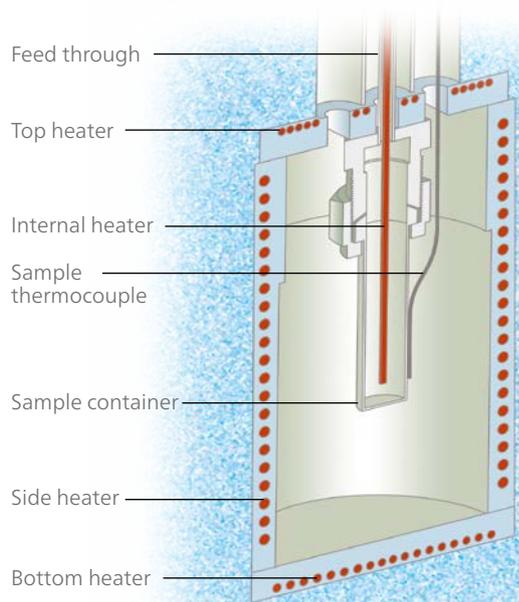
The sample container absorbs some of the energy from its own reaction depending upon its mass and heat capacity.

On a small scale, the thermal mass of the reactor vessel is usually a much larger fraction of the total thermal mass than for large-scale operation. This is taken into account by the sum of the heat capacities of the sample and the container divided by the heat capacity of the sample, which results in what is known as the Φ -factor:

$$\Phi = \frac{T_{ad}}{T_{obs}} = 1 + \frac{m_v \cdot C_{p,v}}{m_s \cdot C_{p,s}}$$

T_{ad} adiabatic
 T_{obs} observed
 m_v mass of the vessel
 m_s mass of the sample
 $C_{p,s}$ specific heat capacity of the sample
 $C_{p,v}$ specific heat capacity of the vessel

Ideally, the Φ -factor amounts to 1.
 In reality, the Φ -factor is always larger than 1.



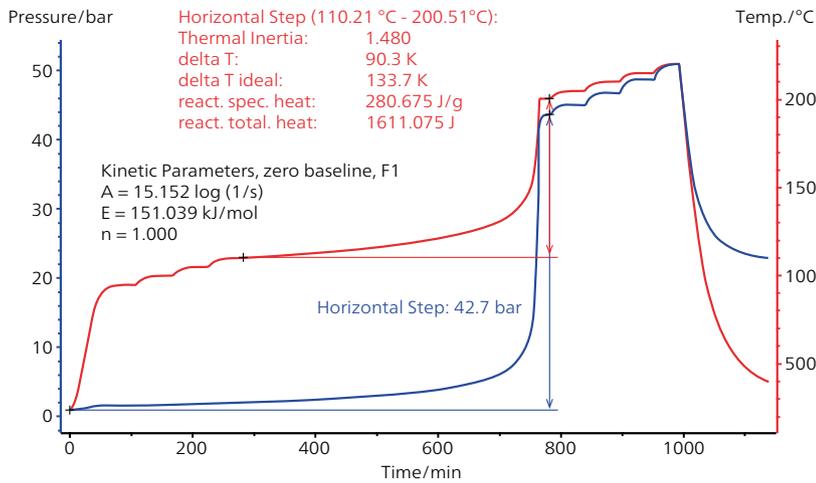
ARC module with optional VariPhi® (internal heater)

Key Features of the ARC Module with VariPhi®

In addition to the key features of the ARC module:

- Compensation for heat loss to the sample container during the test
- Influence of thermal inertia or Φ factor: Scanning and isothermal modes allow for the detection of exo- and endothermic effects
- Scanning mode: Screening to reduce the testing time by 75%
- Fire exposure mode: Simulation of additional heat to the sample

Self-Decomposition Behavior of DTBP in Toluene



Heat-Wait-Search test on 5.74 g of 20% di-tert-butyl-peroxide (DTBP) in toluene in a spherical titanium vessel (mass 10.028 g); exothermal threshold 0.02 K/min, thermal inertia: 1.48

This example shows a test investigating the self-decomposition behavior of DTBP in toluene. At 110°C, heat production from the sample's self-decomposition exceeded the exothermal threshold of 0.02 K/min. Due to this, the HWS mode was changed to an adiabatic mode.

The calorimeter heaters were then tracking the sample temperature. Due to the tracking, the sample temperature and temperature of the surrounding calorimeter were identical and both heat flow and heat loss were avoided. The observed temperature rise of the self-decomposition was evaluated to be 90.3 K. Taking thermal inertia into account, the adiabatic temperature rise was calculated to be 133.7 K.

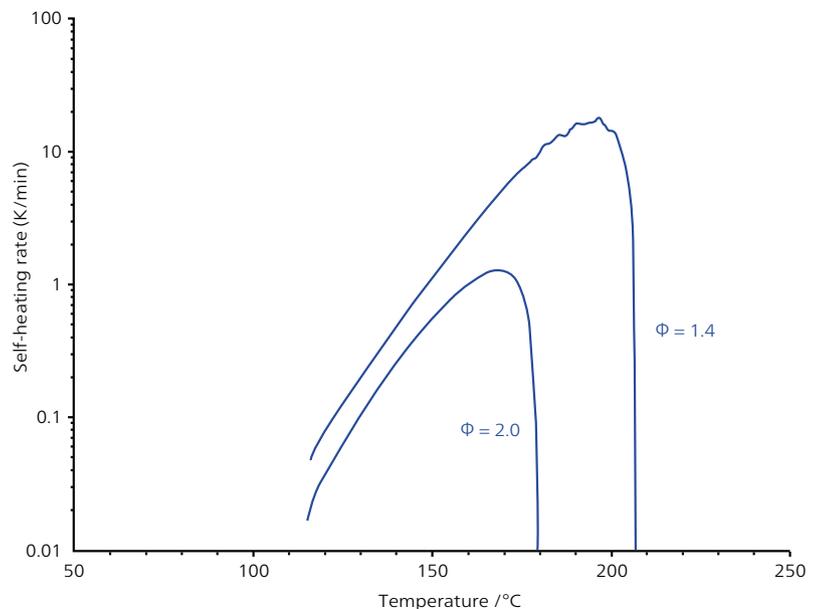
Knowing the specific heat of all of the components allows for determination of the heat of reaction.

The pressure associated with the decomposition reaction was measured to be 42.7 bar. Using a single-step 1st order decomposition mechanism, both the pre-exponential factor and the activation energy can be calculated.

Compensated Φ -Factor

The mass of the container can be completely compensated for when *VariPhi*® is used. This allows for either an adjusted Φ -factor – similar to the real reaction vessel of a plant – or for an ideal Φ -factor close to the value of 1.

This plot shows measurements on 1.25 g of 20% DTBP in toluene with two different Φ -factors. At a Φ -factor of 1.4, the self-heating rate increased by more than one magnitude over what is seen with a Φ -factor of 2.0.



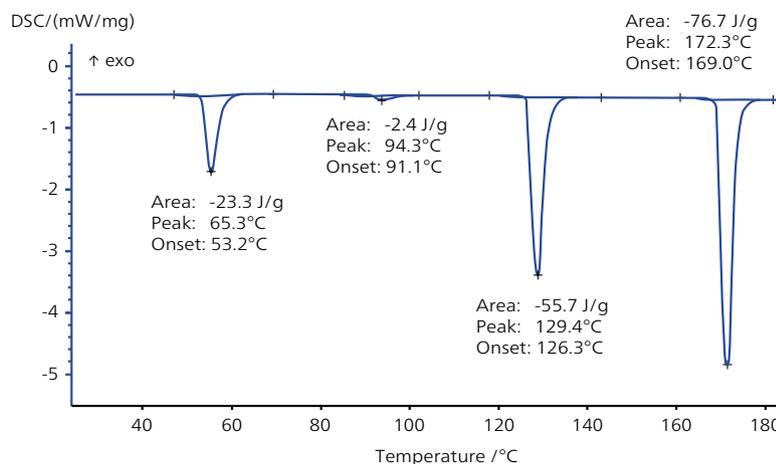
Adjustment of the Φ -factor

MMC with ARC Module and VariPhi in Comparison with Ordinary Differential Scanning Calorimetry (DSC) Tests

Ammonium Nitrate (NH_4NO_3)

DSC Investigation

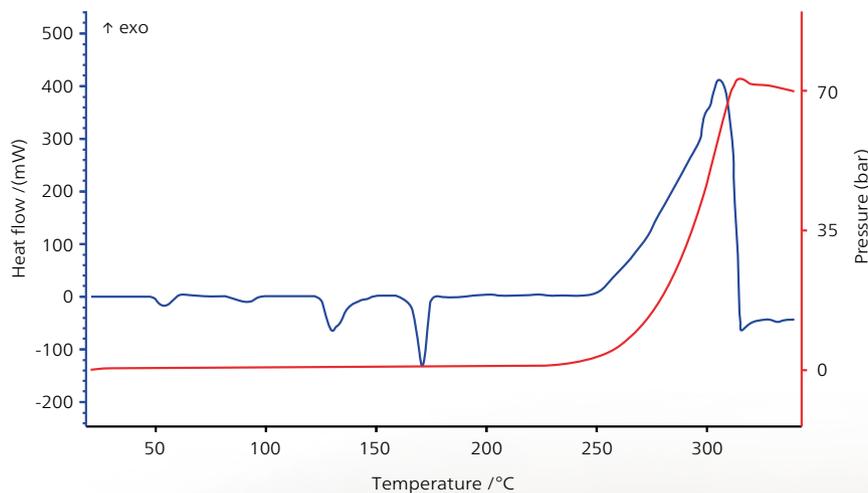
With standard DSC measurements, endo- and exothermal effects are monitored under isothermal or increasing temperature at atmospheric pressure. Usually, the measurement is carried out and evaluated up to the point at which the material melts. The sample is placed in an open crucible or sealed in an aluminum pan with a pierced lid.



DSC 204 F1 Phoenix® measurement on NH_4NO_3 up to 200°C. The sample undergoes three endothermic solid-solid phase transitions followed by melting at 169°C (onset). With increasing temperature after melting, the material will thermally decompose.

Typical MMC Measurements with ARC Module

MMC measurements allow for monitoring a sample's behavior into the decomposition. In contrast with DSC tests, these kinds of measurements are performed in sealed vessels. Decomposition occurs as an exothermal reaction. This is also unlike DSC, in which an endothermic reaction is recorded using an open crucible.



ARC module with VariPhi® plot of NH_4NO_3 with heat flow and pressure versus time

Understanding exothermic reactions is essential for safe processing, storage and transport of chemicals and systems.



The Scanning Module

Screening of Solids and Liquids – Even in Isothermal Tests

The Scanning module has an additional heater which is positioned on the outside of the sample container. This allows for a defined input of power in order to be able to separate endo- and exothermal effects.

The Scanning module is useful for running isothermal and constant temperature ramp tests, especially in experiments where reaction energies are higher.

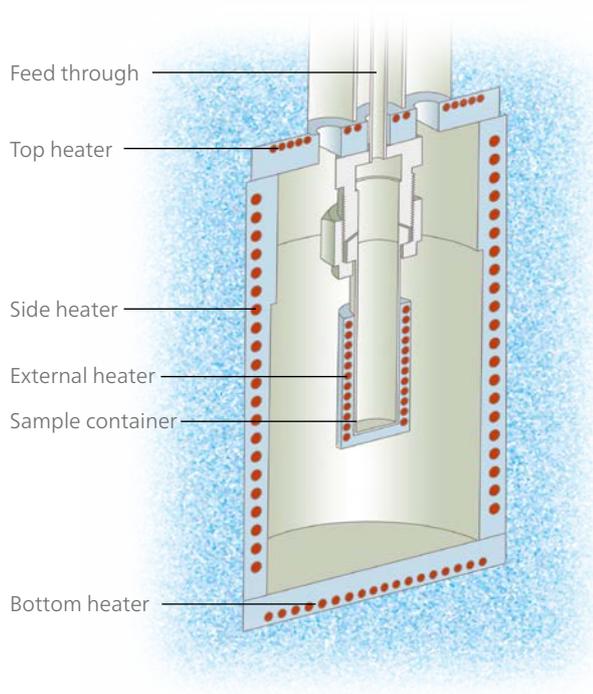
The Scanning module is especially suitable for small masses of highly energetic samples.

Key Technical Data for the Scanning Module

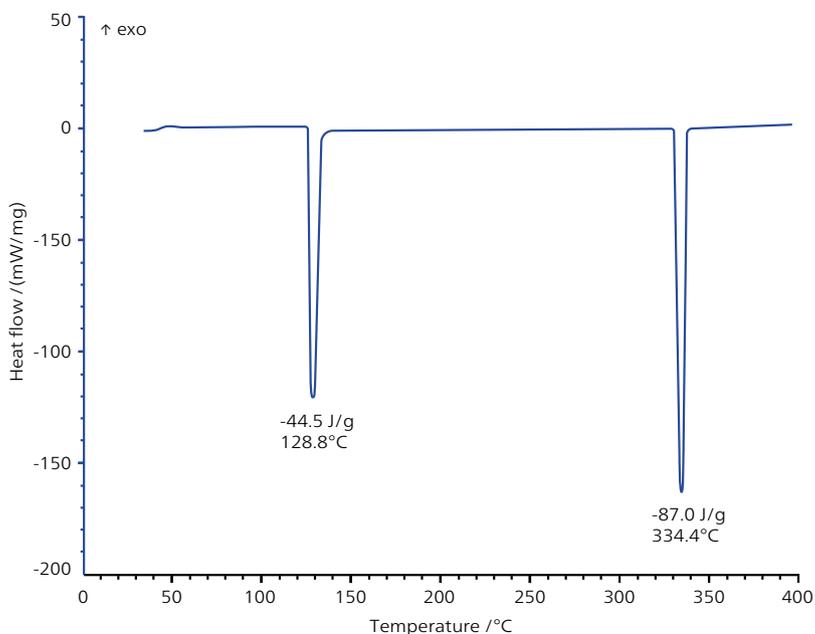
Temperature range	RT to 500°C
Temperature readability	0.01 K
Heating rate	0 to 5 K/min
Pressure limit	150 bar
Pressure readability	0.01 bar
Sample container volume	2.6 ml
Container	Stainless steel, Inconel, Hastelloy, Titanium
Tracking rate	Up to 50 K/min
Modes of operation	Constant rate, constant power, isothermal

Scanning Module – Key Features

- Scanning mode via constant heating rate or constant power
- Isothermal mode
- Determination of endothermal and exothermal enthalpies
- Pressure measurement
- For liquids and solids
- Inhomogeneous samples
- Higher sample masses
- Shorter test times than for Heat-Wait-Search tests



Scanning module with external heater



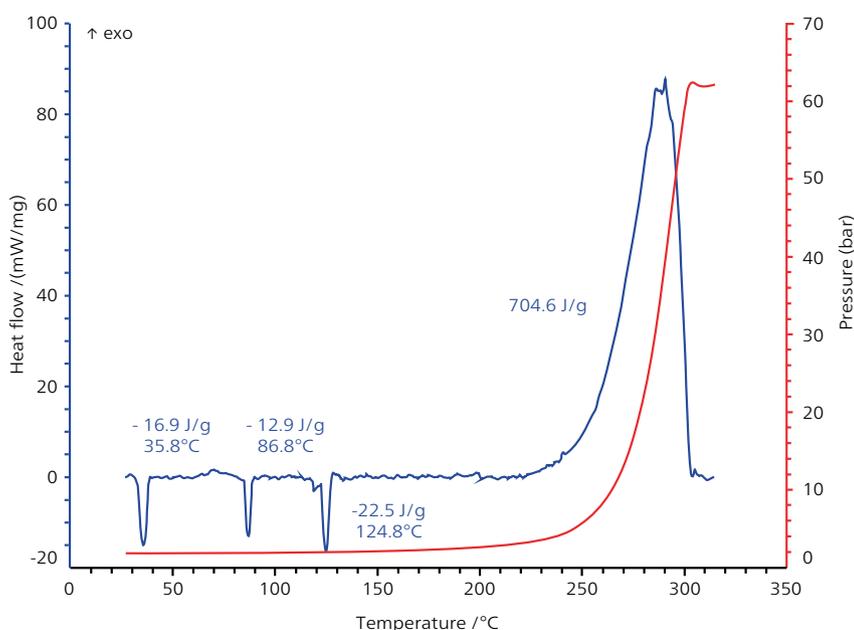
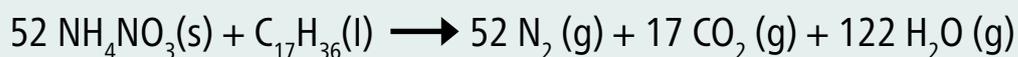
Measurement on potassium nitrate using the Scanning module

Potassium Nitrate (KNO₃)

Besides sulfur and charcoal, KNO₃ is used for the synthesis of gunpowder. It is also used in food preservation (E252) and as an important potassium- and nitrogen-containing fertilizer.

This measurement was carried out in scanning mode with a constant power input of 200 mW. Two endothermic effects, at 129°C (peak area of -45 J/g) and at 334°C (-87 J/g), are related to phase transitions.

Redox reactions that occur rapidly with the production of gases (e.g., nitrogen) are often explosive.



Measurement with Scanning module on an ANFO sample

NH₄NO₃ Fuel Oil (ANC)

ANC is a mixture of solid ammonium nitrate (NH₄NO₃) and fuel oil. It can be safely used in mining, stone quarrying, and tunnel construction. The basic reaction during detonation is the decomposition of the hydrocarbon and the NH₄NO₃ into CO₂, N₂ and H₂O.

For this test, a mixture of NH₄NO₃ and toluene was used. Prior to 150°C, three endothermic phase transitions were observed. For the large exothermic decomposition, an energy release of 705 J/g was detected starting at approx. 235°C.

The Coin Cell Module



Development & Testing
of Batteries, Polymers
and Pharmaceuticals



Advantages of the Coin Cell Module in Battery Testing

- No special sample preparation required
- Analysis of the behavior of a complete coin cell instead of individual components
- Merging of heat generation data with battery data (current, voltage, power)
- Charging and discharging
- Simple interface to a cyclor device

Specifically, an understanding of the heat generation during charging/discharging cycles is crucial for improving the cell efficiency, performance and lifetime of batteries.

Measuring the heat signature of coin cells during cycling provides insight into the underlying processes and provides a quantitative way of comparing changes in chemistry that goes beyond current and voltage measurements.

Some of these processes are reversible, some occur during the initial few cycles, and others take place over a period of weeks, months, or even years. The amount of heat released or absorbed during all these physico-chemical changes and the rate of energy change within the coin cell provide additional pieces of the puzzle and can accelerate the development process.

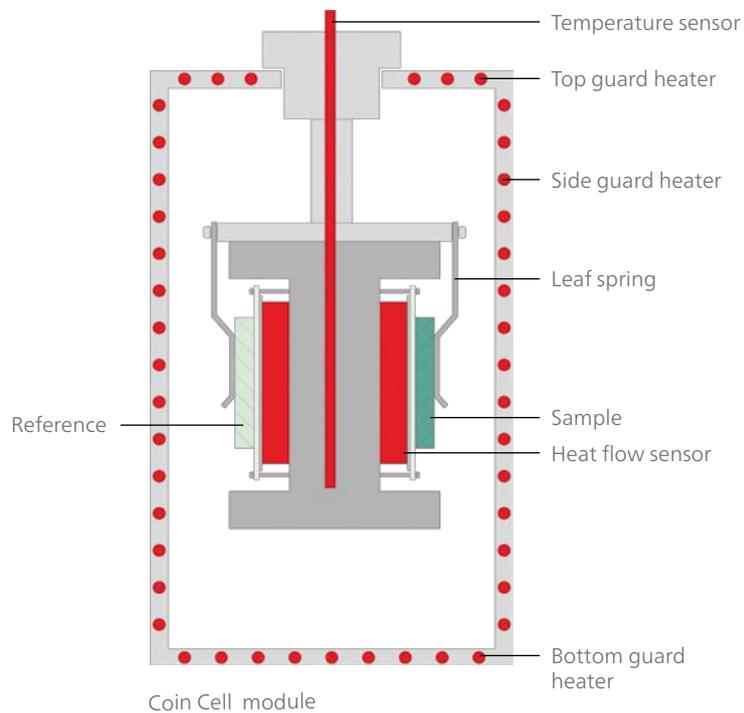
The standard coin cell container, commonly used in battery research, can also serve as a versatile crucible for studying materials other than battery chemicals. The compact coin cell design is ideal for thermal and electrochemical investigations. It can accommodate various substances in a consistent and controlled environment – beyond its traditional role in housing battery components.

The shape of the coin cell allows for a larger sample volume/amount and for measurement over a larger sensor area, making the coin cell a practical tool for a range of thermal and material science applications. For example, in calorimetry studies, coin cells can be filled with alternative materials like pharmaceuticals or polymers.

The Coin Cell module is specially designed for caloric measurements, using coin cell containers for the sample. For battery tests, the instrument can be connected with a fully featured battery analyzer.

The user can perform charge/discharge tests to evaluate battery condition, cycle batteries to improve performance, and gain insight into overall battery condition in an isothermal mode.

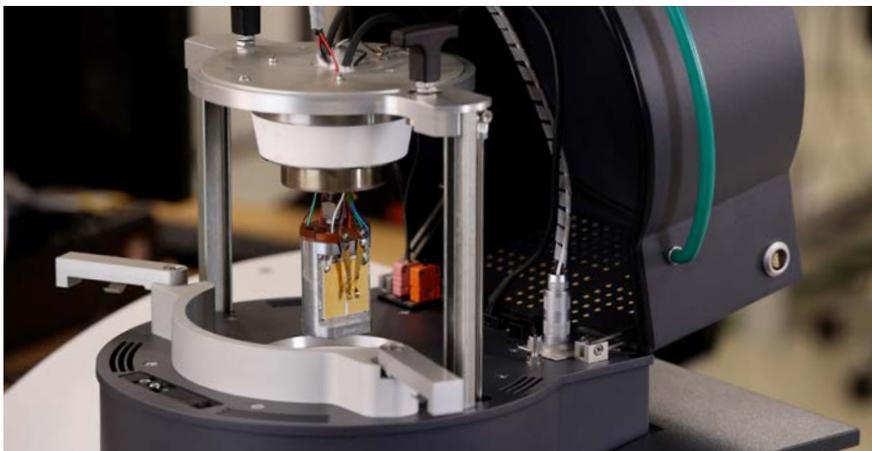
When the coin cell is filled with materials such as pharmaceuticals or polymers, the Coin Cell module acts as a high-volume differential scanning calorimeter (DSC), which characterizes, such points of interest as melting peaks and impurities.



Differential Heat Flow Sensors

The core components of these modules are the sensors, which incorporate an innovative differential measurement design using heat flow sensors to enhance both sensitivity and stability.

When the sample – such as a coin cell – releases or absorbs heat due to charging/discharging or internal physical changes, this thermal response is detected by the sensor. To nullify the effect of external disturbances, the signal coming from the reference heat flow sensor is subtracted from the signal detected by the sample heat flow sensor, thus yielding a true differential measurement, with all its advantages.



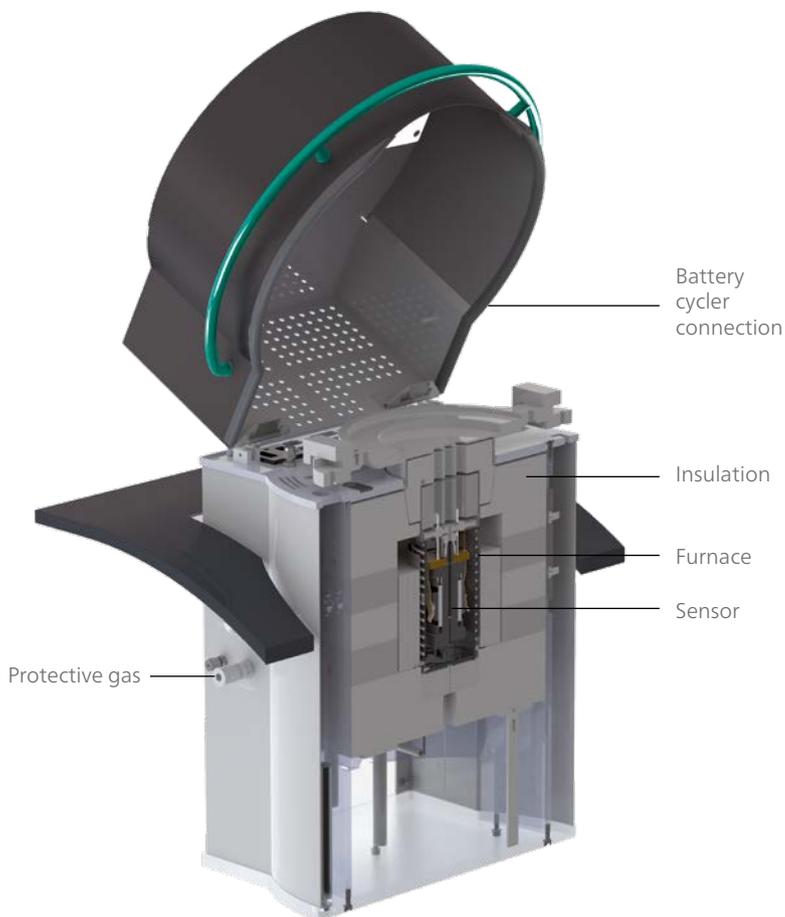
Advantages of the Coin Cell Module as a Differential Scanning Calorimeter

- Better representation: Larger samples provide a more representative thermal profile, which is important for complex or non-uniform materials.
- Enhanced sensitivity: The system allows for highly reproducible measurements, detecting subtle thermal transitions such as melting, crystallization, and polymorphic changes.

The Coin Cell Module for High-Quality Tests on Batteries

Outstanding Value of the Coin Cell Module

- The first calorimeter specifically designed for coin cell measurements
- Unique differential measuring principle for improved stability and sensitivity; captures even weak heat signals from coin cells
- Comprehensive characterization of coin cells to replicate real-world performance
- Easy operation
- Complete characterization of coin cells by isothermal charging/discharging and electrochemical impedance spectroscopy (EIS)



Cross-section of the Coin Cell module



Connection to an External Battery Testing System Made Simple

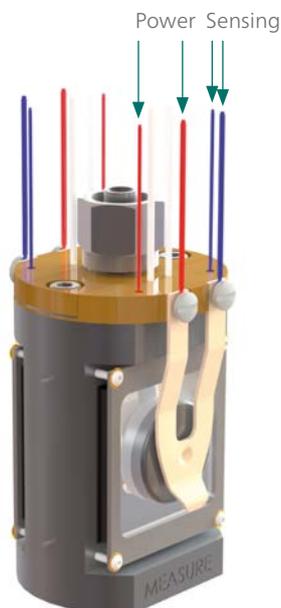
The Coin Cell module for MMC 315 Nexus® is designed to be connected to a battery testing system through a LEMO connector located on the right side of the instrument's hood.

A four-wire system is used to charge and discharge the coin cell: two wires provide electrical power, and the other two measure the exact voltage at the coin cell. This wiring alternatively allows the use of a potentiostat to record electrochemical impedance spectra of the battery under investigation and thus better characterize it. Data coming from the battery testing system can easily be imported into the *Proteus*® analysis software for combined analysis.

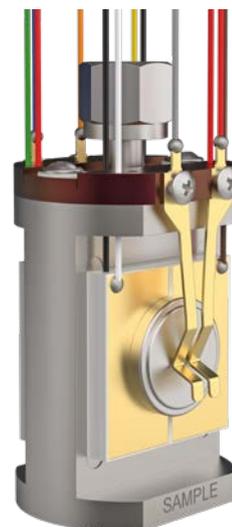
Key Technical Data for the Two Coin Cell Module Variations

	Coin Cell Module with High-Temperature Sensor	Coin Cell Module with High-Sensitivity Sensor
Temperature range	RT to 300°C	RT to 200°C
Temperature readability	0.01 K	0.01 K
Limit of detection	0.1 mW	0.05 mW
Heat flow range	± 4500 mW	± 350 mW
Heating rate	0 to 2 K/min	0 to 2 K/min
Sample sizes	<ul style="list-style-type: none"> ▪ Typically Coin Cell CR2032 ▪ Diameter: 5 to 30 mm ▪ Volume of up to 1 ml ▪ Minipouch 25 x 30 mm ▪ Thickness: 1 to 5 mm 	<ul style="list-style-type: none"> ▪ Typically CR2032 ▪ Diameter: 5 to 30 mm ▪ Volume of up to 1 ml ▪ Minipouch 25 x 30 mm ▪ Thickness: 1 to 5 mm
Modes of Operation	Isothermal, constant heating rate	Isothermal, constant heating rate
Number of sample cell/ reference cell	1/1	1/1

The Coin Cell Module – Superior Performance in Two Variations: High-Temperature (HT) and High-Sensitivity (HS)



Coin Cell – High-Temperature Sensor



Coin Cell – High-Sensitivity Sensor

Lithium Coin Cell

Test parameters of the measurement on the Li coin cell:

- Type of sample: Coin cell LiR2032
- Isothermal mode with temperature set at 35°C
- Charging/Discharging cycle; constant current – constant voltage (CC-CV) – 40 mA from 4.2 V to 3.0 V

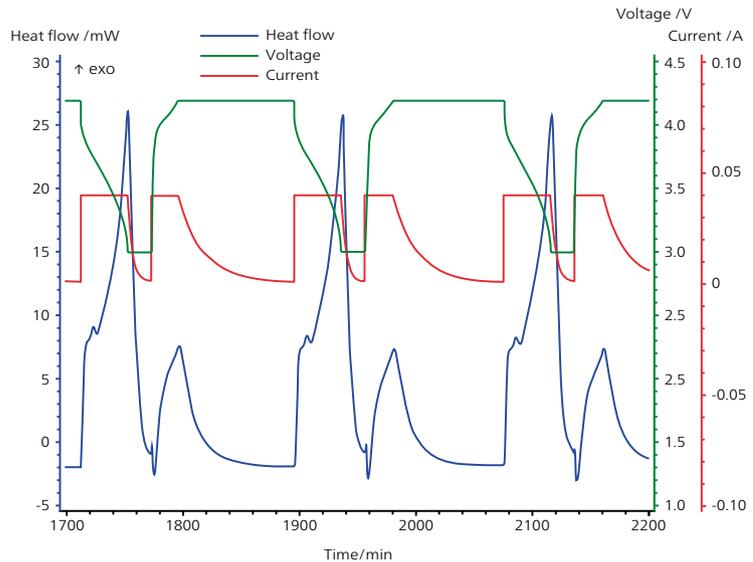
The measurement curves (upper plot) show the exothermal signature of the LiR2032 during three cycles of discharging and charging.

The efficiency is calculated by dividing the electrical energy by the total energy (electrical energy + heat flow) during discharging and charging (lower plot).

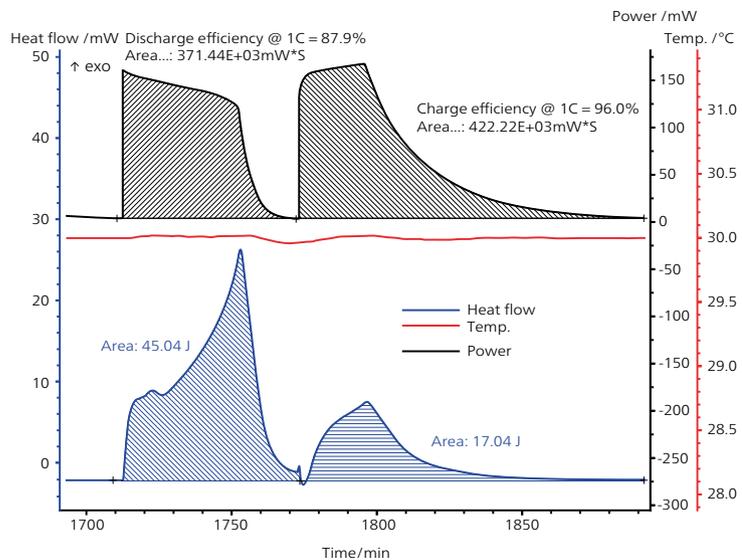
Calculation of efficiency



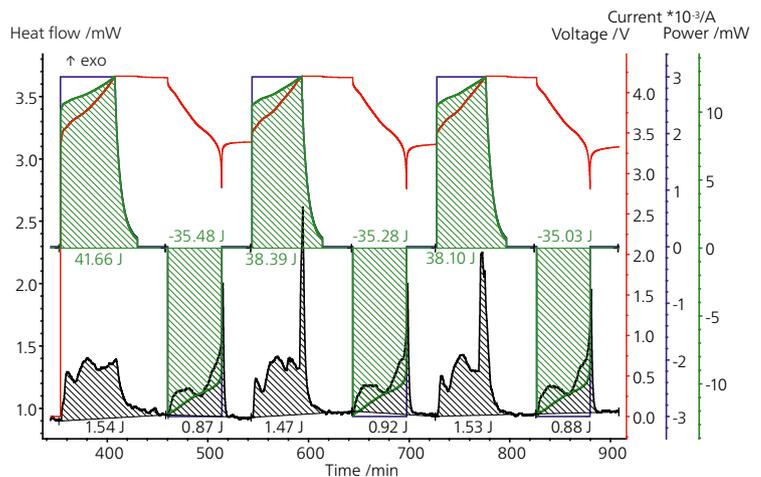
Empty coin cell cases for the Coin Cell module



Three isothermal cycles of discharging and charging of a commercial LiR2032 (40mAh) coin cell



The same data was used to calculate the cell efficiency during discharging and charging

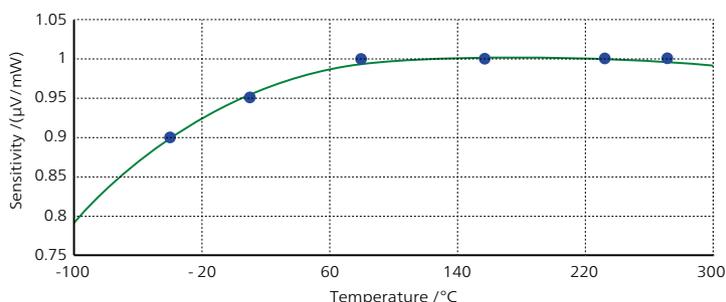


Three isothermal cycles of discharging and charging of an experimental NMC 3 mAh coin cell

Calibration Methods for Scanning and Isothermal Experiments

Temperature and Enthalpy Calibration Over the Entire Temperature Range Using Melting Standards

For calibration of the Coin Cell Module, traditional melting standards have long been relied upon to calibrate instruments in scanning experiments. NETZSCH offers a calibration set containing four reference materials. The lower plot shows a measurement on indium. The peak area and onset temperature for the melting effect are in good correlation with literature data. Enthalpy and temperature calibration are achieved through evaluation of all measurement files for the reference materials; a calibration polynomial is created by the software.

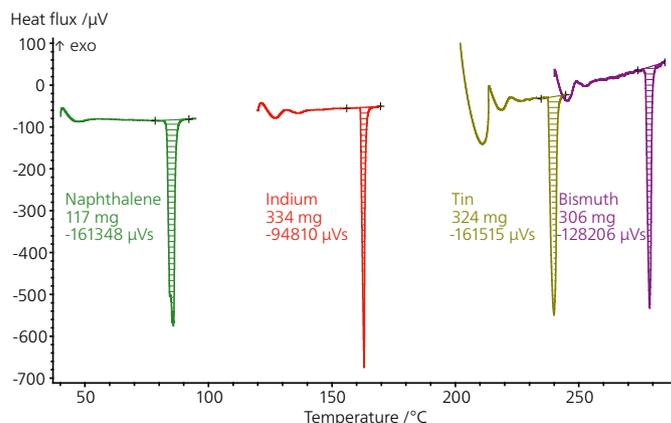


Sensitivity calibration polynomial derived by melting standards

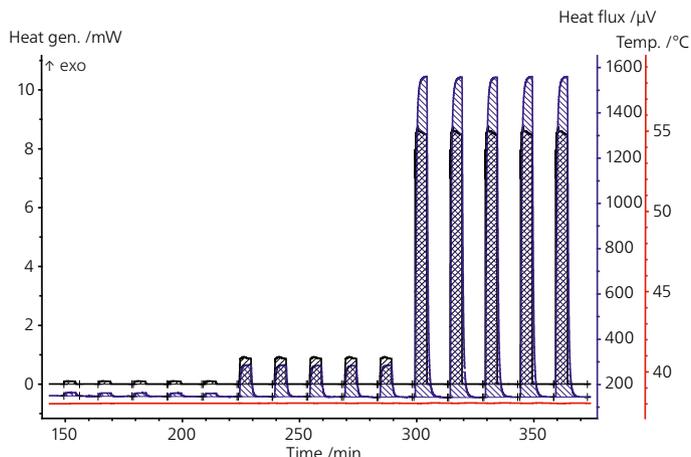
TrueJoule Calibration for Isothermal Experiments

Traditional melting standards are suitable for calibrating measuring instruments for experiments involving dynamic temperature control. However, a different isothermal calibration method is preferred for heat flux sensors in isothermal measurement methods, which are mainly used in research and the quality testing of batteries. This allows for the highest level of accuracy in thermal analysis.

The *TrueJoule* calibration is revolutionizing the field. It uses precise electronic heat pulses with variable power generated by the MMC 315 *Nexus*[®], combined with an in-situ calibration cell, to effectively neutralize the influence of sensor design and cell housing on measurement results. This unique calibration approach allows for isothermal calibration at any temperature and automatically delivers accurate power pulses directly into the calibration cell. It can accommodate various sample containers, including the common 2032 format. The result is the highest level of measurement reliability, which is essential for cutting-edge battery R&D and quality control.

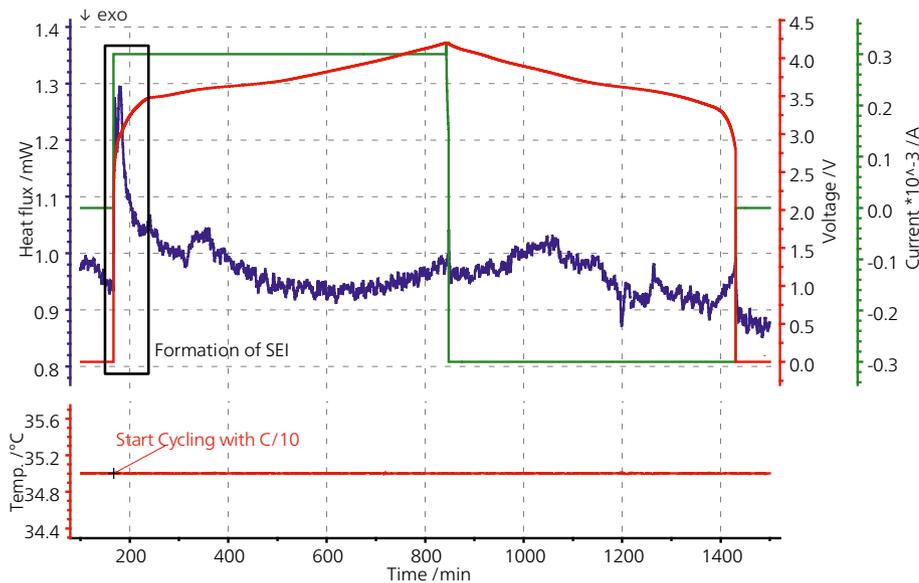


Calibration between 40°C and 270°C using various melting standards



TrueJoule calibration at 35°C, showing five pulses and sensor response at 0.1, 1.0 and 10 mW.

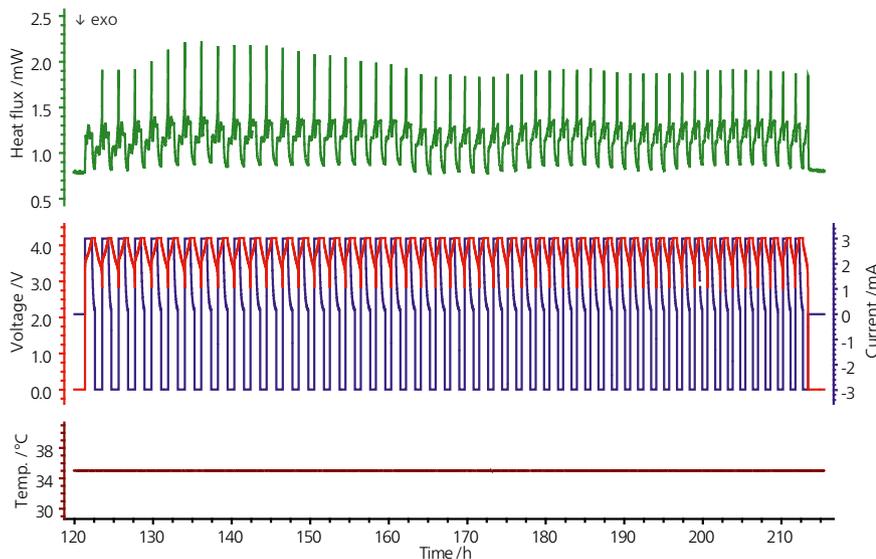
Formation of the Solid Electrolyte Interphase (SEI) in Batteries



Measurement on a newly produced lithium-ion battery using the high sensitivity module

The new module's high calorimetric sensitivity and resolution enable the battery formation process to be studied in detail. For example, it can measure the heat generated when the protective layer known as the solid electrolyte interphase (SEI) forms on the anode of a newly produced lithium-ion battery during the first charge cycles. This helps researchers to better understand and optimize battery performance and lifespan.

Battery Cycling

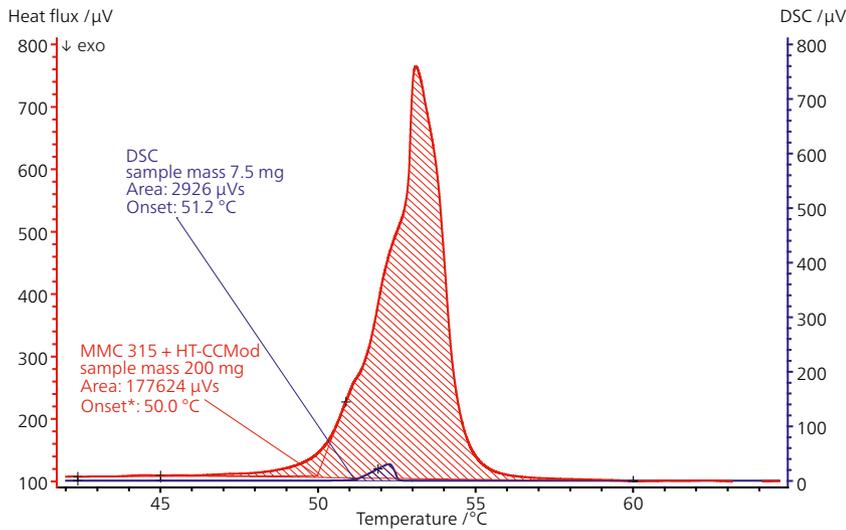


50 times cycling at 1C of a 3 mAh experimental NMC Coin Cell using the high sensitivity module

The High-Sensitivity Coin Cell module provides valuable information about the thermal behavior of new battery chemicals during the cycling process. This allows researchers to understand how these advanced cells perform over time. Long-term cycling tests with this module reveal crucial thermal and electrical characteristics of innovative battery formulations. Thanks to its open electrical design and the *Proteus*® analysis software's versatile import filter, the module can be seamlessly integrated with your preferred potentiostat, offering flexibility and user-friendliness.

You can trust the module to provide accurate, detailed data that supports the development of safer, more efficient batteries while optimizing your existing lab setup and accelerating research outcomes.

Comparison of DSC with MMC Results on (S)-(+)-Ibuprofen



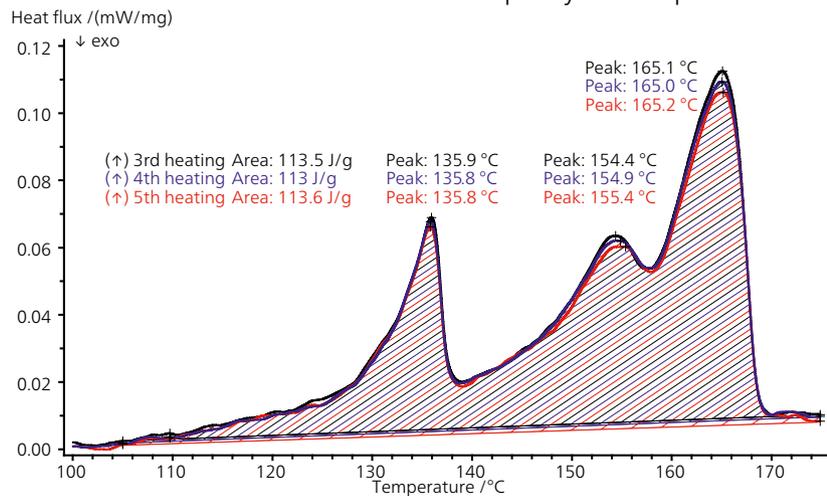
Measurement on (S)-(+)-Ibuprofen; sample mass 200 mg, 0.5 K/min using the MMC high-sensitivity module

Taking small samples from large batches carries the risk of inaccurate or misleading predictions. However, the MMC 315 *Nexus*[®] allows you to test larger, more representative samples, thereby improving the reliability of your results. For instance, experiments employing the High-Sensitivity Coin Cell module to study the melting behavior of (S)-(+)-Ibuprofen revealed exceptional sensitivity and strong comparability with established differential instruments, such as the DSC. The DSC-like sensor can be trusted to deliver precise, high-quality thermal data on bulk samples, thereby enhancing material characterization and ensuring better decision-making in research and quality control processes.

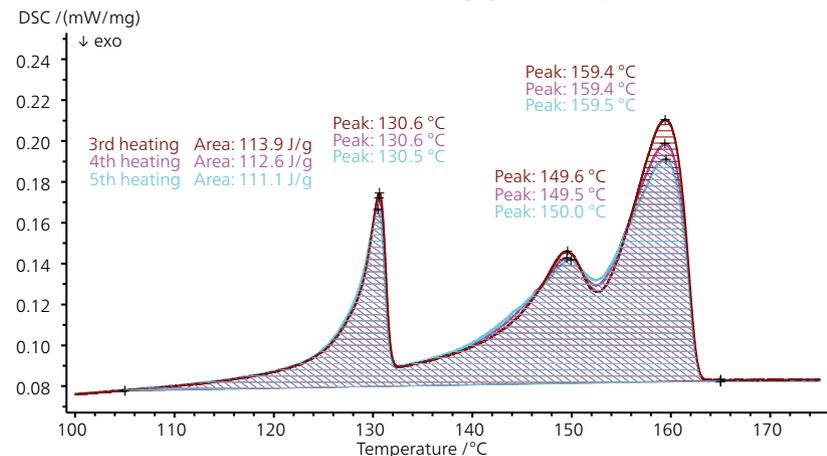
Impurities in Polypropylene

The High-Sensitivity Coin Cell module functions as a high-volume DSC, enabling the analysis of larger sample sizes with exceptional sensitivity. This allows the detection of even smallest impurities in polymer samples.

In this example, the sensor captures detailed temperature scans of polyethylene (PE) and polypropylene (PP) mixtures, revealing substantial differences that smaller sample analyses might miss. The ability to work with larger, more representative samples addresses common challenges in sampling accuracy and yields more reliable, meaningful results. Such precise thermal measurements are essential for quality control and advanced polymer development research, helping optimize material performance and purity.



A mixture of polypropylene and polyethylene powder was measured at a rate of 1 K/min from 80 °C to 175 °C. The mass ratio of the samples was 116.2 mg to 12.0 mg. A standard CR2032 coin cell was used as the sample vessel in the MMC 315 *Nexus*[®] High-Sensitivity Coin Cell module. The third, fourth and fifth heating cycles are depicted.



A mixture of polypropylene and polyethylene powder was measured at a rate of 1 K/min from 80 °C to 175 °C. The mass ratio of the samples was 10.01 mg to 1.00 mg. This was done using a *Concavus* aluminum crucible with a pierced lid in a DSC 300 *Caliris*[®]. The third, fourth and fifth heating cycles are depicted.

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