APPLICATION **NOTE**

About the Calibration of the Coin Cell Module of the MMC 274 *Nexus*®

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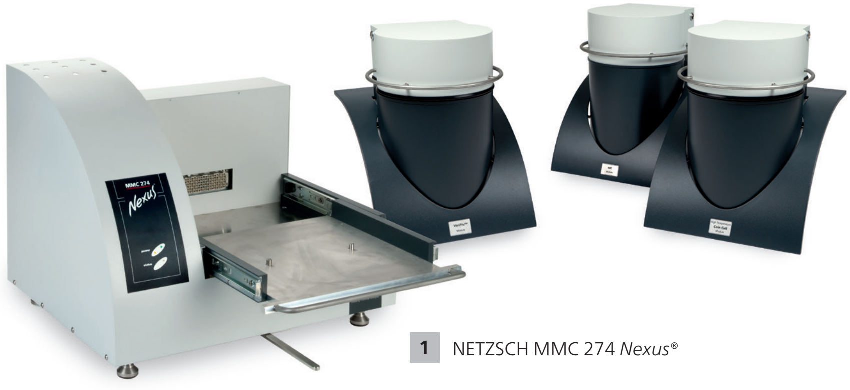
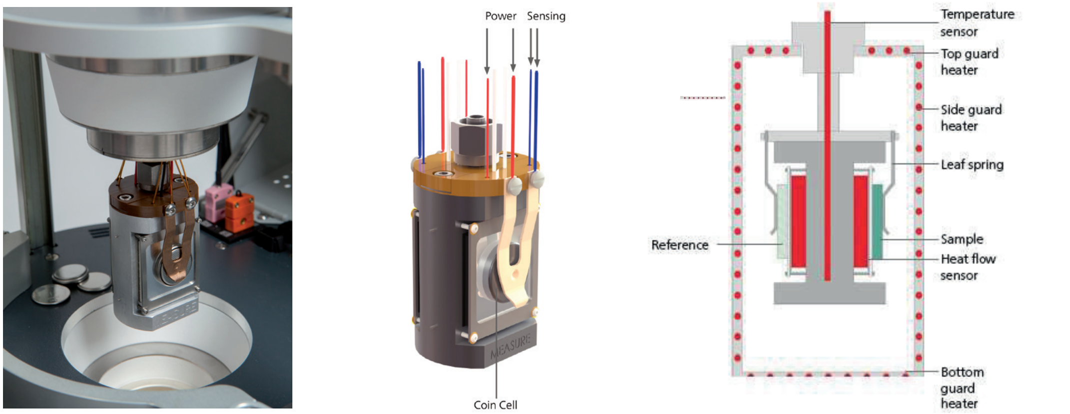


1 NETZSCH MMC 274 *Nexus*®

Introduction

The NETZSCH Multiple Module Calorimeter (MMC) 274 *Nexus*® (Figure 1) offers three different measurement modules. The ARC Module can be used for so-called heat-wait-search (HWS) tests or thermal runaway tests [1][2]; the Scanning Module is suited for such applications as the evaluation of endothermic or exothermic phase transitions as well as thermal hazard screening [3][4]; and the Coin Cell Module is specialized for the investigation of batteries [5]. An external battery cycling unit can easily be connected to the Coin Cell Module via a LEMO connector. Signals for voltage and current can be transferred to the Proteus® evaluation software; the resulting power signal is automatically determined and quantified for charging and discharging independently. By detecting the heat loss during charging and discharging, it is possible to evaluate the efficiency of cycling a battery. To this end, the twin sample carrier offers a DSC-like differential setup (figures 2a, b, c).



2 Schematic drawing of the Coin Cell Module; picture of the sample holder (left), four-wire technique (middle), diagram of the differential set-up (right)

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Since most of the non-destructive isothermal charging and discharging studies of batteries are carried out within a very small temperature range near ambient temperature, it is essential to have the calorimeter calibrated accordingly. For temperature and sensitivity calibration, metals are usually used as reference materials.

Temperature and Sensitivity Calibration

Empty coins (figure 3) can be used in a similar way to DSC crucibles in order to prepare samples or reference materials. The MMC Coin Cell Module allows for scanning at moderate heating rates, which minimizes the dynamic shift and improves comparability to isothermal measurements such as those for cycling a battery.

Typical calibration materials along with the corresponding sample masses are summarized in table 1. A calibration kit established this way for the MMC Coin Cell Module is shown in figure 4.

Gallium is a certified and well-established calibration material for temperature and enthalpy, recommended by several institutions [6]. Nevertheless, it is rarely used since it reacts with aluminum, which is the crucible material most frequently used in DSC. However, its melting temperature is only slightly above ambient temperature. Since the coins are made of steel and the applied heating rates are comparatively low, the above-mentioned drawbacks are not relevant in terms of the MMC Coin Cell Module.

3 Empty 2032 coins (reference) and coins filled with calibration materials

Tab 1. Materials and masses of the calibration kit of the MMC Coin Cell Module

Calibration Material	Sample Mass [mg]	Melting Temperature [°C]	Melting Enthalpy [J/g]
Gallium	473.9	29.76	80.2
Indium	334.0	156.6	28.6
Tin	324.0	231.9	60.5
Bismuth	306.0	271.3	53.8

4 Calibration kit for the MMC Coin Cell Module

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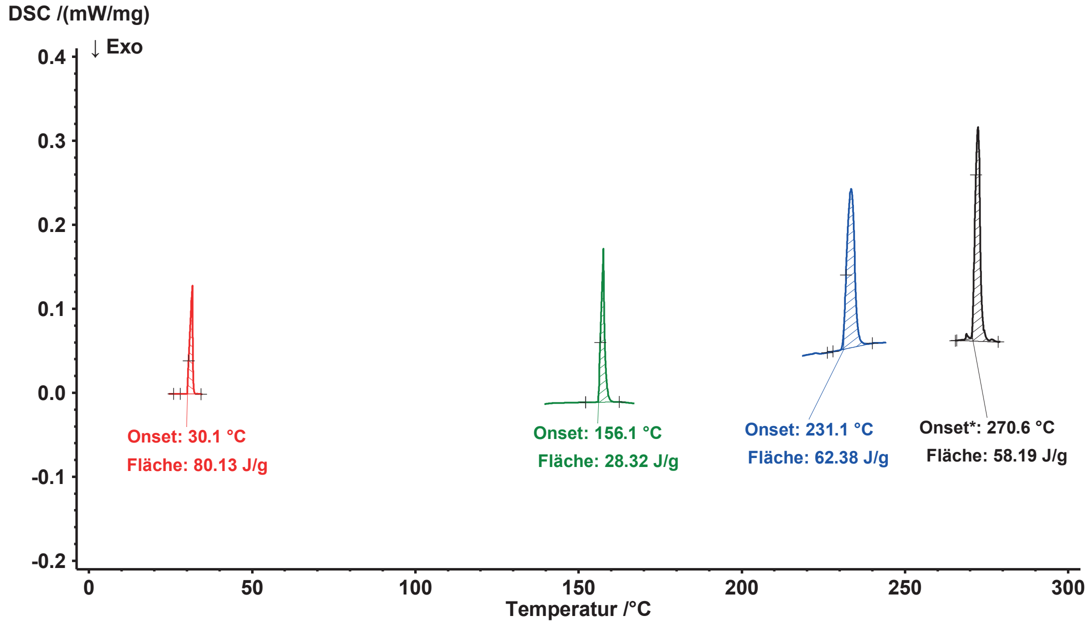
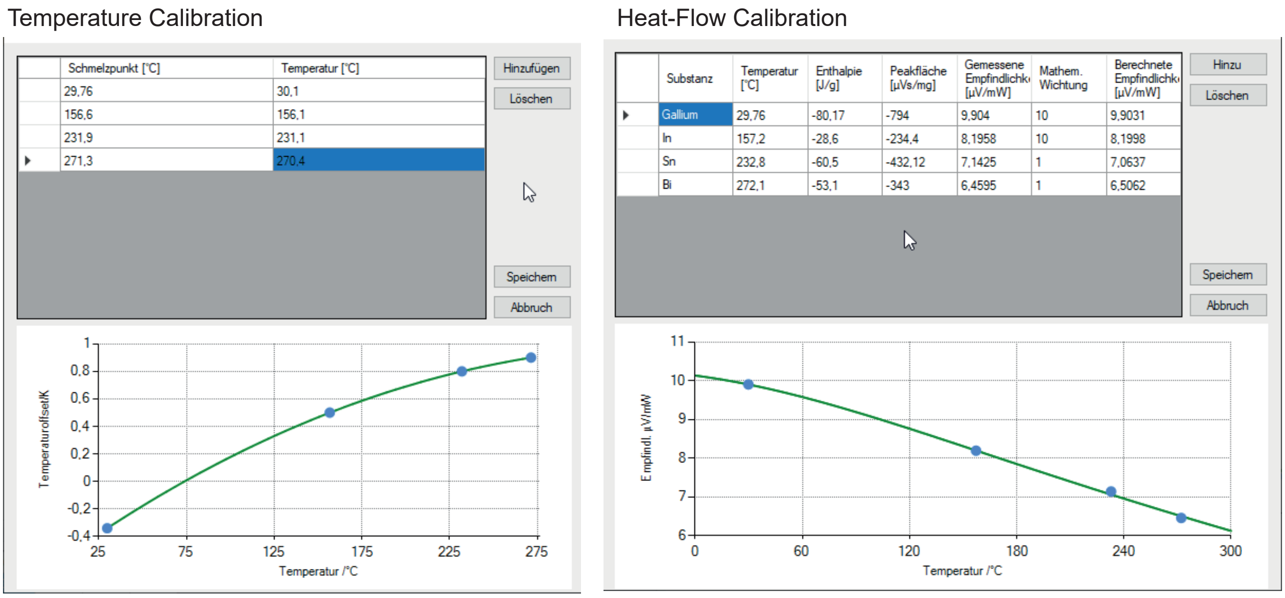
The results for the melting behavior of the above-discussed reference materials are depicted in figure 5. The calculated calibration polynomials for temperature and sensitivity are shown in figure 6. In order to double-check the calibration polynomials for both temperature and sensitivity, naphthalene ($C_{10}H_8$), was used.



5 Measurement results for the melting behavior of gallium, indium, tin and bismuth



6 Calibration polynomials for temperature (left) and sensitivity (right)

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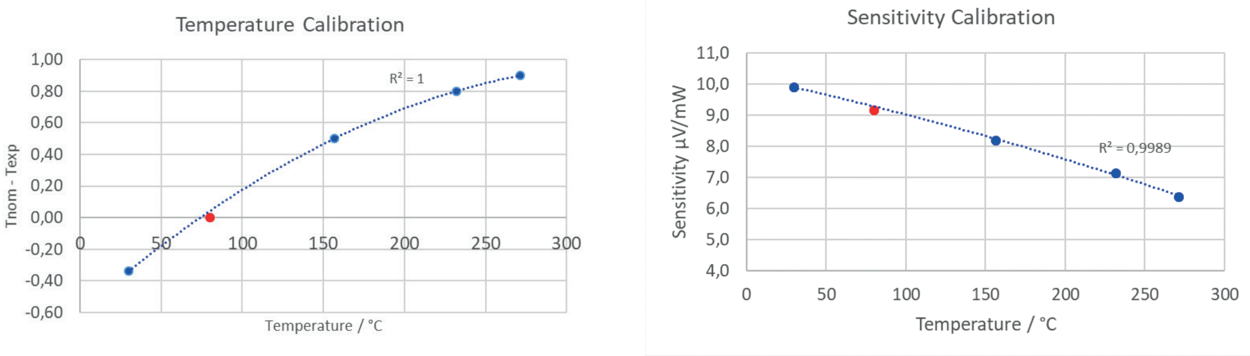

7 Calibration polynomials for temperature (left) and enthalpy (right) with additional results for naphthalene ($C_{10}H_8$) (red)

As the results obtained for naphthalene were in good accordance with the calibration polynomials that were determined using the metal samples, these nicely confirm the validity of the calibration (figure 7).

Conclusion

These results nicely demonstrate the capability of the MMC Coin Cell Module with regard to temperature and enthalpy calibration. The use of gallium as a calibration material is very important since proper calibration near ambient temperature is essential for battery applications. Isothermal cycling of batteries is usually carried out close to or slightly above ambient temperature. More common calibration materials such as indium, for instance, with a melting temperature of 156.6°C, would be too distant from the required range of application.

Literature

[1] Application Note 131, E. Füglein, "Hazard Potential of Decomposoition Reactions Using the Example of Hydrogen Peroxide (H_2O_2)"
[2] Application Note 134, E. Füglein, "*Varify*® – The patented Immersion Heater for Variation of the ϕ-Factor in Thermal Runaway Tests"
[3] Application Note 130, E. Füglein, S. Schmölzer, "Epoxy Curing Investigated by Means of the DSC 214 *Polyma* and MMC 274 *Nexus*®"
[4] Application Note 132, E. Füglein, "Screening of Hydrogen Peroxide Solutions by Means of Scanning Tests and ARC Tests"
[5] Application Note 040, J.-F. Mauger, P. Ralbovsky, G. Widawski, P. Ye, "Coin Cell Cycling in a Novel DSC-Like System"
[6] Sarge M.S., Gmelin E., Höhne G.W.H., Cammenga H.-K., Hemminger W., Eysel W. „The caloric calibration of scanning calorimeters" Thermochimica Acta 247 (1994) 129-168

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