SOFTWARE **INNOVATION**

HFT *MultiCalibration* (for the HFM 446 *Lambda* Eco-Line)

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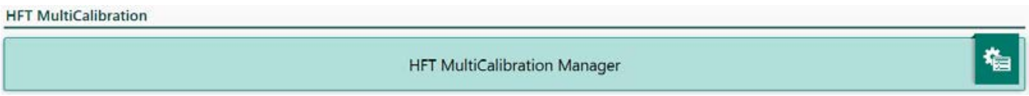
The NETZSCH HFM 446 *Lambda* Eco-line has been released with the new firmware version 6.xx and the *SmartMode* software version 8.5. One innovation is the possibility to create and apply so-called HFT *MultiCalibrations* using the HFT *MultiCalibration* Manager (see figure 1).

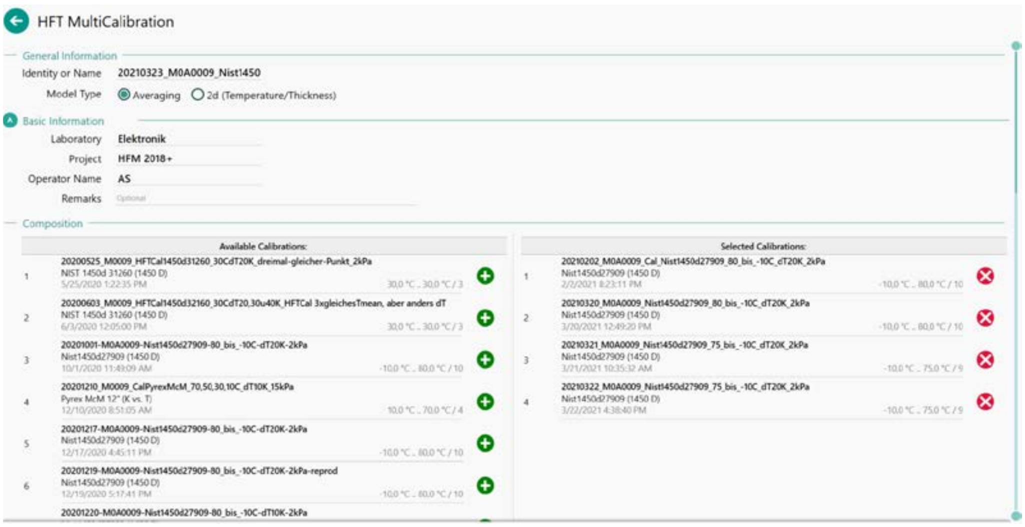
Basic Idea

The heat flux transducers (HFTs) of a heat flow meter (HFM) device need to be calibrated using a standard reference material for thermal conductivity. After an HFT calibration was done and applied, the HFT signals – initially in unit [V] – are shown in [W/m^2] and accurate thermal conductivity or specific heat capacity measurements can be carried out. The principle of HFT *MultiCalibration* is the grouping and mathematical combination of existing HFT calibrations. Figure 2 shows the user interface when creating or editing an HFT *MultiCalibration*.

A name and basic information (laboratory, project, operator, remarks) are entered and a model type for the mathematical combination of the HFT calibrations must be chosen, as explained below. Under "Composition", available HFT calibrations are listed on the left, already selected HFT calibrations are shown on the right. The selected HFT calibrations are included in the HFT *MultiCalibration*.



1 HFT *MultiCalibration* Manager in *SmartMode* 8.5



2 HFT *MultiCalibration SmartMode* 8.5 user interface

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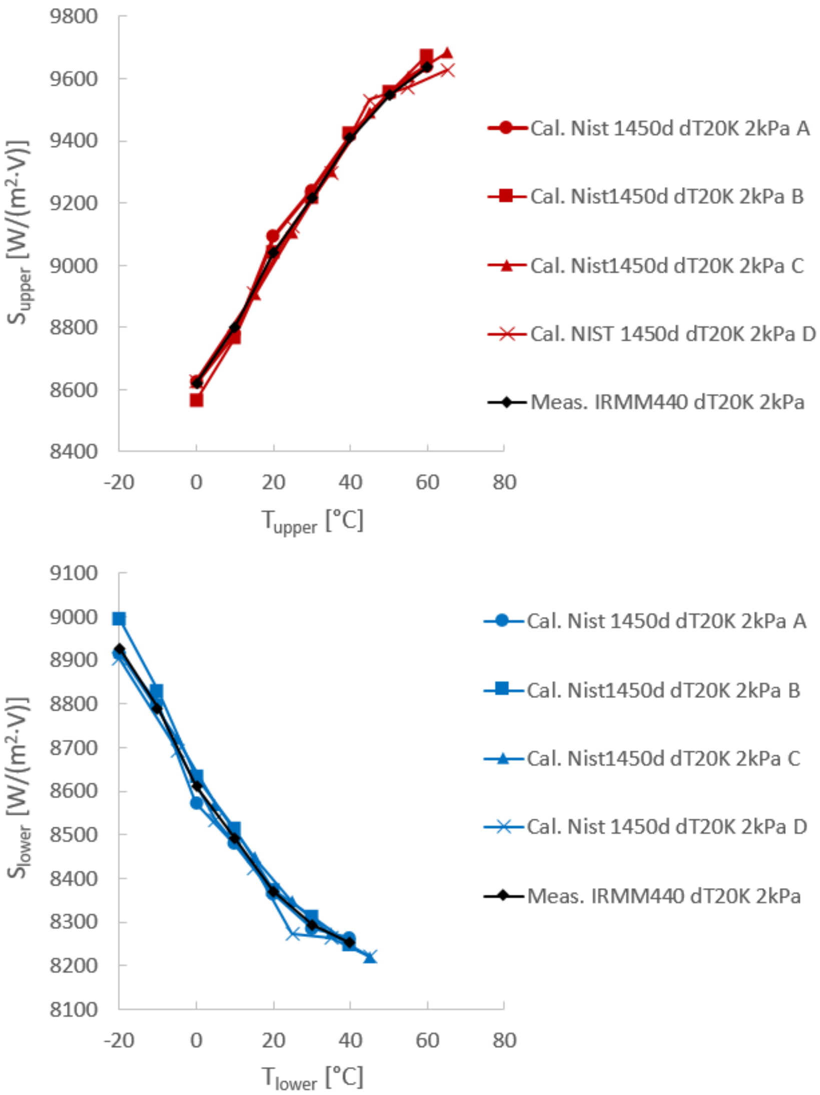
Model Type: Averaging

The model type "Averaging" is illustrated in figure 3. The exemplary HFT *MultiCalibration* contains four single HFT calibrations carried out on the same NIST[1] SRM[2] 1450d sample featuring 30 cm x 30 cm x 2.5 cm in an HFM 446 *Lambda Medium*. All HFT calibrations are represented by the temperature-dependent S-factor curves for the upper and lower plate, respectively.

For a test measurement on the standard reference material from IRMM (Institute for Reference Materials and Measurements, European Commission), called IRMM440, this HFT *MultiCalibration* was applied. The S-factors used for calculating the thermal conductivity λ of the IRMM440 specimen are average values calculated from the four single HFT calibrations of the HFT *MultiCalibration*, as can be seen from figure 3.



3 HFT calibration factors (S-factors) of four different HFT calibrations done with NIST SRM 1450d and those applied for a measurement on an IRMM440 specimen using an HFT *MultiCalibration* (model type: Averaging). The S-factors of the upper and lower plate are shown versus the plate temperatures. The calibrations and the sample measurement were carried out with an HFM 446 *Lambda Medium*.

[1]NIST = National Institute of Standards and Technology, USA
[2]SRM = Standard Reference Material

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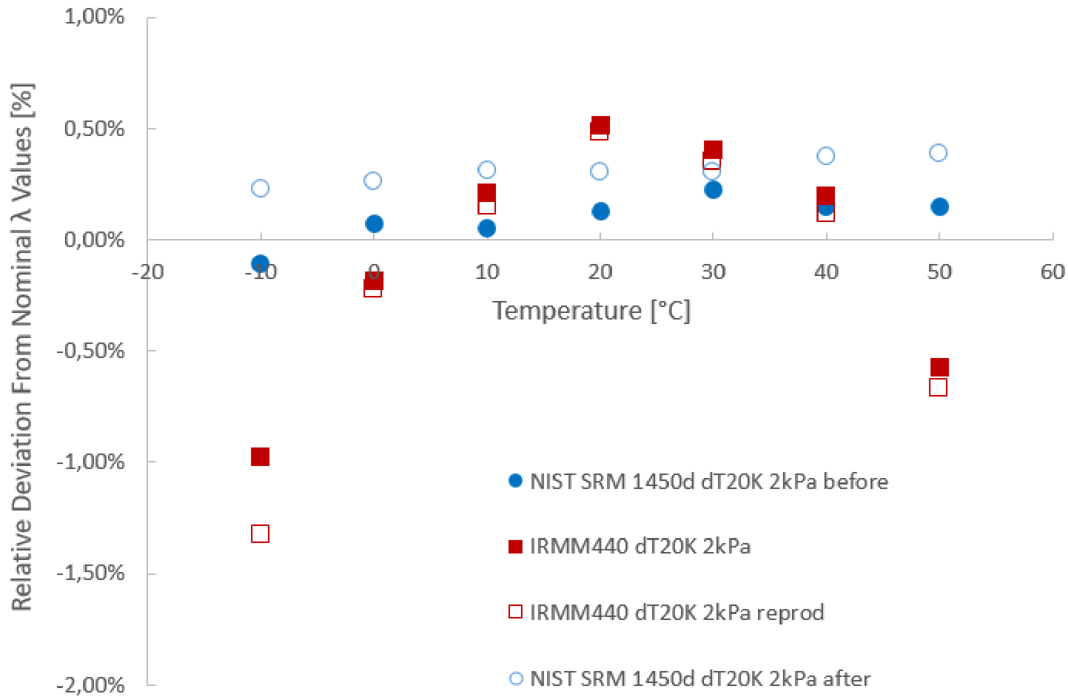
It should be noted that all calibrations and test measurements were performed using a temperature gradient of 20 K and a clamping pressure of 2 kPa. The resulting λ values of IRMM440 depicted in figure 4 are in accordance with the nominal values within ±1.3%. The HFT *MultiCalibration* itself was checked using NIST SRM 1450d before and after testing IRMM440. These measurements agree with the nominal values within ± 0.4% (see figure 4).

What is the purpose of averaging HFT calibrations? On the one hand, the HFM standard ASTM C518 [1] stipulates for the "certification testing of products" that one of the two approaches shall be followed: (1) The average of two calibrations must be used or (2) the short- and long-term stability of the calibrations must be proven. In both cases, the stability of calibrations must be better than 1 %. Furthermore, checking the calibration before and after the certification testing is recommended as demonstrated in this example (see figure 4).



4 Relative deviation from the nominal thermal conductivity values of NIST SRM 1450d and IRMM440 versus mean sample temperature. The measurements were performed on an HFM 446 *Lambda Medium* using the HFT *MultiCalibration* (model type: Averaging).

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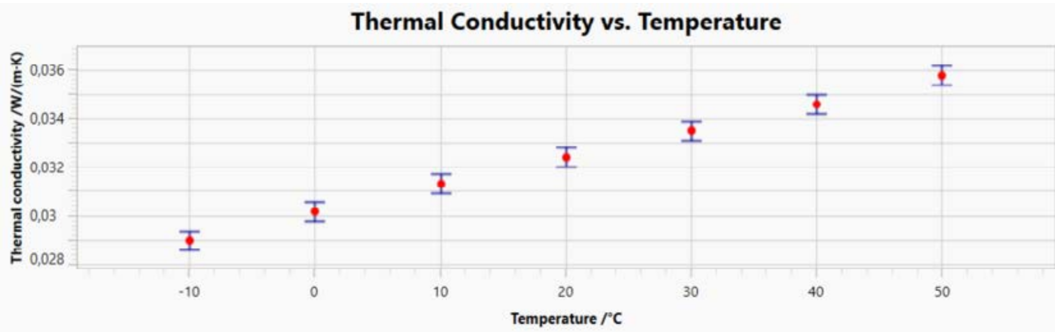
On the other hand, averaging of HFT calibrations and the application of HFT *MultiCalibration* (model type: *Averaging*), respectively, has the advantage that the combined measurement uncertainties of the thermal conductivity values obtained are smaller, because the calibration part of the entire uncertainty is reduced due to averaging. For example, the combined standard measurement uncertainty of λ indicated in the form of error bars in figure 5 is about ± 1.2% for NIST SRM 1450d. The uncertainty values are about 0.3% smaller than values from measurements using a single HFT calibration on NIST SRM 1450d. In general, the thermal conductivity results obtained with the *SmartMode* version 8.5 always include a combined standard measurement uncertainty in accordance with GUM (Guide to the expression of Uncertainty in Measurement) – another demand of ASTM C518 [1].

Model Type: 2d (Temperature/Thickness)

The model type " 2d (Temperature/Thickness)" allows for a 2-dimensional temperature and thickness interpolation of the calibration factors. This procedure is also suggested in ASTM C518 [1], namely when testing standard reference materials with different thicknesses. It should be emphasized that certified reference materials are only available up to a thickness of about 34 mm (IRMM440), at least from national institutes. However, for the certified NIST SRM 1453 (expanded polystyrene), NIST SRM 1450d and IRMM440 reference standards (both are glass fiber insulations) with thicknesses of about 13, 25 and 34 mm, no thickness interpolation is required when using NETZSCH HFM 446 *Lambda* instruments. This is proven by means of thermal conductivity measurements on this type of samples where the same HFT calibration on the basis of an NIST SRM 1450d specimen was applied (see figure 6). All results agree with the nominal values within better than 1.1%. The reproducibility of such tests is typically ±0.5%.



SP#	T_{mean} °C	ΔT K	TC W/(m·K)	U_TC %	TR (m²·K)/W	U_TR %	Time
1	50,0	20,0	0,0358	1,1	0,7055	1,1	00:29:00
2	40,0	20,0	0,0346	1,2	0,7301	1,1	00:32:00
3	30,0	20,0	0,0335	1,2	0,7535	1,1	00:33:00
4	20,0	20,0	0,0324	1,2	0,7789	1,1	00:35:00
5	10,0	20,0	0,0313	1,2	0,8062	1,2	00:34:00
6	0,0	20,0	0,0302	1,3	0,8363	1,2	00:38:00
7	-10,0	20,0	0,0290	1,3	0,8714	1,2	00:35:00

5 Thermal conductivity results for NIST SRM 1450d using an HFM 446 *Lambda Medium* and HFT *MultiCalibration* (average of four calibrations with NIST SRM 1450d). Screenshot from *SmartMode* 8.5.

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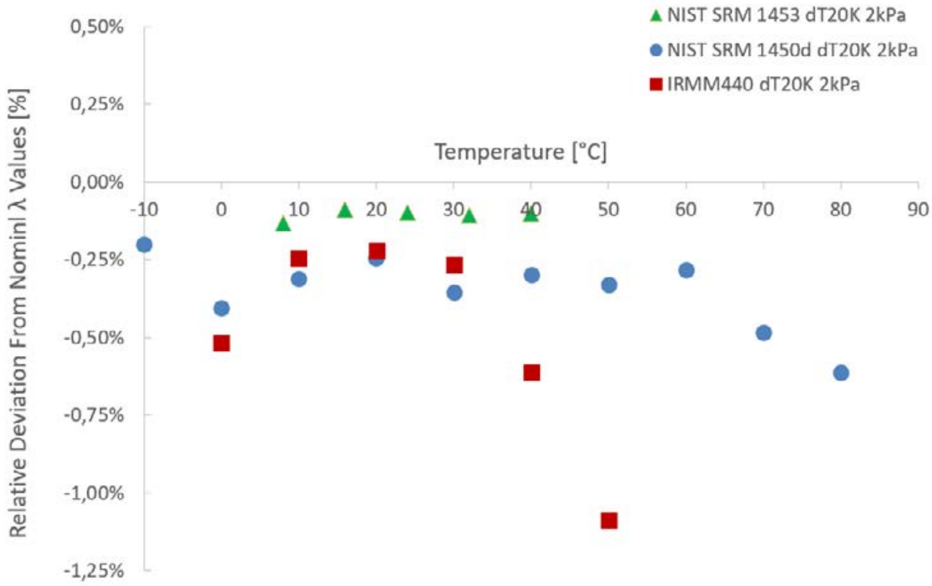

6 Relative deviation from the nominal thermal conductivity values of NIST SRM 1453, NIST SRM 1450d und IRMM440 specimens versus mean sample temperature. The measurements were performed on an HFM 446 *Lambda Medium*.

Summary

The HFT *MultiCalibration* feature, available with firmware 6.xx and *SmartMode* version 8.5, facilitates the fulfillment of HFM standards, such as ASTM C518 [1]. Currently, two model types are available: Averaging and 2d (Temperature/Thickness). Averaging of HFT calibrations reduces the combined measurement uncertainty and thus improves the accuracy of thermal conductivity measurements.

References

[1] ASTM C518-17: Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus.

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