Insights of an isothermal microcalorimeter - Flow and heat transfer simulations as a development tool for calorimeter design and construction

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Any kind of chemical, biological or physical reaction is accompanied by the production or consumption of heat. The research field of calorimetry studies heat phenomena caused by the reactions taking place and calorimeters are the instruments to quantify this heat. Different techniques and instruments are available depending on the amount of heat released or adsorbed. One type of calorimeters are called isothermal microcalorimeters (IMC) and they monitor heat flows in the µW ($\mu J \cdot s^{-1}$) range [1]. The experiments are performed under almost constant temperature. To achieve this condition, precise temperature control systems have to be implemented on the one hand, and good thermal insulation from the environment is required on the other. In practice, the exact spatial temperature distribution and eventual heat flows within the device cannot easily be measured in detail, *i.e.* temperatures can only be recorded at some discrete points. Thus, temperature gradients and undesired heat flows often go undetected. This limits access to essential information for efficient optimization of the instrument during the development phase.

To overcome this problem, one approach to device development is to use simulation tools in which 2D or 3D models are created that represents the aimed device or only selected compounds of it. In this regard, there is a wide range of suitable simulation tools. COMSOL Multiphysics® is a frequently used software and offers various physic interfaces, such as heat transfer, electrical current, fluid flow etc. that can be integrated into the development process of a microcalorimetric test system. Also, different physics can be combined to simulate multiphysics phenomena like Joule heating or conjugate heat transfer (both of which occur in an isothermal microcalorimeter). For this purpose, physical phenomena can be described by fundamental equations (e.g. Navier-Stokes, Fourier's law etc.). These partial and ordinary differential equations (PDE and ODE) are solved numerically using the finite element method (FEM). Performing such simulations might be helpful in revealing the overall temperature distribution of the microcalorimetric system at steady-state. As a result, design and construction changes can be simulated in advance, thus reducing development costs.

In the present study, we simulated flow and heat transfers in a 3D model based on an early-stage engineering microcalorimeter. Briefly, the calorimetric test system has two zones. The temperature in the outer zone is controlled by a fan heater. The inner zone contains the measuring core (heat flow sensor) and the heat sink. The temperature on the heat sink is controlled by two heating foils (pre-tempering) and two Peltier modules. Simulations were performed by COMSOL Multiphysics® version 5.5. This multiphysics issue is simulated by the *non-isothermal* interface, which combines the *laminar flow* and *heat transfer* physics. Additionally, an *event* interface was used to turn off the preheating function of both heating foils after reaching the desired set-point. A *Global ODEs* and *DAEs* interface was used to control the surrounding air temperature, as well as both thermoelectric coolers, in which the PID algorithm was defined.

Finally, the obtained numerical solutions were compared with results from experimental data obtained from the custom-made microcalorimeter under laboratory conditions. Performing these simulations aided us in the development process towards a new custom-made microcalorimeter.

[1] Wadso I, Goldberg RN. Standards in isothermal microcalorimetry (IUPAC technical report). *Pure Applied Chemistry*. **2001**;73(*10*):1625-39