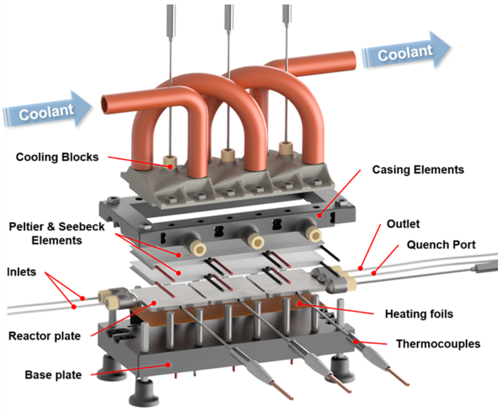
Next-Generation Reaction Calorimetry for Continuous Flow Chemistry

Heidrun Gruber-Woelfler, Sebastian Soritz, Bernhard Beham, Stefan Scheer, Richard Amering

Institute of Process and Particle Engineering, Graz University of Technology, Inffeldgasse 13/3, 8010 Graz, Austria
woelfler@tugraz.at

Reaction calorimetry is essential for quantifying critical safety and scalability parameters, including reaction enthalpy, activation energy, and adiabatic temperature rise. Traditionally, calorimetric measurements are performed using commercially available reaction calorimeters, which are predominantly based on stirred batch reactor designs. These systems typically employ reaction vessels with capacities ranging from 15 mL to 2 L and, for certain transformations, constitute comparatively large-scale laboratory equipment.[1] While traditional batch calorimetry remains state-of-the art, it has several limitations for reaction conditions typically carried out in continuously operated microreactors. To bridge this gap, reaction calorimetry in continuous flow has emerged as a crucial approach, facilitating enhanced yield, improved selectivity and safety, and efficient material usage.

This presentation showcases different versions of novel isothermal heat flow calorimeters (see Figure 1) specifically engineered for continuous flow environments.[2] The devices feature a modular design primarily constructed from 3D-printed components, allowing for rapid reassembly and adaptation to specific user requirements. Key technical highlights include independently temperature-regulated segments managed by a microcontroller, enabling localized, isothermal measurements along the reactor path.



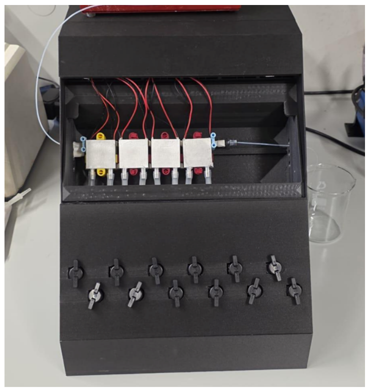


Figure 1: Different versions of isothermal heat flow calorimeters for continuous flow chemistry

We demonstrate the applicability of the calorimeter systems across various chemical transformations, including neutralization, nitration, and nitrosylation.[3] Furthermore, the device shows excellent agreement with literature data for heat capacity and mixing enthalpy measurements, establishing it as a precise tool for modern process development.

[1] Frede, T. A.; Maier, M. C.; Kockmann, N.; Gruber-Woelfler, H., *Org.Proc.Res.Dev.*, **2022**, 267–277

[2] Maier, M. C.; Leitner, M.; Kappe, C. O.; Gruber-Woelfler, H. *React. Chem. Eng.* **2020**, 5 (8), 1410–1420

[3] Soritz, S. Sommitsch A., Irndorfer S., Brouczek D., Schwentenwein M., Priestle IJG, Iosub AV, Krieger JP, Gruber-Woelfler H., *React. Chem. Eng*, **2024,** 9, 1805-1815