

## **Making Quartz Crystal Microbalance/Heat Conduction Calorimetry in Thin Films Reproducible, Precise, and Accurate**

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Quartz crystal microbalance/heat conduction calorimetry [1] is a new measurement technology that permits high sensitivity measurements in real time of three properties of a nanoscale coating or film undergoing chemical reaction: the mass change (to 10 nanograms), the heat generated (to 1 microwatt), and the change in viscoelastic stiffness (loss compliance) of the film. These sensitivities are sufficient to examine the energetics of the formation of a self-assembled monolayer as well as the thermodynamics and kinetics of chemical processes in thin films or coatings (0.001 to 5 microns). To become accepted, however, the technology must be shown to give reproducibly both precise and accurate mass and heat flow measurements that yield thermodynamic quantities in agreement with other methods. In this talk, I will discuss: (a) the conditions under which the quartz crystal microbalance functions as a linear balance; (b) a detailed heat flow analysis of the mass/heat flow sensor and its corresponding sample chamber in the Masscal G1; (c) the calibration of the thermopile as a heat flow sensor in the G1; (d) agreement of loss compliance measurements with dynamic mechanical analysis results on polymers; (e) the difficulty of finding gas-solid processes for which direct calorimetric measurements have been made of sorption enthalpies. Measurement examples discussed will include: (a) hydration and dehydration of polymer and protein films, and (b) the dynamics of drying of microliter-sized droplets of high-purity solvents, of polymer solutions, and of hydrates formed when water evaporates from inorganic salt solutions.

[1] A. L. Smith and H. M. Shirazi, *Thermochimica Acta*, **432**, 202 (2005).