Measurement and modelling of semi-batch reactions using small-scale reaction calorimetry, *in-situ* spectroscopy and gas consumption/production

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The rapid and complete characterization of chemical reaction mechanisms is of the utmost importance in terms of chemical understanding, safety and efficiency. In this presentation a new small volume reaction calorimeter (25-45mL) is described. With this reactor it is possible to make *in-situ* UV-vis and IR measurements, calibration free calorimetry measurements and gas production/consumption measurements. Firstly, the operating principle of the reactor is outlined. Following this, its application to the three-phase hydrogenation of nitrobenzene and ethyl-4-nitrobenzoate is shown along with the type of qualitative interpretation that can rapidly be made from the multiple *in-situ* signals.

The fitting of chemical models to data collected with the reactor is complex and involves the evaluation of the model parameters against multiple objective functions that do not always share the same optimal parameter values. A multiobjective function genetic algorithm has been developed for this task and its application to calorimetric and spectroscopic data measured during the epoxidation of 2,5-di-tert-butyl-1,4-benzoquinone will be shown.

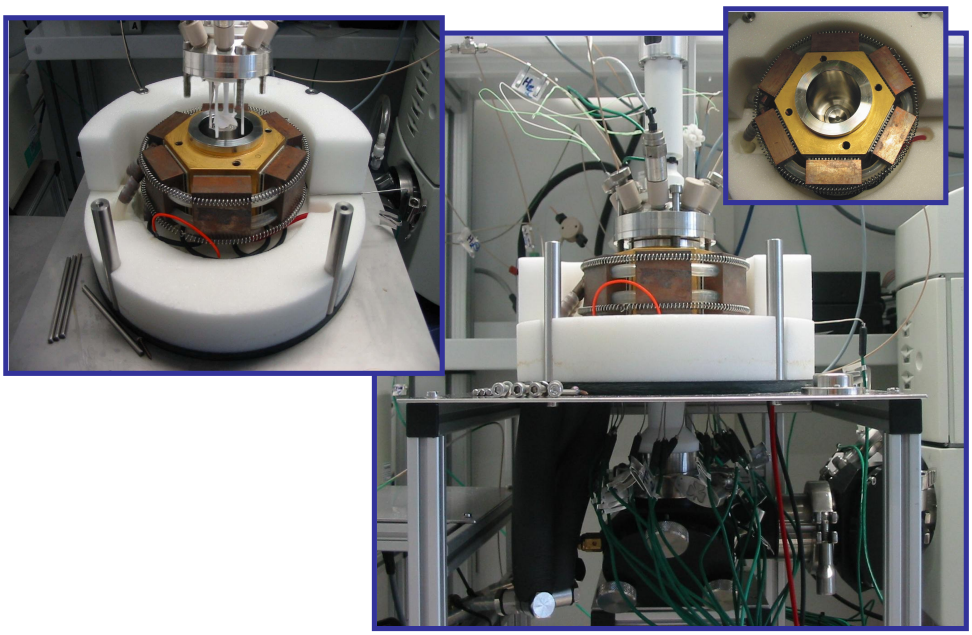

Figure 1: A new small volume reactor (25-45mL) capable of measuring calorimetry, *in-situ* UV-vis and IR spectroscopy and gas production or consumption.