

## Characterization of a CALO-2310

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When a calorimeter is used for the first time, one should get to understand and check the different parts of the equipment. How to start correctly the machine and its software? Which calculations are used by the software to determine the heat flow? In the CALO-2310, the heat flow is calculated either by heat flow calorimetry (HFC) or by heat balance calorimetry (HBC).

To know the accuracy of the calorimeter and its limitations, it is required to test the parameters that can affect the heat flow calculation under different reaction conditions. For this study, a simple reaction, the neutralization of hydrochloric acid with sodium hydroxide, was chosen. The neutralization enthalpy of this reaction is 55.9 kJ/mol at 25°C<sup>1</sup>. The influence of the following reaction conditions was verified: the time span of manual dosing or automatic dosing, the difference between automatic and manual dosing (for the same time span), the variation of the heating rate during the ramp up (for the same temperature range), the temperature range of the ramp up (for the same heating rate), the speed of the stirrer, the filling level and the calibration. Then the influence of different software settings, i.e. the values of the vortex and the torque of the stirrer were tested.

The determination of the heat capacity (Cp) for the considered reaction mixture is very important. With the CALO-2310, it is possible to determine the Cp value of an unknown reaction mixture by comparing it with a reference sample. In the present study, the Cp determination has been performed twice: the first experiment consisted of a Cp determination with ethanol (Cp = 2.4 J/g\*K)<sup>2</sup> using water (Cp = 4.186 J/g\*K)<sup>2</sup> as reference material and the second one with water using ethanol as reference material. In conclusion it is better to use a reference material with an high heat capacity.

Since it is not clear which input parameters are taken into account by the software, it was interesting to compare the results of the software with those manually calculated. E.g. for the HFC calculation, the difference between jacket and reactor temperature is required. Does it take the jacket outlet temperature, the jacket inlet temperature, an average or maybe a logarithmic average of both? The best value in comparison to the software is when the average between the two temperatures was used.

As others<sup>3</sup>, the hydrolysis of the acetic anhydride (enthalpy of reaction: 59 ± 2 kJ/mol at 55°C)<sup>4</sup> was used to verify the repeatability and the precision of the equipment, in this case for the two different modes of evaluation, HFC and HFC/HBC.

To deepen the CALO-2310 system evaluation additional testing with more complex experiments, e.g. the emulsion polymerization of styrene, the esterification of the acetic anhydride with methanol and the variation of viscosity, will be performed.

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<sup>1</sup>Gillette M., Neidig H. and Spencer J., Estimating a heat of neutralization, *Modular Laboratory program in chemistry*, 5, (1999)

<sup>2</sup><http://webbook.nist.gov/chemistry/>

<sup>3</sup>Zogg A., Fischer U., and Hungerbühler K., A New Small-Scale Reaction calorimeter That Combines the Principles of Power Compensation and Heat Balance, *Ind. Eng. Chem. Res.*, 42:4:771 (2003)

<sup>4</sup>Swissi Process Safety GmbH, *Prüf- und Wartungs-Vorschrift*, PS 090, (04-08-2010)