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# A new calorimeter for weak energy transition determination: liquid-liquid phase separation in switchable deep eutectic solvents

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Deep eutectic solvents (DESs) are emerging as an innovative type of solvent for a wide spectrum of applications. In combination with water, DESs may be used as demixing solvents as some go through low critical solution temperatures (LCST) [1]: they are homogeneous at low temperature and biphasic above the separation temperature. Demixing solvents offer an excellent opportunity to improve separation processes because of their capability to segregate the aqueous phase from the non-aqueous phase at a specific temperature.

However, the characterization of liquid-liquid phase separation can be very challenging, as the calorimetric effect associated to the phase separation is really weak in the order of a few joules per gram of solution.

In the present work, we aim to present results obtained using a new calorimeter from CALNEOS, based on sensors and thermal regulators recently developed and patented. This calorimeter has a sensitivity of 550 µV/mW that permits to measure thermal effects in the order of microwatts and can be used on a temperature range of -50°C to 160°C. In isothermal mode the control accuracy of the temperature is claimed to be 100µ°C. The crucibles used for measurements have a very small volume (100 µL). They are hermetically sealed and no leak was observable.

We will present a collection of liquid-liquid phase diagrams for different aqueous demixing systems DES-water. The DES are composed of lidocaine as a hydrogen bond acceptor and different carboxylic acids from C5 to C10 as hydrogen bond donors. Our DSC measurements were compared to data obtained using visual determination through a thermostatic bath described elsewhere [2]. this work on phase separation temperature is completed by the determination of the enthalpy of the transition, and of the heat capacity of the homogeneous samples (below the LCST).

[1] O. Longeras, A. Gautier, K. Ballerat-Busserolles, and J.-M. Andanson, (2020): **Deep Eutectic Solvent with Thermo-Switchable Hydrophobicity**, *ACS Sustainable Chem. Eng.*, vol. 8, no. 33, pp. 12516–12520,

[2] J. Castaneda Corzo, K. Ballerat-Busserolles, J.-Y. Coxam, A. Gautier, and J.-M. Andanson, (2023) **Thermo-switchable hydrophobic solvents formulated with weak acid and base for greener separation processes**, *Journal of Molecular Liquids*, vol. 377, p. 121468

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