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Contribution of Modulated DSC to the investigation of molecular mobility of pharmaceutical materials

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Modulated Differential Scanning Calorimetry (MDSC) is a new technique in which a small sinusoïdal temperature oscillation (frequency ω) is overlaid on the conventional, linear temperature ramp T(t) (linear rate q), or isotherm [1]. It has been shown recently [2,3,4] that MDSC gives the possibility to perform specific heat spectroscopy analysis on one decade of frequency in a range (0.01 Hz to 0.1 Hz) which allows to investigate the dynamics of glass forming liquids just above the transformation domain of the glasses. The drop and peak of respectively the real C' and imaginary C'' part of the complex heat capacity $Cp^*(\omega)$ occur at the temperature where the most probable relaxation time of the enthalpy of the system equal ω^{-1}. In a way similar to that of dielectric spectroscopy, this measurement allows to determine the enthalpic relaxation times of a compound [2]. MDSC is thus susceptible to offer an attractive way to check for a correlation between the dynamic and the thermodynamic behavior of glass-formers.

The objective of the present paper are :

• to test the reliability of the measured activation energies. MDSC provides a very useful tool to quickly estimate the fragility index :

$$m = \left. \frac{d \log_{10} \langle \tau \rangle}{d\,(Tg/T)} \right|_{T=Tg}$$

• To present a confrontation of dielectric and MDSC spectroscopic data that we have obtained on several molecular compounds. This reveals the coherency of both types of measurements. Furthermore MDSC proves to be an excellent substitute method to characterise the molecular mobility in all the cases where the electric conductivity is important which hides the low frequency dielectric relaxation signal. It is particularly the case of the hydrated compounds. Special attention is payed to pharmaceutical and food materials.

[1] M. Reading, Trends Polym. Sci., 1, (1993), 248

[2] O. Bustin, M. Descamps, J. Chem. Phys., 120, 22, (1999), 10982

[3] J. E. K. Shawe, Thermochim. Acta, 260, (1995), 1

[4] J. M. Hutchinson, Thermochim. Acta, 364, (1998), 165