Transitiometry and Spectro-Calorimetry for Research Laboratories and Industry

Jean-Pierre E. Grolier^a, Florin Dan^b

^b Department of Macromolecular Chemistry, Gh. Asachi Technical University, Iasi, Romania.

Newly developed technologies in *pVT*-scanning calorimetry or transitiometry, and in spectro-calorimetry have yield a series of instruments which help to investigate in depth temperature and pressure effects on fundamental physical chemistry phenomena as well as on industrial processes. On the one hand, transitiometry is at the centre of several experimental investigations; its principle allows to scan in the working cell of a very sensitive calorimeter one of the independent variables (p, V or T) while the other independent variable is kept constant. The change of the dependent variable is recorded simultaneously with the thermal effect associated with the process or the system under investigation. In the case of a non-reacting system which remains in a homogeneous state, both the mechanical and thermal outputs thus obtained give straightforward access to different pairs of thermomechanical coefficients: a_p and \mathbf{k}_T , \mathbf{b}_V and \mathbf{k}_T , C_p and \mathbf{a}_p , C_V and \mathbf{b}_V , depending on the couple of selected independent variables. When the system or the material sample goes through a chemical reaction or a phase change, the recorded information yields the corresponding heat and *pVT* characteristics. The actual operating ranges of scanning transitiometry are respectively 173 K < T < 673 K and 0.1 MPa (or400 MPa). On the other hand, with the use of optical fibers, probes for *in situ* spectroscopic readings from UV to NIR and other types of probes (pH, ultrasounds, ...) as well as injection and stirring devices, reaction calorimetry has become a multipurpose technique to investigate reacting systems.

With such equipments bulk properties, transitions as well as reactions (for example polymerization) can be advantageously studied. Selected examples all dealing with polymeric systems (including biopolymers) will be shown, namely measurements of thermomechanical coefficients (thermal expansion, compressibility), characterization of transitions (fusion, crystallization, glass transition, gelatinization) and particle synthesis. All examples show that scanning transitiometry and instrumented reaction calorimetry are versatile techniques to fully characterize thermophysical properties as well as thermodynamic behavior of a large variety of systems and materials.

References

- 1. Transitiometric investigation of asphaltenic fluids under in-well temperature and pressure conditions. Ch. Stachowiak, J-P.E. Grolier, S.L. Randzio. *Energy and Fuel*, 2001, 15, 1033-1037.
- 2. Transitiometric determination of the three-phase curve in asymmetric binary systems. S.L. Randzio, Ch. Stachowiak, J-P.E. Grolier. *J. Chem. Thermodynamics*, 2003, 35, 639-648.
- 3. The use of scanning transitiometry to investigate thermodynamic properties of polymeric systems over extended T and p ranges. J-P.E. Grolier, F. Dan, S.A.E. Boyer, M. Orlowska, S.L. Randzio. *Int. J. Thermophysics (submitted).*
- 4. Transitiometric *in situ* measurements of pressure effects on the phase transition during starch gelatinization. M. Orlowska, S.L. Randzio, J-P.E. Grolier, *in Advances in High Pressure Bioscience and Biotechnology II (R..Winter Ed.), Springer Verlag, (Heidelberg), 2003, 395-398.*

^a Laboratory of Thermodynamics of Solutions and Polymers, Blaise Pascal University, Aubière, France.