On-Line Determination of Solution Supersaturation of Fast Precipitating Systems via SR ATR FTIR Spectroscopy

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Abstract No. Robe9575
Beamline(s): U2B

Introduction: This study aims to apply SR ATR FTIR spectroscopy to an examination of the crystallisation kinetics associated with fast precipitating reactions of organic specialty chemical products, notably pharmaceuticals.

Methods and Materials: The experimental system comprising an AXIOM Dipper-210 ATR immersion probe connected to the NICOLET MAGNA FTIR spectrometer at beamline U2B is linked to a 400 ml computer controlled batch reactor. This enables in-situ internal reflection measurements using SR IR radiation to monitor supersaturation during precipitation and crystallisation processes.

Results: The experiments carried out have been directed towards characterising a poorly understood real pharmaceutical system: an organic compound, produced by Glaxo Wellcome, in aqueous solution. Calibration measurements and bench marking tests against existing lab instrumentation were successfully carried out. As can be seen in Figure 1, using a globar source no reliable calibration curves, relating the measured ratio of two IR peaks RA to concentration of the solute and temperature, could be established. Using SR IR light as a source produced much more quantifiable results as can be seen in Figure 2. The fact, that the 30°C calibration curve dropped below the 15°C one, is probably due to inadequate control in experimental procedures rather than being due to the analytical technique itself.

Conclusions: This first data on a new 2-year programme is most encouraging, particularly the data on the improved base line linearity using SR ATR FTIR. Future work will develop this work and take forward improvement in micro-probe sensor technology.

Acknowledgments: Work supported by EPSRC. Pharmaceutical compound provided by Glaxo Wellcome.