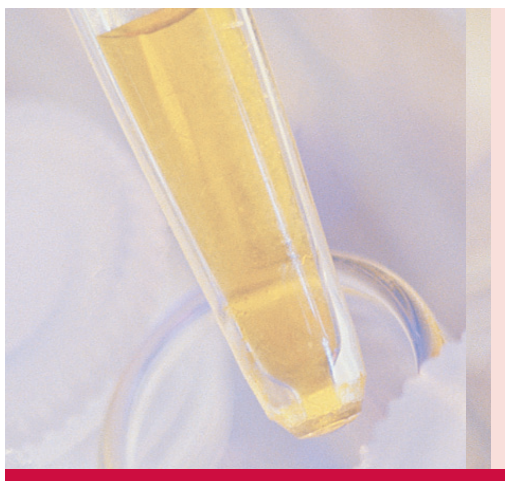

Laboratory Product Focus

CRYSTALLISATION TOOLS FOR THE RESEARCH CHEMIST

Jasbir Singh, HEL Ltd

The need to study crystallisation is extremely widespread in the chemical industry due to the importance of solids as the final form of many products. The most basic information required is the solubility of crystals – proceeding without this basic information is like trying to locate a house without knowing the address – it can be done but will take a long time.

When the crystallisation is to be scaled up into a process, you also need the so-called metastable zone width (MSZW). MSZW is important in crystallisation processing as it represents the difference between the saturation temperature and the temperature during cooling at which crystallisation actually commences. – essentially a road map of the temperature region over which the crystallisation process will be run. During research knowledge of these parameters is needed at a range of concentrations for common solvents and solvent mixtures.

> ***"IT RECORDS TEMPERATURE AND TURBIDITY TO GIVE THE SOLUBILITY AND MSZW FOR THE SOLVENT AND CRYSTALS BEING STUDIED"***

Author Details:

Jasbir Singh

HEL Limited
50 Moxon Street, Barnet.
Herts. EN5 5TS.

CRYSTALEYES – A BASIC TOOL

When crystals are added to a solvent and heated, the solution will become clear when the crystals have dissolved; as it is cooled down again the crystals will reappear. The point at which these two events occur can be estimated by simply looking into the vessel and noting the relevant temperatures – a procedure that is likely to be time consuming and unreliable.

An simple but effective alternative is to insert a turbidity probe in the vessel and this will reproducibly do the same job. If these measurements can be integrated with temperature, and the data is automatically recorded, then of course you have the ideal system.

This is in fact exactly what crystalEYES does – it records temperature and turbidity to give the solubility and MSZW for the solvent and crystals being studied – it is linked to a PC with pre-loaded software so the information is captured without effort. This can be hooked on to existing stirred reactors or simply vials on hot plates to record the necessary data. CrystalEYES is also records pH if necessary, as this can sometimes affect solubility.

Typical data is shown in figure 1 below – this is a screen shot of the live data display as you heat and cool a mixture.

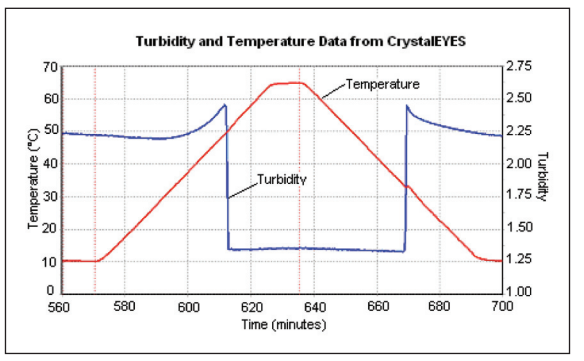

Figure 1. Typical data from CrytalEYES

If this cycle is repeated at different concentrations this will give the all the necessary information for that crystal/solvent combination and the classic plot shown in figure 2 can be generated.

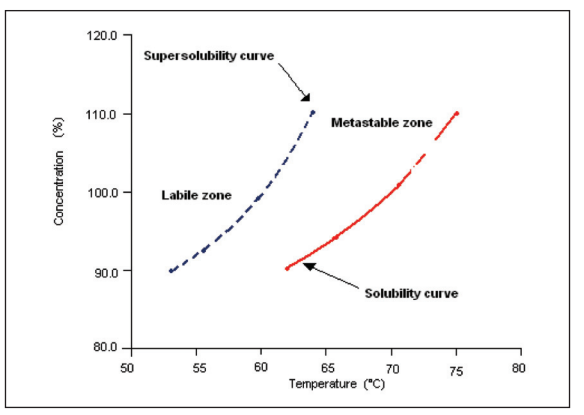

Figure 2. Metastable zone width (MSZW) plot.

CrystalEYES basically consists of temperature and turbidity probes plugged into a signal conditioning unit which in turns is connected to a PC. The system

comes ready to run – all that needs to be decided is the size of the probes. Different interchangeable probes can be supplied to allow sample volumes from 1ml upwards to be studied.

PARALLEL STUDIES FOR HIGHER THROUGHPUT

Some of the larger companies or even smaller ones that have lots of samples to evaluate need multiple crystalEYES units working in parallel. CrystalSCAN is exactly such a system, designed to handle 4 or 8 samples in parallel with sample sizes from around 1ml up to 100ml.

In order to permit a range of concentrations to be studied automatically (for each sample), it is necessary to dilute the samples after successive heat./cool cycles. The crystalSCAN is unique in being able to achieves this by using one or more precision syringe pump with a multi-way rotary valve.

The type of data that is generated by crystalSCAN for each vial is shown in figure 3, for potassium nitrate in water – this is very similar to figure 2 in terms of the heat/cool cycles but with dilutions after each cycle.

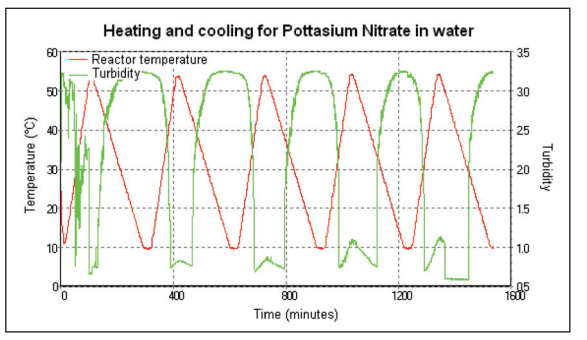

Figure 3. Typical data from CrystalSCAN with successive dilutions

When the appropriate crystallisation and re-crystallisation points are collated, MSZW plot such as in figure 2 is produced.

QUALITY OF DATA

In order to confirm the accuracy of the above measurements, the data can be compared to literature solubility results for potassium nitrate (Crystallization, JW Mullin, 2001). This is shown in the figure 4.

Good agreement is clearly observed.

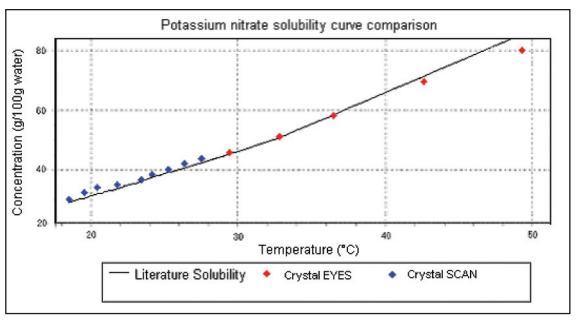

Figure 4. Comparison of result with literature

CONCLUSIONS

It is clear that crystallisation studies in parallel can be performed at sample sizes as low as 1ml or as large as several hundred millilitres. The data scales up reliably and thus provides an excellent early insight into the key variables. While CrystalSCAN appeals to situations where large numbers of samples need to be studied, CrystalEYES can generate exactly the same data on a single portable platform. They use the same detection method which is both reliable and accurate.