QUANTITATIVE ANALYSIS OF MIXTURES USING HIGH THROUGHPUT INSTRUMENTATION WITHOUT THE USE OF STANDARDS

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The PolySNAP computer program [1-4] uses the full powder diffraction pattern coupled with linear least squares or singular value decomposition to carry out the quantitative analysis of mixtures from powder diffraction data. The reference powder patterns for the pure phases are assumed to be available and collected on a similar diffraction instrument. It does not, in general, however, use calibration methods or standards (although it can do so). It is capable of achieving an accuracy of *ca*. 1% under favourable conditions. We present here the results of experiments involving 2- and 3-phase mixtures of organic samples carried out on a Bruker D8 GADDS system (which is designed for high throughput crystallography) and on a Bruker D4 and the TOPAS software as a reference. Our intention with this method is not to obtain very accurate analysis of mixtures, but to get mixture compositions in a high throughput environment to within 10% of the true value quickly and without user intervention. A typical result for a 2-phase mixture is shown in Figure 1.

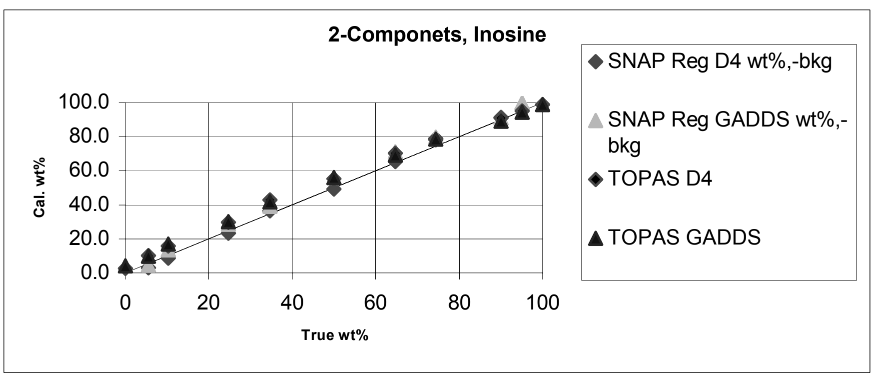

Figure 1. The results of quantitative analysis of a binary mixture of inosine and fluconazole using a D4 GADDS.

It can be seen that the 10% requirement is easily met in this case. Other examples are equally encouraging although the use of calibration samples can be very useful with 3-phase mixtures.

1. Gilmore, C.J., Barr, G & Paisley, J. (2004). *J. Appl. Cryst.* **37**, 231-242.
2. Barr, G., Dong, W. & Gilmore, C.J. (2004). *J. Appl. Cryst.* **37**, 243-252.
3. Barr, G., Gilmore, C.J. & Paisley, J. (2004). *J. Appl. Cryst.* **37**, 231-242.
4. Barr, G., Dong, W. & Gilmore, C.J. (2004). *J. Appl. Cryst.* **37**, 658-664.