CRYSTAL STRUCTURE SOLUTION FROM POWDER MIXTURES: THE PXRD-BTEM-RIETVELD METHOD.

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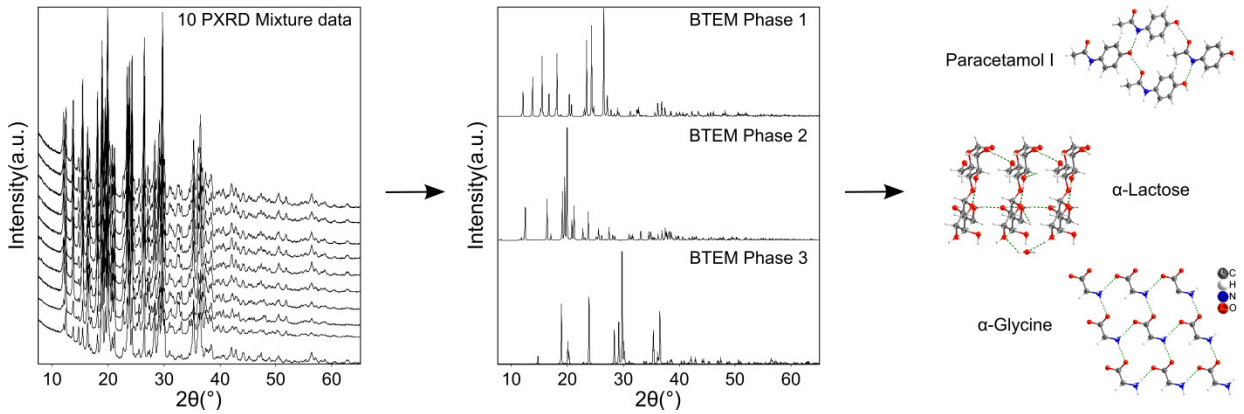
Complex mixtures are commonly encountered in the chemical sciences. About 10 years ago, we developed an algorithm to untangle the complex mixture spectra obtained during organic syntheses in order to provide pure component spectra of the individual components without recourse to libraries or any a priori information. This algorithm, Band-Target Entropy Minimization (BTEM), untangles the spectra by searching for the simplest spectral patterns i.e. those with lowest signal entropy [1]. BTEM has been repeatedly used with vibrational spectroscopic measurements (FTIR, Raman) in order to identify new intermediates present at trace levels [2]. In 2004, we showed that BTEM can also be applied to the raw PXRD patterns from powder mixtures, in order to obtain the individual pure phase powder patterns [3].

In this talk, we discuss the combination of PXRD measurements from powder mixtures with BTEM analysis followed by structure solution and Rietveld analysis. Both inorganic and organic powder mixtures are investigated. We demonstrate that in many cases, the BTEM pure powder patterns are sufficiently accurate to obtain reasonably good crystal structure solutions. Details of the procedure are explained and a wide variety of signal issues are addressed. Also, future directions are explored and suggestions for enhancing the PXRD-BTEM-Rietveld method are discussed. The present development opens new opportunities for powder mixture analysis.



[1] (a) C. Wee, E. Widjaja, M. Garland *Organometallics*, 2002, *21*, 1982–1990 (b) E. Widjaja, C. Wee, M. Garland *Organometallics*, 2002, *21*, 1991–1998. [2] C. Li, E. Widjaja, W. Chew; M. Garland, *Angew. Chem. Int. Eng.* 2002, *20*, 3785-3789. (b) C. Li, E. Widjaja, M. Garland, *J. Am. Chem. Soc.* 2003, *125*, 5540–5548. (c) C. Li, L. Chen, M. Garland, *J. Am. Chem. Soc.* 2007, *129*, 133327–13334 [3] L. Guo, F. Kooli, M. Garland, *Analytica Chimica Acta*, 2004, *517*, 229-236

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