powder patterns. The best accuracy is achieved with calculated corundum numbers for the best matching reference spectrums. The second method has developed for simultaneous QPA of group of powder patterns with identical qualitative phase composition of samples. The method is development of J.Rius [1] and K.Zangalis [2] methods but differ by iterative refinement mass absorption coefficients of samples and calibrate constants or corundum numbers of phases which made by least squares or simplex methods.

The QPA accuracy of both methods are discussed on data of Round Robin on Quantitative Phase Analysis CPD IUCr [3]. Identification possibilities of system are discussed on data of Search-Match Round Robin - 2002 CPD IUCr [4] where only the Q&QPA Retrieve identified all phase compositions exactly.

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MS31 P09

Detections of the Pattern Deformations in the Search/Match Procedure Akihiro Himeda, Hiroki Yoshida, *X-Ray Research Laboratory, Rigaku Corporation, Japan.* E-mail: himeda@rigaku.co.jp

Keywords: powder patterns, phase identification, lattice distortion

Phase identification with the powder patterns is important as a starting point for the quantifications or the whole pattern fitting analyses. Although there are a lot of search/match programs, they often fail when the powder patterns are distorted from those in the database. Two main reasons of the deformations are the lattice distortion and the intensity deformation due to the preferred orientations. Especially, when the lattice distortion is anisotropic, which is often the case in organic compounds, it is hard to identify the correct phase.[1]

We have developed a new search/match algorithm which detects and evaluates the anisotropic lattice distortion and intensity deformation due to the preferred orientations. In Figure 1, overlay of the observed powder pattern downloaded from the Search-Match Round Robin-2002 web site [1] and the pattern registered in the ICDD database [2] is shown. The inconsistency between these patterns is due to the lattice distortion of the observed pattern with its lattice constants (a, c)=(9.09, 13.47) from the ICDD pattern (a, c)=(9.19, 13.40). Our search/match procedure automatically detects the anisotropic deformation while searching the card from the ICDD database. After finding the appropriate pattern, the correct lattice constants are readily obtained. In Figure 2, the modified pattern is shown and it agrees well with the experimental pattern.

[1] See the result of Sample 2 in the Search-Match Round Robin-2002, http://sdpd.univ-lemans.fr/smrr/
[2] John Faber and Tim Fawcett. (2002) *Acta Cryst*. B58, 325-332.

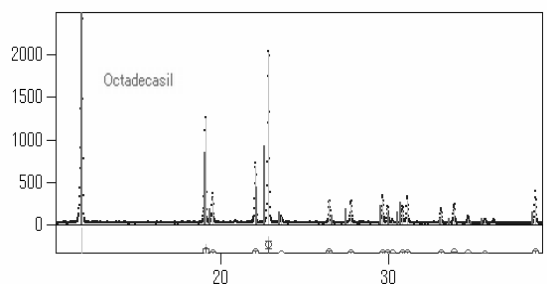

Figure 1 Overlay of the observed powder pattern(dots) and the pattern(spikes) registered in the ICDD database**.**

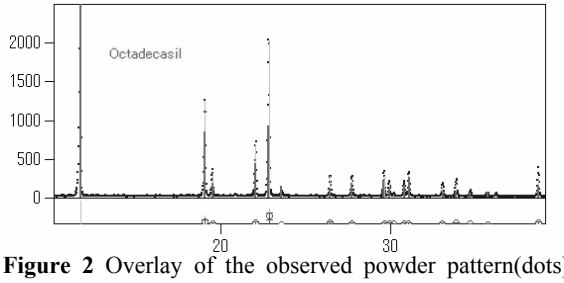

Figure 2 Overlay of the observed powder pattern(dots) and the modified pattern(spikes) based on the detected anisotropic lattice distortion.

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Protein powder diffraction – pH variation studies of insulin. Lisa Knight[a], Irene Margiolaki[a], Andy Fitch[a], Jon Wright[a], Mathias Norrman[b], Gerd Schluckebier[b].
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Keywords: Powder Diffraction Techniques, Protein Crystallography, pH.

Modern developments of the powder diffraction technique have allowed the investigations of systems with large unit cells like proteins [1-3]. One of the main advantages of powder diffraction is the flexibility in crystallisation conditions, as well as being able to observe phase transitions and multiple phases in situ. Variatiation in the structure of microcrystals is important particularly in the treatment of diabetes where medication is often administered as insulin microcrystals. Dependent on the crystal form, the medication can have different time/action profiles. The present study aims to investigate the effect of pH on the lattice parameteres of various forms of insulin microcrystals at room temperature. Variation of the cell axes with increasing pH has been seen in the hexagonal and cubic forms, these variations along with phase transitions observed in orthorhombic forms will be presented. Preliminary data interepretation correlating the variations with the structural and microstructural characteristics of the systems under study will be shown.

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