Crystallography

4B2/2010P005

Structure determination of oligopeptides based on powder diffraction data

Tamiko KIYOTANI*[1, 2], Yoko SUGAWARA[2]
[1]Showa Pharmaceutical University, Machida, Tokyo 194-8543, Japan
[2]Kitasato University, Sagamihara, Kanagawa 252-0373, Japan

Introduction

Oligopeptides are one of the physiologically active substances and used as medicines, sweeteners, and food additives. They frequently crystallize as hydrates, and crystal structural transformations are induced by hydration and dehydration processes. Under such backgrounds, we aim to carry out *ab initio* crystal structure determination of oligopeptides from powder diffraction data. We have begun with powder diffraction analysis of several baseic dipeptides.

Experimental

Glycylglycine, glycyl-L-phenylalanine and L-leucyl-glycine were purchased from Peptide Institute INC. and recrystallized from aqueous solutions. Samples sealed in glass capillaries were used for synchrotron X-ray data collection using the multiple-detector system (MDS) installed at the BL-4B2 beam line of the Photon Factory, KEK, Tsukuba. The measurements were carried out with monochromatized 1.197369(7) Å x-ray at room temperature. Data analysis was performed using the program for crystal structure solution from powder diffraction data, DASH, developed by CCDC and CCLRC.

Results and discussion

Figure 1(a) shows the powder diffraction pattern of glycylglycine. Crystal data of glycylglycine and glycyl-L-phenylalanine based on the powder diffraction data were coincide with the reported values determined by single crystal analysis [1,2]. Crystal structure determination of glycylglycine by the simulated annealing method was carried out. The solution (Figure 1(b)) agreed with the structure determined by single crystal data (Figure 1(c)) [1].

Hitherto, L-leucylglycine dihydrate had obtained from an aqueous-acetone solution, and only the crystal data were reported (triclinic, space group $P1$, a = 5.98, b = 7.95, c = 6.89 Å, α = 90, β = 108, γ = 96°) [3]. We obtained a new crystal form from an aqueous solution (Figure 2). The crystal data were determined to be: orthorhombic, a = 24.36, b = 15.53, c = 8.718 Å. Structure determination of both two forms is in progress. In the case of the new form of L-leucylglycine, slight changes of the diffraction pattern occurred during intensity measurements, which would attribute to desorption of small amount of crystal water molecules. The data collection under the more strictly humidity- and temperature-controlled condition will be designed.

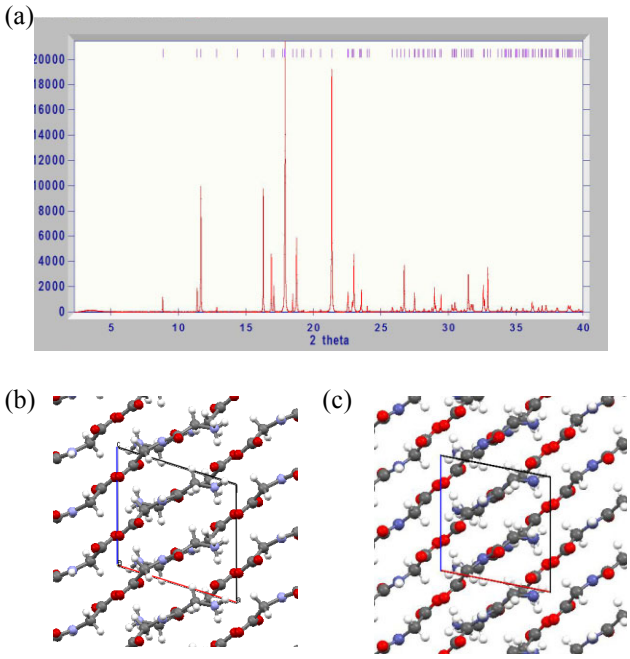

Fig. 1 Powder diffraction pattern of glycylglycine (a), and crystal structures determined by the simulated annealing method (b) and single crystal analysis [1] (c).

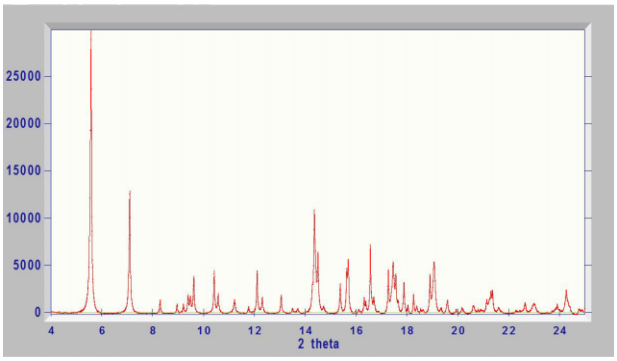

Fig. 2 Powder diffraction pattern of the new crystal form of L-leucyl-glycine hydrate.

References

[1] S. A. Moggach et al., *Acta Crystallogr. Sect. B*, **62**, 310 (2006).
[2] D. Mukkamala et al., *J. Am. Chem. Soc.*, **129**, 7385 (2007).
[3] J. E. Leonard et al., *Acta Cryatallogr.* **5**, 150 (1952).

* kiyotani@ac.shoyaku.ac..jp