Powder X-ray diffraction of capecitabine, $C_{15}H_{22}FN_3O_6$

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Capecitabine (Xeloda) is a chemotherapy drug used to treat breast, gastric, and colorectal cancers. Commercial capecitabine crystallizes in the orthorhombic space group $P2_12_12_1$ (#19) with $a = 5.20587(3)$, $b = 9.52324(4)$, $c = 34.79574(21)$ Å, $V = 1725.062(12)$ Å^3, and $Z = 4$. A reduced cell search in the Cambridge Structural Database (Groom C. R., Bruno, I. J., Lightfoot, M. P., and Ward, S. C. (2016) *Crystallogr. Sect. B: Struct. Sci., Cryst. Eng. Mater.* **72**, 171–179) yielded three previous structure determinations (Rohlicek, J., Husak, M., Gavenda, A., Jegorov, A., Kratochvil, B., and Fitch, A. (2016). *Acta Cryst. Sect. E: Crystallgr. Commun.* **72**, 879–880, BOVDUM; Malińska, M., Krzeczyński, P., Czerniec-Michalik, E., Trzcińska, K., Cmoch, P., Kutner, A., and Woźniak, K. (2014). *J. Pharm. Sci.* **103**, 587–593, BOVDUM01 and BOVDUM02), using synchrotron powder data and later single crystal data at two temperatures. In this work, the sample was ordered from United States Pharmacopeial Convention (lot # G0J205), and analyzed as-received. The room temperature (295 K) crystal structure was refined using synchrotron ($\lambda = 0.413914$ Å) powder diffraction data, density functional theory (DFT), and Rietveld refinement techniques. Hydrogen positions were included as part of the structure, and were re-calculated during the refinement. The diffraction data were collected on a beamline 11-BM at the Advanced Photon Source, Argonne National Laboratory and the powder X-ray diffraction pattern of the compound is provided. The agreement of the Rietveld-refined and DFT-optimized structures is poorest in the pentyl side chain, consistent with the disorder observed previously. © *2019 International Centre for Diffraction Data.* [doi:10.1017/S0885715619000575]

Key words: capecitabine, Xeloda®, X-ray diffraction, powder diffraction file

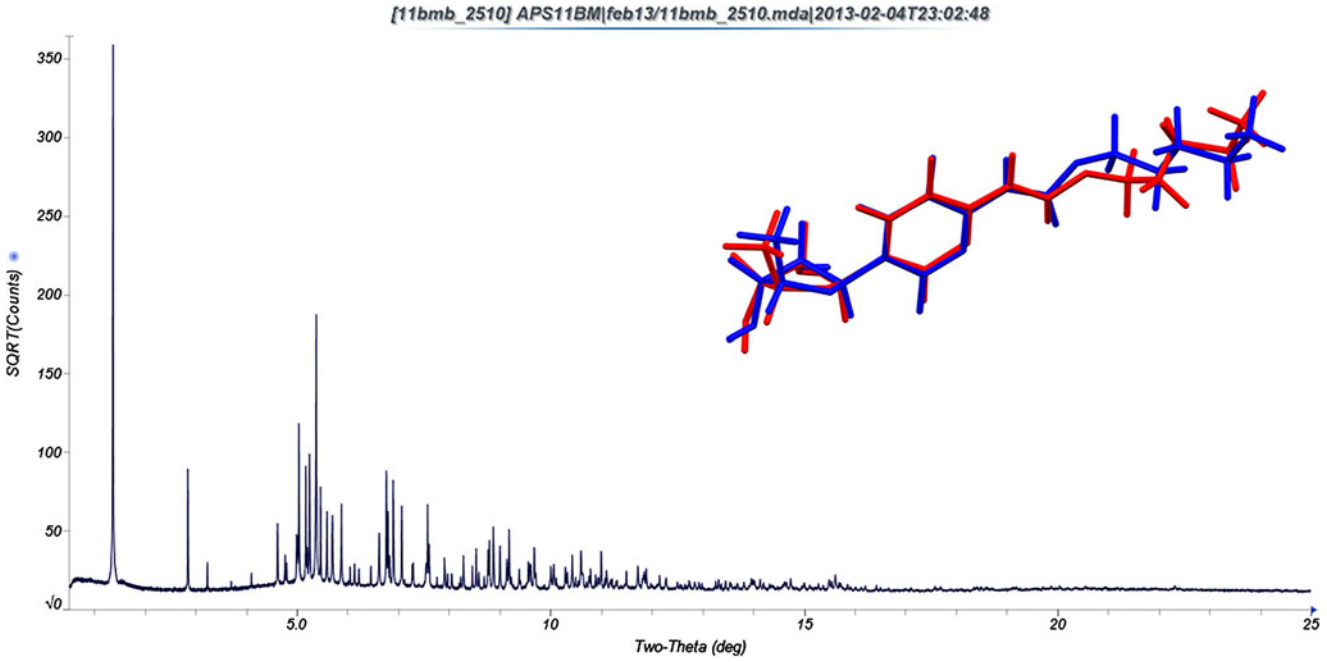

Figure 1. (Color online) Powder X-ray diffraction pattern of capecitabine. The Rietveld-refined structure is indicated in red, and the DFT-optimized structure is indicated in blue.

* Figure 1 has been corrected. A correction notice detailing this change has also been published (doi:10.1017/S0885715619000642).

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(#19) with $a = 5.20587(3)$, $b = 9.52324(4)$, $c = 34.79574(21)$ Å, $V = 1725.062(12)$ Å^3, and $Z = 4$. A reduced cell search in the Cambridge Structural Database (Groom *et al.*, 2016) yielded three previous structure determinations (Rohlicek *et al.*, 2016, BOVDUM; Malińska *et al.*, 2014, BOVDUM01 and BOVDUM02), using synchrotron powder data and later single-crystal data at two temperatures. The lattice parameters of the room temperature studies BOVDUM and BOVDUM01 are in excellent agreement with those determined here. The lattice parameters of BOVDUM02 (determined at 100 K) are smaller, by 2.43, 0.45, and 3.64% for *a*, *b*, and *c*, respectively. In this work, the sample was ordered from United States Pharmacopeial Convention (lot # G0J205), and analyzed as-received. The room temperature (295 K) crystal structure was refined using synchrotron ($\lambda = 0.413914$ Å) powder diffraction data, density functional theory (DFT), and Rietveld refinement techniques. Hydrogen positions were included as part of the structure, and were re-calculated during the refinement. The diffraction data were collected on a beamline 11-BM at the Advanced Photon Source, Argonne National Laboratory. Figure 1 shows the powder X-ray diffraction pattern of the compound and the agreement of the refined and optimized structures. The agreement of the Rietveld-refined and DFT-optimized structures is poorest in the pentyl side chain, consistent with the disorder observed previously in BOVDUM and BOVDUM01. Pairwise comparisons of the two structures of this study and the three previous determinations shows that the root-mean-square Cartesian displacements of the non-hydrogen atoms lie in the range 0.18–0.53 Å, and that the differences are mainly in the side chains. The pattern is included in the Powder Diffraction File as entry 00-064-1493.

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SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715619000575

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