Powder X-ray diffraction of 3-(4-Morpholinyl)-1-(4-nitrophenyl)-5,6-dihydro-2(1H)-pyridinone, $C_{15}H_{17}N_3O_4$

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X-ray powder diffraction data for 3-(4-Morpholinyl)-1-(4-nitrophenyl)-5,6-dihydro-2(1H)-pyridinone, $C_{15}H_{17}N_3O_4$, are reported [$a = 7.112(1)$ Å, $b = 33.360(2)$ Å, $c = 6.265(1)$ Å, $\alpha = 90°$, $\beta = 94.037(1)°$, $\gamma = 90°$, $V = 1483.08$ Å^3, $Z = 4$, $\rho_{cal} = 1.358$ g cm^{-3} and space group $P2_1$]. All measured lines were indexed and are consistent with the $P2_1$ space group. No detectable impurities were observed. © *2015 International Centre for Diffraction Data*. [doi:10.1017/S0885715615000640]

Key words: 3-(4-Morpholinyl)-1-(4-nitrophenyl)-5, 6-dihydro-2(1H)-pyridinone, pharmaceutical intermediate, anticoagulant, apixaban

3-(4-Morpholinyl)-1-(4-nitrophenyl)-5,6-dihydro-2(1H)-pyridinone is an intermediate in the synthesis of the anticoagulant, Apixaban (Jiang and Ji, 2013; Zikria and Ansell, 2009). The sample was prepared using 3,3-Dichloro-1-(4-nitrophenyl)-2-piperidinone and was recrystallized in ethanol and dried. The sample was then ground into powder (HPLC ≥ 98%, $\rho = 1.371$ g cm^{-3}, $T_{melt} = 166$–168 °C) and mounted on a flat zero background plate. X-ray powder diffraction measurement was performed at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., The Netherlands) with a PIXcel 1D detector and Cu$K\alpha$ radiation (generator setting: 40 kV and 40 mA). The diffraction data were collected over the angular range from 4° to 50°2θ with a step size of 0.013 13°2θ and a counting time of 30 ms step^{-1}. The software package Material Studio 8.0 (Accelrys Co., Ltd., CA, USA) was used to process the data in the Analytical & Testing Center (Sichuan University, China). The X-ray powder diffraction pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results were obtained by DICVOL91 method (Boultif and Louër, 1991). The following figures of merit were achieved: $F_{20} = 36.0$ (0.0103, 54) (Smith and Snyder, 1979) and $M_{20} = 19.1$ (de Wolff, 1968). The preliminary cell from indexing was refined using the Pawley method (Pawley, 1981). Pawley refinement results confirmed that the sample crystallizes in the monoclinic space group $P2_1$ (4), with $a = 7.112(1)$ Å, $b = 33.360(2)$ Å, $c = 6.265(1)$ Å, $\alpha = 90°$, $\beta = 94.037(1)°$, $\gamma = 90°$, $V = 1483.08$ Å^3, $Z = 4$, $\rho_{cal} = 1.358$ g cm^{-3}. Figure 1 shows the powder X-ray diffraction pattern of the compound.

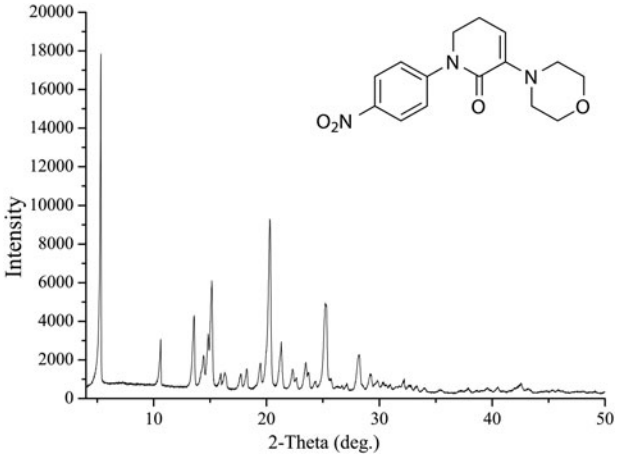

Figure 1. Powder x-ray diffraction pattern of 3-(4-morpholinyl)-1-(4-nitrophenyl)-5,6-dihydro-2(1H)-pyridinone.

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SUPPLEMENTARY MATERIALS

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