Powder X-ray diffraction of 1-(4-Nitrophenyl)-2-piperidinone, $C_{11}H_{12}N_2O_3$

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X-ray powder diffraction data for 1-(4-Nitrophenyl)-2-piperidinone, $C_{11}H_{12}N_2O_3$, are reported [$a = 9.514(3)$ Å, $b = 12.308(6)$ Å, $c = 9.175(1)$ Å, $\alpha = 90°$, $\beta = 91.811(2)°$, $\gamma = 90°$, $V = 1073.94$ Å^3, $Z = 4$, $\rho_{cal} = 1.362$ g cm^{-3} and space group $P2_1/n$]. All measured lines were indexed and are consistent with the $P2_1/n$ space group. No detectable impurities were observed. © *2015 International Centre for Diffraction Data*. [doi:10.1017/S0885715615000639]

Key words: 1-(4-Nitrophenyl)-2-piperidinone, X-ray powder diffraction, crystal structure, apixaban

1-(4-Nitrophenyl)-2-piperidinone is an intermediate in the synthesis of the anticoagulant, Apixaban (Jiang and Ji, 2013; Zikria and Ansell, 2009). Commercial 1-(4-Nitrophenyl)-2-piperidinone was purchased from Haoyuan Chemexpress Co., Ltd., (Shanghai, China), recrystallized in methanol and dried. The sample was then ground into powder (HPLC >98%, $\rho = 1.327$ g cm^{-3}, $T_{melt} = 95$–96 °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: $F_{20} = 36.2$ (0.0094, 59) (Smith and Snyder, 1979) and $M_{20} = 18.8$ (de Wolff, 1968). The preliminary cell from indexing was refined using the Pawley method (Pawley, 1981). The indexing results were then refined with the type of Pawley. Pawley refinement results confirmed that the sample crystallizes in the monoclinic space group $P2_1/n$ (14), with $a = 9.514(3)$ Å, $b = 12.308(6)$ Å, $c = 9.175(1)$ Å, $\alpha = 90°$, $\beta = 91.811(2)°$, $\gamma = 90°$, $V = 1073.94$ Å^3, $Z = 4$, and $\rho_{cal} = 1.362$ g cm^{-3}. The crystal structure result [$a = 9.163\,19(19)$ Å, $b = 12.2880(4)$ Å, $c = 9.506\,85(18)$ Å, $\alpha = 90°$, $\beta = 91.843(2)°$, $\gamma = 90°$, $V = 1069.89(4)$ Å^3, $Z = 4$, and $\rho_{cal} = 1.367$ g cm^{-3}] from single-crystal X-ray diffraction was also obtained. Results showed that both single-crystal and powder diffraction methods can get similar structure data. Figure 1 shows the Powder X-ray diffraction pattern of the compound.

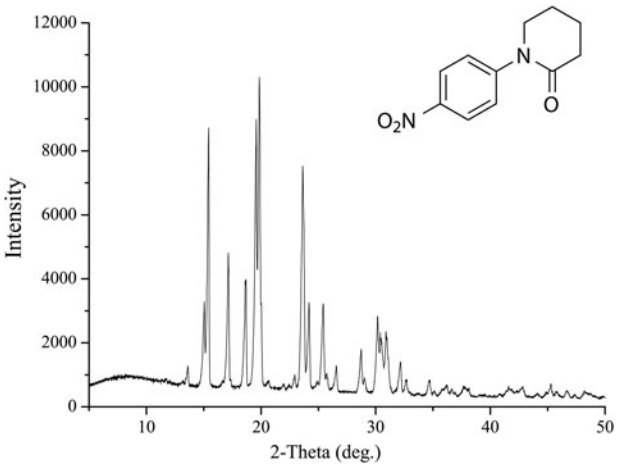

Figure 1. Powder X-ray diffraction pattern of 1-(4-Nitrophenyl)-2-piperidinone.

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SUPPLEMENTARY MATERIALS

For supplementary material for this article, please visit http://dx.doi.org/10.1017/S0885715615000639

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