X-ray powder diffraction data for azilsartan, $C_{25}H_{20}N_4O_5$

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X-ray powder diffraction data, unit-cell parameters and space group for azilsartan, $C_{25}H_{20}N_4O_5$, are reported [$a = 9.641(3)$ Å, $b = 11.301(9)$ Å, $c = 20.010(8)$ Å, $\alpha = 90°$, $\beta = 90.351(5)°$, $\gamma = 90°$, unit-cell volume $V = 2196.735(4)$ Å^3, $Z = 4$, $\rho_{cal} = 1.379$ g • cm^{-3}, and space group $P2_1/c$]. All measured lines were indexed and are consistent with the $P2_1/c$ space group. No detectable impurities were observed.
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Key words: X-ray powder diffraction, azilsartan

I. INTRODUCTION

The title compound azilsartan (Figure 1), systematic name 2-Ethoxy-1-[[2′-(4,5-dihydro-5-oxo-12,4-oxadiazol-3-yl)biphenyl-4-yl]methyl]benzimidazole-7-carboxylic acid, is a new angiotensin II (AII) receptor blocker (ARB) that inhibits the binding of AII to AII type 1 (AT_1) receptors selectively, and is thus expected to exert a more potent and sustained blood pressure lowering effect than existing ARBs (Hiromi *et al.*, 2012).

The single crystallographic data of azilsartan [$a = 9.6590$ (19) Å, $b = 11.329$ (2) Å, $c = 20.046$ (4) Å, $\alpha = 90°$, $\beta = 90.30$ (3)°, $\gamma = 90°$, unit-cell volume $V = 2193.54$ Å^3, $Z = 4$, and space group $P2_1/c$] was obtained by Ge *et al.* (2016). The Powder X-ray diffraction (PXRD) data have recorded in Powder Diffraction File (PDF)-4/Organic 2016 database with the PDF numbers 00-063-1112. In this study, we provided a calculated PXRD pattern and Fourier transform infrared spectroscopy (FTIR) spectrum for azilsartan. The experimental data can collaborate with the earlier data.

II. EXPERIMENTAL

A. Sample preparation

The sample (Figure 1) was purchased from Tianjin Heowns Biochem LLC (China). The melting point and measured density of the title compound are 200–201°C and 1.383 g cm^{-3}, respectively. The FTIR spectrum of the title compound was presented in Figure SI. Crystallization of azilsartan at room temperature was successful using methanol as solvent. Then, part of crystals were dried and ground into powder.

B. Diffraction data collection and reduction

The PXRD 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 0.01313° 2θ and a counting time of 30 ms step^{-1} (Figure 2).

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, Chengdu, China). The PXRD pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results were obtained by the X-Cell method

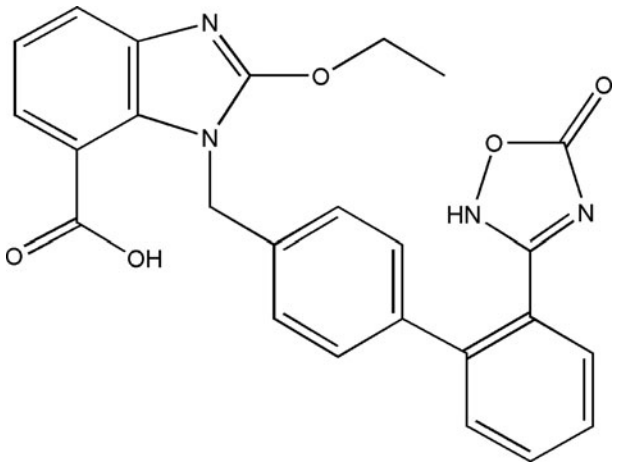

Figure 1. Structural formula of azilsartan.

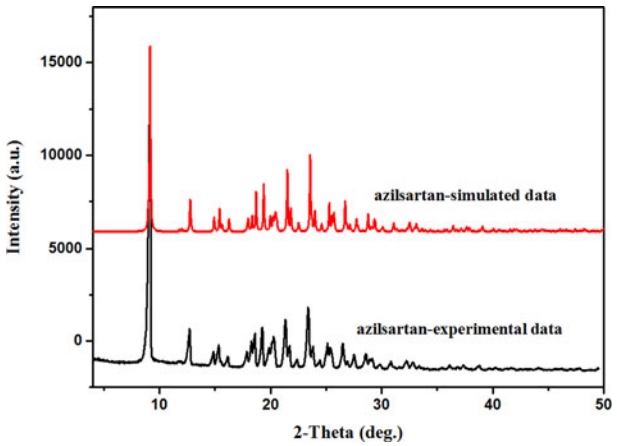

Figure 2. (Colour online) XRD pattern of azilsartan using Cu$K\alpha$ radiation (bottom line) and the simulated pattern of ours (top line).

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TABLE I. Indexed X-ray powder diffraction data for azilsartan

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
9.1699	9.6410	100	1	0	0	9.1650	9.6412	0.0049
12.7807	6.9241	11	1	0	2	12.7795	6.9213	0.0012
14.9341	5.9302	3	1	1	−2	14.9302	5.9288	0.0039
15.3937	5.7542	4	0	1	3	15.4124	5.7443	−0.0188
16.2734	5.4451	2	0	2	1	16.2855	5.4383	−0.0121
17.9147	4.9497	3	1	1	−3	17.9180	4.9463	−0.0033
18.0328	4.9176	3	0	2	2	18.0132	4.9204	0.0197
18.3874	4.8235	8	2	0	0	18.3893	4.8206	−0.0020
18.6894	4.7463	6	1	2	−1	18.7044	4.7401	−0.0151
19.3590	4.5836	7	0	1	4	19.3879	4.5745	−0.0289
20.0024	4.4376	5	1	0	4	20.0298	4.4293	−0.0274
20.2650	4.3807	5	1	2	2	20.2704	4.3773	−0.0054
20.3832	4.3555	6	2	0	−2	20.3835	4.3533	−0.0003
20.4357	4.3444	7	2	1	−1	20.4738	4.3343	−0.0381
21.4598	4.1394	10	1	1	−4	21.4357	4.1419	0.0242
21.5124	4.1294	10	1	1	4	21.5301	4.1239	−0.0178
22.5365	3.9440	2	1	2	−3	22.5377	3.9418	−0.0011
23.5476	3.7769	10	0	1	5	23.5631	3.7725	−0.0155
24.0071	3.7056	4	0	3	1	24.0168	3.7023	−0.0097
24.6111	3.6160	2	2	2	−1	24.6377	3.6104	−0.0266
25.2545	3.5253	4	0	3	2	25.2392	3.5257	0.0153
25.3070	3.5181	4	1	1	−5	25.2800	3.5201	0.0270
25.5696	3.4826	4	2	0	−4	25.5616	3.4819	0.0080
26.7120	3.3362	5	0	0	6	26.7076	3.3351	0.0043
27.1453	3.2839	2	0	3	3	27.1622	3.2803	−0.0170
27.7361	3.2153	3	3	0	0	27.7357	3.2137	0.0004
28.7734	3.1017	3	1	2	−5	28.7935	3.0980	−0.0201
29.1673	3.0607	3	3	1	−1	29.1819	3.0577	−0.0146
29.3511	3.0419	3	1	1	−6	29.3423	3.0413	0.0089
30.1258	2.9655	2	2	1	5	30.1400	2.9626	−0.0142
32.5287	2.7517	2	1	2	6	32.5489	2.7487	−0.0203
33.0408	2.7102	2	2	3	3	33.0495	2.7082	−0.0088

The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

(Neumann, 2003). The indexing results were then refined using Pawley (Pawley, 1981), which involves assigning the Miller indices (h, k, l) to each observed peak in the experimental PXRD pattern.

C. Single-crystal XRD

XRD data for azilsartan were collected on an Xcalibur, Eos diffractometer. The crystal was kept at 293.15 K during data collection. The structure was solved with olex2 (Dolomanov *et al.*, 2009), a structure solution program using charge flipping and refined with the ShelXL (Sheldrick, 2008). Structure solution program using Direct Methods and refined with the ShelXL (Sheldrick, 2015) refinement package using least-squares minimization.

III. RESULTS

Pawley refinement results confirmed that the title compound is monoclinic with space group $P2_1/c$ and unit-cell parameters: $a = 9.641(3)$ Å, $b = 11.301(9)$ Å, $c = 20.010(8)$ Å, $\alpha = 90°$, $\beta = 90.351(5)°$, $\gamma = 90°$, unit-cell volume $V = 2196.735(4)$ Å^3, $Z = 4$, and $\rho_{cal} = 1.379$ g cm^{-3}. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, and $\Delta 2\theta$ are listed in Table I. The results were in good agreement with the single crystallographic data of ours [$a = 9.6649(3)$ Å, $b = 11.3218(3)$ Å, $c = 20.0104(7)$ Å, $\alpha = 90°$, $\beta = 90.285(3)°$, $\gamma = 90°$, unit-cell volume $V = 2189.60(12)$ Å^3, $Z = 4$, $\rho_{cal} = 1.385$ g cm^{-3}]. The detail single crystallographic data of azilsartan and the experimental data were listed in Table SI. The comparison of the experimental PXRD pattern with the simulated pattern of ours is shown in Figure 2. Results showed that both single-crystal and powder diffraction methods can get similar structure data.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715618000210.

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