X-ray powder diffraction data for rivaroxaban, $C_{19}H_{18}ClN_3O_5S$

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(Received 11 June 2021; accepted 7 November 2021)

X-ray powder diffraction data, unit-cell parameters, and space group for rivaroxaban, $C_{19}H_{18}ClN_3O_5S$, are reported [$a = 9.010(3)$ Å, $b = 10.986(6)$ Å, $c = 11.230(1)$ Å, $\alpha = 63.439(5)°$, $\beta = 74.355(4)°$, $\gamma = 78.133(3)°$, unit-cell volume $V = 952.87$ Å^3, $Z = 2$, $\rho_{cal} = 1.519$ g cm^{-3}, and space group $P1$]. All measured lines were indexed and are consistent with the $P1$ space group. No detectable impurities were observed. © *The Author(s), 2021. Published by Cambridge University Press on behalf of International Centre for Diffraction Data*. [doi:10.1017/S0885715621000634]

Key words: rivaroxaban, factor Xa inhibitor, X-ray powder diffraction

I. INTRODUCTION

Rivaroxaban (Figure 1), a novel oral direct factor Xa inhibitor, has been widely used for nonvalvular atrial fibrillation (NVAF) and deep venous thrombosis or venous embolism (Akao *et al.*, 2021). It can inhibit the formation of thrombin by inhibiting factor Xa activity and ultimately inhibit thrombus formation and enlargement (Ajmal *et al.*, 2021).

The single crystallographic data of rivaroxaban [$a = 9.0184$ (6) Å, $b = 10.9980$ (8) Å, $c = 11.2386$ (8) Å, $\alpha = 63.426$ (2)°, $\beta = 74.414$ (3)°, $\gamma = 78.144$ (2)°, unit-cell volume $V = 955.56$ (12) Å^3, $Z = 2$, $\rho_{cal} = 1.515$ g cm^{-3}, and space group $P1$] had been deposited with the Cambridge Crystallographic Data Center (CCDC) with a supplementary publication number of CCDC-1810879 (Shen *et al.*, 2017). To date, the detailed X-ray powder diffraction (PXRD) data for rivaroxaban have not been reported.

II. EXPERIMENTAL

A. Sample preparation

The sample was purchased from Energy Chemical (Shanghai, People's Republic of China). The melting point and measured density of rivaroxaban are 227–228 °C and 1.491 g cm^{-3}, respectively. Recrystallization of rivaroxaban at room temperature was successful using methanol as a solvent. Then, the crystals were dried, smashed, screened through 75 μm mesh size, and pressed flat to a zero background plate.

B. Diffraction data collection and reduction

XRD measurement was performed at room temperature using a Bruker D8 Advance diffractometer (Bruker, AXS, Germany) with a LynxEye array detector and Cu$K\alpha$ radiation (generator setting: 40 kV and 40 mA). The diffraction data were collected over the angular range from 5° to 50° 2θ with a step size of 0.019757° 2θ and a counting time of 37 ms/step. Some peaks in the diffraction pattern display asymmetry which is possibly because of stacking faults (Langford and Louër, 1991).

The software package Material Studio 8.0 (Accelrys Co., Ltd., CA, USA) was used to process the data. The PXRD pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Peaks were located in the diffraction pattern using automated peak searching. Automatic indexing results were obtained by X-cell method (Neumann, 2003). The indexing results were then refined using Pawley ($R_{wp} = 6.51\%$) (Pawley, 1981), which involves assigning the Miller indices (h, k, l) to each observed peak in the experimental PXRD pattern. Line shift correction was turned on during the cell parameter refinement. All measured lines in the raw data were indexed. The method of determination of intensity was peak height above background.

III. RESULTS

Pawley refinement results confirmed that rivaroxaban is triclinic with space group $P1$ and unit-cell parameters: $a = 9.010(3)$ Å, $b = 10.986(6)$ Å, $c = 11.230(1)$ Å, $\alpha = 63.439(5)°$, $\beta = 74.355(4)°$, $\gamma = 78.133(3)°$, unit-cell volume $V = 952.87$ Å^3, $Z = 2$, $\rho_{cal} = 1.519$ g cm^{-3}. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, $\Delta 2\theta$ are listed in Table I. The results are in good agreement with the single-crystal data and the deviations of the unit-cell parameters and unit-cell volume were between 0.02% and 0.28%. The comparison of PXRD pattern (raw data file deposited with ICDD) with the simulated pattern is shown in Figure 2.

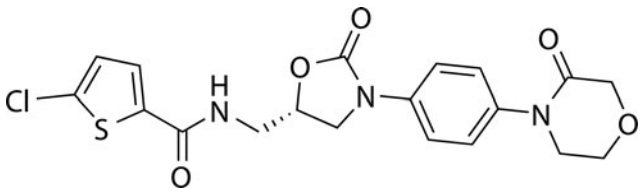


Figure 1. Molecular diagram of rivaroxaban.

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https://doi.org/10.1017/S0885715621000634 Published online by Cambridge University Press

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

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal}(Å)	$\Delta 2\theta$
9.0304	9.7848	13	0	1	0	9.0354	9.7792	−0.0050
12.0335	7.3488	5	1	1	1	12.0097	7.3632	0.0238
14.3648	6.1610	7	1	−1	0	14.3273	6.1769	0.0375
15.2144	5.8188	4	0	1	−1	15.1884	5.8286	0.0260
16.4788	5.3751	11	0	1	2	16.4560	5.3823	0.0228
16.5578	5.3496	23	0	2	1	16.5420	5.3545	0.0158
16.8344	5.2623	3	1	1	2	16.8152	5.2682	0.0193
17.5062	5.0619	7	1	2	1	17.4616	5.0746	0.0446
17.8618	4.9619	3	1	−1	1	17.8149	4.9747	0.0469
18.0594	4.9080	6	0	0	2	18.0226	4.9179	0.0368
18.1186	4.8921	4	0	2	0	18.1276	4.8896	−0.0089
18.8892	4.6943	3	1	1	−1	18.8703	4.6988	0.0189
19.5807	4.5300	26	0	2	2	19.5545	4.5359	0.0262
19.9758	4.4413	21	1	2	0	19.9485	4.4472	0.0273
21.7342	4.0858	13	1	−2	0	21.7365	4.0853	−0.0023
22.5640	3.9374	100	2	1	2	22.5400	3.9414	0.0240
23.3938	3.7996	12	0	1	−2	23.3624	3.8045	0.0314
24.1643	3.6801	3	2	2	2	24.1539	3.6816	0.0104
24.5594	3.6218	4	1	−2	−2	24.5628	3.6212	−0.0033
24.7768	3.5905	6	0	1	3	24.7748	3.5907	0.0019
25.1917	3.5323	4	1	3	1	25.1892	3.5326	0.0025
25.6658	3.4681	23	0	2	3	25.6436	3.4710	0.0222
25.7449	3.4577	15	0	3	2	25.7376	3.4585	0.0073
26.4561	3.3663	5	2	1	−1	26.4143	3.3714	0.0418
26.6932	3.3369	30	1	1	−2	26.6804	3.3384	0.0128
27.2069	3.2751	6	0	0	3	27.1766	3.2786	0.0303
28.7677	3.1008	3	1	−3	−1	28.7287	3.1049	0.0390
29.3011	3.0456	3	1	−1	−3	29.2793	3.0477	0.0219
29.4394	3.0316	3	2	3	1	29.4070	3.0348	0.0324
29.5184	3.0237	4	0	3	3	29.5148	3.0239	0.0036
29.8543	2.9904	2	2	−1	−2	29.8745	2.9884	−0.0202
30.0914	2.9674	5	1	−3	−2	30.0945	2.9670	−0.0031
30.3482	2.9428	3	1	−2	−3	30.3435	2.9432	0.0047
30.7236	2.9077	3	2	0	3	30.6863	2.9111	0.0373
31.0792	2.8753	3	3	0	0	31.0478	2.8780	0.0315
31.8498	2.8074	3	3	2	2	31.8146	2.8104	0.0352
32.1461	2.7822	2	0	1	−3	32.1748	2.7798	−0.0287
33.6872	2.6584	3	0	1	4	33.6588	2.6605	0.0284
34.0428	2.6314	2	1	−3	−3	34.0078	2.6340	0.0350
34.5367	2.5949	2	3	2	0	34.5373	2.5948	−0.0005
34.9714	2.5637	2	2	1	4	34.9529	2.5649	0.0185
35.7814	2.5075	2	2	−1	3	35.7820	2.5074	−0.0006
36.7495	2.4436	2	0	4	0	36.7298	2.4448	0.0197
37.5595	2.3927	2	2	−2	−3	37.5628	2.3925	−0.0033
38.2708	2.3499	2	2	2	−2	38.2781	2.3494	−0.0073
39.0808	2.3030	2	2	−3	1	39.0759	2.3033	0.0049
39.3179	2.2897	2	3	2	−1	39.2893	2.2912	0.0286
39.8316	2.2613	3	1	−3	−4	39.8083	2.2625	0.0233
40.1279	2.2453	2	1	2	5	40.1581	2.2436	−0.0302
41.5505	2.1717	2	3	−2	−2	41.5512	2.1716	−0.0007
41.7085	2.1638	2	0	2	5	41.7321	2.1626	−0.0236
42.8149	2.1104	2	0	5	3	42.8424	2.1091	−0.0275
43.2298	2.0911	2	3	0	4	43.2340	2.0909	−0.0042
43.8225	2.0642	2	3	−3	−1	43.8166	2.0644	0.0059
44.1386	2.0502	2	2	−1	−4	44.1319	2.0504	0.0067
44.3955	2.0389	3	1	1	−4	44.4002	2.0386	−0.0048
44.9091	2.0167	2	2	3	−2	44.9267	2.0160	−0.0175
45.8970	1.9756	2	1	−2	−5	45.8590	1.9771	0.0380
46.5687	1.9487	2	1	5	0	46.5478	1.9494	0.0209
46.7465	1.9417	1	1	−1	−5	46.7527	1.9414	−0.0061
46.8848	1.9363	2	1	−3	−5	46.9304	1.9344	−0.0455
49.5323	1.8388	2	4	−1	3	49.5547	1.8380	−0.0224
49.7101	1.8326	2	2	1	−4	49.6995	1.8330	0.0106
49.9472	1.8245	2	3	−3	2	49.9449	1.8245	0.0023

The d-values were calculated using CuK$\alpha_1$radiation ($\lambda = 1.5405981$ Å).

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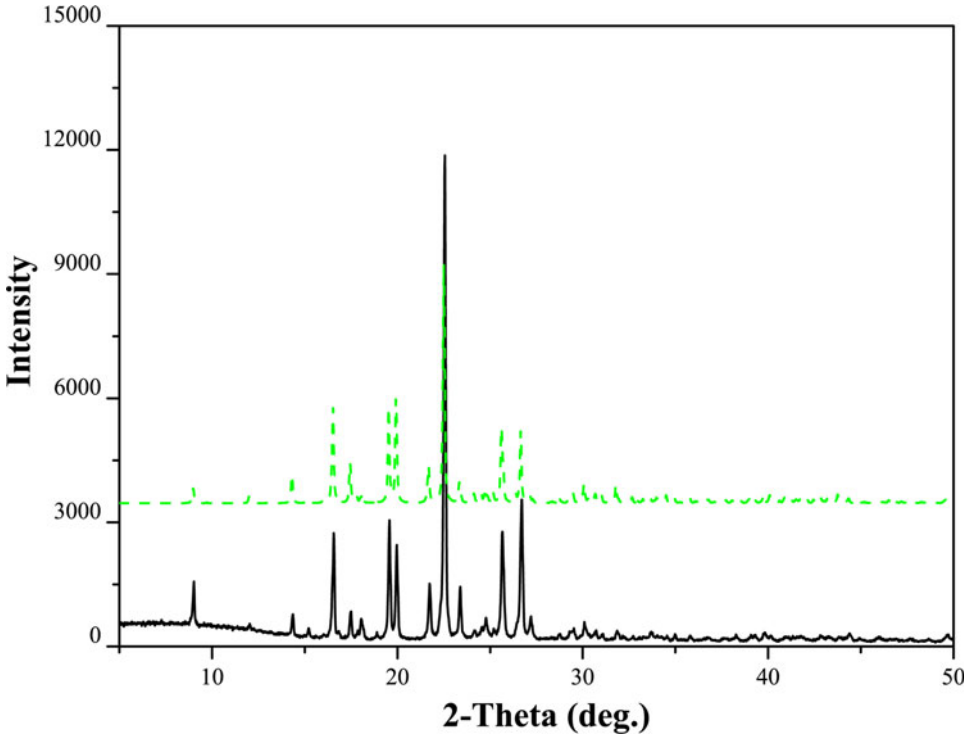


Figure 2. X-ray powder diffraction pattern of rivaroxaban using CuKα radiation (black solid line) and the simulated pattern of the crystal structure (green dash line).

IV. DEPOSITED DATA

The rivaroxaban, the raw data file has been deposited with ICDD. You may request this data from ICDD at info@icdd.com.

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