X-ray powder diffraction data for Palbociclib, $C_{24}H_{29}N_7O_2$

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X-ray powder diffraction (XRPD) data for Palbociclib, $C_{24}H_{29}N_7O_2$, are reported [$a = 18.182(2)$ Å, $b = 11.508(1)$ Å, $c = 5.041(1)$ Å, $\alpha = 81.282(7)°$, $\beta = 97.423(7)°$, $\gamma = 102.415(2)°$, unit-cell volume $V = 1013.1(4)$ Å^3, $Z = 2$, and space group P-1. All XRPD measured lines were indexed and no detectable impurities were observed. © *2016 International Centre for Diffraction Data*.
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Key words: X-ray powder diffraction, Palbocilib

I. INTRODUCTION

Palbociclib [PD-0332991, chemical name 6-acetyl-8-cyclopentyl-5-methyl-2-((5-(piperazin-1-yl)pyridin-2-yl)amino) pyrido[2,3-d]pyrimidin-7(8H)-one] (Figure 1) developed as IBRANCE by Pfizer, is a kinase inhibitor indicated in combination with letrozole for the treatment of postmenopausal women with estrogen receptor-positive, human epidermal growth factor receptor 2-negative advanced breast cancer as initial endocrine-based therapy for their metastatic disease. This indication is approved under accelerated approval based on progression-free survival by FDA on February 2015.

Presently, the crystal structure of Palbocilib has not been reported.

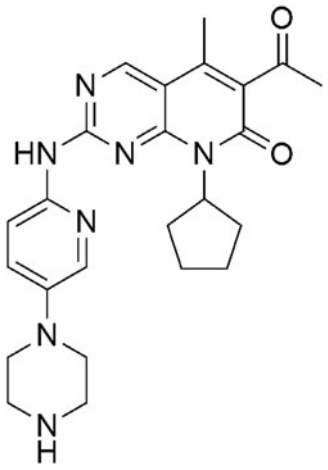

Figure 1. The structure of Palbociclib.

II. EXPERIMENTAL

A. Sample preparation

The title compound was purchased from Sichuan CheCO Pharmaceutical Technology Co., Ltd, China. It was recrystallized in acetone and water, and then dried and ground into powder. The structure of Palbociclib was characterized by high-performance liquid chromatography (HPLC), high-resolution mass spectrum (HRMS), Fourier transform infrared spectroscopy, and ^{1}H-NMR.

The purity of sample is 99.5% measured by HPLC, HRMS showed $m/z = 448.2417$ peak, the melting point is 269–271 °C, the infrared spectrum is consistent with the structure, ^{1}H-NMR (see supporting information) is consistent with literature (Chekal and Ide, 2014).

B. X-ray powder diffraction (XRPD) data collection and reduction

The diffraction pattern for the title compound was collected at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd, The Netherlands) with an X'celerator detector and Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å, 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}. Data evaluation was performed using the software package Material Studio 4.2 (Accelrys Co., Ltd, USA).

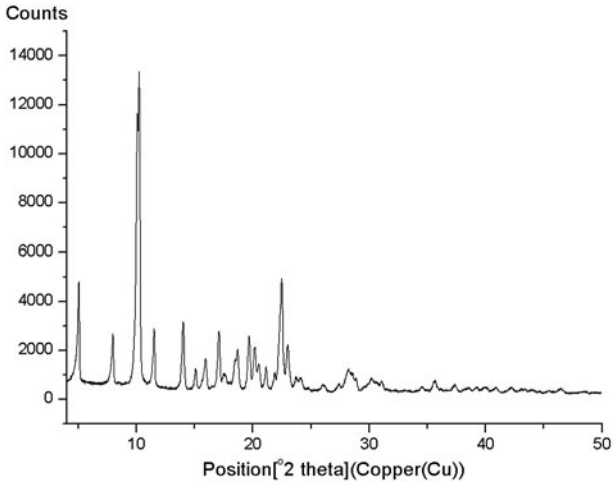

Figure 2. XRPD pattern of Palbocilib.

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TABLE I. Indexed XRPD data of Palbociclib, $C_{24}H_{29}N_7O_2$

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
4.9913	17.6902	28	1	0	0	4.9977	17.6678	0.0063
7.9325	11.1365	18	0	1	0	7.9252	11.1468	−0.0074
9.9940	8.8435	62	2	0	0	10.0048	8.8339	0.0109
10.1909	8.6730	100	1	1	0	10.1841	8.6788	−0.0068
11.4646	7.7122	18	2	−1	0	11.4647	7.7121	0.0001
13.9593	6.3390	20	2	1	0	13.9666	6.3357	0.0073
15.0360	5.8874	8	3	0	0	15.0313	5.8893	−0.0047
15.6794	5.6473	6	1	−2	0	15.6771	5.6481	−0.0023
15.8895	5.5731	12	0	2	0	15.8886	5.5734	−0.0009
17.0187	5.2058	18	2	−2	0	17.0205	5.2052	0.0019
17.6095	5.0324	8	1	2	0	17.6003	5.0350	−0.0092
17.8721	4.9590	0	0	0	1	17.8764	4.9579	0.0042
18.0691	4.9054	6	1	0	−1	18.0787	4.9028	0.0096
18.3711	4.8255	10	3	1	0	18.3729	4.8250	0.0018
18.3842	4.8220	10	1	−1	−1	18.3825	4.8225	−0.0017
18.6074	4.7647	13	0	1	1	18.6196	4.7616	0.0121
19.6185	4.5214	17	3	−2	0	19.6104	4.5232	−0.0081
19.6316	4.5184	18	2	0	−1	19.6204	4.5209	−0.0112
20.0780	4.4189	14	4	0	0	20.0870	4.4170	0.0090
20.1568	4.4018	16	1	1	1	20.1585	4.4015	0.0017
20.4457	4.3403	10	2	2	0	20.4499	4.3394	0.0042
21.0628	4.2145	8	1	1	−1	21.0626	4.2145	−0.0002
21.7587	4.0812	6	3	−1	−1	21.7534	4.0822	−0.0054
21.8244	4.0691	8	1	−2	−1	21.8423	4.0658	0.0179
22.2314	3.9955	18	3	0	−1	22.2347	3.9949	0.0033
22.4021	3.9655	32	0	2	1	22.4076	3.9645	0.0055
22.9404	3.8736	15	2	−1	1	22.9486	3.8722	0.0082
23.4394	3.7923	5	1	−3	0	23.4443	3.7915	0.0049
23.9383	3.7143	6	0	3	0	23.9301	3.7156	−0.0082
24.1090	3.6884	6	3	−2	−1	24.1003	3.6897	−0.0088
25.7372	3.4587	3	1	−2	1	25.7326	3.4593	−0.0046
26.0917	3.4125	4	3	1	1	26.0906	3.4126	−0.0011
27.0240	3.2968	3	5	−2	0	27.0286	3.2963	0.0047
27.3391	3.2595	4	1	−3	−1	27.3336	3.2602	−0.0055
27.8249	3.2037	5	2	3	0	27.8200	3.2043	−0.0049
28.0875	3.1744	8	0	3	1	28.0874	3.1744	−0.0001
28.3501	3.1456	8	4	0	1	28.3522	3.1453	0.0021
28.7046	3.1075	6	4	1	−1	28.7067	3.1073	0.0020
29.9651	2.9796	5	4	1	1	29.9602	2.9801	−0.0050
30.3197	2.9456	6	6	0	0	30.3293	2.9446	0.0096
30.5691	2.9221	5	4	−3	−1	30.5616	2.9228	−0.0075
30.8843	2.8930	5	3	3	0	30.8848	2.8929	0.0005
31.2388	2.8610	4	5	−3	0	31.2360	2.8612	−0.0027
32.9457	2.7165	3	6	1	0	32.9474	2.7164	0.0017
33.1952	2.6967	3	5	−3	−1	33.1976	2.6965	0.0024
33.5103	2.6720	2	1	4	0	33.5119	2.6719	0.0016
33.9699	2.6369	3	1	−4	−1	33.9645	2.6373	−0.0053
34.4294	2.6028	4	4	−4	0	34.4318	2.6026	0.0024
34.8102	2.5752	3	7	−1	0	34.8167	2.5747	0.0065
35.1122	2.5537	3	5	−2	1	35.1187	2.5532	0.0065
35.5324	2.5245	5	7	0	0	35.5395	2.5240	0.0071
35.9788	2.4942	3	7	−2	0	35.9871	2.4936	0.0083
36.0182	2.4915	3	2	−1	−2	36.0252	2.4911	0.0070
36.3990	2.4663	3	6	−3	−1	36.4005	2.4662	0.0016
36.7010	2.4467	3	6	1	−1	36.7097	2.4462	0.0088
36.7141	2.4459	3	3	3	−1	36.7178	2.4456	0.0037
37.1080	2.4208	3	3	−1	−2	37.1007	2.4213	−0.0073
37.2656	2.4109	4	2	−2	−2	37.2607	2.4112	−0.0049
38.4604	2.3387	3	5	3	0	38.4679	2.3383	0.0074
39.1432	2.2995	4	2	−4	1	39.1462	2.2993	0.0029
39.5765	2.2753	3	4	−2	−2	39.5808	2.2751	0.0043
39.8260	2.2616	3	6	−4	0	39.8266	2.2616	0.0006
40.0492	2.2496	4	7	−3	−1	40.0587	2.2490	0.0095
40.4300	2.2292	3	3	1	−2	40.4297	2.2293	−0.0003
40.7582	2.2120	3	3	−3	−2	40.7520	2.2124	−0.0062
40.7976	2.2100	3	8	−2	0	40.8058	2.2096	0.0082

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
41.2047	2.1891	2	5	−1	−2	41.2011	2.1893	−0.0035
41.5460	2.1719	2	4	−5	0	41.5436	2.1720	−0.0024
41.8086	2.1589	3	8	−1	−1	41.8180	2.1584	0.0094
41.9531	2.1518	3	4	−4	1	41.9512	2.1519	−0.0019
42.1500	2.1422	4	8	−2	−1	42.1557	2.1419	0.0056
42.4652	2.1270	3	1	3	2	42.4873	2.1259	0.0221
42.7803	2.1120	3	6	3	0	42.7831	2.1119	0.0028
42.9641	2.1034	3	5	3	−1	42.9650	2.1034	0.0009
43.2530	2.0901	3	8	−3	0	43.2480	2.0903	−0.0050
43.7782	2.0662	3	3	4	−1	43.7772	2.0662	−0.0010
44.0277	2.0551	3	5	−5	−1	44.0299	2.0550	0.0022
45.3670	1.9975	3	6	0	−2	45.3668	1.9975	−0.0002
45.7346	1.9823	3	0	4	2	45.7346	1.9823	0.0000
46.1416	1.9657	3	6	−3	−2	46.1485	1.9654	0.0069
46.2992	1.9594	3	3	5	0	46.2931	1.9596	−0.0061
46.4699	1.9526	3	4	2	2	46.4575	1.9531	−0.0124
46.6275	1.9464	3	8	0	1	46.6294	1.9463	0.0020
47.1527	1.9259	2	8	2	0	47.1531	1.9259	0.0004
47.4021	1.9163	2	7	−1	−2	47.4048	1.9162	0.0027
49.0040	1.8574	2	7	−3	−2	49.0044	1.8574	0.0004

Only the peaks with I_{rel} of 2 or greater are reported $a = 18.182(2)$ Å, $b = 11.508(1)$ Å, $c = 5.041(1)$ Å, $\alpha = 81.282(7)°$, $\beta = 97.423(7)°$, $\gamma = 102.415(2)°$, unit-cell volume $V = 1013.1(4)$ Å^3, $Z = 2$, and space group P-1. All measured lines were indexed. The d-values were calculated using Cu$K_{\alpha 1}$ radiation ($\lambda = 1.54056$ Å).

The powder diffraction pattern was pretreated by subtracting the background and smoothing. Through analyzing the peak positions in the XRPD pattern by the X-Cell method from "Powder Indexing", the preliminary unit-cell parameters were obtained. The indexing results were then refined using the Pawley refinement (Pan *et al.*, 2012), which involves assigning the Miller indices (*h, k, l*) to each observed peak in the experimental powder XRD pattern (Harris, 2012). After Pawley refinement, the final R_{wp} of the spectrogram was converged to 6.95%.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Pawley refinement confirms that the lattice type of Palbociclib is triclinic with space group P-1 and unit-cell parameters: [$a = 18.182(2)$ Å, $b = 11.508(1)$ Å, $c = 5.041(1)$ Å, $\alpha = 81.282(7)°$, $\beta = 97.423(7)°$, $\gamma = 102.415(2)°$, unit-cell volume $V = 1013.1(4)$ Å^3, $Z = 2$]. 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.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at http://dx.doi.org/10.1017/S0885715616000233.

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