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DATA REPORT

X-ray powder diffraction data for alogliptin benzoate, $C_{18}H_{21}N_5O_2 \cdot C_7H_6O_2$

Xia Zeng, Qing Wang, Xin Nuo Xiong, Qiao Hong Du, and Hui Li[a)]
College of Chemical Engineering, Sichuan University, Chengdu 610065, China

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X-ray powder diffraction data, unit-cell parameters, and space group for alogliptin benzoate, $C_{18}H_{21}N_5O_2 \cdot C_7H_6O_2$, are reported [$a = 28.8260(8)$ Å, $b = 9.9654(4)$ Å, $c = 8.1477(8)$ Å, $\alpha = 90$, $\beta = 90$, $\gamma = 90°$, unit-cell volume $V = 2340.57$ Å^3, $Z = 4$, $\rho_{cal} = 1.3097$ g cm^{-3}, and space group $P2_12_12_1$]. All measured lines were indexed and are consistent with the $P2_12_12_1$ space group, No detectable impurities were observed. © *2016 International Centre for Diffraction Data.*
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Key words: X-ray powder diffraction, alogliptin benzoate

I. INTRODUCTION

Alogliptin benzoate (Nesina®) (Figure 1), systematic name 2-[6-[3(R)-Aminopiperidin-1-yl]-3-methyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ylmethyl]benzonitrile benzoate, is a newly developed DPP-4 inhibitor (Saisho, 2015). The efficacy of the compound as a highly selective dipeptidyl peptidase-4 (DPP-4) inhibitor has been demonstrated (Feng *et al.*, 2007). DPP-4 inhibitors (McIntosh *et al.*, 2005) metabolize the insulin-increasing hormones glucagon-like peptide-1 (GLP-1) and glucose-dependent insulinotropic peptide. Therefore, alogliptin benzoate has become one of the most promising type-2 diabetes mellitus drugs.

The single crystallographic data of alogliptin benzoate was reported by Gelbrich *et al.* (2013), the values of cell parameters were $a = 28.4593(6)$ Å, $b = 9.8727(2)$ Å, $c = 8.0666(2)$ Å, $\alpha = 90$, $\beta = 90$, $\gamma = 90°$, unit-cell volume $V = 2266.47(9)$ Å^3, $Z = 4$, $\rho_{cal} = 1.353$ g cm^{-3}, and space group $P2_12_12_1$. Presently, the detailed X-ray powder diffraction data for alogliptin benzoate have not been reported.

II. EXPERIMENTAL

A. Sample preparation

The title compound alogliptin benzoate, with purity of more than 99%, was from Wuhan E-ternity Technologies Co., Ltd. (Shanghai, China). It was recrystallized in methanol and dried. The sample melting point and measured density of the title compound are 183–184 °C and 1.353 g cm^{-3}, respectively. Moreover, the sample was then ground into powder and mounted on a flat zero background plate.

B. Diffraction data collection and reduction

X-ray powder diffraction measurement was performed at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with a PIXcel one-dimensional (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 50 ms step^{-1}. Figure 2 shows the Powder X-ray diffraction pattern of the compound.

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 X-ray powder diffraction pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component (Li *et al.*, 2014). Automatic indexing results were obtained by the DICVOL91 method (Boultif and Louër, 1991). Then the indexing result was refined using the Pawley method (Wang *et al.*, 2015). Pawley refinement converged to $R_{wp} = 6.46\%$.

III. RESULTS

The figure-of-merit was achieved: $F_{24} = 44.7$ (0.0080, 76) (Smith and Snyder, 1979) and $M_{24} = 16.8$ (de Wolff, 1968). Pawley refinement results confirmed that the title compound is orthorhombic with space group $P2_12_12_1$ and unit-cell parameters: $a = 28.8260(8)$ Å, $b = 9.9654(4)$ Å, $c = 8.1477(8)$ Å, $\alpha = 90$, $\beta = 90$, $\gamma = 90°$, unit-cell volume $V = 2340.57$ Å^3, $Z = 4$, and $\rho_{cal} = 1.3097$ g cm^{-3}. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, I_{cal}, and $\Delta 2\theta$ are listed in Table I.

The comparison of this pattern with the calculated pattern of single-crystal data is shown in online Figure S1. The results showed that both single-crystal and powder diffraction methods can get similar structure data.

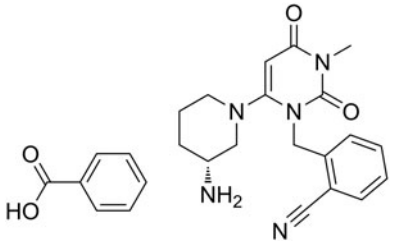


Figure 1. Structural formula of alogliptin benzoate.

[a)]Author to whom correspondence should be addressed. Electronic mail: lihuilab@sina.com

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TABLE I. X-ray powder diffraction data for the alogliptin benzoate, $C_{18}H_{21}N_5O_2 \cdot C_7H_6O_2$. $a = 28.8260(8)$ Å, $b = 9.9654(4)$ Å, $c = 8.1477(8)$ Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$, unit-cell volume $V = 2340.57$ Å^3, $Z = 4$, $\rho_{cal} = 1.3097$ g cm^{-3}, and space group $P2_12_12_1$. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$ (°)
9.4162	9.3845	96	1	1	0	9.3822	9.4185	0.0340
10.8212	8.1691	48	2	1	0	10.7843	8.1969	0.0369
11.2939	7.8282	17	1	0	1	11.2760	7.8406	0.0179
12.5019	7.0744	17	2	0	1	12.4691	7.0929	0.0328
12.7776	6.9223	10	3	1	0	12.7874	6.9171	−0.0098
14.0381	6.3035	10	0	1	1	14.0284	6.3078	0.0097
14.3664	6.1602	10	1	1	1	14.3620	6.1620	0.0044
15.1936	5.8266	41	4	1	0	15.1595	5.8396	0.0341
15.3117	5.7819	7	2	1	1	15.3204	5.7786	−0.0087
16.8217	5.2661	9	3	1	1	16.7993	5.2731	0.0224
17.7802	4.9843	53	0	2	0	17.7861	4.9827	−0.0059
18.0691	4.9053	10	1	2	0	18.0520	4.9099	0.0171
18.6994	4.7414	100	4	1	1	18.6792	4.7464	0.0202
18.8700	4.6989	57	5	0	1	18.8403	4.7062	0.0297
20.0649	4.4217	26	3	2	0	20.0572	4.4234	0.0077
20.5245	4.3237	10	6	1	0	20.5053	4.3277	0.0192
20.8790	4.2511	77	5	1	1	20.8567	4.2555	0.0223
21.1285	4.2014	51	1	2	1	21.1084	4.2054	0.0201
21.4698	4.1354	16	6	0	1	21.4537	4.1385	0.0161
21.8112	4.0714	36	0	0	2	21.7979	4.0739	0.0133
21.9951	4.0378	13	1	0	2	22.0172	4.0338	−0.0221
22.8617	3.8867	11	3	2	1	22.8572	3.8874	0.0045
23.2556	3.8217	12	6	1	1	23.2539	3.8220	0.0017
23.6101	3.7651	19	5	2	0	23.5801	3.7698	0.0300
23.8070	3.7344	13	1	1	2	23.7769	3.7391	0.0301
24.3979	3.6453	12	2	1	2	24.3786	3.6482	0.0193
25.3695	3.5079	6	3	1	2	25.3516	3.5103	0.0179
25.8422	3.4448	18	7	1	1	25.8157	3.4482	0.0265
26.6563	3.3414	6	4	1	2	26.6579	3.3412	−0.0016
26.7876	3.3253	11	5	0	2	26.7731	3.3271	0.0145
27.0240	3.2967	32	8	0	1	27.0353	3.2954	−0.0113
27.5229	3.2381	18	2	3	0	27.5328	3.2370	−0.0099
28.0219	3.1816	11	6	2	1	28.0037	3.1836	0.0182
28.2713	3.1541	7	0	2	2	28.2727	3.1539	−0.0014
28.4683	3.1327	20	8	1	1	28.5047	3.1288	−0.0364
28.7440	3.1033	9	6	0	2	28.7069	3.1072	0.0371
29.0066	3.0758	15	0	3	1	29.0043	3.0760	0.0023
29.2036	3.0555	12	1	3	1	29.1726	3.0586	0.0310
29.5581	3.0196	6	4	3	0	29.5868	3.0168	−0.0287
29.6894	3.0066	11	2	3	1	29.6723	3.0083	0.0171
29.9257	2.9833	5	9	0	1	29.9514	2.9809	−0.0257
30.0964	2.9668	6	6	1	2	30.1014	2.9663	−0.0050
30.2015	2.9567	6	7	2	1	30.1912	2.9577	0.0103
30.4903	2.9294	6	3	3	1	30.4886	2.9295	0.0017
30.5954	2.9196	6	8	2	0	30.5928	2.9198	0.0026
31.0549	2.8774	5	5	3	0	31.0457	2.8782	0.0092
31.6195	2.8273	7	4	3	1	31.5993	2.8291	0.0202
32.1448	2.7823	4	7	1	2	32.1590	2.7811	−0.0142
32.3155	2.768	4	5	2	2	32.3280	2.7669	−0.0125
32.9457	2.7165	5	10	0	1	32.9321	2.7175	0.0136
32.9851	2.7133	5	5	3	1	32.9779	2.7139	0.0072
33.2214	2.6945	4	9	2	0	33.2247	2.6943	−0.0033
33.9830	2.6359	4	6	2	2	33.9739	2.6366	0.0091
34.2587	2.6153	9	3	0	3	34.2824	2.6135	−0.0237
34.3506	2.6085	10	1	1	3	34.3357	2.6096	0.0149
34.3900	2.6056	9	8	1	2	34.3960	2.6052	−0.0060
34.5739	2.5922	5	6	3	1	34.5964	2.5905	−0.0225
34.8233	2.5742	5	0	3	2	34.8192	2.5745	0.0041
35.2698	2.5426	5	4	0	3	35.2864	2.5414	−0.0166
35.3748	2.5353	7	2	3	2	35.3884	2.5343	−0.0136
35.4667	2.5289	9	3	1	3	35.4796	2.5280	−0.0129
35.8475	2.5029	4	7	2	2	35.8330	2.5039	0.0145
35.9657	2.4950	6	10	2	0	35.9632	2.4951	0.0025
36.0182	2.4915	6	0	4	0	36.0197	2.4914	−0.0015
36.0707	2.4880	7	3	3	2	36.0888	2.4867	−0.0181

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$ (°)
36.1495	2.4827	7	1	4	0	36.1586	2.4821	−0.0091
36.5303	2.4577	4	5	0	3	36.5419	2.4569	−0.0116
36.5565	2.4560	5	2	4	0	36.5726	2.4550	−0.0161
36.7666	2.4424	4	9	1	2	36.7863	2.4412	−0.0197
37.2393	2.4125	4	3	4	0	37.2538	2.4116	−0.0145
37.3969	2.4027	5	12	0	0	37.4056	2.4022	−0.0087
37.6726	2.3858	6	10	2	1	37.6723	2.3858	0.0003
37.7120	2.3834	6	0	4	1	37.7266	2.3825	−0.0146
37.8696	2.3738	8	1	4	1	37.8601	2.3744	0.0095
37.8958	2.3722	8	8	2	2	37.8794	2.3732	0.0164
38.2635	2.3503	12	2	4	1	38.2583	2.3506	0.0052
39.1169	2.3009	4	6	1	3	39.1257	2.3004	−0.0088
39.7997	2.2630	4	4	4	1	39.8173	2.2621	−0.0176
40.1280	2.2453	4	12	1	1	40.1347	2.2449	−0.0067
40.7976	2.2099	5	7	1	3	40.7822	2.2107	0.0154
40.9027	2.2045	5	11	0	2	40.9134	2.2040	−0.0107
40.9421	2.2025	6	5	4	1	40.9539	2.2019	−0.0118
41.3228	2.1831	3	7	3	2	41.3245	2.1830	−0.0017
41.6905	2.1647	4	13	1	0	41.6944	2.1645	−0.0039
42.2682	2.1364	4	6	2	3	42.2757	2.1360	−0.0075
42.3601	2.1320	4	7	4	0	42.3674	2.1316	−0.0073
42.9641	2.1034	4	10	3	1	42.9647	2.1034	−0.0006
43.0823	2.0979	4	1	3	3	43.1007	2.0970	−0.0184
43.2136	2.0918	4	12	2	1	43.2239	2.0913	−0.0103
43.5812	2.0750	4	3	4	2	43.5759	2.0753	0.0053
43.8570	2.0626	5	7	4	1	43.8659	2.0622	−0.0089
44.4347	2.0371	5	0	0	4	44.4388	2.0369	−0.0041
44.6185	2.0292	5	9	1	3	44.6429	2.0281	−0.0244
44.7104	2.0252	5	13	2	0	44.6954	2.0258	0.0150
44.9205	2.0162	4	11	2	2	44.9354	2.0156	−0.0149
45.1437	2.0068	4	9	3	2	45.1476	2.0066	−0.0039
45.6033	1.9876	5	8	4	1	45.6097	1.9873	−0.0064
46.4568	1.9531	6	3	1	4	46.4335	1.9540	0.0233
46.4962	1.9515	5	3	5	0	46.4948	1.9515	0.0014
47.2315	1.9228	4	4	1	4	47.2189	1.9233	0.0126
47.3365	1.9188	6	2	5	1	47.3369	1.9188	−0.0004
47.5597	1.9103	5	12	2	2	47.5413	1.9110	0.0184
47.9011	1.8975	4	3	5	1	47.8908	1.8979	0.0103
48.1637	1.8877	5	7	4	2	48.1381	1.8887	0.0256
48.2294	1.8853	5	10	4	0	48.2405	1.8849	−0.0111
48.5576	1.8734	4	7	3	3	48.5775	1.8726	−0.0199
48.6758	1.8691	4	2	2	4	48.6628	1.8696	0.0130
49.4636	1.8411	4	6	5	0	49.4688	1.8410	−0.0052
49.6343	1.8352	4	5	5	1	49.6319	1.8353	0.0024

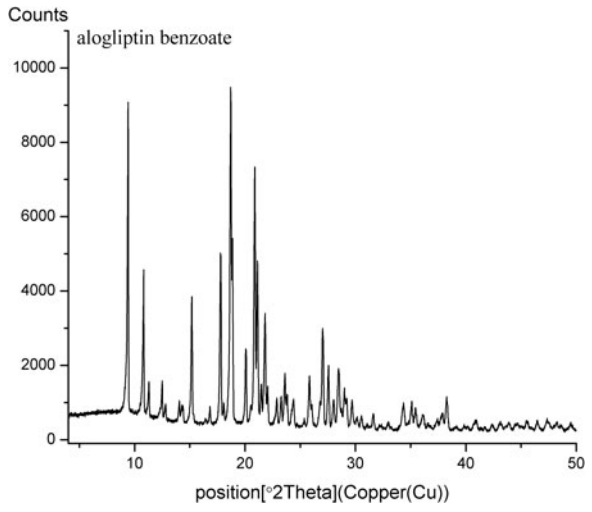


Figure 2. Powder X-ray diffraction pattern of alogliptin benzoate, using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715616000592.

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