X-ray powder diffraction data for olmesartan medoxomil, $C_{29}H_{30}N_6O_6$

Xiaochun Chen,[1] Peixiao Tang,[2] Shanshan Li,[2] Kailin Xu,[2] and Hui Li[2,a)]
[1]*Research Center of Analytical Instrumentation, Key laboratory of Bio-resource and Eco-environment, Ministry of Education, College of Life Science, Sichuan University, Chengdu 610064, China*
[2]*College of Chemical Engineering, Sichuan University, Chengdu, Sichuan 610065, China*

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X-ray powder diffraction data, unit-cell parameters, and space group for olmesartan medoxomil, C29H30N6O6, are reported [a = 12.3969(7) Å, b = 21.2667(3) Å, c = 10.9603(5) Å, $\alpha = \gamma = 90°$, $\beta = 101.38(9)°$, unit-cell volume V = 2832.72 Å3, Z = 4 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. © *2016 International Centre for Diffraction Data*. [doi:10.1017/S0885715615000925]

Key words: X-ray powder diffraction, olmesartan medoxomil

I. INTRODUCTION

Olmesartan medoxomil (OLM, Figure 1) [systematic name (5-methyl-2-oxo-2H-1,3-dioxol-4-yl)-methyl-4-(2-hydroxypropan-2-yl)-2-propyl-1-(-[2-(2H-1,2,3,4-tetrazol-5-yl)-phenyl]phenylmethyl)-1H-imidazole-5-carboxylate] is an ester prodrug of olmesartan, which shows excellent pharmacological actions and good tolerance in clinical trials performed on hypertensive patients (Makoto *et al*., 1995; Smith *et al*., 2005).

The unit-cell parameters determined by single-crystal methods were reported by Hiroaki *et al*. (1996) and Richard *et al*. (2013). The average values of cell parameters are a = 12.358(8), b = 21.232(9), c = 10.909(7) Å, $\alpha = \gamma = 90°$, β = 101.05(5)°, unit-cell volume V = 2809.48 Å^3, Z = 4, and space group $P2_1/c$. Until now, the detailed X-ray powder diffraction data have not been reported.

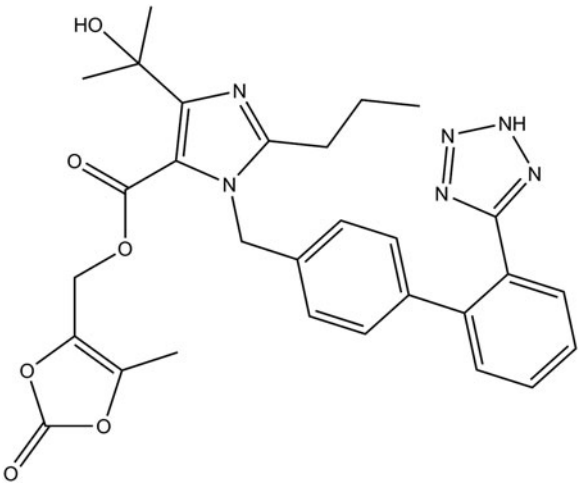


Figure 1. Structural formula of OLM.

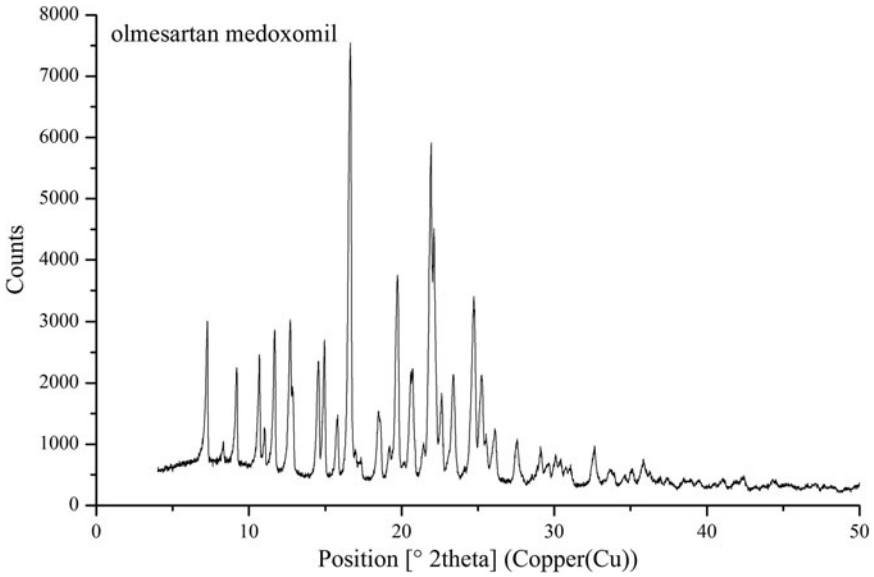


Figure 2. X-ray powder diffraction pattern of the OLM recrystallized in acetone–water solution, using Cu$K\alpha_1$ radiation (λ = 1.540 56 Å).

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

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TABLE I. Indexed X-ray powder diffraction data of OLM, $C_{29}H_{30}N_6O_6$. Only the peaks with I_{obs} of 1 or greater are reported [$a = 12.3969(7)$, $b = 21.2667(3)$, $c = 10.9603(5)$ Å, $\alpha = \gamma = 90°$, $\beta = 101.38(9)°$, unit-cell volume $V = 2832.72$ Å^3, $Z = 4$, and space group $P2_1/c$]. All measured lines were indexed and are consistent with the $P2_1/c$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	K	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
7.2697	12.1649	40	1	0	0	7.2682	12.1528	0.0015
8.3727	10.5575	10	1	1	0	8.3730	10.5515	−0.0003
9.2130	9.5922	29	0	1	1	9.2142	9.5901	−0.0012
10.6836	8.2693	33	1	1	−1	10.6883	8.2706	−0.0047
11.0513	7.9938	16	1	2	0	11.0473	8.0025	0.0040
11.6947	7.5534	38	0	2	1	11.6994	7.5579	−0.0047
12.7188	6.9444	40	1	1	1	12.7163	6.9557	0.0025
12.8895	6.8523	24	1	2	−1	12.8954	6.8595	−0.0059
14.5571	6.0666	31	2	0	0	14.5658	6.0764	−0.0087
14.6227	6.0393	17	1	2	1	14.6248	6.0521	−0.0021
14.9641	5.9014	33	0	3	1	14.9602	5.9171	0.0039
15.8176	5.5827	18	2	1	−1	15.8175	5.5983	0.0001
16.4872	5.3558	51	0	0	2	16.4875	5.3723	−0.0003
16.6448	5.3051	100	1	0	−2	16.6595	5.3172	−0.0147
16.6710	5.2967	89	0	4	0	16.6611	5.3167	0.0099
16.7892	5.2594	16	2	2	0	16.7912	5.2758	−0.0020
17.0124	5.1903	11	0	1	2	17.0092	5.2086	0.0032
17.1700	5.1427	9	1	1	−2	17.1761	5.1584	−0.0061
17.3538	5.0882	9	1	3	1	17.3539	5.1060	−0.0001
18.4830	4.7771	20	0	2	2	18.4887	4.7950	−0.0057
18.6012	4.7467	18	0	4	1	18.6055	4.7652	−0.0043
18.6406	4.7367	17	1	2	−2	18.6428	4.7557	−0.0022
19.2183	4.5942	13	2	3	0	19.2230	4.6135	−0.0047
19.3890	4.5538	12	1	4	−1	19.3880	4.5746	0.0010
19.7566	4.4690	48	2	3	−1	19.7555	4.4903	0.0011
19.9667	4.4219	8	2	2	1	19.9643	4.4438	0.0024
20.2031	4.3702	9	2	1	−2	20.2030	4.3919	0.0001
20.5970	4.2866	28	1	4	1	20.5912	4.3099	0.0058
20.7283	4.2594	30	0	3	2	20.7288	4.2816	−0.0005
20.8596	4.2326	14	1	3	−2	20.8669	4.2536	−0.0073
21.4767	4.1109	12	2	2	−2	21.4710	4.1353	0.0057
21.9231	4.0272	77	3	0	0	21.9234	4.0509	−0.0003
22.1201	3.9913	60	1	5	0	22.1246	4.0146	−0.0045
22.1989	3.9771	41	2	4	0	22.1990	4.0013	−0.0001
22.3302	3.9537	17	3	1	0	22.3226	3.9794	0.0076
22.4615	3.9306	16	0	5	1	22.4636	3.9548	−0.0021
22.6584	3.8964	22	2	4	−1	22.6646	3.9201	−0.0062
23.0655	3.8276	10	1	3	2	23.0663	3.8528	−0.0008
23.1311	3.8168	12	1	5	−1	23.1211	3.8437	0.0100
23.4069	3.7718	28	3	2	−1	23.4112	3.7968	−0.0043
23.4856	3.7591	20	3	2	0	23.4816	3.7855	0.0040
23.5250	3.7528	16	0	4	2	23.5231	3.7790	0.0019
23.6432	3.7341	9	1	4	−2	23.6457	3.7596	−0.0025
24.1422	3.6569	8	1	5	1	24.1471	3.6827	−0.0049
24.5229	3.6001	21	2	1	2	24.5279	3.6264	−0.0050
24.7199	3.5714	44	2	4	1	24.7123	3.5997	0.0076
24.7593	3.5657	43	3	0	−2	24.7556	3.5935	0.0037
24.8249	3.5563	33	1	1	−3	24.8252	3.5836	−0.0003
25.1138	3.5153	22	3	1	−2	25.1122	3.5433	0.0016
25.2320	3.4989	27	3	3	−1	25.2363	3.5262	−0.0043
25.2976	3.4898	27	3	3	0	25.3019	3.5172	−0.0043
25.5471	3.4557	16	2	5	0	25.5430	3.4845	0.0041
25.5996	3.4486	13	2	2	2	25.5927	3.4779	0.0069
25.8754	3.4119	10	1	2	−3	25.8786	3.4401	−0.0032
25.9541	3.4015	12	2	5	−1	25.9520	3.4305	0.0021
26.1511	3.3759	16	3	2	−2	26.1548	3.4044	−0.0037
26.2299	3.3657	12	0	2	3	26.2351	3.3941	−0.0052
27.0177	3.2676	6	1	6	−1	27.0243	3.2968	−0.0066
27.5429	3.2052	14	1	3	−3	27.5507	3.2350	−0.0078
27.6086	3.1976	13	3	4	−1	27.6017	3.2291	0.0069
27.6611	3.1915	11	3	4	0	27.6621	3.2222	−0.0010
27.8843	3.1660	7	0	3	3	27.8874	3.1967	−0.0031
28.0025	3.1526	5	3	3	1	27.9911	3.1851	0.0114
28.5802	3.0889	6	1	2	3	28.5759	3.1212	0.0043

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	K	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
28.8953	3.0552	9	2	5	−2	28.8957	3.0874	−0.0004
29.1579	3.0277	10	2	3	−3	29.1597	3.0600	−0.0018
29.4993	2.9926	9	2	4	2	29.4948	3.0260	0.0045
29.6831	2.9741	9	4	1	0	29.6789	3.0077	0.0042
29.9851	2.9441	9	3	4	−2	29.9892	2.9773	−0.0041
30.1164	2.9313	10	4	2	−1	30.1150	2.9651	0.0014
30.1427	2.9287	10	3	1	−3	30.1346	2.9632	0.0081
30.4184	2.9022	10	3	1	2	30.4123	2.9368	0.0061
30.4447	2.8997	9	3	5	0	30.4484	2.9334	−0.0037
30.7992	2.8663	8	4	0	−2	30.7916	2.9015	0.0076
31.0881	2.8396	7	4	1	−2	31.0840	2.8748	0.0041
31.2982	2.8206	5	3	2	2	31.2918	2.8562	0.0064
32.1516	2.7457	5	2	6	−2	32.1521	2.7817	−0.0005
32.4536	2.7201	9	3	3	−3	32.4503	2.7569	0.0033
32.6637	2.7026	12	0	5	3	32.6602	2.7396	0.0035
32.7162	2.6983	11	3	3	2	32.7103	2.7355	0.0059
32.9394	2.6800	6	1	1	−4	32.9416	2.7169	−0.0022
33.2677	2.6536	6	2	7	−1	33.2603	2.6915	0.0074
33.5960	2.6276	7	0	1	4	33.6019	2.6650	−0.0059
33.6879	2.6205	8	2	0	−4	33.6848	2.6586	0.0031
33.6879	2.6205	8	0	8	0	33.6880	2.6583	−0.0001
33.9505	2.6002	7	2	1	−4	33.9549	2.6381	−0.0044
34.3575	2.5694	5	3	4	−3	34.3618	2.6077	−0.0043
34.6070	2.5508	6	3	4	2	34.6091	2.5897	−0.0021
34.7383	2.5412	6	2	7	1	34.7316	2.5808	0.0067
35.0928	2.5155	8	1	3	−4	35.0916	2.5552	0.0012
35.1979	2.5080	6	4	4	−2	35.2092	2.5469	−0.0113
35.4079	2.4931	5	1	7	2	35.4031	2.5334	0.0048
35.5524	2.4830	6	3	6	−2	35.5468	2.5235	0.0056
35.7231	2.4711	8	2	4	3	35.7255	2.5113	−0.0024
35.8544	2.4621	10	1	1	4	35.8543	2.5025	0.0001
35.8806	2.4603	9	1	8	1	35.8879	2.5003	−0.0073
35.9069	2.4585	8	4	5	−1	35.9121	2.4986	−0.0052
36.0513	2.4486	8	2	3	−4	36.0517	2.4893	−0.0004
36.3008	2.4318	7	4	5	0	36.3085	2.4723	−0.0077
36.6159	2.4109	5	1	2	4	36.6173	2.4521	−0.0014
36.9704	2.3878	6	4	1	2	36.9672	2.4297	0.0032
37.4169	2.3593	6	1	6	3	37.4184	2.4014	−0.0015
37.4431	2.3576	6	5	0	−2	37.4487	2.3996	−0.0056
37.7189	2.3404	4	0	8	2	37.7255	2.3826	−0.0066
37.7977	2.3355	5	1	8	−2	37.8056	2.3777	−0.0079
38.5198	2.2917	6	2	8	1	38.5126	2.3357	0.0072
38.7824	2.2762	5	3	7	−2	38.7824	2.3201	0.0000
39.0188	2.2624	6	4	6	0	39.0154	2.3067	0.0034
39.4652	2.2368	5	2	1	4	39.4620	2.2817	0.0032
39.5965	2.2294	5	3	6	2	39.5951	2.2743	0.0014
39.9510	2.2096	4	2	5	−4	39.9590	2.2544	−0.0080
40.1348	2.1995	5	4	0	−4	40.1468	2.2443	−0.0120
40.1480	2.1987	4	3	4	−4	40.1551	2.2439	−0.0071
40.5156	2.1788	4	3	8	−1	40.5144	2.2248	0.0012
40.8439	2.1613	5	4	5	−3	40.8524	2.2072	−0.0085
41.0671	2.1495	6	4	2	−4	41.0707	2.1959	−0.0036
41.1984	2.1427	5	4	6	1	41.2065	2.1890	−0.0081
41.4085	2.1318	4	5	2	−3	41.4138	2.1785	−0.0053
41.6186	2.1210	5	1	5	4	41.6226	2.1681	−0.0040
41.8024	2.1117	5	1	9	−2	41.7989	2.1593	0.0035
41.8812	2.1077	5	2	8	2	41.8878	2.1550	−0.0066
42.0781	2.0979	5	1	8	−3	42.0734	2.1459	0.0047
42.3670	2.0836	6	3	7	−3	42.3621	2.1319	0.0049
42.4983	2.0771	5	3	5	3	42.5011	2.1253	−0.0028
42.5377	2.0752	5	5	3	−3	42.5378	2.1235	−0.0001
42.8134	2.0618	4	5	5	0	42.8174	2.1103	−0.0040
43.0760	2.0493	4	4	7	−2	43.0747	2.0983	0.0013
43.2598	2.0406	4	5	5	−2	43.2559	2.0899	0.0039
43.7063	2.0197	4	1	10	−1	43.7069	2.0694	−0.0006
43.9951	2.0065	4	6	1	−1	43.9977	2.0564	−0.0026

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	K	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
44.3497	1.9904	5	3	9	0	44.3447	2.0411	0.0050
44.5729	1.9804	5	5	6	−1	44.5753	2.0311	−0.0024
44.9930	1.9620	4	4	6	2	44.9971	2.0130	−0.0041
45.2688	1.9500	4	5	1	−4	45.2663	2.0017	0.0025
45.2950	1.9489	4	4	8	0	45.2909	2.0006	0.0041
45.5576	1.9376	4	6	2	0	45.5538	1.9897	0.0038
45.6102	1.9354	4	5	6	−2	45.6182	1.9870	−0.0080
45.9909	1.9194	4	5	5	−3	45.9848	1.9720	0.0061
46.2798	1.9074	4	4	8	−2	46.2824	1.9601	−0.0026
46.4505	1.9004	4	6	3	−2	46.4431	1.9537	0.0074
46.5949	1.8945	4	6	3	0	46.5975	1.9475	−0.0026
46.7000	1.8902	4	2	8	3	46.6954	1.9437	0.0046
46.9495	1.8802	4	3	4	−5	46.9565	1.9335	−0.0070
47.0545	1.8760	5	1	10	2	47.0565	1.9296	−0.0020
47.2383	1.8687	4	4	8	1	47.2446	1.9224	−0.0063
47.6979	1.8507	4	4	7	2	47.6985	1.9051	−0.0006
48.0524	1.8370	4	6	2	1	48.0545	1.8918	−0.0021
48.1181	1.8345	4	0	8	4	48.1182	1.8895	−0.0001
48.3807	1.8246	4	2	8	−4	48.3812	1.8798	−0.0005
48.4726	1.8211	4	4	9	−1	48.4725	1.8765	0.0001
49.1553	1.7958	4	4	8	−3	49.1512	1.8522	0.0041
49.7199	1.7754	4	4	9	−2	49.7219	1.8322	−0.0020
49.9563	1.7670	5	1	0	−6	49.9515	1.8243	0.0048

II. EXPERIMENTAL

A. Sample preparation

OLM (99.2% purity) was obtained from Wuhan Biocar Biology Technology Co., Ltd. The single crystal of OLM was obtained in acetone–water solution (V/V = 2:1) at room temperature. The crystal was then grinded into powder (T_{melt} = 173 °C) and sieved through a 200-mesh screen. The experimental density of the title compound is 1.338 g cm^{-3} at 25 °C. The elemental analysis showed that the content of C, N, and H was 15.06, 62.36, and 5.04%, respectively.

B. Diffraction data collection and reduction

An X'Pert PRO diffractometer (PANalytical Co., Ltd., The Netherlands) equipped with a PIXcel one-dimensional (1D) detector and Cu$K\alpha_1$ radiation (generator setting: 40 kV and 40 mA) was used to obtain the X-ray powder diffraction data. The diffractometer was operated in 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}. All data were collected at relative humidity of 60% and temperature of 25 °C.

Data evaluation was carried out using the software package Material Studio 4.2 (Accelrys Co., Ltd., USA). The diffractogram was indexed by discover tools (Xu *et al.*, 2014; Zhang *et al.*, 2014) and refined according to the Pawley methods (Engel *et al.*, 1999). After refinement, the final R_{wp} was converged at 5.59%.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results confirmed that OLM is monoclinic with space group $P2_1/c$ and unit-cell parameters: a = 12.3969(7), b = 21.2667(3) Å, c = 10.9603(5) Å, $\alpha = \gamma = 90°$, β = 101.38(9)°, unit-cell volume V = 2832.72 Å^3, and Z = 4 (Table I). Using Pawley refinement, the powder pattern of OLM was successfully indexed. These results are in good agreement with those single-crystal data of OLM.

SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit http://dx.doi.org/10.1017/S0885715615000925

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