X-ray powder diffraction data for drospirenone, $C_{24}H_{30}O_3$

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(Received 15 August 2015; accepted 27 September 2015)

X-ray powder diffraction data, unit-cell parameters, and space group for drospirenone, $C_{24}H_{30}O_3$, are reported [$a = 12.897(1)$ Å, $b = 12.618(1)$ Å, $c = 12.252(1)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1994.13$ Å^3, $Z = 4$, $\rho_{cal} = 1.229$ 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.
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Key words: X-ray powder diffraction, drospirenone

I. INTRODUCTION

The title compound drospirenone (Figure 1), systematic name 6,7,15,16-dimethylen-3-oxo-17-pregn-4-ene-2,17-carbolactone, is a novel progestogen with anti-mineralocorticoid and anti-androgenic characteristics (Muhn *et al*., 1995; Cagnacci *et al*., 2014). Drospirenone can counteract the estrogen-induced stimulation of the reninangiotensin–aldosterone system and blocks testosterone from binding to androgen receptors (Krattenmacher, 2000). Hence, it has the potential to reduce body weight, blood pressure, and low-density lipoprotein levels and to enhance high-density lipoprotein levels.

The single crystallographic data of drospirenone [$a = 12.236(2)$ Å, $b = 12.592(2)$ Å, $c = 12.879(2)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1984.25$ Å^3, $Z = 4$, $\rho_{cal} = 1.227$ g cm^{-3}, and space group $P2_12_12_1$] was obtained by Parisi *et al*. (2013). To date, the detailed X-ray powder diffraction (XRD) data for drospirenone have not been reported.

II. EXPERIMENTAL

A. Sample preparation

The title compound was purchased from Zhejiang Xianju Pharmaceutical Co., Ltd., China (high-performance liquid chromatography, HPLC ≥ 98%) and characterized by ultraviolet and Fourier transform infrared spectrometer. The melting point and measured density of drospirenone are 200–201 °C and 1.236 g cm^{-3}, respectively. Crystallization of drospirenone at room temperature was successful using ethyl acetate as solvent. Then, part of crystals was dried and ground into powder.

B. Diffraction data collection and reduction

XRD measurement was performed at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with a PIXcel one-dimensional 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 0.013 13°2θ and a counting time of 30 ms step^{-1}.

The software package Material Studio 4.2 (Accelrys Co., Ltd., USA) was used to process the data in the State Key Laboratory of Polymer Materials Engineering (Sichuan University, China). The XRD 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 X-Cell method (Neumann, 2003; Wu *et al*., 2014). The indexing results were then refined using Pawley, which involves assigning the Miller indices (*h, k, l*) to each observed peak in the experimental powder XRD pattern (Wang *et al*., 2013).

C. Single-crystal XRD

XRD data for drospirenone 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) refinement package using least-squares minimization.

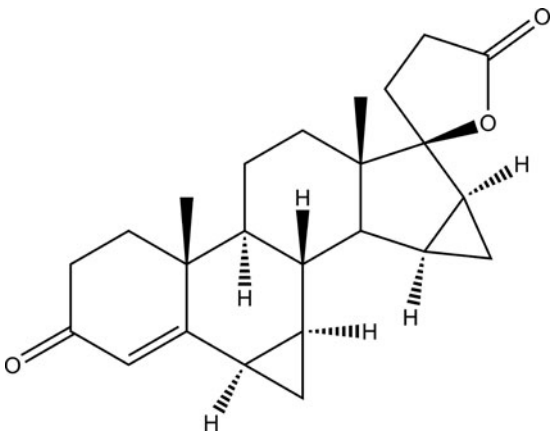


Figure 1. Structural formula of drospirenone.

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https://doi.org/10.1017/S0885715615000822 Published online by Cambridge University Press

TABLE I. Indexed XRD data for drospirenone, $C_{24}H_{30}O_3$. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
9.8122	9.0067	24	1	1	0	9.7993	9.0186	0.0130
9.9304	8.8997	21	1	0	1	9.9479	8.8842	−0.0174
12.1494	7.2788	83	1	1	1	12.1745	7.2639	−0.0251
13.6988	6.4588	5	2	0	0	13.7210	6.4484	−0.0222
14.0533	6.2967	4	0	2	0	14.0278	6.3081	0.0255
14.4472	6.1259	12	0	0	2	14.4423	6.1279	0.0049
15.4320	5.7371	24	2	1	0	15.4191	5.7419	0.0129
15.4977	5.7129	22	2	0	1	15.5147	5.7067	−0.0170
15.6290	5.6652	10	1	2	0	15.6253	5.6666	0.0037
15.7734	5.6137	7	0	2	1	15.7874	5.6087	−0.0140
15.9966	5.5359	15	1	0	2	15.9994	5.5349	−0.0028
16.0229	5.5268	14	0	1	2	16.0660	5.5121	−0.0431
17.0339	5.2010	40	2	1	1	17.0388	5.1995	−0.0049
17.2177	5.1459	100	1	2	1	17.2261	5.1434	−0.0084
17.4672	5.0730	12	1	1	2	17.4824	5.0686	−0.0152
19.9751	4.4413	10	2	0	2	19.9717	4.4421	0.0034
20.1720	4.3984	22	0	2	2	20.1860	4.3954	−0.0140
20.9467	4.2375	5	2	2	1	20.9744	4.2319	−0.0277
21.1962	4.1882	9	2	1	2	21.1870	4.1900	0.0092
21.3406	4.1601	4	1	2	2	21.3392	4.1604	0.0015
21.8921	4.0566	3	3	0	1	21.8918	4.0566	0.0004
22.2204	3.9974	2	1	3	0	22.2158	3.9982	0.0046
22.8769	3.8841	7	0	1	3	22.8621	3.8866	0.0148
23.0213	3.8601	9	3	1	1	23.0103	3.8619	0.0110
23.3890	3.8002	18	1	3	1	23.3839	3.8010	0.0050
23.9010	3.7200	4	1	1	3	23.8924	3.7213	0.0087
24.4919	3.6315	3	2	2	2	24.4892	3.6319	0.0027
25.2535	3.5237	8	2	3	0	25.2624	3.5225	−0.0089
25.6736	3.4670	4	0	3	2	25.6705	3.4674	0.0031
25.7655	3.4548	4	2	0	3	25.7945	3.4510	−0.0290
26.0938	3.4121	5	3	2	1	26.0947	3.4120	−0.0009
26.3170	3.3837	7	2	3	1	26.3031	3.3854	0.0140
26.5927	3.3492	16	1	3	2	26.5986	3.3485	−0.0058
26.8816	3.3139	4	1	2	3	26.8817	3.3139	−0.0001
28.2866	3.1524	3	0	4	0	28.2716	3.1540	0.0150
28.5229	3.1268	3	4	1	0	28.5508	3.1238	−0.0279
29.2057	3.0552	4	0	4	1	29.2129	3.0545	−0.0072
29.4683	3.0286	7	2	2	3	29.4787	3.0276	−0.0105
30.0460	2.9717	4	1	4	1	30.0398	2.9723	0.0062
30.1248	2.9641	3	3	0	3	30.1529	2.9614	−0.0281
30.4793	2.9304	3	0	3	3	30.4809	2.9303	−0.0015
30.5844	2.9206	3	3	3	1	30.5945	2.9196	−0.0102
31.1358	2.8701	3	4	2	0	31.1266	2.8709	0.0092
31.2934	2.8560	3	1	3	3	31.2773	2.8574	0.0161
32.1337	2.7832	3	4	1	2	32.1355	2.7831	−0.0017
32.4489	2.7569	2	0	2	4	32.4591	2.7561	−0.0103
32.6458	2.7407	2	1	4	2	32.6505	2.7403	−0.0047
33.1316	2.7016	4	2	1	4	33.1121	2.7032	0.0195
33.3942	2.6810	3	3	2	3	33.3981	2.6807	−0.0039
34.8254	2.5740	2	2	4	2	34.8577	2.5717	−0.0323
35.2719	2.5424	2	3	4	0	35.2638	2.5430	0.0081
35.5082	2.5261	2	5	1	0	35.4933	2.5271	0.0150
35.8365	2.5037	3	4	3	1	35.8210	2.5047	0.0155
36.0334	2.4904	3	3	4	1	36.0404	2.4900	−0.0070
36.2960	2.4730	3	0	5	1	36.3208	2.4714	−0.0248
36.7031	2.4465	2	3	1	4	36.6851	2.4477	0.0180
36.9263	2.4322	3	1	3	4	36.9307	2.4320	−0.0044
37.0970	2.4214	2	3	3	3	37.0995	2.4213	−0.0026
37.3465	2.4058	2	0	1	5	37.3411	2.4062	0.0054
38.0817	2.3611	2	4	3	2	38.0801	2.3612	0.0016
38.3050	2.3478	3	3	4	2	38.2884	2.3488	0.0165
38.5282	2.3347	3	5	1	2	38.5023	2.3362	0.0259
38.8958	2.3135	2	2	3	4	38.9259	2.3118	−0.0301
39.2635	2.2927	2	2	0	5	39.2895	2.2912	−0.0260
39.9331	2.2558	2	4	4	0	39.9539	2.2546	−0.0208
40.0776	2.2480	2	1	2	5	40.0451	2.2497	0.0324
40.4715	2.2270	2	5	2	2	40.5167	2.2246	−0.0452
41.1148	2.1936	3	2	5	2	41.1077	2.1940	0.0071
41.2593	2.1863	2	4	1	4	41.2369	2.1874	0.0223
41.3643	2.1810	2	5	0	3	41.3631	2.1810	0.0013
41.4825	2.1750	3	3	5	0	41.4613	2.1761	0.0212
41.6401	2.1672	4	1	4	4	41.6538	2.1665	−0.0137
41.8370	2.1574	3	3	4	3	41.8065	2.1589	0.0305
42.0734	2.1458	4	3	3	4	42.0734	2.1458	0.0000
42.6773	2.1169	3	4	4	2	42.6961	2.1160	−0.0188
43.1763	2.0935	2	4	2	4	43.1451	2.0950	0.0311
43.2945	2.0881	2	6	1	1	43.2973	2.0880	−0.0028
43.4520	2.0809	2	2	4	4	43.4670	2.0802	−0.0149
43.6096	2.0737	2	0	6	1	43.6390	2.0724	−0.0294
43.8722	2.0619	2	5	2	3	43.8864	2.0613	−0.0142
44.1479	2.0497	2	3	5	2	44.1267	2.0506	0.0213
44.5025	2.0342	2	6	2	0	44.4928	2.0346	0.0097
44.9620	2.0145	2	0	1	6	44.9165	2.0164	0.0455
45.0277	2.0117	2	2	3	5	45.0202	2.0120	0.0075
46.5114	1.9509	2	4	0	5	46.5009	1.9513	0.0105
46.6427	1.9457	2	2	0	6	46.6026	1.9473	0.0400
47.0235	1.9308	2	6	2	2	47.0204	1.9310	0.0031
48.8223	1.8638	2	4	2	5	48.8128	1.8642	0.0095

III. RESULTS

Pawley refinement results confirmed that drospirenone is orthorhombic with space group $P2_12_12_1$ and unit-cell parameters: $a = 12.897(1)$ Å, $b = 12.618(1)$ Å, $c = 12.252(1)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1994.13$ Å^3, $Z = 4$, and $\rho_{cal} = 1.229$ 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 single crystallographic data of drospirenone [$a = 12.2379(5)$ Å, $b = 12.6069(8)$ Å, $c = 12.8921(8)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1989.01(19)$ Å^3, $Z = 4$, and $\rho_{cal} = 1.224$ g cm^{-3}] and the experimental data are listed in Table SI.

The comparison of XRD pattern with the simulated patterns of single-crystal structure and Parisi's is shown in Figure 2. Results showed that both single-crystal and powder diffraction methods can get similar structure data.

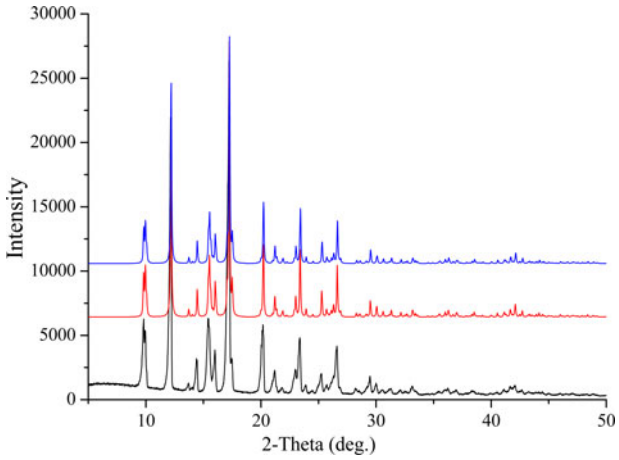


Figure 2. (Colour online) XRD pattern of drospirenone using Cu$K\alpha$ radiation (black line) and the simulated pattern of single-crystal structure (red line) and Parisi's (blue line).

https://doi.org/10.1017/S0885715615000822 Published online by Cambridge University Press

SUPPLEMENTARY MATERIALS AND METHODS

The supplementary material for this article can be found at http://dx.doi.org/10.1017/S0885715615000822

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