X-ray powder diffraction data for 17-hydroxy-16-methyl-9,11-epoxypregna-1,4,6-triene-3,20-dione, $C_{22}H_{26}O_4$

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X-ray powder diffraction data, unit-cell parameters, and space group for 17- hydroxy-16-methyl-9,11-epoxypregna-1,4,6-triene-3,20-dione, $C_{22}H_{26}O_4$, are reported [$a = 18.435(1)$ Å, $b = 12.269(3)$ Å, $c = 8.251(5)$ Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$, unit-cell volume $V = 1866.43$ Å^3, $Z = 4$, $\rho_{cal} = 1.261$ 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. © *2019 International Centre for Diffraction Data*. [doi:10.1017/S088571561900054X]

Key words: pharmaceutical intermediate, anticoagulant, dexamethasone

1. INTRODUCTION

Dexamethasone is a glucocorticoid commonly used for treatment of many inflammatory and autoimmune conditions, such as rheumatoid arthritis and bronchospasm (Hossain, *et al*., 2018; Formica, *et al*., 2019). The title compound (Figure 1) is an intermediate in the synthesis of dexamethasone.

In order to better study the effect of the minor difference in the structure on the steroids binding to human serum albumin, Lin *et al*. (2017) analyzed the single-crystal structures of some novel steroids. The single crystallographic data of the title compound [$a = 8.26215(13)$ Å, $b = 12.27870(19)$ Å, $c = 18.4797(3)$ Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$, unit-cell volume $V = 1874.73(5)$ Å^3, $Z = 4$, $\rho_{cal} = 1.256$ g cm^{-3} and space group $P2_12_12_1$] were deposited with the Cambridge Crystallographic Data Center (CCDC) with a supplementary publication number of CCDC-1471422. To date, the detailed X-ray powder diffraction (PXRD) data for the title compound have not been reported.

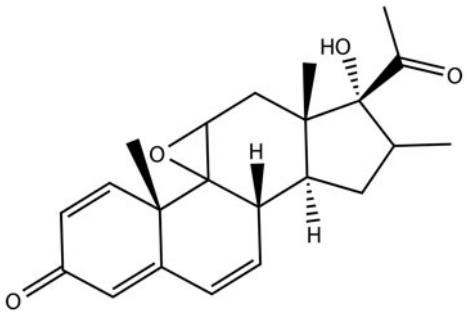

Figure 1. Molecular diagram of the title compound.

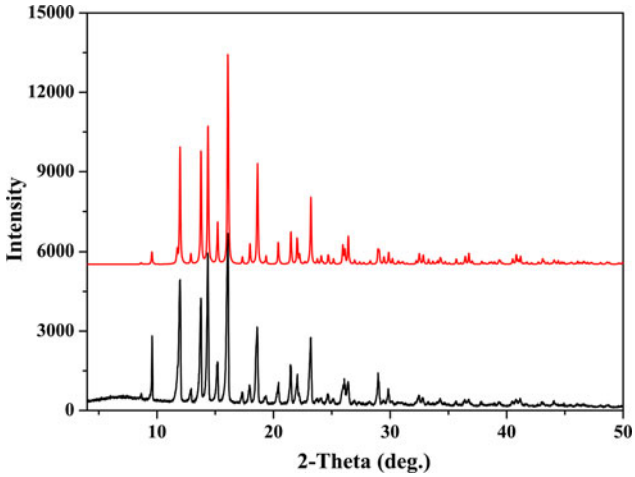

Figure 2. (colour online) X-ray powder diffraction pattern of the title compound using Cu$K\alpha$ radiation (black line) and the simulated pattern of the crystal structure (red line).

II. EXPERIMENTAL

A. Sample preparation

The sample was purchased from J&K Scientific (Beijing, People's Republic of China). The melting point and measured density of the title compound are 244–245 °C and 1.253 g cm^{-3}, respectively. Crystallization of the title compound at room temperature was successful using methanol as solvent. The crystals have a prismatic and transparent crystal structure. Then, part of crystals was dried, smashed, and screened through 75 μm mesh size.

B. Diffraction data collection and reduction

PXRD measurement was performed at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with a PIXcel 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 0.01313° 2θ and a counting time of 30 ms step^{-1} (Figure 2).

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TABLE I. Indexed X-ray powder diffraction data for the title compound. 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)	$\Delta 2\theta$
8.6516	10.2122	10	1	1	0	8.6500	10.2140	0.0015
9.5838	9.2208	42	2	0	0	9.5872	9.2175	−0.0034
11.7503	7.5251	24	1	0	1	11.7401	7.5317	0.0102
11.9735	7.3853	74	2	1	0	11.9993	7.3695	−0.0257
12.9320	6.8400	12	0	1	1	12.9183	6.8472	0.0137
13.7724	6.4245	64	1	1	1	13.7847	6.4188	−0.0123
14.3632	6.1615	88	2	0	1	14.3948	6.1481	−0.0316
14.4026	6.1447	63	3	0	0	14.4020	6.1450	0.0006
15.1904	5.8278	28	1	2	0	15.2086	5.8209	−0.0182
16.0964	5.5018	100	2	1	1	16.1116	5.4966	−0.0152
17.3438	5.1088	10	2	2	0	17.3498	5.1070	−0.0060
18.0003	4.9239	12	0	2	1	18.0028	4.9232	−0.0025
18.6174	4.7620	44	1	2	1	18.6393	4.7565	−0.0218
19.2477	4.6075	7	4	0	0	19.2424	4.6088	0.0053
19.3921	4.5735	7	3	1	1	19.3928	4.5734	−0.0007
20.4294	4.3436	16	3	2	0	20.4392	4.3415	−0.0098
20.5607	4.3161	5	4	1	0	20.5690	4.3144	−0.0083
21.4930	4.1310	25	0	0	2	21.5199	4.1259	−0.0270
22.0444	4.0289	20	4	0	1	22.0731	4.0237	−0.0287
22.2414	3.9936	8	1	3	0	22.2466	3.9927	−0.0052
23.1474	3.8393	34	3	2	1	23.1300	3.8422	0.0174
23.2393	3.8244	25	4	1	1	23.2455	3.8234	−0.0062
23.7776	3.7390	7	2	3	0	23.7818	3.7383	−0.0042
24.1190	3.6868	7	5	0	0	24.1178	3.6870	0.0012
24.6968	3.6019	9	2	1	2	24.7093	3.6001	−0.0126
24.7624	3.5925	6	1	3	1	24.7511	3.5941	0.0113
25.2088	3.5299	5	5	1	0	25.2004	3.5310	0.0085
25.9967	3.4246	14	3	0	2	25.9908	3.4254	0.0059
26.1411	3.4060	15	3	3	0	26.1518	3.4047	−0.0108
26.4562	3.3662	13	5	0	1	26.4556	3.3663	0.0006
27.0208	3.2971	5	3	1	2	27.0031	3.2992	0.0177
27.4541	3.2461	4	5	1	1	27.4521	3.2463	0.0020
28.3470	3.1458	4	3	3	1	28.3333	3.1473	0.0137
29.0560	3.0706	16	6	0	0	29.0380	3.0725	0.0180
29.0823	3.0679	9	0	4	0	29.0879	3.0673	−0.0056
29.4893	3.0265	6	1	4	0	29.4968	3.0257	−0.0075
29.8438	2.9914	12	3	2	2	29.8498	2.9908	−0.0060
29.9357	2.9824	8	4	1	2	29.9410	2.9819	−0.0053
30.2509	2.9520	4	5	2	1	30.2599	2.9512	−0.0091
30.7630	2.9040	4	0	3	2	30.7569	2.9046	0.0060
31.0649	2.8765	5	6	0	1	31.0330	2.8794	0.0320
32.2861	2.7704	5	2	3	2	32.2876	2.7703	−0.0015
32.5093	2.7519	8	0	0	3	32.5255	2.7506	−0.0162
32.6800	2.7379	5	5	3	0	32.6734	2.7385	0.0066
32.9032	2.7199	5	1	0	3	32.8957	2.7205	0.0075
33.7304	2.6550	4	1	1	3	33.7182	2.6560	0.0122
34.1112	2.6263	4	3	3	2	34.1144	2.6260	−0.0033
34.3869	2.6058	5	6	2	1	34.3771	2.6065	0.0098
34.7677	2.5782	4	2	1	3	34.7846	2.5769	−0.0169
35.6999	2.5129	5	3	0	3	35.7350	2.5106	−0.0351
36.4221	2.4648	6	6	0	2	36.4295	2.4643	−0.0074
36.5009	2.4596	5	3	1	3	36.5012	2.4596	−0.0003
36.7766	2.4418	6	1	4	2	36.8056	2.4399	−0.0290
37.9190	2.3708	4	2	5	0	37.9113	2.3713	0.0076
38.7987	2.3191	4	4	1	3	38.7941	2.3193	0.0045
39.0613	2.3041	3	8	0	0	39.0560	2.3044	0.0052
39.4158	2.2842	5	3	4	2	39.3995	2.2851	0.0163
40.5713	2.2217	5	7	0	2	40.6067	2.2199	−0.0354
40.9127	2.2040	5	5	0	3	40.8995	2.2047	0.0132
41.0702	2.1959	5	3	5	1	41.0553	2.1967	0.0150
41.2803	2.1852	5	7	1	2	41.2958	2.1844	−0.0155
43.1448	2.0950	4	1	5	2	43.1364	2.0954	0.0084
44.1690	2.0488	4	6	0	3	44.1556	2.0494	0.0134
44.1690	2.0488	4M	9	0	0	44.1786	2.0483	−0.0096
44.7992	2.0214	3	1	1	4	44.7826	2.0221	0.0167
45.0356	2.0113	3	8	0	2	45.0232	2.0119	0.0123
46.2436	1.9616	3	9	1	1	46.2224	1.9624	0.0211

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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 XRD pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results were obtained by DICVOL91 method (Boultif and Louër, 1991). The indexing results were then refined using Pawley ($R_{wp} = 9.63\%$) (Pawley, 1981), which involves assigning the Miller indices (*h, k, l*) to each observed peak in the experimental PXRD pattern.

III. RESULTS

Pawley refinement results confirmed that the title compound is orthorhombic with space group $P2_12_12_1$ and unit-cell parameters: $a = 18.435(1)$ Å, $b = 12.269(3)$ Å, $c = 8.251(5)$ Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$, unit-cell volume $V = 1866.43$ Å^3, $Z = 4$, $\rho_{cal} = 1.261$ 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 0.07% and 0.44%. The comparison of PXRD pattern (Supplementary Material) with the simulated pattern is shown in Figure 2.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S088571561900054X.

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