X-ray powder diffraction data for estra-4,9-diene-3,17-dione, $C_{18}H_{22}O_2$

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X-ray powder diffraction data for estra-4,9-diene-3,17-dione, $C_{18}H_{22}O_2$, are reported [$a = 9.236(7)$ Å, $b = 10.294(4)$ Å, $c = 15.471(1)$ Å, unit cell volume $V = 1471.11$ Å^3, $Z = 4$, 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. The single-crystallographic data of the compound are also reported [$a = 9.2392(7)$ Å, $b = 10.2793(5)$ Å, $c = 15.4822(7)$ Å, unit cell volume $V = 1470.37(15)$ Å^3, $Z = 4$, and space group $P2_12_12_1$]. Both single-crystal and powder diffraction methods can get the similar structure data. © *2020 International Centre for Diffraction Data.* [doi:10.1017/S0885715620000512]

Key words: steroid, pharmaceutical intermediate, X-ray powder diffraction, crystal structure

I. INTRODUCTION

Steroids display a variety of biological functions in the human organism, such as decreasing inflammatory and immune responses (Zeelen, 1997). Estra-4,9-diene-3,17-dione (Figure 1), a designer steroid, is designed based on the structure of trenbolone (Clarke *et al.*, 2010). This compound shows strong metabolic activation to HuH7 cells (Cooper *et al.*, 2017). The major metabolite of this compound is considered to be an isomer of 17-hydroxy-estra-4,9-dien-3-one. Hydroxylation and reduction followed by hydroxylation are the metabolic pathways (Scarth *et al.*, 2010). So far, the crystal structure of estra-4,9-diene-3,17-dione has not been reported.

II. EXPERIMENTAL

A. Sample preparation

Estra-4,9-diene-3,17-dione (Figure 1) was purchased from J&K Scientific (Beijing, People's Republic of China). The melting point and measured density of estra-4,9-diene-3,17-dione are 145–146 °C and 1.197 g cm^{-3}, respectively. Crystallization of estra-4,9-diene-3,17-dione at room temperature was successful using methanol as a solvent. The crystals are transparent and have a prismatic structure. A portion of the crystals were dried, smashed, screened through 75 μm mesh size, and mounted on a flat zero background plate.

B. Diffraction data collection and reduction

The X-ray powder diffraction measurement was performed at 298 K 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 sample was mounted on a flat zero background plate. 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}.

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 pretreated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results were obtained by the X-cell method (Neumann, 2003). The indexing results were then refined using Pawley ($R_{wp} = 10.36\%$) (Pawley, 1981), which involves assigning the Miller indices (hkl) to each observed peak in the experimental PXRD pattern.

C. Single-crystal X-ray diffraction

X-ray diffraction data for estra-4,9-diene-3,17-dione were collected on a New Gemini, Dual, Cu at zero, EosS2 diffractometer. The crystal was kept at 293.15 K during data collection. The structure was solved with Olex2 (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015), using charge flipping, and refined with the SHELXL (Sheldrick, 2015) refinement package using least-squares minimization.

III. RESULTS

Pawley refinement results confirmed that estra-4,9-diene-3,17-dione is orthorhombic with the space group $P2_12_12_1$ and unit cell parameters: $a = 9.236(7)$ Å, $b = 10.294(4)$ Å, $c = 15.471(1)$ Å, unit cell volume $V = 1471.11$

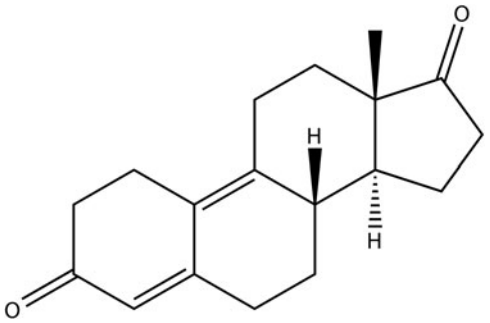

Figure 1. Molecular diagram of estra-4,9-diene-3,17-dione.

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TABLE I. X-ray powder diffraction data for estra-4,9-diene-3,17-dione, $C_{18}H_{22}O_2$. The *d*-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	*l*	*k*	*h*	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
10.3060	8.5763	15	0	1	1	10.3130	8.5704	−0.0070
11.4352	7.7318	28	0	0	2	11.4296	7.7355	0.0056
12.9058	6.8539	8	1	1	0	12.8659	6.8750	0.0399
14.0875	6.2815	20	1	1	1	14.0849	6.2826	0.0026
14.3107	6.1840	45	0	1	2	14.3104	6.1842	0.0003
17.2125	5.1474	39	0	2	0	17.2135	5.1471	−0.0010
18.1579	4.8815	6	0	2	1	18.1488	4.8839	0.0091
19.2083	4.6169	7	2	0	0	19.2016	4.6185	0.0067
19.2346	4.6106	7	0	1	3	19.2338	4.6108	0.0008
19.7073	4.5011	22	1	0	3	19.6998	4.5028	0.0075
19.7073	4.5011	22M	1	2	0	19.7289	4.4962	−0.0216
20.0486	4.4252	100	2	0	1	20.0474	4.4255	0.0013
20.5739	4.3134	10	1	2	1	20.5538	4.3176	0.0200
20.7183	4.2837	5	0	2	2	20.7108	4.2852	0.0075
21.0465	4.2176	13	2	1	0	21.0656	4.2138	−0.0190
21.8081	4.0720	55	2	1	1	21.8422	4.0657	−0.0341
22.3596	3.9728	27	2	0	2	22.4015	3.9655	−0.0419
22.8717	3.8850	6	1	2	2	22.8581	3.8873	0.0135
22.9636	3.8697	5	0	0	4	22.9750	3.8678	−0.0114
24.0271	3.7007	7	2	1	2	24.0291	3.7004	−0.0020
24.4210	3.6419	6	0	2	3	24.4132	3.6431	0.0079
24.5392	3.6246	10	0	1	4	24.5667	3.6206	−0.0275
24.9331	3.5683	6	1	0	4	24.9376	3.5676	−0.0045
25.8391	3.4452	5	2	0	3	25.8751	3.4405	−0.0360
26.2724	3.3893	8	1	2	3	26.2749	3.3890	−0.0025
26.4168	3.3711	5	1	1	4	26.4183	3.3709	−0.0015
26.5875	3.3499	4	0	3	1	26.5862	3.3500	0.0013
27.3097	3.2629	4	2	1	3	27.3085	3.2630	0.0012
28.2945	3.1515	4	1	3	1	28.3150	3.1493	−0.0205
28.4126	3.1387	4	0	3	2	28.4313	3.1367	−0.0187
28.8722	3.0898	4	0	2	4	28.8502	3.0921	0.0220
29.5418	3.0212	9	3	0	1	29.5566	3.0198	−0.0148
30.0670	2.9696	4	1	3	2	30.0624	2.9701	0.0046
30.1196	2.9646	5	0	1	5	30.1335	2.9632	−0.0140
30.3034	2.9470	4	3	1	0	30.2735	2.9499	0.0299
30.4741	2.9309	4	1	2	4	30.4608	2.9322	0.0133
31.2356	2.8612	5	3	0	2	31.2408	2.8607	−0.0051
31.3538	2.8506	5	2	1	4	31.3676	2.8494	−0.0138
32.4305	2.7584	6	3	1	2	32.4567	2.7563	−0.0262
33.8617	2.6450	5	3	2	0	33.8976	2.6423	−0.0359
34.5445	2.5943	5	2	3	2	34.5374	2.5948	0.0070
34.7414	2.5800	7	0	0	6	34.7628	2.5785	−0.0213
34.9121	2.5678	4	0	3	4	34.9257	2.5669	−0.0136
35.0172	2.5604	4	3	1	3	35.0143	2.5606	0.0029
35.8706	2.5014	4	0	1	6	35.8726	2.5012	−0.0019
35.9625	2.4952	4	2	1	5	35.9797	2.4940	−0.0172
36.9736	2.4292	3	2	3	3	36.9684	2.4296	0.0051
37.2231	2.4135	3	1	1	6	37.2110	2.4143	0.0121
38.0765	2.3614	4	1	4	2	38.0851	2.3609	−0.0086
38.2210	2.3528	3	3	2	3	38.2409	2.3516	−0.0199
38.9825	2.3086	3	4	0	0	38.9705	2.3092	0.0120
39.1795	2.2974	3	0	3	5	39.1700	2.2979	0.0094
39.4421	2.2827	3	4	0	1	39.4200	2.2839	0.0221
39.7047	2.2682	3	3	3	1	39.7279	2.2669	−0.0232
40.0198	2.2511	6	4	1	0	39.9797	2.2532	0.0401
40.0198	2.2511	6M	2	0	6	40.0140	2.2514	0.0058
40.0723	2.2483	4	2	4	0	40.0750	2.2481	−0.0027
40.3481	2.2335	3	1	4	3	40.3320	2.2344	0.0160
40.4137	2.2300	4	1	3	5	40.4151	2.2300	−0.0014
40.5319	2.2238	3	2	4	1	40.5143	2.2247	0.0176
41.6873	2.1648	4	4	1	2	41.7170	2.1633	−0.0297
41.8055	2.1590	4	2	4	2	41.8090	2.1588	−0.0035
42.0025	2.1493	3	1	0	7	41.9986	2.1495	0.0038
42.1206	2.1435	4	0	4	4	42.1398	2.1426	−0.0191
42.2913	2.1353	3	3	1	5	42.2955	2.1351	−0.0041
42.8691	2.1078	3	4	0	3	42.8742	2.1076	−0.0051

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	l	k	h	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
43.3418	2.0859	3	1	4	4	43.3143	2.0872	0.0275
43.9720	2.0575	4	2	3	5	43.9750	2.0573	−0.0030
45.5083	1.9915	4	1	5	1	45.4776	1.9928	0.0307
45.8103	1.9791	2	0	4	5	45.8220	1.9786	−0.0117
45.9809	1.9722	3	3	3	4	45.9950	1.9716	−0.0140
46.9132	1.9351	4	0	0	8	46.9448	1.9339	−0.0316
47.5303	1.9114	3	0	5	3	47.5121	1.9121	0.0182
47.7929	1.9015	2	4	3	1	47.7992	1.9013	−0.0062
48.9615	1.8588	2	2	2	7	48.9555	1.8591	0.0060

Å^3, $Z = 4$, $\rho_{cal} = 1.221$ 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 single-crystal experiment was carried out at the temperature of 293.15 K and the structure solution was obtained [$a = 9.2392(7)$ Å, $b = 10.2793(5)$ Å, $c = 15.4822(7)$ Å, unit cell volume $V = 1470.37(15)$ Å^3, $Z = 4$, $\rho_{cal} = 1.221$ g cm^{-3}, and space group $P2_12_12_1$]. The detailed single-crystal data of estra-4,9-diene-3,17-dione and the experimental data are listed in Table II. The figures were drawn with ORTEP-3 (Oak Ridge Thermal Ellipsoid Plot) and Mercury (Figure 2). Estra-4,9-diene-3,17-dione contains three chiral centers and is arranged in a head-to-tail fashion. The compound is arranged without intramolecular and intermolecular H-bonding, but it has two strong acceptors, namely, C1=O1 and C9=O2.

The comparison of PXRD pattern (Deposited Data) with the simulated pattern is shown in Figure 3. Results showed that both single-crystal and powder diffraction methods can get the similar structure data and the deviations of the unit cell parameters and unit cell volume were between 0.03% and 0.15%.

TABLE II. Crystal and experimental data of estra-4,9-diene-3,17-dione.

Empirical formula	$C_{18}H_{22}O_2$
Formula weight	270.35
Temperature (K)	293.15
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a (Å)	9.2392(7)
b (Å)	10.2793(5)
c (Å)	15.4822(7)
α (°)	90
β (°)	90
γ (°)	90
Volume (Å^3)	1470.37(15)
Z	4
ρ_{calc}(g cm^{-3})	1.221
μ (mm^{-1})	0.078
F(000)	584.0
Radiation	MoKα ($\lambda = 0.71073$)
2θ range for data collection (°)	5.928–52.742
Index ranges	$-5 \le h \le 11$, $-12 \le k \le 12$, $-18 \le l \le 19$
Reflections collected	4449
Independent reflections	2754 [$R_{int} = 0.0129$, $R_{sigma} = 0.0315$]
Data/restraints/parameters	2754/0/1825
Goodness of fit on F^2	1.058
Final R indexes [$I \ge 2\sigma(I)$]	$R_1 = 0.0585$, $wR_2 = 0.1371$
Final R indexes (all data)	$R_1 = 0.0751$, $wR_2 = 0.1499$
Largest diffraction peak/hole (e Å^{-3})	0.27/−0.19

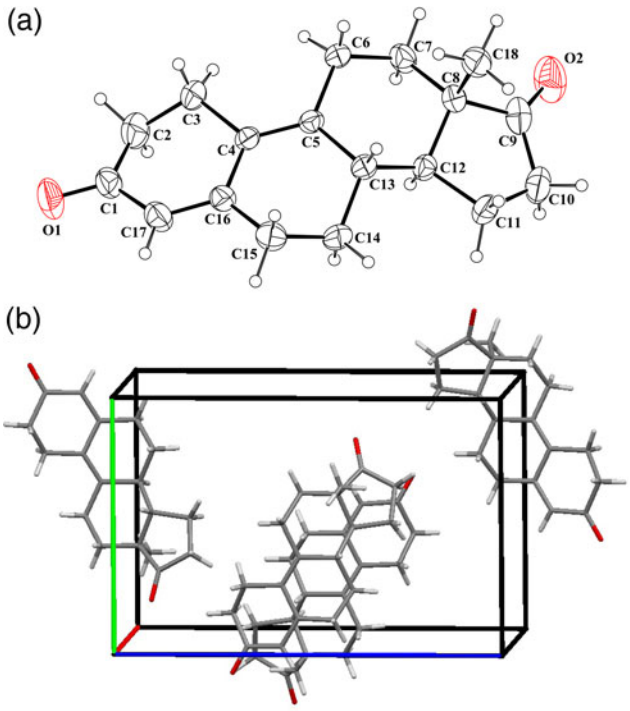

Figure 2. (a) ORTEP drawing one of the two independent molecules in the asymmetric unit of estra-4,9-diene-3,17-dione with the labeling of non-H atoms. (b) Crystal packing of estra-4,9-diene-3,17-dione.

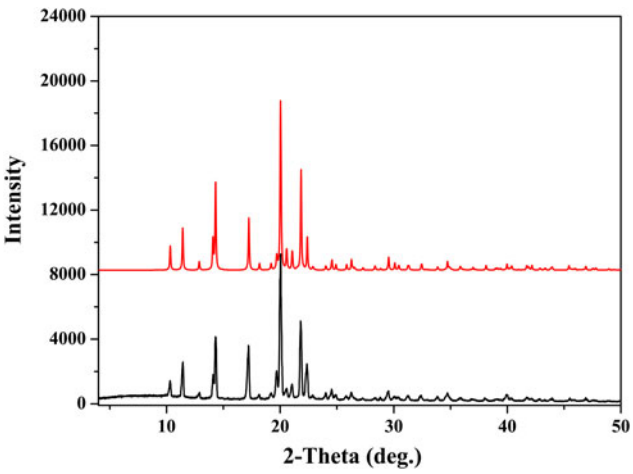

Figure 3. X-ray powder diffraction pattern of estra-4,9-diene-3,17-dione using CuKα radiation (black line) and the simulated pattern of the crystal structure (red line).

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IV. DEPOSITED DATA

CIF and/or RAW data files were deposited with ICDD. You may request this data from ICDD at info@icdd.com.

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