X-ray powder diffraction data for norandrostenedione

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X-ray powder diffraction data, unit-cell parameters, and space group for norandrostenedione, $C_{18}H_{24}O_2$, are reported [$a = 26.3955(15)$ Å, $b = 8.0476(4)$ Å, $c = 7.3002(3)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1550.71$ 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 impurity was observed. © *2013 International Centre for Diffraction Data.* [doi:10.1017/S088571561300047X]

Key words: X-ray powder diffraction, norandrostenedione, anabolic steroid

I. INTRODUCTION

The title compound norandrostenedione (Figure 1), systematic name 19-norandrost-4-ene-3, 17-dione, an anabolic steroid whose use in sport is prohibited by the World Anti-Doping Agency (WADA), is an intermediate for contraceptive drugs such as norethindrone, androlin, and nandrolone benzyl propionate. In the process of synthesizing norethindrone from the norandrostenedione matrix, norandrostenedione makes the synthesized compounds impure and leaves residues in norethisterone tablets (Walker, 2009). Therefore, it is very important to detect and control the percentage of norandrostenedione as well as make certain of its crystal structure.

Our lab has reported the crystal structure of normethisterone and gestonoronacetat (Wu *et al.*, 2013). Both of them are steroid progestational hormones. The three compounds have a similar parent structure, but their crystal structures have many differences.

Presently, the crystal structure of norandrostenedione has not been reported.

II. EXPERIMENTAL

A. Sample preparation

The title compound was obtained from Zhejiang Guangersheng Import & Export Co., Ltd. It was re-crystallized in 95% ethanol, then, dried and ground into powder. The structure of norandrostenedione was characterized by IR, UV, and high-performance liquid chromatography (HPLC).

B. Diffraction data collection and reduction

The diffraction pattern for the title compound was collected at room temperature using an X'Pert PRO diffractometer (PANalytical) with an X'celerator detector and Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å, generator setting: 40 kV, 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 10.16 ms/step. Data evaluation was performed using the software package Material Studio 4.2 (Accelrys Co. Ltd, USA).

The first stage of structure determination involves determination of the unit cell {*a, b, c, α, β, γ*} by analysis of the peak positions in the powder XRD pattern [this task is referred to as "indexing" the powder pattern, and involves assigning the Miller indices (*h, k, l*) to each observed peak in the experimental powder XRD pattern] (Harris, 2012). Indexing was carried out using peak positions obtained from the powder diffraction profiles by the X-cell method then the indexing result was refined

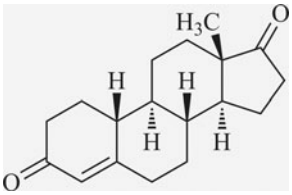


Figure 1. Structural formula of norandrostenedione.

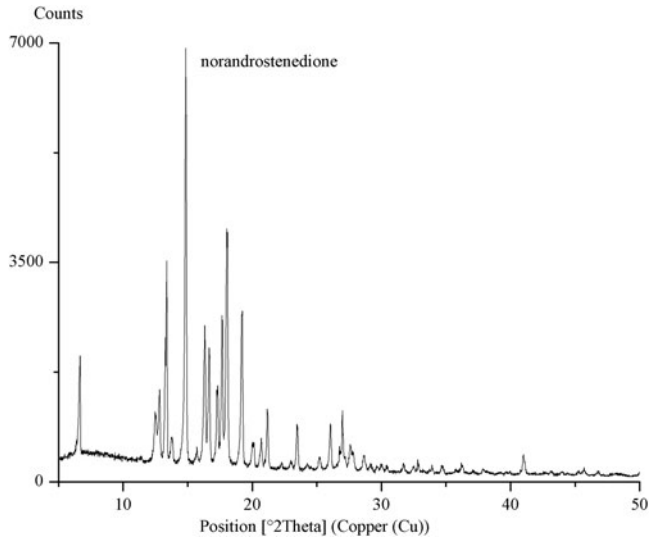


Figure 2. X-ray powder diffraction pattern of norandrostenedione, using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

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with the type of Pawley (Pan *et al.*, 2012). MC/SA search algorithm in Powder Solve package (Engle *et al.*, 1999) was used to constantly adjust the conformation, position, and orientation of the trial model in the unit cell of norandrostenedione, and then the result of Powder Solve was refined by Rietveld refinement techniques based on the experimental X-ray powder diffraction pattern. In the Rietveld refinement (Young, 1993), variables defining the structural model and the powder diffraction profile were adjusted by least squares methods in order to obtain an optimal fit between the experimental pattern and the calculated pattern. After Rietveld refinement, the final R_{wp} of the structure was converged at 9.66%.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results show that norandrostenedione is orthorhombic with the space group $P2_12_12_1$ and unit-cell parameters: $a = 26.3955(15)$ Å, $b = 8.0476(4)$ Å, $c = 7.3002(3)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1550.71$ Å^3, $Z = 4$,

TABLE I. Indexed X-ray powder diffraction data of norandrostenedione, $C_{18}H_{24}O_2$. Only the peaks with I_{rel} of 1 or greater are presented reported [$a = 26.3955$ (15) Å, $b = 8.0476(4)$ Å, $c = 7.3002(3)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1550.71$ 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. 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}(°)	$\Delta 2\theta$
6.6872	13.2069	21	2	0	0	6.6908	13.1999	−0.0035
12.5696	7.0364	14	1	0	1	12.5705	7.0359	−0.0009
12.8716	6.8720	20	2	1	0	12.8712	6.8722	0.0004
13.4099	6.5973	46	4	0	0	13.4044	6.6000	0.0055
13.8564	6.3857	7	2	0	1	13.8512	6.3881	0.0052
14.9068	5.9380	100	3	1	0	14.9036	5.9393	0.0032
15.7603	5.6183	3	3	0	1	15.7601	5.6184	0.0002
16.3774	5.4080	37	0	1	1	16.3795	5.4073	−0.0021
16.7188	5.2983	31	1	1	1	16.7220	5.2973	−0.0033
17.3622	5.1034	21	4	1	0	17.3615	5.1036	0.0007
17.7167	5.0021	36	2	1	1	17.7109	5.0037	0.0058
18.1106	4.8942	69	4	0	1	18.1048	4.8957	0.0057
19.2529	4.6063	45	3	1	1	19.2494	4.6071	0.0035
20.0933	4.4155	5	5	1	0	20.0960	4.4149	−0.0027
20.1589	4.4013	4	6	0	0	20.1647	4.4000	−0.0058
20.7498	4.2772	8	5	0	1	20.7451	4.2782	0.0047
21.2225	4.1830	15	4	1	1	21.2242	4.1827	−0.0017
22.3254	3.9788	2	1	2	0	22.3266	3.9786	−0.0012
23.0213	3.8601	1	6	1	0	23.0170	3.8608	0.0043
23.0870	3.8493	2	2	2	0	23.0849	3.8496	0.0021
23.5334	3.7772	13	5	1	1	23.5304	3.7777	0.0030
24.2950	3.6605	1	3	2	0	24.2985	3.6600	−0.0036
25.2535	3.5237	3	0	2	1	25.2485	3.5244	0.0050
25.2929	3.5183	1	2	0	2	25.2959	3.5179	−0.0031
26.0938	3.4121	11	6	1	1	26.0877	3.4129	0.0061
26.1463	3.4054	2	2	2	1	26.1485	3.4051	−0.0022
26.8028	3.3234	3	0	1	2	26.7974	3.3241	0.0054
27.0129	3.2981	12	1	1	2	27.0126	3.2981	0.0003
27.2361	3.2715	3	3	2	1	27.2339	3.2718	0.0022
27.6563	3.2228	7	2	1	2	27.6501	3.2235	0.0062
27.8533	3.2004	4	5	2	0	27.8501	3.2008	0.0032
27.9058	3.1945	1	4	0	2	27.9106	3.1940	−0.0048
28.6805	3.1100	2	3	1	2	28.6832	3.1097	−0.0027
28.6936	3.1086	3	4	2	1	28.6907	3.1089	0.0029
28.8380	3.0934	1	7	1	1	28.8385	3.0933	−0.0005
29.2188	3.0539	2	8	1	0	29.2247	3.0533	−0.0059
29.7309	3.0025	1	5	0	2	29.7315	3.0024	−0.0006
30.0723	2.9691	2	6	2	0	30.0665	2.9697	0.0058
30.4662	2.9316	1	5	2	1	30.4688	2.9314	−0.0026
31.7398	2.8169	1	8	1	1	31.7393	2.8169	0.0005
31.7792	2.8135	1	5	1	2	31.7833	2.8131	−0.0041
32.5276	2.7504	2	6	2	1	32.5228	2.7508	0.0048
32.8822	2.7216	3	9	0	1	32.8791	2.7218	0.0030
33.9326	2.6397	1	10	0	0	33.9282	2.6400	0.0043
34.7598	2.5787	1	9	1	1	34.7643	2.5784	−0.0045
36.2829	2.4739	3	2	3	1	36.2858	2.4737	−0.0029
37.9242	2.3705	2	9	2	0	37.9244	2.3705	−0.0002
41.0361	2.1976	4	7	2	2	41.0409	2.1974	−0.0048
41.0623	2.1963	3	4	1	3	41.0604	2.1964	0.0019
45.2903	2.0006	1	5	3	2	45.2905	2.0006	−0.0002
45.7498	1.9816	2	7	1	3	45.7468	1.9817	0.0030
50.0040	1.8225	2	5	4	1	50.0510	1.8209	−0.0470

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and space group $P2_12_12_1$ (Table I). After Rietveld refinement, the structure of norandrostenedione was solved. The final results were compared with single-crystal data, which were collected on an Oxford Diffraction Xcalibur Nova system with Mo$K\alpha$ radiation ($\lambda = 0.710\ 73$ Å) at room temperature and the θ from 3.05–28.77°, the deviations of the two methods were between 0.011% and 0.039%. All lines of powder and single-crystal data were indexed and are consistent with the $P2_12_12_1$ space group.

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