X-ray powder diffraction data for peiminine

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X-ray powder diffraction data, unit-cell parameters and space group for peiminine, $C_{27}H_{43}NO_3$, are reported ($a = 30.2026$ Å, $b = 5.8468$ Å, $c = 14.4344$ Å, $\beta = 96.9456°$, unit-cell volume $V = 2530.23$ Å^3, $Z = 2$ and space group $P2_1$). All measured lines were indexed and are consistent with the $P2_1$ space group. No detectable impurity was observed. © *2013 International Centre for Diffraction Data.*
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Key words: X-ray powder diffraction, peiminine

I. INTRODUCTION

Peiminine (Figure 1), also named verticinone, is an important isosteroidal alkaloids that are responsible for the antitussive activity of Bulbus *Fritillariae* (Xu *et al.*, 1990; Ding *et al.*, 1996). Peiminine has strong biological activities and pharmacological properties, such as antitussive, expectorant, anti-inflammatory effects (Wang *et al.*, 2011), inhibition of the liveness of angiotensin-converting enzyme (Oh *et al.*, 2003), and antimicrobial activity (Xiao *et al.*, 1992).

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

II. EXPERIMENTAL

A. Sample preparation

The title compound was extracted, isolated, purified from bulbs of *Fritillariae thunbergii miq*. and the natural sample was characterized by high-performance liquid chromatography (HPLC), IR, MS (positive electrospray, $[M+H]^+ = 430.3321$), as well as by nuclear magnetic resonance (^{1}H NMR and ^{13}C NMR). The pure peiminine was then re-crystallized in methanol.

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 a PIXcel detector, Cu$K\alpha$ radiation and 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 50 s per step. Data evaluation was performed using the software package Material Studio 4.2 (Accelrys Co., Ltd, USA).

The software package Material Studio 4.2 Reflex model was used to smooth the data, to fit the background and to eliminate the $K\alpha_2$ component. X-Cell method was applied to index the pretreated powder diffraction pattern using selected peak positions. The indexing result was then refined with the type of Pawley (Pan *et al.*, 2012). MC/SA search algorithm in Powder Solve package (Engel *et al.*, 1999) was used to constantly adjust the conformation, position and orientation of the trial model in the unit cell of peiminine. Based on the

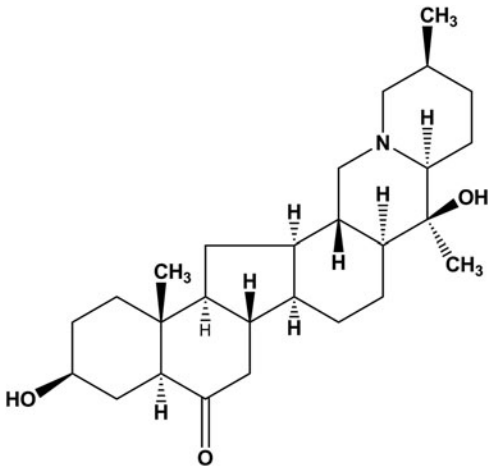

Figure 1. Molecular structure of peiminine.

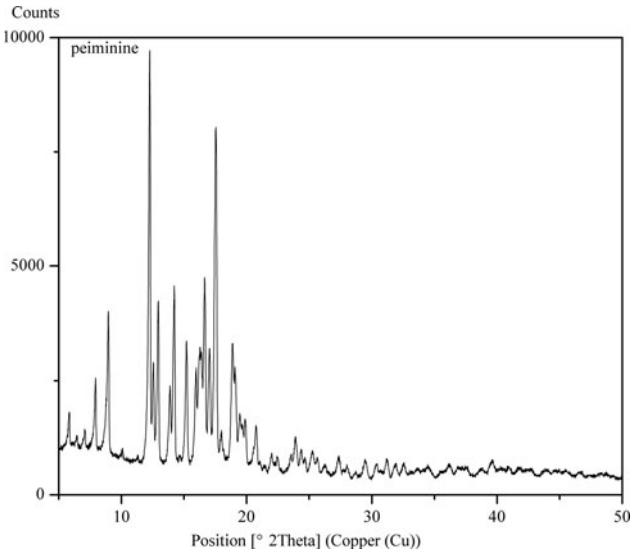

Figure 2. X-ray powder diffraction pattern of peiminine.

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TABLE I. Indexed X-ray powder diffraction data of peiminine, $C_{27}H_{43}NO_3$. Only the peaks with I_{rel} of 1 or greater are presented reported ($a = 30.2026$ Å, $b = 5.8468$ Å, $c = 14.4344$ Å, $\beta = 96.9456°$, unit-cell volume $V = 2530.23$ Å^3, $Z = 2$ and space group $P2_1$). All measured lines were indexed and are consistent with the $P2_1$ 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} (Å)	$\Delta 2\theta$
5.8469	15.1030	19	2	0	0	5.8908	14.9905	−0.0439
6.1620	14.3313	12	0	0	1	6.1633	14.3285	−0.0013
6.4378	13.7181	13	1	0	−1	6.5020	13.5827	−0.0642
7.1074	12.4271	15	1	0	1	7.1463	12.3595	−0.0389
7.9477	11.1149	26	2	0	−1	7.9969	11.0466	−0.0492
8.8799	9.9500	41	3	0	0	8.8411	9.9937	0.0388
9.0376	9.7769	16	2	0	1	9.0312	9.7838	0.0064
10.0880	8.7611	11	3	0	−1	10.1523	8.7057	−0.0643
11.3485	7.7906	9	3	0	1	11.3818	7.7679	−0.0333
11.7818	7.5051	8	4	0	0	11.7974	7.4952	−0.0156
12.2545	7.2166	99	1	0	−2	12.3402	7.1667	−0.0857
12.2676	7.2089	100	0	0	2	12.3445	7.1642	−0.0769
12.5827	7.0291	30	4	0	−1	12.6387	6.9981	−0.0560
12.9504	6.8304	44	2	0	−2	13.0250	6.7914	−0.0746
13.0292	6.7892	14	1	0	2	13.0373	6.7850	−0.0081
13.9614	6.3379	25	4	0	1	13.9694	6.3343	−0.0080
14.2240	6.2215	47	3	0	−2	14.3021	6.1877	−0.0781
14.2634	6.2044	38	2	0	2	14.3205	6.1798	−0.0571
14.7624	5.9958	8	5	0	0	14.7614	5.9962	0.0010
15.1694	5.8358	31	0	1	0	15.1408	5.8468	0.0286
15.3007	5.7860	14	5	0	−1	15.3003	5.7862	0.0004
15.4189	5.7419	8	1	1	0	15.4277	5.7387	−0.0088
15.9835	5.5404	29	4	0	−2	16.0332	5.5233	−0.0497
16.0491	5.5179	20	3	0	2	16.0563	5.5154	−0.0072
16.2592	5.4470	32	2	1	0	16.2590	5.4471	0.0002
16.3643	5.4123	31	0	1	1	16.3609	5.4134	0.0034
16.6531	5.3191	49	5	0	1	16.6897	5.3075	−0.0366
17.1258	5.1733	33	2	1	−1	17.1449	5.1676	−0.0191
17.5460	5.0503	83	3	1	0	17.5594	5.0465	−0.0134
17.6510	5.0205	30	2	1	1	17.6568	5.1890	−0.0058
18.0449	4.9118	14	6	0	−1	18.0657	4.9062	−0.0208
18.7934	4.7179	34	2	0	−3	18.7896	4.7188	0.0038
19.1479	4.6313	29	1	0	3	19.1512	4.6305	−0.0033
19.4630	4.5570	18	6	0	1	19.4925	4.5502	−0.0295
19.5812	4.5297	15	0	1	2	19.5811	4.5298	0.0001
19.5943	4.5268	16	3	0	−3	19.5907	4.5276	0.0036
19.7781	4.4851	13	4	1	−1	19.7702	4.4869	0.0079
20.7892	4.2692	15	4	0	−3	20.7922	4.2686	−0.0030
20.8811	4.2506	10	3	1	−2	20.8858	4.2497	−0.0047
20.8942	4.2480	8	2	1	2	20.8986	4.2471	−0.0044
21.2093	4.1856	7	5	1	0	21.2067	4.1861	0.0026
22.3517	3.9742	7	7	0	1	22.3539	3.9738	−0.0022
23.5991	3.7668	9	6	1	−1	23.6536	3.7583	−0.0545
23.9404	3.7139	13	1	1	−3	23.9426	3.7136	−0.0022
24.6626	3.6068	8	1	0	−4	24.6500	3.6086	0.0126
25.2665	3.5219	10	8	0	1	25.2594	3.5229	0.0071
25.7655	3.4548	7	7	1	0	25.7635	3.4551	0.0020
27.3281	3.2607	8	6	0	3	27.3233	3.2613	0.0048
27.3937	3.2531	9	5	0	−4	27.3927	3.2532	0.0010
28.1159	3.1711	5	9	0	−2	28.1189	3.1708	−0.0030
29.5084	3.0246	8	5	1	3	29.5133	3.0241	−0.0049
30.4268	2.9354	7	2	1	4	30.4379	2.9343	−0.0111
31.1752	2.8666	8	10	0	1	31.1826	2.8659	−0.0074
31.9236	2.8011	7	2	2	1	31.9242	2.8010	−0.0006
32.6589	2.7396	6	11	0	−1	32.6669	2.7390	−0.0080
34.4578	2.6006	7	9	0	−4	34.4486	2.6013	0.0092
36.1779	2.4808	7	11	1	−1	36.1859	2.4803	−0.0080
39.6311	2.2723	8	1	2	−4	39.6449	2.2715	−0.0138
40.9442	2.2024	6	7	0	−6	40.9494	2.2021	−0.0052

experimental X-ray powder diffraction pattern, the result of Powder Solve was subsequently refined by Rietveld refinement techniques. In order to obtain an optimal fit between the experimental and calculated pattern, variables defining the structural model and the powder diffraction profile were adjusted by least squares methods in the Rietveld refinement

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(Young, 1993). After Rietveld refinement, the final R_{wp} was converged at 6.74%.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results show that peiminine is monoclinic with space group $P2_1$ and unit-cell parameters: $a = 30.2026$ Å, $b = 5.8468$ Å, $c = 14.4344$ Å, $\beta = 96.9456°$, unit-cell volume $V = 2530.23$ Å^3, and $Z = 2$. After Rietveld refinement, the structure of peiminine was solved, and the final R_{wp} was converged at 6.74%. 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.

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