X-ray powder diffraction data for deoxyschisandrin

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X-ray powder diffraction data, unit-cell parameters, and space group for deoxyschisandrin, $C_{24}H_{32}O_6$, are reported [$a = 13.083(3)$ Å, $b = 19.563(9)$ Å, $c = 8.805(6)$ Å, $\beta = 90.472(0)°$, unit-cell volume $V = 2253.82$ Å^3, $Z = 4$, 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*. [doi:10.1017/S0885715613000067]

Key words: X-ray powder diffraction, deoxyschisandrin

I. INTRODUCTION

Deoxyschisandrin, systematic name (6R, 7S, 12R)-5, 6,7,8-tetrahydro-1,2,3,10,11,12-hexamethoxy-6,7-dimethyldibenzo[a, c]cyclooctene, is an important lignan for antihepatotoxicity of *Schisandra chinensis* (Turcz.) Baill, which was described by Ikeya *et al.* (1980). Deoxyschisandrin (Figure 1) has strong biological activities and pharmacological properties, such as elevating the content of hepatic mitochondrial glutathione and protecting liver against CCl_4 damage (Huang *et al.*, 2005; Chien *et al.*, 2011), enhancing renal mitochondrial antioxidant status, promoting the generation of liver glycogen evidently.

At present, the crystal structure of deoxyschisandrin has not been reported.

II. EXPERIMENTAL

A. Sample preparation

The title compound was extracted, isolated, purified from *S. chinensis*, and the natural sample was characterized by high-performance liquid chromatography (HPLC), as well as by UV, IR, and MS (positive electrospray, $[M + H]^+ = 403.2121$ Da). Then, pure deoxyschisandrin was re-crystallized in petroleum ether–ethyl acetate (8:1, v/v).

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, 30 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).

Indexing was carried out using peak positions obtained from the powder diffraction profiles by the X-Cell method and then the indexing result was 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 deoxyschisandrin, and then the results of Powder Solve were 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 and the calculated patterns. After Rietveld refinement, the final R_{wp} of the structure was converged at 5.07%.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results show that deoxyschisandrin is monoclinical with space group $P2_1$ and unit-cell parameters: $a = 13.083(3)$ Å, $b = 19.563(9)$ Å, $c = 8.805(6)$ Å, $\beta = 90.472(0)°$, unit-cell volume $V = 2253.82$ Å^3, $Z = 4$, and space

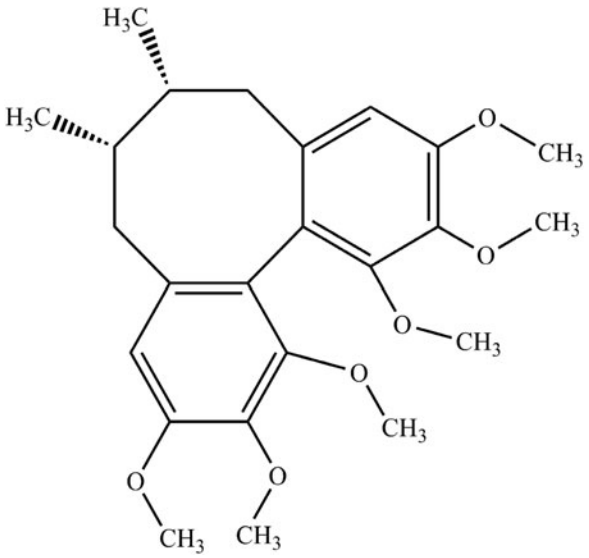


Figure 1. Structural formula of deoxyschisandrin.

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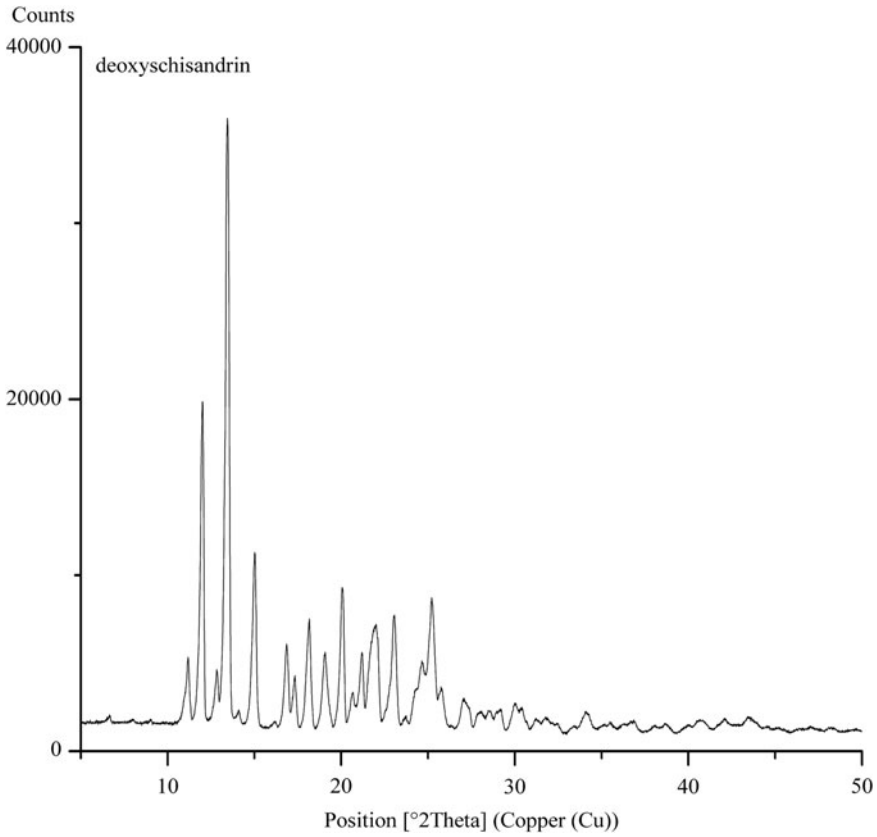


Figure 2. X-ray powder diffraction pattern of deoxyschisandrin, using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

TABLE I. Indexed X-ray powder diffraction data of deoxyschisandrin, $C_{24}H_{32}O_6$. Only the peaks with I_{rel} of 1 or greater are presented [$a = 13.083(3)$ Å, $b = 19.563(9)$ Å, $c = 8.805(6)$ Å, $\beta = 90.472(0)°$, unit-cell volume $V = 2253.82$ Å^3, $Z = 4$, 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} (°)	$\Delta 2\theta$
6.7550	13.0745	1	1	0	0	6.7601	13.0648	−0.0051
9.0660	9.7463	1	0	2	0	9.0651	9.7472	0.0009
11.0092	8.0299	5	0	1	1	11.0141	8.0264	−0.0049
11.3112	7.8162	10	1	2	0	11.3166	7.8125	−0.0054
12.0203	7.3567	53	1	0	−1	12.0634	7.3305	−0.0431
12.1516	7.2775	3	1	0	1	12.1544	7.2758	−0.0028
12.8343	6.8919	7	1	1	−1	12.8915	6.8614	−0.0572
13.5302	6.5389	100	0	2	1	13.5386	6.5349	−0.0084
13.5434	6.5326	98	2	0	0	13.5438	6.5324	−0.0004
15.1059	5.8602	29	1	2	−1	15.1101	5.8586	−0.0042
16.9441	5.2284	14	0	3	1	16.9420	5.2290	0.0021
16.9573	5.2243	11	2	0	1	16.9499	5.2266	0.0074
18.1652	4.8796	18	0	4	0	18.1876	4.8736	−0.0224
18.2309	4.8621	11	1	3	−1	18.2291	4.8626	0.0018
18.2834	4.8483	6	1	3	1	18.2898	4.8466	−0.0064
19.1369	4.6339	10	2	2	−1	19.1371	4.6339	−0.0002
19.2551	4.6058	5	2	2	1	19.2532	4.6062	0.0019
19.4257	4.5657	1	1	4	0	19.4235	4.5662	0.0022
20.0823	4.4179	22	0	0	2	20.1471	4.4038	−0.0648
20.6863	4.2902	4	0	1	2	20.6606	4.2955	0.0257
20.8176	4.2635	1	0	4	1	20.8134	4.2643	0.0042
20.8832	4.2502	1	3	1	0	20.8833	4.2502	−0.0001
21.2246	4.1826	10	1	0	−2	21.2211	4.1833	0.0035
21.3296	4.1623	1	1	0	2	21.3263	4.1629	0.0033
21.7367	4.0852	12	1	1	−2	21.7099	4.0902	0.0268
21.8811	4.0586	14	1	4	−1	21.8816	4.0885	−0.0005
21.9336	4.0490	15	1	4	1	21.9324	4.0492	0.0012
22.1306	4.0134	11	0	2	2	22.1316	4.0132	−0.0010
22.6821	3.9170	4	3	0	−1	22.6858	3.9164	−0.0037
22.8396	3.8904	7	3	0	1	22.8341	3.8913	0.0055

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Table I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (°)	$\Delta 2\theta$
23.0760	3.8511	19	1	2	−2	23.1177	3.8442	−0.0417
23.2204	3.8274	5	1	2	2	23.2145	3.8284	0.0059
24.2577	3.6661	4	2	0	−2	24.2635	3.6652	−0.0058
24.4021	3.6447	3	0	3	2	24.3966	3.6455	0.0055
24.6910	3.6027	7	2	1	−2	24.6952	3.6021	−0.0042
24.9667	3.5635	6	2	4	1	24.9606	3.5644	0.0061
25.2950	3.5180	18	1	3	−2	25.2996	3.5174	−0.0046
25.3869	3.5055	7	1	3	2	25.3884	3.5053	−0.0015
25.8596	3.4425	3	1	5	−1	25.8610	3.4423	−0.0014
25.8990	3.4373	2	1	5	1	25.9046	3.4366	−0.0056
27.2777	3.2667	5	4	0	0	27.2815	3.2660	−0.0038
28.0917	3.1738	2	2	3	2	28.0873	3.1743	0.0044
28.5382	3.1252	2	2	5	1	28.5379	3.1252	0.0003
28.6826	3.1098	1	3	0	−2	28.6888	3.1091	−0.0062
29.0502	3.0712	3	3	1	−2	29.0593	3.0703	−0.0091
29.2209	3.0537	3	3	4	−1	29.2296	3.0528	−0.0087
29.2735	3.0483	1	0	6	1	29.2737	3.0483	−0.0002
30.0481	2.9715	4	1	6	−1	30.0593	2.9704	−0.0112
30.1401	2.9626	3	3	2	−2	30.1455	2.9621	−0.0054
30.4289	2.9352	3	0	0	3	30.4220	2.9358	0.0069
30.4946	2.9290	2	2	4	−2	30.4912	2.9293	0.0034
31.1380	2.8699	1	1	0	−3	31.1437	2.8694	−0.0057
31.3349	2.8523	1	1	5	−2	31.3363	2.8522	−0.0014
31.4006	2.8465	1	1	5	2	31.4097	2.8457	−0.0091
31.5975	2.8292	1	1	1	3	31.5977	2.8292	−0.0002
31.7945	2.8121	2	0	2	3	31.8065	2.8111	−0.0120
31.8864	2.8042	2	3	3	−2	31.8822	2.8046	0.0042
32.0965	2.7864	1	3	3	2	32.0984	2.7862	−0.0019
32.2146	2.7764	1	4	3	−1	32.2171	2.7762	−0.0025
32.5035	2.7524	1	1	2	−3	32.5010	2.7526	0.0025
33.3438	2.6849	1	2	0	−3	33.3313	2.6859	0.0125
34.0135	2.6336	2	4	0	−2	34.0132	2.6336	0.0003
34.1185	2.6257	3	1	3	−3	34.1294	2.6249	−0.0109
34.1842	2.6208	2	3	4	−2	34.1804	2.6211	0.0038
34.2630	2.6150	2	0	6	2	34.2693	2.6145	−0.0063
34.3286	2.6101	1	4	1	−2	34.3316	2.6099	−0.0030
35.5235	2.5250	1	4	2	2	35.5353	2.5242	−0.0118
36.7577	2.4430	1	3	0	−3	36.7502	2.4435	0.0075
36.9415	2.4313	2	2	6	−2	36.9412	2.4313	0.0003
37.9263	2.3704	1	3	2	−3	37.9294	2.3702	−0.0031
38.8192	2.3179	1	1	7	−2	38.8138	2.3182	0.0054
39.9484	2.2549	1	5	0	−2	39.9436	2.2552	0.0048
40.6574	2.2172	2	2	7	−2	40.6543	2.2174	0.0031
42.0361	2.2176	2	0	2	4	42.0330	2.1478	0.0031
42.2593	2.1368	1	1	9	0	42.2576	2.1369	0.0017
43.4804	2.0796	2	2	1	−4	43.4782	2.0797	0.0022
43.5329	2.0772	1	4	3	−3	43.5331	2.0772	−0.0002

group $P2_1$ (Table I). After Rietveld refinement, the structure of deoxyschisandrin 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 to 28.77°, the deviations of the two methods were between 0.543 and 0.808%. All lines of powder and single-crystal data were indexed and are consistent with the $P2_1$ space group.

Chien, C. F., Wu, Y. T., and Tsai, T. H. (**2011**). "Biological analysis of herbal medicines used for the treatment of liver diseases," Biomed. Chromatogr. **25**, 21–38.

Engel, G. E., Wilke, S., König, O., Harris, K. D. M., and Leusen, F. J. J. (**1999**). "Powder Solve – a complete package for crystal structure solution from powder diffraction patterns," J. Appl. Crystallogr. **32**, 1169–1179.

Huang, T. H., Shen, P. N., and Shen, Y. J. (**2005**). "Preparative separation and purification of deoxyschisandrin andγ-schisandrin from Schisandra chinensis (Turcz.) Baill by high-speed counter-current chromatography," J. Chromatogr. A **1066**, 239–242.

Ikeya, Y., Taguchi, H., Sasaki, H., Nakajima, K., and Yosioka, I. (**1980**). "The constituents of *Schizandrin chinensis* Baill. ^{13}C nuclear magnetic resonance spectroscopy of dibenzocyclooctadience lignans," Chem. Pharm. Bull. **28**, 2414–2421.

Pan, Q. Q., Guo, P., Duan, J., Cheng, Q., and Li, H. (**2012**). "Comparative crystal structure determination of griseofulvin: powder X-ray diffraction versus single-crystal X-ray diffraction," Chin. Sci. Bull. **57**, 3867–3871.

Young, R. A. (**1993**). *The Rietveld Method, IUCr Monographies of Crystallography* (Wiley, Oxford).

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