X-ray powder diffraction data for schisanhenol

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(Received 24 July 2013; accepted 14 August 2013)

Experimental X-ray powder diffraction data, unit-cell parameters and space group for schisanhenol, $C_{23}H_{30}O_6$, are reported [$a = 14.6157$ Å, $b = 12.8801$ Å, $c = 11.4907$ Å, unit-cell volume $V = 2163.14$ Å^3, $Z = 4$, and space group $P2_12_12_1$]. All of the measured lines were indexed and are consistent with the $P2_12_12_1$ space group. No detectable impurities were observed. © *2013 International Centre for Diffraction Data*. [doi:10.1017/S0885715613000778]

Key words: X-ray powder diffraction, schisanhenol, *Schisandra chinensis*, Rietveld refinement

I. INTRODUCTION

Schisanhenol, systematic name (6*S*, 7*R*,)-2,3,10,11,12-pentamethoxy-6,7-dimethyl-5,6,7,8-tetrahydrodibenzo[a,c] [8]annulen-1-ol, is an important lignan of *Schisandra chinensis* (Turcz.) Baill, which was first described by Liu *et al.* (1978). Schisanhenol (Figure 1) has strong biological activities and pharmacological properties, which was shown to be the most active one in inhibiting microsome lipid oxidation (Yu *et al.*, 2004). Schisanhenol absolutely inhibited the preoxidative damages of brain mitochondria and membrane of rats. The swelling and disintegration of brain mitochondria, as well as the reduction of brain fluidity were also restrained by schisanhenol (Xue *et al.*, 1992). In addition, schisanhenol exhibits various beneficial activities including antihepatitis, antitumor, and anti-HIV effects (Chen *et al.*, 2006).

The crystal structure of schisanhenol was solved by Xu *et al.* (1982), which was confirmed by single-crystal measurement.

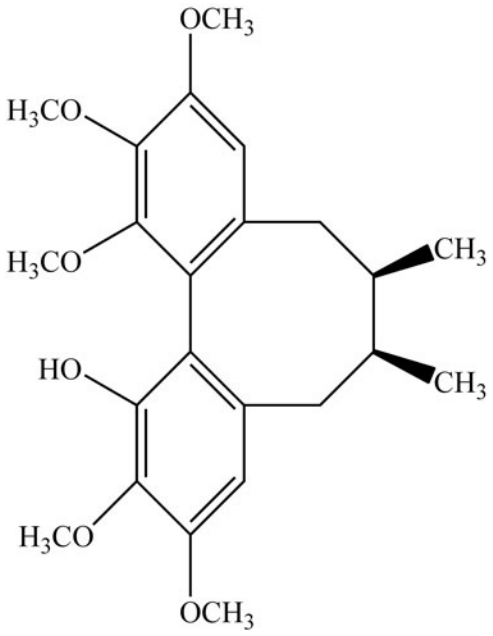

Figure 1. Structural formula of schisanhenol.

II. EXPERIMENTAL

A. Sample preparation

The title compound was extracted, isolated, and purified from *S. chinensis*, and the natural sample was characterized by high-performance liquid chromatography (HPLC), as well as by UV, IR, and MS. Then, the pure schisanhenol (>98% purity) was recrystallized in petroleum ether–ethyl acetate (5:1, v/v).

B. Diffraction data collection and reduction

The diffraction pattern for the target compound was collected at room temperature using an X'Pert PRO diffractometer (PANalytical) with an X'celerator detector and Cu $K\alpha_1$ radiation ($\lambda = 1.54056$ 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 0.01313°2θ and a counting time of 10.16 ms step^{-1}. Data evaluation was

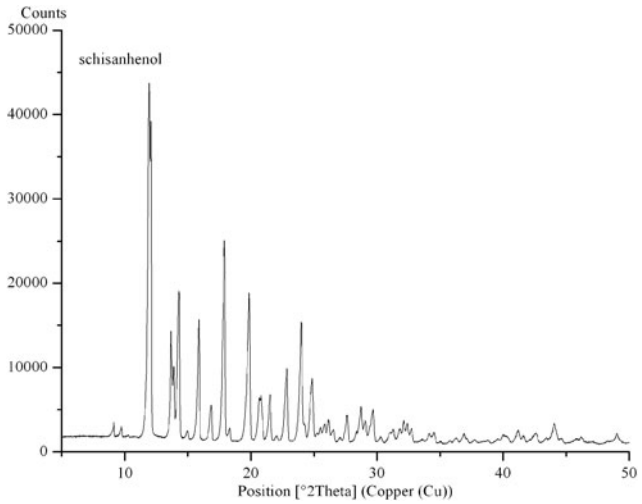

Figure 2. X-ray powder diffraction pattern of schisanhenol, using Cu $K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

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TABLE I. Indexed X-ray powder diffraction data of schisanhenol, $C_{23}H_{30}O_6$. Only the peaks with I_{rel} of 1 or greater are reported [$a = 14.6157$ Å, $b = 12.8801$ Å, $c = 11.4907$ Å, unit-cell volume $V = 2163.14$ Å^3, $Z = 4$, and space group $P2_12_12_1$]. All of the 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 ($\lambda = 1.54056$ Å).

$2\theta_{obs}$(°)	d_{obs}(Å)	I_{obs}	h	k	l	$2\theta_{cal}$(°)	d_{cal}(Å)	$\Delta 2\theta$
9.1447	9.6625	4	1	1	0	9.1440	9.6633	0.0007
9.7487	9.0652	3	1	0	1	9.7832	9.0333	−0.0345
10.3002	8.5811	1	0	1	1	10.3081	8.5745	−0.0079
11.9415	7.4051	100	1	1	1	11.9567	7.3957	−0.0152
12.0728	7.3248	89	2	0	0	12.1009	7.3079	−0.0281
13.6747	6.4702	30	0	2	0	13.7388	6.4401	−0.0641
13.8979	6.3667	20	2	1	0	13.9213	6.3561	−0.0234
14.3312	6.1752	41	2	0	1	14.3518	6.1664	−0.0206
15.0140	5.8959	2	1	2	0	15.0206	5.8933	−0.0066
15.7624	5.6176	14	0	2	1	15.7615	5.6179	0.0009
15.8937	5.5715	33	2	1	1	15.9212	5.5619	−0.0275
16.8785	5.2486	9	0	1	2	16.8835	5.247	−0.0050
16.8916	5.2445	9	1	2	1	16.8936	5.2439	−0.0020
17.9158	4.9469	56	1	1	2	17.9470	4.9384	−0.0312
18.3228	4.8380	3	2	2	0	18.3471	4.8316	−0.0243
19.4651	4.5565	3	3	1	0	19.4640	4.5568	0.0012
19.6358	4.5173	12	2	0	2	19.6389	4.5166	−0.0031
19.7671	4.4876	27	3	0	1	19.7769	4.4854	−0.0098
19.8722	4.4641	41	2	2	1	19.9182	4.4539	−0.0460
20.6731	4.2929	11	0	3	0	20.6708	4.2934	0.0023
20.8044	4.2661	12	2	1	2	20.8238	4.2622	−0.0194
20.9489	4.2370	4	3	1	1	20.9546	4.2359	−0.0057
21.5397	4.1221	12	1	3	0	21.5547	4.1193	−0.0150
21.5791	4.1147	9	1	2	2	21.5833	4.1139	−0.0042
22.8659	3.8860	19	3	2	0	22.8693	3.8854	−0.0034
22.9184	3.8772	11	1	3	1	22.9153	3.8777	0.0031
23.9294	3.7156	25	3	0	2	23.9282	3.7158	0.0013
23.9951	3.7056	33	1	0	3	23.9983	3.7051	−0.0032
24.0213	3.7016	31	2	3	0	24.0200	3.7018	0.0013
24.2183	3.6719	4	0	1	3	24.2226	3.6713	−0.0043
24.3365	3.6544	2	4	0	0	24.3397	3.6539	−0.0032
24.8879	3.5746	16	3	1	2	24.9194	3.5702	−0.0314
24.9799	3.5617	5	1	1	3	24.9869	3.5607	−0.0071
25.2425	3.5252	1	2	3	1	25.2551	3.5235	−0.0126
25.5445	3.4842	3	4	0	1	25.5604	3.4821	−0.0159
25.8858	3.4391	4	0	3	2	25.8847	3.4392	0.0011
26.2141	3.3967	4	2	0	3	26.2474	3.3925	−0.0333
26.4898	3.3620	2	4	1	1	26.4946	3.3614	−0.0048
26.5949	3.3490	2	1	3	2	26.6042	3.3478	−0.0093
27.6716	3.2210	6	0	4	0	27.6807	3.22	−0.0091
27.6847	3.2195	5	3	2	2	27.6939	3.2185	−0.0092
27.7503	3.2121	1	1	2	3	27.7554	3.2115	−0.0051
28.3675	3.1436	2	1	4	0	28.3581	3.1446	0.0093
28.7482	3.1028	9	3	3	1	28.7606	3.1015	−0.0124
29.0634	3.0699	5	4	2	1	29.1301	3.063	−0.0667
29.4442	3.0310	3	1	4	1	29.4237	3.0331	0.0205
29.6411	3.0113	7	3	0	3	29.6436	3.0111	−0.0025
31.0986	2.8735	2	0	0	4	31.1070	2.8727	−0.0084
31.2824	2.8570	3	0	3	3	31.2688	2.8582	0.0136
31.3218	2.8535	2	2	4	1	31.3126	2.8543	0.0091
31.7157	2.8189	1	1	0	4	31.7185	2.8187	−0.0028
31.8733	2.8054	2	1	3	3	31.8775	2.805	−0.0042
32.1359	2.7830	5	4	3	0	32.1410	2.7826	−0.0052
32.4247	2.7589	4	1	4	2	32.4295	2.7585	−0.0048
32.4772	2.7546	2	1	1	4	32.4889	2.7536	−0.0116
32.8055	2.7277	2	3	2	3	32.8060	2.7277	−0.0005
34.1448	2.6237	1	0	2	4	34.1481	2.6235	−0.0034
34.5650	2.5928	1	5	2	1	34.5610	2.5931	0.0040
36.9153	2.4329	1	3	4	2	36.9082	2.4334	0.0071
40.0140	2.2514	1	4	3	3	40.0176	2.2512	−0.0035
41.2614	2.1862	1	5	2	3	41.2686	2.1858	−0.0072
42.5876	2.1211	1	1	4	4	42.5919	2.1209	−0.0043
44.0319	2.0548	4	7	0	1	44.0437	2.0543	−0.0118
44.0975	2.0519	4	3	1	5	44.0956	2.052	0.0019
49.0477	1.8558	1	1	4	5	49.0554	1.8555	−0.0078

https://doi.org/10.1017/S0885715613000778 Published online by Cambridge University Press

performed using the software package Material Studio 4.2 (Accelrys Co., Ltd. USA).

Indexing was conducted using peak positions obtained from the powder diffraction profiles by X-cell method 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 schisanhenol, and then the result of Powder Solve was refined by Rietveld refinement techniques. 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} was converged at 7.60%.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results show that schisanhenol is orthorhombic with space group $P2_12_12_1$ and unit-cell parameters: $a = 14.6157$ Å, $b = 12.8801$ Å, $c = 11.4907$ Å, unit-cell volume $V = 2163.14$ Å^3, $Z = 4$, and space group $P2_12_12_1$ (Table I). All of the lines were indexed and are consistent with the $P2_12_12_1$ space group.

After Rietveld refinement, the structure of schisanhenol was solved. At the same time, single-crystal data were collected on an Oxford Diffraction Xcalibur Nova system with Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å) at room temperature and θ from 3.05° to 28.77°, in that way to verify whether the powder data are accurate. A comparison of the unit-cell parameters from powder data and from single-crystal data reported by Xu *et al.* (1982) shows significant consistency, which indicates that X-ray powder diffraction as a means of crystal structure solution is credible.

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https://doi.org/10.1017/S0885715613000778 Published online by Cambridge University Press