X-ray powder diffraction data for tectoridin, $C_{22}H_{22}O_{11}$

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X-ray powder diffraction data, unit-cell parameters, and space group for tectoridin, $C_{22}H_{22}O_{11}$, are reported [$a = 13.577$ (2) Å, $b = 10.466$ (8) Å, $c = 9.454$ (7) Å, $\alpha = 85.709$ (4)°, $\beta = 94.855$ (3)°, $\gamma = 101.485$ (3)°, unit-cell volume $V = 1309.50$ Å^3, $Z = 2$, $\rho = 1.179 \times 10^3$ kg m^{-3}, and space group $P1$]. No detectable impurity was observed. © *2015 International Centre for Diffraction Data.*
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Key words: X-ray powder diffraction, tectoridin

I. INTRODUCTION

The title compound tectoridin (Figure 1), systematic name 5-hydroxy-3-(4-hydroxyphenyl)-6-methoxy-7-[(2S,3R, 4S,5S,6R)-3,4,5-trihydroxy-6-(hydroxymethyl)oxan-2-yl] oxychromen-4-one, is an isoflavonoid compound extracted from the rhizome of *Belamcanda chinensis* (L.) Redouté, which has properties of heat-clearing, detoxifying, eliminating phlegm, and relieving sore throat. It is commonly used as anti-inflammation antipyretic, antiviral, and diuretic (Qu *et al.*, 2012). As time flies, there are a number of different methods to extract tectoridin from *B. chinensis* (Lee *et al.*, 2011). At present, about 20 isoflavonoid glycosides including tectoridin can be separated from *B. chinensis.* The purity of tectoridin could be influenced by those materials, such as tectorigenin, iridin, irisflorentin, belamcanidin, etc. Hence, it is necessary for the qualitative analysis by powder diffraction data of tectoridin for the purpose of identifying tectoridin easily.

Up to now, both the single-crystal structure and powder diffraction data of tectoridin have not been published.

II. EXPERIMENTAL

A. Sample preparation

The title compound was extracted from *B. chinensis*. It was re-crystallized by slow evaporation of a compound solution in 3:2 (v/v) ethanol and water at room temperature. Then the crystallized product with a needle-like shape was dried and ground into powder. Three parallel determinations showed that the experimental density of the title compound was 1.204×10^3 kg m^{-3} at 25 °C. The structure of tectoridin was characterized by Fourier transform infrared spectrometer (FTIR, Thermo Fisher Scientific, USA; Figure 2), ultraviolet (UV, Beijing Purkinje General Instrument, China; Figure 3) and high-performance liquid chromatography (HPLC, Agilent Technologies, USA; Figure 4). The purity of isolated tectoridin was proven using microscope, melting point test, and HPLC (detailed in supplementary information).

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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 Co. Ltd., the Netherlands) with a PIXcel one-dimensional detector and Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å, generator setting: 40 kV and 40 mA). The diffraction data were collected over the angular range from 4 to 50°2θ with a step size of 0.013 13°2θ and a counting time of 30 ms step^{-1}. Data evaluation was performed using the software package Material Studio 4.2 (Accelrys Co., Ltd., USA) in the State Key Laboratory of Polymer Materials Engineering (Sichuan University, China), which we used to successfully solve organic crystal structures such as normethisterone, gestonoronacetat (Wu *et al.*, 2013), and norandrostenedione (Tang *et al.*, 2013).

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 X-ray diffraction 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 pattern (Harris, 2012). 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). After Pawley refinement of the unit cell, the final R_{wp} and R_p of the structure were converged at 7.47 and 5.10%, respectively.

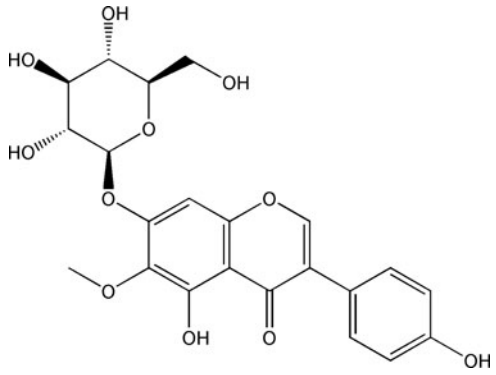


Figure 1. Structural formula of tectoridin.

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TABLE I. Indexed X-ray powder diffraction data of tectoridin, $C_{22}H_{22}O_{11}$. Only the peaks with I_{rel} of 1 or greater are presented reported [$a = 13.577$ (2) Å, $b = 10.466$ (8) Å, $c = 9.454$ (7) Å, $\alpha = 85.709$ (4)°, $\beta = 94.855$ (3)°, $\gamma = 101.485$ (3)°, unit-cell volume $V = 1309.50$ Å^3, $Z = 2$, $\rho = 1.179 \times 10^3$ kg ·m^{-3}, and space group $P1$]. 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}(°)	$\Delta 2\theta$
9.4031	9.3976	100	0	0	1	9.3970	9.4037	−0.0061
11.8847	7.4403	12	1	1	0	11.8877	7.4384	0.0030
11.8979	7.4321	35	1	0	1	11.9064	7.4268	0.0085
15.2067	5.8216	70	1	1	−1	15.1975	5.8251	−0.0092
16.8742	5.2499	85	2	0	1	16.8797	5.2482	0.0055
17.3075	5.1194	21	0	2	0	17.3082	5.1192	0.0007
18.8569	4.7021	44	0	0	2	18.8579	4.7019	0.0010
18.9751	4.6731	24	1	−2	−1	18.9815	4.6715	0.0064
19.2246	4.6130	23	0	2	1	19.2263	4.6126	0.0017
19.5528	4.5363	12	1	0	−2	19.5585	4.5350	0.0057
20.3932	4.3512	6	1	−1	−2	20.3978	4.3502	0.0046
20.9972	4.2274	73	2	−2	−1	21.0054	4.2258	0.0082
21.2335	4.1809	12	0	1	−2	21.2393	4.1798	0.0058
22.1526	4.0094	59	1	2	−1	22.1479	4.0103	−0.0047
22.3890	3.9677	50	1	1	−2	22.3823	3.9688	−0.0067
22.7435	3.9066	10	2	−2	1	22.7407	3.9071	−0.0028
23.9383	3.7142	18	2	0	2	23.9438	3.7134	0.0055
24.9231	3.5697	31	0	2	2	24.9203	3.5701	−0.0028
25.3433	3.5114	6	2	1	−2	25.3409	3.5118	−0.0024
25.5927	3.4778	50	3	1	1	25.5980	3.4771	0.0052
25.7634	3.4551	78	2	2	−1	25.7655	3.4548	0.0020
25.9867	3.4259	51	2	−2	−2	25.9822	3.4265	−0.0045
26.4200	3.3707	15	3	−1	−2	26.4140	3.3715	−0.0060
26.6432	3.3430	25	3	0	−2	26.6415	3.3432	−0.0017
26.9320	3.3078	9	2	−3	0	26.9376	3.3071	0.0056
27.3916	3.2533	15	4	−1	−1	27.3882	3.2537	−0.0034
27.8512	3.2007	12	4	0	−1	27.8454	3.2013	−0.0058
28.0744	3.1757	18	1	−3	1	28.0693	3.1763	−0.0050
28.2188	3.1598	20	1	3	0	28.2112	3.1606	−0.0076
28.7965	3.0977	7	2	−2	2	28.7931	3.0981	−0.0034
28.9804	3.0785	3	3	−2	−2	28.9863	3.0779	0.0059
29.2692	3.0488	6	0	1	3	29.2734	3.0483	0.0042
30.3459	2.9430	9	3	−3	−1	30.3423	2.9433	−0.0036
30.5166	2.9269	7	3	2	−1	30.5176	2.9268	0.0010
30.7792	2.9025	4	3	2	1	30.7750	2.9029	−0.0042
31.1206	2.8715	3	1	−1	3	31.1190	2.8716	−0.0016
33.8517	2.6458	2	2	1	3	33.8576	2.6453	0.0059
34.2062	2.6192	5	4	−1	2	34.2108	2.6188	0.0046
34.3375	2.6095	2	1	−4	0	34.3414	2.6092	0.0038
34.5476	2.5941	5	2	−3	2	34.5518	2.5938	0.0042
35.2698	2.5426	4	5	−2	−1	35.2673	2.5428	−0.0025
35.3485	2.5371	7	1	2	−3	35.3413	2.5376	−0.0072
35.4930	2.5271	5	2	−4	−1	35.4932	2.5271	0.0003
35.7687	2.5083	2	5	0	1	35.7711	2.5081	0.0024
36.2545	2.4758	2	3	0	3	36.2568	2.4756	0.0023
36.3202	2.4714	2	4	1	2	36.3205	2.4714	0.0003
36.5565	2.4560	4	5	1	0	36.5537	2.4562	−0.0029
36.7929	2.4408	8	3	−1	3	36.7905	2.4409	−0.0024
37.0555	2.4241	3	1	−3	−3	37.0591	2.4238	0.0036
37.1080	2.4208	2	3	−3	2	37.1194	2.4200	0.0114
39.0250	2.3061	3	0	4	2	39.0222	2.3063	−0.0029
39.1432	2.2994	9	3	−4	1	39.1442	2.2994	0.0010
39.4189	2.2840	11	3	−3	−3	39.4127	2.2843	−0.0062
39.6028	2.2738	15	2	−1	−4	39.6002	2.2740	−0.0026
39.6815	2.2695	18	1	3	3	39.6877	2.2691	0.0062
40.8501	2.2072	2	3	2	−3	40.8513	2.2072	0.0012
41.1521	2.1917	6	4	−1	3	41.1503	2.1918	−0.0018
42.2945	2.1351	5	4	−3	−3	42.2967	2.1350	0.0022
42.5046	2.1251	5	4	3	−1	42.5007	2.1252	−0.0038
43.2398	2.0906	3	5	−2	−3	43.2359	2.0908	−0.0039
44.1458	2.0498	5	3	4	0	44.1460	2.0498	0.0001
47.7304	1.9039	1	2	−4	3	47.7353	1.9037	0.0049
48.6758	1.8691	3	3	4	−2	48.6752	1.8691	−0.0006
48.9384	1.8597	1	4	4	0	48.9400	1.8596	0.0016

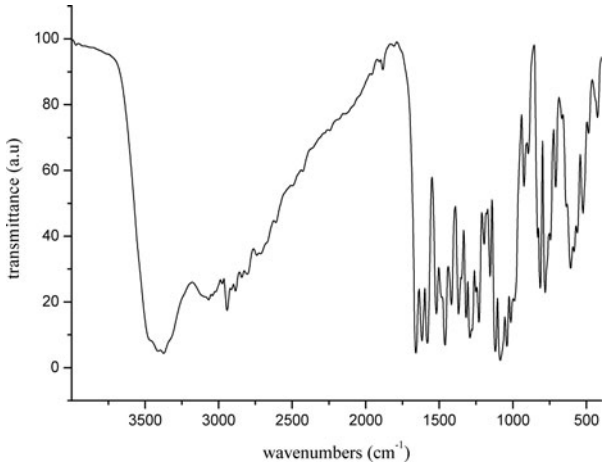


Figure 2. FT-IR spectra of tectoridin recorded by employing Nicolet 6700 FT-IR spectrometer, scanning from 4000 to 400 cm^{-1}.

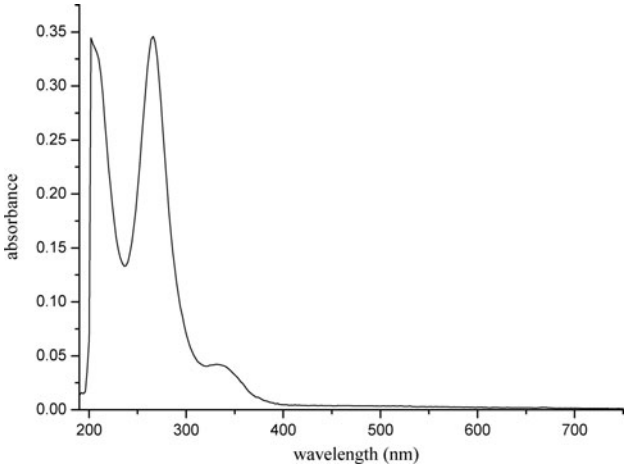


Figure 3. UV absorption spectra of tectoridin from 190 to 750 nm.

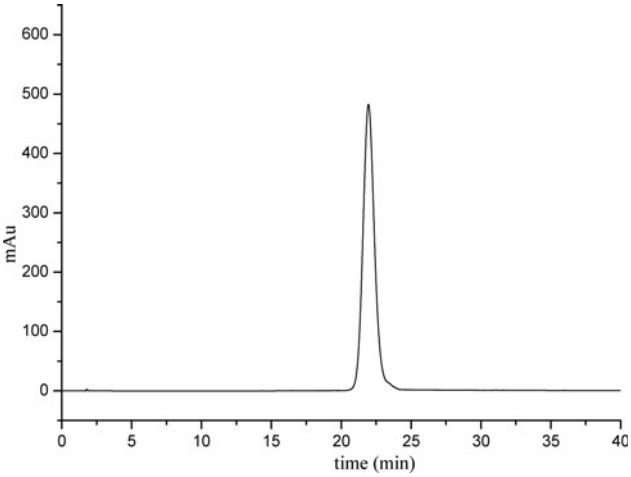


Figure 4. HPLC analysis of tectoridin use Agilent 1200 DAD detector at 265 nm with mobile phase in 32:68 (v/v) methanol and water.

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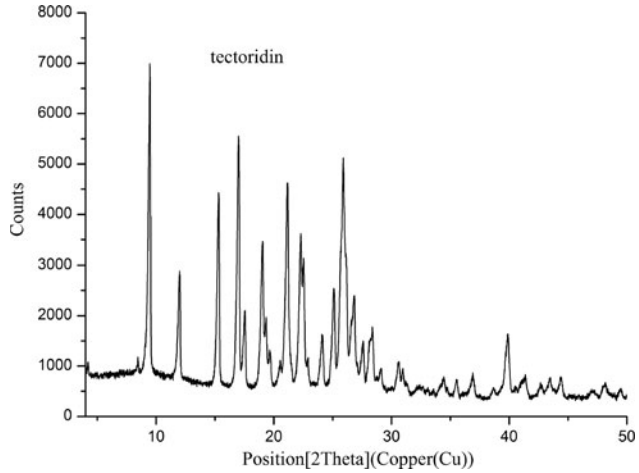


Figure 5. X-ray powder diffraction pattern of the tectoridin, using Cu$K\alpha_1$ radiation ($\lambda = 1.54\,056$ Å).

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 5. Indexing results show that tectoridin is triclinic with space group $P1$ and unit-cell parameters: $a = 13.577$ (2) Å, $b = 10.466$ (8) Å, $c = 9.454$ (7) Å, $\alpha = 85.709$ (4)°, $\beta = 94.855$ (3)°, $\gamma = 101.485$ (3)°, unit-cell volume $V = 1309.50$ Å^3, $Z = 2$, $\rho = 1.179 \times 10^3$ kg m^{-3}, and space group $P1$ (Table I). After Pawley refinement, the unit-cell parameters of tectoridin were solved. All lines of powder data were indexed and consistent with the $P1$ space group.

SUPPLEMENTARY MATERIALS AND METHODS

The supplementary material for this article can be found at http://dx.doi.org/10.1017/S0885715615000810

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