X-ray powder diffraction data for inclusion complex of β-cyclodextrin with fraxinellone

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X-ray powder diffraction data, unit-cell parameters, and space group for inclusion complex of β-cyclodextrin with fraxinellone, $C_{42}H_{70}O_{35}\cdot C_{14}H_{16}O_3\cdot 3H_2O$, are reported [$a = 19.294(2)$ Å, $b = 26.639(1)$ Å, $c = 16.467(3)$ Å, $\beta = 110.451(9)°$, cell volume $V = 7930.34$ Å^3, $Z = 4$ and space group $C2$]. All measured lines were indexed and are consistent with the $C2$ space group. No detectable impurities were observed. © *2013 International Centre for Diffraction Data.*
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Key words: X-ray powder diffraction, β-cyclodextrin, fraxinellone, inclusion complex

I. INTRODUCTION

Fraxinellone (Figure 1), $C_{14}H_{16}O_3$, is formed by the natural degradation of limonoids isolated from the Rutaceae and Meliaceae plants (Trudeau and Morken, 2005). It has been reported that fraxinellone possesses a variety of therapeutic activities such as antifertility, antiplatelet aggregation, vascular relaxation, and fungicidal activity (Lü *et al.*, 2010). Considering the poor solubility of fraxinellone, the inclusion complex of β-cyclodextrin with fraxinellone can increase its solubility. Generally, the inclusion complexes of cyclodextrins with drugs are formed by non-covalent bond (Saenger and Steiner, 1998). The crystal structure of β-cyclodextrin-*p*-aminobenzoic acid inclusion complex (Guo *et al.*, 2011) has been determined by our research group. This paper continues our work on the crystal structure determination of supramolecular compound directly from X-ray powder diffraction. The inclusion complex of β-cyclodextrin with fraxinellone has been prepared, whose molecular is more complex than *p*-aminobenzoic acid inclusion complex, and the crystal structure of the inclusion complex directly from powder X-ray diffraction data using a direct space global optimization method and refined by Rietveld refinement techniques has not been reported in the literature.

II. EXPERIMENTAL

A. Sample preparation

β-CD was purchased from Tianjin Bodi Chemical Holding Co. Ltd., China, and recrystallized twice from distilled water. Fraxinellone was isolated from the root bark of *Dictamnus dasycarpus* (Turcz.) and was identified by UV, FT-IR, MS, ^{1}H-NMR, and ^{13}C-NMR. The isolated fraxinellone was checked by RP-HPLC external standard method and the purity was 99.4%.

Fraxinellone (0.5000 g) was dissolved in acetone (4 ml), and then the solution was added to saturated β-CD (55 °C) aqueous solution drop by drop. The mixture was stirred at 50 to 55 °C for 1.5 h, then cooled down at 5 °C for 12 h. The solution was filtered and washed with water to remove the residual β-CD. The inclusion complex of fraxinellone-β-cyclodextrin was characterized by elemental analysis, FT-IR, and TG-DTA.

B. Diffraction data collection and reduction

X-ray powder diffraction measurement was performed on an X'Pert PRO (PANalytical, Almelo, Netherlands) diffractometer using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å) with an X'celerator detection system, operating at 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 11.22 ms per step at 295 K.

All the structure solution work was performed on Materials Studio 4.2 (Accelrys Co., Ltd., USA) in the State Key Laboratory of Polymer Materials Engineering (Sichuan University, China). The powder diffraction pattern was pretreated by subtracting the background, stripping off $K\alpha_2$ peaks, and smoothing. X-Cell method was used to index the pretreated powder diffraction pattern (Neumann, 2003; Pan *et al.*, 2012). MC/SA search algorithm in Powder Solve package

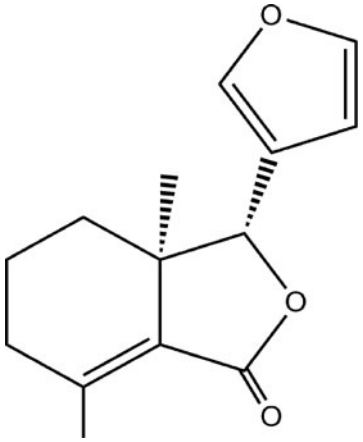

Figure 1. Structural formula of fraxinellone.

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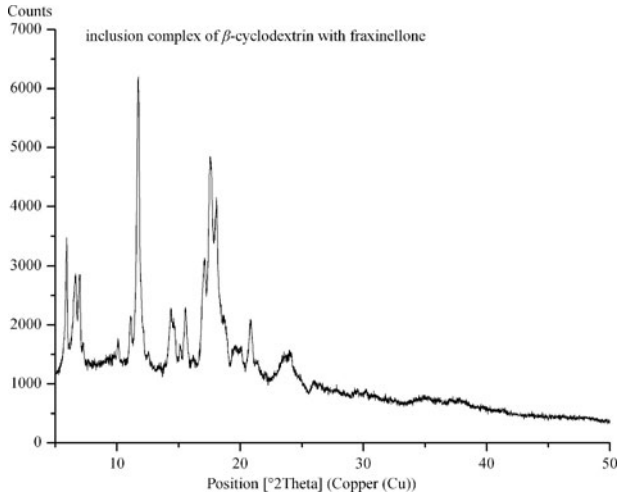

Figure 2. X-ray powder diffraction pattern of inclusion complex of β-cyclodextrin with fraxinellone using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

(Engel *et al.*, 1999) was used to constantly adjust the conformation, position, and orientation of the molecular groups in the unit cell, which was selected by the indexing step, in order to reduce the difference between the calculated and the measured diffraction data. The results obtained from the Powder Solve step were subsequently refined by Rietveld refinement techniques. In the Rietveld refinement, cell parameters, atomic fraction coordinates, thermal vibration, and preferred orientation parameters were optimized simultaneously to obtain the optimum crystal structure. Only the indexing results are reported below and the crystal-structure results obtained by Rietveld refinement will not be reported in this paper.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results show that the inclusion complex is monoclinical with space group $C2$ and unit-cell parameters: $a = 19.294(2)$ Å, $b = 26.639(1)$ Å, $c = 6.467(3)$ Å, $\beta = 110.451(9)°$, unit-cell volume $V = 7930.34$ Å^3, $Z = 4$. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, I_{cal}, $\Delta 2\theta$ are listed in Table I.

TABLE I. Indexed X-ray powder diffraction data for inclusion complex of β-cyclodextrin with fraxinellone.

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (°)	$\Delta 2\theta$
5.9388	14.8699	43	1	1	0	5.9034	14.9587	0.0354
6.6610	13.2592	31	0	2	0	6.6306	13.3195	0.0304
6.9892	12.6372	31	1	1	−1	6.9343	12.7369	0.0549
9.8122	9.0069	4	2	0	0	9.7771	9.0390	0.0351
11.1121	7.9560	16	1	3	0	11.0922	7.9701	0.0199
11.6767	7.5726	87	1	3	−1	11.6761	7.5728	0.0006
11.7424	7.5304	100	2	2	0	11.8224	7.4794	−0.0800
14.1649	6.2475	7	3	1	−1	14.1677	6.2461	−0.0028
14.3488	6.1678	20	1	1	2	14.3471	6.1684	0.0017
14.5654	6.0766	16	2	2	1	14.5664	6.0760	−0.0010
14.6836	6.0279	13	1	3	−2	14.6868	6.0265	−0.0032
15.1103	5.8587	8	3	1	0	15.0612	5.8775	0.0491
15.5633	5.6891	20	3	1	−2	15.5175	5.7057	0.0458
17.0208	5.2051	29	3	3	−1	17.0194	5.2054	0.0014
17.3359	5.1112	23	1	5	0	17.3379	5.1105	−0.0020
17.5132	5.0599	62	2	0	2	17.5137	5.0596	−0.0005
17.5657	5.0449	67	0	4	2	17.5781	5.0412	−0.0124
17.7232	5.0004	51	1	5	−1	17.7205	5.0010	0.0027
17.8742	4.9585	37	3	1	1	17.8718	4.9590	0.0024
18.0843	4.9013	51	2	4	−2	18.0706	4.9049	0.0137
18.4126	4.8147	16	4	0	−1	18.4117	4.8148	0.0009
18.6424	4.7559	12	3	1	−3	18.6401	4.7563	0.0023
18.7474	4.7294	12	2	2	2	18.7455	4.7298	0.0019
19.5877	4.5284	5	4	2	−1	19.5890	4.5280	−0.0013
19.9817	4.4400	6	0	6	0	19.9821	4.4398	−0.0004
20.7366	4.2800	13	4	2	0	20.7372	4.2798	−0.0006
20.8023	4.2667	18	0	6	1	20.8016	4.2667	0.0007
21.2947	4.1691	4	2	4	−3	21.2989	4.1682	−0.0042
21.6426	4.1029	2	2	0	−4	21.6419	4.1029	0.0007
22.0497	4.0280	2	2	4	2	22.0449	4.0288	0.0048
22.3254	3.9789	1	4	0	1	22.3277	3.9784	−0.0023
22.6668	3.9197	1	2	0	3	22.6647	3.9200	0.0021
22.7718	3.9019	2	4	4	−1	22.7713	3.9019	0.0005
22.8441	3.8897	2	3	1	−4	22.8431	3.8898	0.0010
23.0344	3.8580	3	0	0	4	23.0381	3.8573	−0.0037
23.1657	3.8364	5	0	6	2	23.0940	3.8481	0.0717
23.2642	3.8204	6	1	5	−3	23.2749	3.8186	−0.0107
23.3693	3.8035	7	4	2	1	23.3157	3.8120	0.0536
23.4809	3.7857	8	2	6	−2	23.4756	3.7864	0.0053
23.6319	3.7618	8	2	2	3	23.6396	3.7605	−0.0077
23.7763	3.7393	8	4	4	0	23.7730	3.7397	0.0033

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Table I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (°)	$\Delta 2\theta$
23.8879	3.7221	8	2	6	1	23.8871	3.7221	0.0008
24.0126	3.7030	11	1	3	−4	24.0128	3.7029	−0.0002
24.5313	3.6259	4	4	0	−4	24.5333	3.6255	−0.0020
24.7414	3.5956	3	3	3	−4	24.7433	3.5952	−0.0019
25.0171	3.5566	3	4	4	−3	25.0463	3.5524	−0.0292
25.8443	3.4446	3	1	7	−2	25.7840	3.4524	0.0603
26.1266	3.408	4	4	4	1	26.0683	3.4154	0.0583
26.4155	3.3714	4	2	4	3	26.3605	3.3782	0.0550
26.5534	3.3542	3	0	6	3	26.4994	3.3608	0.0540
26.967	3.3037	2	4	2	2	26.9386	3.307	0.0284
27.0589	3.2927	2	2	0	−5	27.0528	3.2933	0.0061
27.5841	3.2311	2	1	5	−4	27.5385	3.2363	0.0456
27.6826	3.2199	2	3	1	−5	27.6711	3.2211	0.0115
28.3916	3.1411	2	2	8	−1	28.4283	3.137	−0.0367
28.6476	3.1136	1	5	5	−1	28.6813	3.1099	−0.0337
29.5471	3.0208	4	2	8	−2	29.4943	3.026	0.0528
29.6718	3.0084	1	0	2	5	29.6930	3.0062	−0.0212
29.8491	2.9909	2	5	5	−3	29.8699	2.9888	−0.0208
30.1117	2.9654	3	4	6	1	30.1372	2.9629	−0.0255
30.3743	2.9404	2	6	2	0	30.3902	2.9388	−0.0159
30.5253	2.9262	1	4	0	3	30.5296	2.9257	−0.0043
30.6763	2.9121	2	0	6	4	30.6779	2.9119	−0.0016
30.8404	2.897	2	1	9	−1	30.8058	2.9001	0.0346
30.9323	2.8886	2	5	1	−5	30.9898	2.8833	−0.0575
31.1818	2.8661	1	6	4	−1	31.1983	2.8645	−0.0165
31.3656	2.8497	1	6	2	−4	31.3295	2.8528	0.0361
31.6151	2.8278	2	6	4	−3	31.6597	2.8238	−0.0446
31.7989	2.8118	2	3	1	4	31.7647	2.8147	0.0342
32.4817	2.7543	1	5	3	−5	32.4477	2.757	0.0340
33.5387	2.6698	1	6	0	−5	33.5021	2.6726	0.0366
33.8078	2.6492	2	2	0	5	33.7845	2.6509	0.0233
34.0836	2.6284	2	7	3	−2	34.0332	2.6321	0.0504
34.1295	2.625	2	0	10	1	34.1267	2.6251	0.0028
34.4118	2.6041	1	1	9	−3	34.4132	2.6039	−0.0014
34.8648	2.5713	2	0	0	6	34.8606	2.5715	0.0042
35.2062	2.5471	2	5	5	−5	35.2011	2.5474	0.0051
35.2587	2.5434	2	4	6	−5	35.2597	2.5433	−0.0010
35.994	2.4931	1	6	6	0	35.9937	2.4931	0.0003
36.0794	2.4874	2	4	2	4	36.1091	2.4854	−0.0297
36.9066	2.4336	2	3	5	−6	36.9098	2.4333	−0.0032
37.1692	2.417	2	6	0	−6	37.1678	2.417	0.0014
37.2086	2.4145	2	3	1	5	37.2092	2.4144	−0.0006
37.7075	2.3837	1	4	8	2	37.7064	2.3837	0.0011
38.0161	2.3651	2	4	4	4	38.0176	2.3649	−0.0015
38.0555	2.3627	2	8	2	−3	38.0544	2.3627	0.0011
38.3575	2.3448	2	1	3	6	38.3681	2.3441	−0.0106
39.5655	2.2759	1	2	6	5	39.5632	2.2760	0.0023
39.8609	2.2597	2	8	0	0	39.8588	2.2598	0.0021
40.1629	2.2434	1	6	2	3	40.1627	2.2434	0.0002
41.1936	2.1897	1	7	3	−6	41.1957	2.1895	−0.0021
42.2047	2.1395	1	8	4	0	42.1956	2.1399	0.0091
43.8853	2.0614	1	4	6	−7	43.8885	2.0612	−0.0032

Only the peaks with I_{rel} of 1 or greater are presented [$a = 19.294(2)$ Å, $b = 26.639(1)$ Å, $c = 16.467(3)$ Å, $\beta = 110.451(9)°$, cell volume $V = 7930.34$ Å^3 and $Z = 4$, space group $C2$]. All measured lines were indexed and are consistent with the $C2$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

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