X-ray powder diffraction data for calcium(II)–naproxen complex ($C_{28}H_{26}CaO_6 \cdot 2H_2O$)

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(Received 8 July 2014; accepted 20 February 2015)

X-ray powder diffraction data, unit-cell parameters, and space group for calcium(II)–naproxen complex, $C_{28}H_{26}CaO_6 \cdot 2H_2O$, are reported [$a = 36.918(2)$ Å, $b = 5.655(6)$ Å, $c = 12.505(6)$ Å, $\beta = 91.263(2)°$, cell volume $V = 2610.47$ 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. © *2015 International Centre for Diffraction Data*. [doi:10.1017/S0885715615000160]

Key words: X-ray powder diffraction, naproxen, complex

I. INTRODUCTION

Calcium(II)–naproxen complex (Figure 1) is a new potential anti-inflammatory drug synthesized recently, which is a white powder at room temperature. Metal complexes of the anti-inflammatory drug naproxen as auxiliary ligand have been widely studied since they were found to be more active and desirable drugs than their parent drugs themselves (Sharma *et al.*, 2003; Abuhijleh and Khalaf, 2010). The title compound is also expected to have some biological activities and medicinal value.

Presently, the crystal structure of calcium(II)–naproxen complex has not been reported.

II. EXPERIMENTAL

A. Sample preparation

Sodium naproxen, prepared by adding naproxen (2.3 g or 0.01 mol) to a 100 ml ethanolic solution of NaOH (0.4 g or 0.01 mol) and drying under vacuum at 40 °C, was dissolved in water (100 ml). To it, $CaCl_2$ (0.56 g) was added with constant stirring. The precipitates of the calcium(II)–naproxen complex (final yield, 76%), thus, formed were filtered, washed with cold water, and dried under vacuum to a constant weight. The sample was characterized by UV–vis, FTIR (KBr, cm^{-1}): ν(O–H): 3415, ν_{asy}(COO): 1604, ν_{sy}(COO): 1395, ν(Ca–O): 484; TG-DTA: mass change: -6.76%, melting point: 137°C; mass spectrometry [the UV–vis; FTIR and TG-DTA spectra are as supplementary material (Fig. 1S–3S)].

B. Diffraction data collection and reduction

The X-ray powder diffraction measurements were performed on an X'Pert PRO diffractometer (PANalytical Co., Ltd., the Netherlands) equipped with a PIXcel one-dimensional (1D) detection system and Cu$K\alpha$ radiation (generator setting, 40 kV and 40 mA). The diffraction data were recorded at room temperature with a step size of 0.013 13°2θ within 5° to 50° in 2θ. Data evaluation was mostly conducted using the Reflex module in the software package Material Studio 4.2 (Accelrys Co., Ltd., San Diego, CA) in the State Key Laboratory of Polymer Materials Engineering (Sichuan University, Chengdu, Sichuan, China).

The powder diffraction pattern was pretreated by subtracting the background, stripping off $K\alpha_2$ peaks, and smoothing. 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 using Pawley refinement

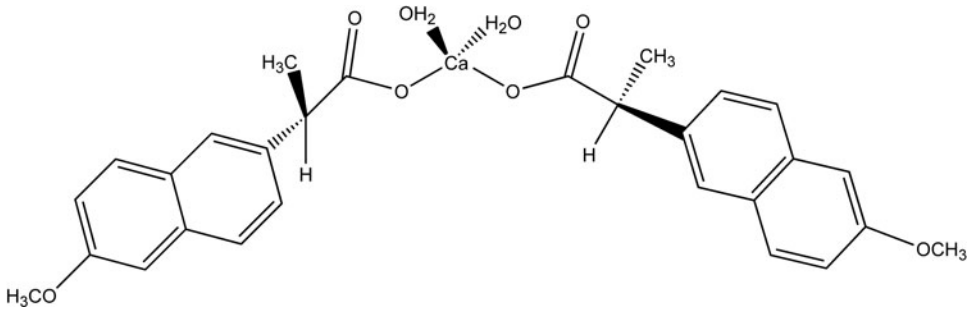

Figure 1. Structural formula of calcium(II)–naproxen complex.

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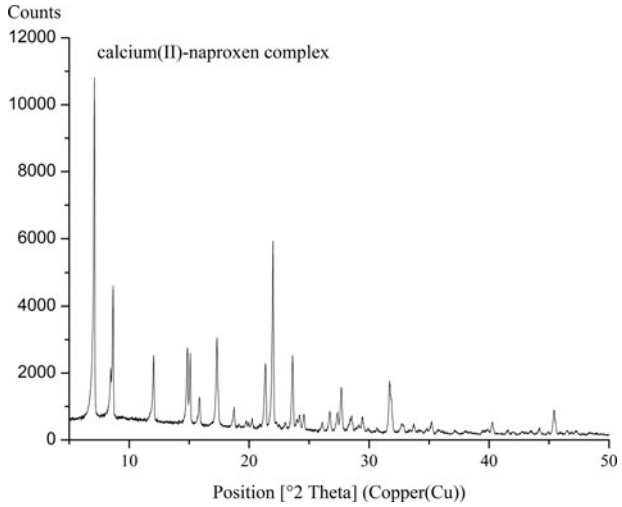

Figure 2. X-ray powder diffraction pattern of calcium(II)–naproxen complex.

(Harris, 2012; Pan *et al.*, 2012). MC/SA search algorithm in Powder Solve package (Engel *et al.*, 1999; Wu *et al.*, 2013) 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.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results show that calcium(II)–naproxen complex is monoclinic with space group *C*2 and unit-cell parameters: $a = 36.918(2)$ Å, $b = 5.655(6)$ Å, $c = 12.505(6)$ Å, $\beta = 91.263(2)°$, unit-cell volume $V = 2610.47$ Å^3, space group *C*2, and $Z = 4$ (Table I). After Pawley refinement, the unit-cell parameters of calcium(II)–naproxen complex were solved. All lines of powder data were indexed and consistent with the *C*2 space group.

TABLE I. Indexed X-ray powder diffraction data of calcium(II)–naproxen complex, $C_{28}H_{26}CaO_6{\cdot}2H_2O$. Only the peaks with I_{obs} of 1 or greater are reported [$a = 36.918(2)$ Å, $b = 5.655(6)$ Å, $c = 12.505(6)$ Å, $\beta = 91.263(2)°$, unit-cell volume $V = 2610.47$ Å^3, $Z = 4$, and space group *C*2]. All measured lines were indexed and are consistent with the *C*2 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*	$2\theta_{cal}$(Å)	d_{cal}(Å)	$\Delta 2\theta$
4.8075	18.3657	9	2	0	0	4.7843	18.4546	0.0232
7.1053	12.4307	100	0	0	1	7.0644	12.5026	0.0409
8.4577	10.4458	20	2	0	−1	8.4475	10.4584	0.0102
8.6547	10.2085	43	2	0	1	8.6226	10.2465	0.0321
11.7666	7.5148	8	4	0	−1	11.7839	7.5037	−0.0174
12.0423	7.3433	23	4	0	1	12.0357	7.3473	0.0066
14.1300	6.2627	5	0	0	2	14.1559	6.2513	−0.0259
14.4320	6.1323	6	6	0	0	14.3866	6.1515	0.0454
14.8653	5.9545	25	2	0	−2	14.8494	5.9609	0.0160
15.0885	5.8669	24	2	0	2	15.0508	5.8816	0.0378
15.8238	5.5959	11	1	1	0	15.8397	5.5904	−0.0159
15.8632	5.5821	12	6	0	−1	15.9024	5.5684	−0.0392
16.9530	5.2256	5	4	0	−2	16.9414	5.2292	0.0117
17.3207	5.1155	28	1	1	−1	17.3404	5.1098	−0.0197
18.5812	4.7713	5	3	1	−1	18.5927	4.7683	−0.0115
18.7256	4.7348	8	3	1	1	18.7145	4.7376	0.0111
19.7760	4.4856	5	5	1	0	19.7589	4.4894	0.0171
19.9730	4.4418	5	6	0	−2	20.0096	4.4338	−0.0366
21.3648	4.1555	21	1	1	2	21.3402	4.1602	0.0246
21.7456	4.0836	8	2	0	−3	21.7403	4.0845	0.0053
21.9951	4.0378	55	2	0	3	21.9498	4.0460	0.0453
22.2971	3.9838	5	3	1	−2	22.2751	3.9877	0.0220
23.0323	3.8583	5	7	1	0	23.0421	3.8566	−0.0098
23.6232	3.7631	23	4	0	3	23.5977	3.7671	0.0255
24.0171	3.7022	6	7	1	−1	24.0177	3.7021	−0.0006
24.0959	3.6903	5	10	0	0	24.0919	3.6909	0.0040
24.2141	3.6726	7	8	0	2	24.2069	3.6737	0.0072
24.5555	3.6223	7	5	1	2	24.5466	3.6236	0.0089
26.0786	3.4141	5	6	0	3	26.0675	3.4155	0.0111
26.7220	3.3333	8	1	1	3	26.7008	3.3359	0.0212
26.8139	3.3221	5	9	1	0	26.8308	3.3200	−0.0170
27.3785	3.2549	8	7	1	2	27.3441	3.2589	0.0344
27.4047	3.2518	6	3	1	−3	27.4073	3.2515	−0.0025
27.6805	3.2200	15	3	1	3	27.6602	3.2223	0.0203
28.3370	3.1469	4	10	0	2	28.3264	3.1481	0.0106
28.5208	3.1270	6	8	0	−3	28.5203	3.1271	0.0005
28.5602	3.1228	7	0	0	4	28.5338	3.1256	0.0264
29.1642	3.0595	4	8	0	3	29.1666	3.0593	−0.0024
29.4005	3.0354	6	5	1	3	29.4134	3.0341	−0.0128

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https://doi.org/10.1017/S0885715615000160 Published online by Cambridge University Press

TABLE I. Continued

$2\theta_{obs}$(°)	d_{obs}(Å)	I_{obs}	h	k	l	$2\theta_{cal}$(Å)	d_{cal}(Å)	$\Delta 2\theta$
31.6983	2.8204	16	11	1	−1	31.6625	2.8236	0.0359
31.7902	2.8125	13	6	0	−4	31.7995	2.8117	−0.0093
31.8559	2.8069	11	7	1	3	31.8412	2.8081	0.0146
32.7094	2.7355	5	12	0	2	32.7035	2.7360	0.0059
32.7488	2.7323	4	1	1	−4	32.7522	2.7321	−0.0035
32.8275	2.7260	4	2	2	1	32.8283	2.7259	−0.0008
32.8669	2.7228	4	1	1	4	32.8481	2.7243	0.0188
33.7204	2.6558	5	3	1	4	33.6709	2.6596	0.0495
34.2194	2.6182	3	9	1	−3	34.2001	2.6196	0.0193
34.8102	2.5751	3	9	1	3	34.8192	2.5745	−0.0089
35.0334	2.5592	3	8	0	4	34.9993	2.5616	0.0341
35.0859	2.5555	4	2	2	−2	35.0946	2.5549	−0.0086
35.2173	2.5463	5	2	2	2	35.1848	2.5485	0.0324
35.3223	2.5389	3	13	1	0	35.3445	2.5374	−0.0223
35.7293	2.5109	3	6	2	1	35.7111	2.5122	0.0183
36.1101	2.4853	3	2	0	−5	36.1125	2.4852	−0.0024
37.2262	2.4133	3	10	0	−4	37.2538	2.4116	−0.0276
37.9877	2.3667	3	13	1	−2	37.9759	2.3674	0.0118
39.3927	2.2855	2	1	1	−5	39.3933	2.2854	−0.0006
39.5109	2.2789	3	16	0	−1	39.5359	2.2775	−0.0250
39.8129	2.2623	3	4	2	3	39.8272	2.2615	−0.0143
39.8654	2.2594	3	16	0	1	39.8613	2.2597	0.0041
39.8916	2.2580	3	8	2	−2	39.8880	2.2582	0.0037
40.2330	2.2396	5	8	2	2	40.2110	2.2408	0.0221
41.5460	2.1718	3	5	1	5	41.5393	2.1722	0.0067
41.5854	2.1699	3	12	0	4	41.6120	2.1686	−0.0266
42.7409	2.1139	2	7	1	−5	42.7437	2.1137	−0.0028
43.4368	2.0816	3	12	2	0	43.4341	2.0817	0.0027
43.4631	2.0804	3	2	2	4	43.4691	2.0801	−0.0060
44.1196	2.0509	3	4	2	−4	44.1093	2.0514	0.0103
44.1590	2.0492	3	10	0	5	44.1568	2.0493	0.0022
45.4326	1.9947	8	17	1	1	45.4413	1.9943	−0.0087
45.5245	1.9909	6	13	1	−4	45.5635	1.9892	−0.0390
46.4962	1.9515	3	13	1	4	46.5020	1.9513	−0.0059
46.5224	1.9505	3	1	1	6	46.5237	1.9504	−0.0013
47.2971	1.9203	3	12	0	5	47.3144	1.9196	−0.0173
48.3738	1.8800	2	16	0	−4	48.4866	1.8759	−0.1129
49.0434	1.8559	2	18	0	−3	49.0372	1.8561	0.0062
49.2929	1.8471	2	15	1	−4	49.2889	1.8473	0.0040

SUPPLEMENTARY MATERIALS AND METHODS

The supplementary material for this article can be found at http://www.journals.cambridge.org/PDJ.

ACKNOWLEDGEMENT

We acknowledge financial support by the National Development and Reform Commission and Education of China (Grant No. 2014BW011).

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