X-ray powder diffraction data for bisacodyl, $C_{22}H_{19}NO_4$

Shan Shan Li, Xiao Qing Wu, Qing Wang, Pei Xiao Tang, and Hui Li[a)]
College of Chemical Engineering, Sichuan University, Chengdu 610065, China

(Received 7 March 2014; accepted 30 April 2014)

In this paper, X-ray powder diffraction data, unit-cell parameters, and space group for bisacodyl, $C_{22}H_{19}NO_4$, are reported [$a = 9.081(3)$ Å, $b = 10.631(5)$ Å, $c = 11.549(6)$ Å, $\alpha = 111.492(4)°$, $\beta = 108.082(3)°$, $\gamma = 101.501(3)°$, unit-cell volume $V = 922.368$ Å^3, $Z = 2$, and space group P-1]. All measured lines were indexed, and no detectable impurity was observed. © *2014 International Centre for Diffraction Data*. [doi:10.1017/S0885715614000475]

Key words: X-ray powder diffraction, bisacodyl

I. INTRODUCTION

Bisacodyl (Figure 1), systematic name 4,4′-(2-pyridylmethylene) bisphenol diacetate, is a poorly absorbed diphenylmethane which acts locally on the colon as a peristaltic stimulant (Adams *et al.*, 1994). Bisacodyl can affect the prostaglandin, kinase, and ATP from the colon, and restrain the absorption of water. As a mild laxative, bisacodyl has been commonly studied in gastroenterology for bowel preparation (Clark *et al.*, 2013).

At present, the crystal structure of bisacodyl by single-crystal diffraction or powder diffraction has not been reported. Although there was already a reference pattern for bisacodyl in the ICDD Powder Diffraction File (00-043-1744), there were no precise unit-cell data, only *d*-spacings and intensities were reported.

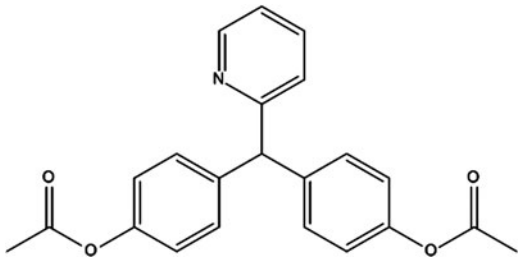

Figure 1. Structural formula of bisacodyl.

II. EXPERIMENTAL

A. Sample preparation

The title compound was purchased from Heowns Biochem Technologies LLC., China, with 98% purity and characterized by UV and FTIR. It was recrystallized in acetone, dried for 6 h at 40 °C, then ground into powder, and sieved through a 200-mesh screen.

B. Diffraction data collection and reduction

X-ray powder diffraction measurement was performed at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with a PIXcel 1D detector and Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å, generator setting: 40 kV and 40 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 30 s step^{-1}. The experimental X-ray powder diffraction pattern is depicted in Figure 2.

The software package Material Studio 4.2 (Accelrys Co. Ltd., USA) was used to process the data in the State Key Laboratory of Polymer Materials Engineering (Sichuan University, China). The X-ray powder diffraction pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results were obtained by the X-Cell method (Neumann, 2003) and the indexing results were refined using Pawley refinement ($R_{wp} = 6.73\%$). Direct-space approach based on Monte Carlo algorithm in Powder Solve package (Engel *et al.*, 1999) was used for structure solution. It allowed for the optimized bisacodyl molecule searching conformation, position, and orientation in the refined cell to maximize the agreement between the calculated and the measured diffraction data. In the Rietveld refinement (Young, 1993; Li *et al.*, 2014),

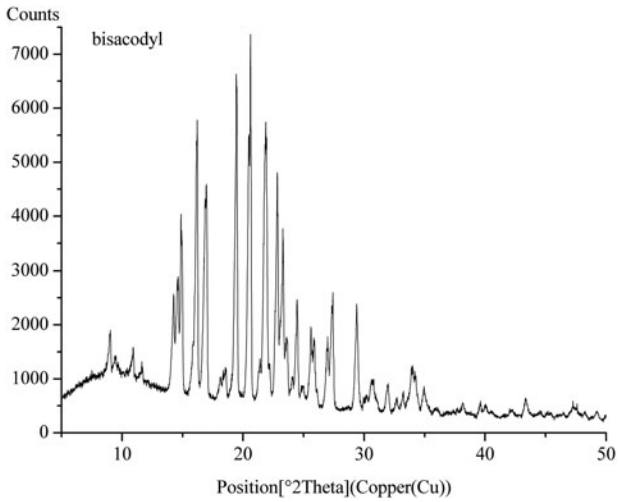

Figure 2. X-ray powder diffraction pattern of the bisacodyl, using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

[a)]Author to whom correspondence should be addressed. Electronic mail: lihuilab@sina.com

TABLE I. Indexed X-ray powder diffraction data of bisacodyl, $C_{22}H_{19}NO_4$. Only the peaks with I_{rel} of 1 or greater are reported [$a = 9.081(3)$ Å, $b = 10.631(5)$ Å, $c = 11.549(6)$ Å, $\alpha = 111.492(4)°$, $\beta = 108.082(3)°$, $\gamma = 101.501(3)°$, unit-cell volume $V = 922.368$ Å^3, $Z = 2$, and space group P-1]. All measured lines were indexed and are consistent with the P-1 space group. The d-values were calculated using CuK α_1 radiation ($\lambda = 1.54056$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
9.0355	9.7791	26	0	0	1	9.0629	9.7496	−0.0274
9.5082	9.2940	19	0	1	0	9.5517	9.2518	−0.0435
9.7051	9.1058	17	0	1	−1	9.6846	9.1251	0.0205
10.9262	8.0908	21	1	0	0	10.9496	8.0735	−0.0234
11.6878	7.5652	17	1	−1	0	11.7125	7.5493	−0.0248
14.2876	6.1940	34	1	1	−1	14.3093	6.1846	−0.0218
14.6290	6.0502	39	1	−1	−1	14.6977	6.0220	−0.0688
14.9178	5.9337	51	1	−1	1	14.9589	5.9175	−0.0411
16.2177	5.4609	79	0	1	−2	16.1796	5.4737	0.0381
16.8742	5.2499	59	1	0	−2	16.8202	5.2666	0.0541
16.9793	5.2176	62	1	1	−2	17.0011	5.2110	−0.0218
18.1610	4.8807	14	0	0	2	18.1831	4.8748	−0.0221
18.4630	4.8015	15	1	−2	0	18.4074	4.8159	0.0556
18.5943	4.7679	16	1	−2	1	18.5828	4.7708	0.0115
19.4346	4.5636	90	0	2	−2	19.4393	4.5625	−0.0047
19.9467	4.4476	10	2	0	−1	19.9394	4.4492	0.0073
20.4982	4.3292	75	2	−1	−1	20.5137	4.3259	−0.0155
20.6164	4.3046	100	2	−1	0	20.6407	4.2996	−0.0243
21.8769	4.0594	78	1	2	−1	21.8389	4.0663	0.0380
21.9294	4.0498	74	1	2	−2	21.9280	4.0500	0.0014
22.8223	3.8933	65	1	−2	2	22.7909	3.8986	0.0314
23.2818	3.8175	51	1	1	−3	23.2697	3.8194	0.0121
23.5969	3.7672	24	2	−2	0	23.5497	3.7746	0.0472
23.6626	3.7569	23	2	1	−1	23.6816	3.7539	−0.0190
24.1222	3.6864	14	2	−1	−2	24.1609	3.6805	−0.0388
24.4635	3.6357	34	0	1	−3	24.4851	3.6325	−0.0215
24.9625	3.5641	12	1	0	2	24.9740	3.5625	−0.0115
25.1857	3.5330	9	0	2	−3	25.2081	3.5300	−0.0224
25.3433	3.5114	10	1	2	0	25.3318	3.5130	0.0115
25.6059	3.4760	27	1	−3	1	25.5924	3.4778	0.0134
25.9079	3.4362	23	0	3	−1	25.9688	3.4283	−0.0610
26.1048	3.4107	11	0	3	−2	26.1206	3.4087	−0.0158
26.9846	3.3015	24	2	1	0	27.0235	3.2968	−0.0390
27.3391	3.2595	32	1	−3	2	27.3712	3.2557	−0.0321
27.4179	3.2503	35	0	0	3	27.4213	3.2499	−0.0035
28.8622	3.0908	6	1	−2	−2	28.8515	3.0919	0.0107
29.3874	3.0368	32	1	3	−2	29.3922	3.0363	−0.0048
29.6237	3.0131	10	2	−2	−2	29.6445	3.0110	−0.0208
29.7025	3.0053	8	1	−1	−3	29.7068	3.0048	−0.0043
29.7944	2.9962	7	2	−3	1	29.7342	3.0021	0.0603
30.1621	2.9605	10	2	−2	2	30.1807	2.9587	−0.0186
30.2540	2.9517	9	2	2	−1	30.2538	2.9517	0.0002
30.3984	2.9380	9	3	0	−2	30.4250	2.9355	−0.0266
30.7267	2.9074	13	1	3	−1	30.7099	2.9089	0.0168
30.9762	2.8845	10	1	1	2	30.9968	2.8827	−0.0206
31.1206	2.8715	8	3	−1	0	31.1263	2.8710	−0.0057
31.2782	2.8574	6	1	−3	−1	31.2766	2.8575	0.0016
31.9872	2.7956	12	3	−2	0	31.9856	2.7958	0.0016
32.7094	2.7355	9	0	2	−4	32.6935	2.7368	0.0158
32.7619	2.7313	8	2	1	−4	32.7650	2.7310	−0.0031
33.1295	2.7018	8	0	1	3	33.1238	2.7023	0.0057
33.2477	2.6925	11	3	0	0	33.2641	2.6912	−0.0164
34.0093	2.6339	17	2	0	−4	34.0170	2.6333	−0.0078
34.2850	2.6133	14	3	1	−1	34.2900	2.6130	−0.0050
34.9546	2.5648	12	1	3	−4	34.9743	2.5634	−0.0196
35.1647	2.5500	8	3	−1	1	35.1544	2.5507	0.0103
35.7950	2.5065	6	0	4	−3	35.8081	2.5056	−0.0131
36.0970	2.4862	6	2	−2	−3	36.1129	2.4851	−0.0159
37.3575	2.4052	6	1	−2	4	37.3313	2.4068	0.0262
38.0271	2.3643	7	3	2	−2	38.0253	2.3644	0.0019
38.7230	2.3234	5	1	2	2	38.7243	2.3234	−0.0012
39.6290	2.2724	8	1	1	3	39.6461	2.2714	−0.0171
40.1017	2.2467	7	2	2	1	40.1198	2.2457	−0.0181
42.2813	2.1358	6	4	−1	0	42.2822	2.1357	−0.0009

Continued

https://doi.org/10.1017/S0885715614000475 Published online by Cambridge University Press

Table I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
43.3449	2.0858	9	3	1	−5	43.3544	2.0853	−0.0096
45.1175	2.0079	5	3	−3	−3	45.1301	2.0073	−0.0126
47.2577	1.9218	8	3	−4	−2	47.2599	1.9217	−0.0022
49.2010	1.8504	5	3	−5	2	49.2151	1.8499	−0.0141

a pseudo-Voigt (Sánchez-Bajo *et al.*, 1997) peak-shape function was employed, and variables such as cell parameters, atomic coordinates, thermal vibration, and preferred orientation were adjusted by least-squares methods to obtain the final crystal structure. After Rietveld refinement, the R_{wp} was 9.11%. The crystal structure results of bisacodyl from single-crystal X-ray diffraction were also obtained, but not been reported in this paper. Results showed that both single-crystal and powder diffraction methods can get similar structure data.

III. RESULTS

Pawley refinement results confirmed that bisacodyl is triclinic with space group P-1 and unit-cell parameters: $a =$ 9.081(3) Å, $b = 10.631(5)$ Å, $c = 11.549(6)$ Å, $\alpha = 111.492$ (4)°, $\beta = 108.082(3)°$, $\gamma = 101.501(3)°$, unit-cell volume $V =$ 922.368 Å^3, and $Z = 2$. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, and $\Delta 2\theta$ are listed in Table I.

ACKNOWLEDGEMENT

The authors would like to thank the State Key Laboratory of Polymer Materials Engineering in Sichuan university for the software providing.

SUPPLEMENTARY MATERIALS AND METHODS

The supplementary material referred to in this article can be found at http://www.journals.cambridge.org/pdj

Adams, W. J., Meagher, A. P., Lubowski, D. Z., and King, D. W. (**1994**). "Bisacodyl reduces the volume of polyethylene glycol solution required for bowel preparation," Dis. Colon Rectum **37**, 229–234.

Clark, R. E., Godfrey, J. D., Choudhary, A., Ashraf, I., Matteson, M. L., and Bechtold, M. L. (**2013**). "Low-volume polyethylene glycol and bisacodyl for bowel preparation prior to colonoscopy: a meta-analysis," Ann. Gastroenterol. **26**, 319–324.

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.

Li, S. S., Wu, X. Q., Pan, Q. Q., Cheng, Q., and Li, H. (**2014**). "Comparison of the accuracy of powder and single-crystal X-ray diffraction techniques in determining organic crystal structure," Chin. Sci. Bull. **59**, 497–501.

Neumann, M. A. (**2003**). "*X-Cell*: a novel indexing algorithm for routine tasks and difficult cases," J. Appl. Crystallogr. **36**, 356–365.

Sánchez-Bajo, F., and Cumbrera, F. L. (**1997**). "The use of the Pseudo-Voigt function in the variance method of X-ray line-broadening analysis," J. Appl. Crystallogr. **30**, 427–430.

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

https://doi.org/10.1017/S0885715614000475 Published online by Cambridge University Press