X-ray powder diffraction data for meloxicam, $C_{14}H_{13}N_3O_4S_2$

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

(Received 27 November 2013; accepted 26 February 2014)

X-ray powder diffraction data, unit-cell parameters, and space group for meloxicam, $C_{14}H_{13}N_3O_4S_2$, are reported [$a = 6.997(2)$ Å, $b = 8.113(2)$ Å, $c = 13.604(4)$ Å, $\alpha = 85.774(2)°$, $\beta = 88.311(1)°$, $\gamma = 74.994(1)°$, unit-cell volume $V = 743.821$ 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/S0885715614000153]

Key words: X-ray powder diffraction, meloxicam

I. INTRODUCTION

Meloxicam (Figure 1), is a non-steroidal anti-inflammatory drug of the enolic acid class of compounds, used to treat chronic rheumatic diseases and mastitis (Fitzpatrick *et al.*, 2013). Meanwhile, it shows a high potency in animal tests for chronic musculoskeletal disorders (Johnston, 2013).

The single crystallographic data of meloxicam were reported by Fabiola *et al.* (1998) and Luger *et al.* (1996), recorded separately in the Cambridge Structural Database with the reference code SEDZOQ and SEDZOQ01. The average values of cell parameters were $a = 6.999(6)$ Å, $b = 8.114(1)$ Å, $c = 13.607(6)$ Å, $\alpha = 85.711(1)°$, $\beta = 88.336(1)°$, $\gamma = 74.871(1)°$, unit-cell volume $V = 743.908$ Å^3, $Z = 2$, and space group P-1. To date, detailed X-ray powder diffraction data for meloxicam have not been reported in the literature.

II. EXPERIMENTAL

A. Sample

Meloxicam, in powder form of more than 99% purity was purchased from Sichuan Sunnyhope Pharmaceutical Co., Ltd., China. The meloxicam sample was also characterized by melting point at 255.1 °C and IR measurements.

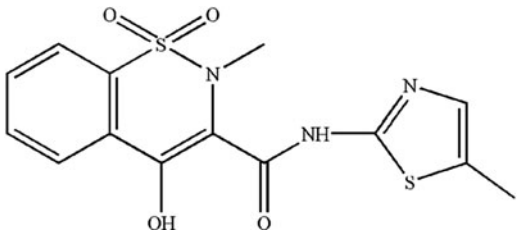


Figure 1. Structural formula of meloxicam.

B. Powder diffraction data collection and reduction

The X-ray powder diffraction measurements were performed on an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) equipped with a PIXcel 1D detection system and Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å, 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 5–50° in 2θ. The structural calculations were mostly conducted using the Reflex module in the software package Material Studio 4.2 (Accelrys Co., Ltd., USA) in the State Key Laboratory of Polymer Materials Engineering (Sichuan University, China).

The unit-cell dimensions can be determined by indexing and analyzing the peak positions in the powder diffraction patterns using X-Cell method (David *et al.*, 2002). The indexing result was then refined Pawley refinement to fit the experimental powder pattern (Harris, 2012; Wu *et al.*, 2013). After Pawley refinement of the unit cell, the final R_{wp} of the structure was converged at 3.49%.

III. RESULTS

The experimental powder diffraction pattern is shown in Figure 2. Pawley refinement results show that meloxicam is

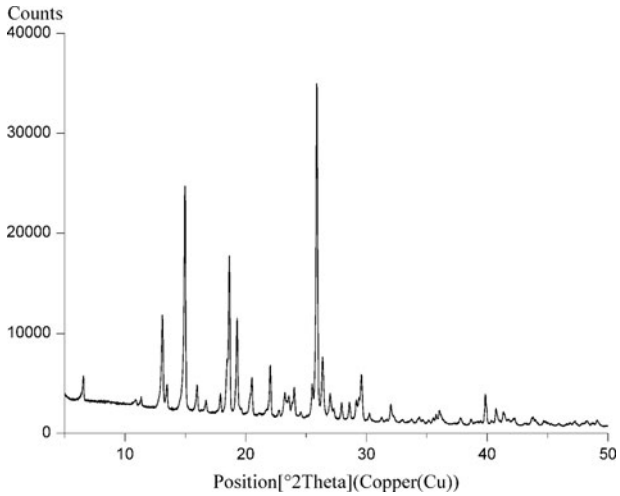


Figure 2. X-ray powder diffraction pattern of the meloxicam, using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

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

https://doi.org/10.1017/S0885715614000153 Published online by Cambridge University Press

TABLE I. Indexed X-ray powder diffraction data of meloxicam, $C_{14}H_{13}N_3O_4S_2$. Only the peaks with relative I_{obs} of 1 or greater are reported [$a = 6.997(2)$ Å, $b = 8.113(2)$ Å, $c = 13.604(4)$ Å, $\alpha = 85.774(2)°$, $\beta = 88.311(1)°$, $\gamma = 74.994(1)°$, unit-cell volume $V = 743.821$ 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 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} (Å)	$\Delta 2\theta$
6.5712	13.4399	17	0	0	1	6.5101	13.5659	0.0611
11.3506	7.7892	11	0	1	0	11.3090	7.8178	0.0416
13.0969	6.7542	34	1	0	0	13.0895	6.7581	0.0075
13.4909	6.5579	14	0	1	−1	13.4422	6.5815	0.0487
14.9746	5.9113	71	1	1	0	14.9455	5.9227	0.0291
15.9856	5.5396	14	1	1	1	15.9433	5.5542	0.0423
16.7078	5.3018	10	0	1	2	16.6938	5.3062	0.0140
17.9026	4.9505	11	0	1	−2	17.8747	4.9582	0.0279
18.4541	4.8038	21	1	0	2	18.4167	4.8135	0.0374
18.6511	4.7535	51	1	0	−2	18.6183	4.7618	0.0328
19.2944	4.5965	33	1	1	2	19.2655	4.6033	0.0290
20.5156	4.3255	16	1	1	−2	20.4858	4.3318	0.0298
22.0387	4.0299	19	0	1	3	22.0061	4.0358	0.0326
22.7477	3.9059	7	0	2	0	22.7300	3.9089	0.0177
23.2335	3.8253	12	0	2	1	23.2270	3.8264	0.0065
23.5880	3.7686	11	1	2	1	23.5966	3.7673	−0.0085
24.0082	3.7036	13	1	1	3	23.9966	3.7054	0.0116
24.5859	3.6179	6	1	2	−1	24.5355	3.6252	0.0505
25.5182	3.4878	14	0	2	2	25.4891	3.4917	0.0291
25.8858	3.4391	100	2	1	0	25.8689	3.4413	0.0169
26.3717	3.3768	22	2	0	0	26.3538	3.3790	0.0179
26.9888	3.3010	12	2	1	−1	26.9684	3.3034	0.0204
27.3039	3.2636	7	1	−1	−3	27.2830	3.2660	0.0209
27.9473	3.1899	9	0	1	4	27.9278	3.1921	0.0195
28.5907	3.1196	9	2	1	2	28.5763	3.1211	0.0144
29.1553	3.0604	10	1	−2	0	29.1658	3.0593	−0.0106
29.5755	3.0179	17	1	0	−4	29.5726	3.0182	0.0029
30.2451	2.9526	6	1	−2	1	30.2333	2.9537	0.0118
31.2561	2.8593	5	0	2	−3	31.2273	2.8619	0.0289
32.0177	2.7930	8	2	1	3	32.0019	2.7944	0.0158
32.9762	2.7140	4	0	0	5	32.9865	2.7132	−0.0103
33.7509	2.6535	4	0	2	4	33.7557	2.6531	−0.0049
34.3811	2.6063	5	0	3	0	34.3856	2.6059	−0.0045
35.1427	2.5515	4	2	2	3	35.1632	2.5501	−0.0205
35.5366	2.5241	5	1	1	5	35.5093	2.5260	0.0273
35.7729	2.5080	5	1	0	−5	35.7651	2.5085	0.0078
36.0356	2.4903	7	0	3	2	36.0359	2.4903	−0.0004
37.7688	2.3799	4	1	3	3	37.7615	2.3804	0.0073
38.6485	2.3277	4	0	3	3	38.6477	2.3278	0.0008
39.8433	2.2606	11	0	0	6	39.8370	2.2610	0.0063
40.7231	2.2138	7	0	1	6	40.7435	2.2128	−0.0204
41.3402	2.1822	6	3	1	−2	41.3296	2.1827	0.0105
42.2856	2.1356	4	3	2	2	42.2752	2.1361	0.0103
43.7168	2.0689	5	1	1	−6	43.6823	2.0705	0.0345
44.6753	2.0267	4	3	0	3	44.7131	2.0251	−0.0378
45.9358	1.9740	3	3	3	0	45.9293	1.9742	0.0064
47.2619	1.9216	4	2	−2	−4	47.2369	1.9226	0.0251
48.2730	1.8837	3	2	4	2	48.2757	1.8836	−0.0027
49.0608	1.8553	4	2	1	−6	49.0707	1.8550	−0.0099

triclinic with space group P-1 and unit-cell parameters: $a = 6.997(2)$ Å, $b = 8.113(2)$ Å, $c = 13.604(4)$ Å, $\alpha = 85.774(2)°$, $\beta = 88.311(1)°$, $\gamma = 74.994(1)°$, unit-cell volume $V = 743.821$ Å^3, and $Z = 2$ (Table I). The results are in good agreement with the single-crystal data and the deviations of the two methods were between 0.01 and 1.60%. All lines of powder and single-crystal data were indexed and are consistent with the P-1 space group.

David W. I. F., Shankland K., McCusker L. B., and Baerlocher C. (Eds.) (**2002**). *Structure Determination from Powder Diffraction Data* (Oxford University Press, Oxford)

Fabiola, G. F., Pattabhi, V., Manjunatha, S. G., Rao, G. V., and Nagarajan, K. (**1998**). "4-Hydroxy-2-methyl-N-(5-methyl-1,3-thiazol-2-yl)-2H-1, 2-benzothiazine-3-carboxamide 1, 1-dioxide," Acta Crystallogr. C, **54**, 2001–2003.

Fitzpatrick, C. E., Chapinal, N., Petersson-Wolfe, C. S., DeVries, T. J., Kelton, D. F., Duffield, T. F., and Leslie, K. E. (**2013**). "The effect of meloxicam on pain sensitivity, rumination time, and clinical signs in dairy cows with endotoxin-induced clinical mastitis," J. Dairy Sci., **96**, 2847–2856.

Harris, K. D. M. (**2012**). "Powder diffraction crystallography of molecular solids," Top. Curr. Chem., **315**, 133–178.

Johnston, L. (**2013**). "Use of meloxicam for the long-term treatment of musculoskeletal disorders in cats," U.S. Patent, Appl. no. 201300 35330.

https://doi.org/10.1017/S0885715614000153 Published online by Cambridge University Press

Luger, P., Daneck, K., Engel, W., Trummlitz, G., and Wagner, K. (**1996**). "Structure and physicochemical properties of meloxicam, a new NSAID," Eur. J. Pharm. Sci., **4**, 175–187.

Wu, X. Q., Tang, P. X., Pan, Q. Q., Cheng, Q., and Li, H. (**2013**). "Crystal structure determination of three polycyclic compounds and comparative Rietveld refinement between MS and GSAS programs," Chin. Sci. Bull., **58**, 2430–2434.

https://doi.org/10.1017/S0885715614000153 Published online by Cambridge University Press