X-ray powder diffraction data for loratadine ($C_{22}H_{23}ClN_2O_2$)

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X-ray powder diffraction data, unit-cell parameters, and space group for loratadine ($C_{22}H_{23}ClN_2O_2$) are reported [$a = 28.302(18)$ Å, $b = 4.996(3)$ Å, $c = 29.154(19)$ Å, $\beta = 109.158(2)°$, unit-cell volume $V = 3894.25$ Å^3, $Z = 8$, and space group $C2/c$]. All measured lines were indexed and are consistent with the $C2/c$ space group. No detectable impurities were observed. © *2014 International Centre for Diffraction Data*. [doi:10.1017/S0885715614000104]

Key words: X-ray powder diffraction, loratadine

I. INTRODUCTION

Loratadine (Figure 1), systematic name ethyl 4-(8-chloro-5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene)-1-piperidinecarboxylate, is a second-generation H_1 histamine antagonist drug used to treat allergies such as hay fever (allergic rhinitis), urticaria (hives), and other skin allergies (Haria *et al.*, 1994). The oral loratadine is well absorbed from the gastrointestinal tract with rapid first-pass hepatic metabolism (Ghosal *et al.*, 2009).

The single crystallographic data of loratadine [$a = 28.299(3)$ Å, $b = 4.993(1)$ Å, $c = 29.137(3)$ Å, $\beta = 109.189(9)°$, unit-cell volume $V = 3888.20$ Å^3, $Z = 8$, and space group $C2/c$] was obtained by Kaminski *et al.* (1999). To date, the detailed X-ray powder diffraction data for loratadine have not been reported.

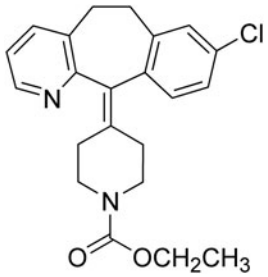

Figure 1. Structural formula of loratadine.

II. EXPERIMENTAL

A. Sample preparation

The title compound was purchased from J&K Chemical Co., Ltd., China and characterized by high-performance liquid chromatography (HPLC), UV and IR. It was recrystallized in methanol, then dried, and ground into powder.

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., Netherlands) with an X'celerator detector and Cu$K\alpha_1$ radiation ($\lambda = 1.54\,056$ 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 0.01 313° 2θ and a counting time of 30 s/step. Data evaluation was performed using the software package Material Studio 4.2 (Accelrys Co., Ltd. USA).

The powder diffraction pattern was pre-treated by subtracting the background, smoothing, and eliminating the $K\alpha_2$ component. Indexing was carried out using peak positions obtained from the powder diffraction profiles by the X-Cell method. Then the best indexing results with 1094 for the value of figure-of-merit were refined using Pawley refinement (Pan *et al.*, 2012). In the indexing step, MC/SA search algorithm in the Powder Solve package (Engel *et al.*, 1999) was used to constantly adjust the conformation, position, and orientation of the trial model in a unit cell of loratadine. The result of Powder Solve ($R_{wp} = 6.74\%$) was refined by Rietveld refinement techniques based on the experimental X-ray powder diffraction pattern. In the Rietveld refinement (Young, 1993), variables defining the structural model and the powder diffraction profiles were adjusted by least-squares

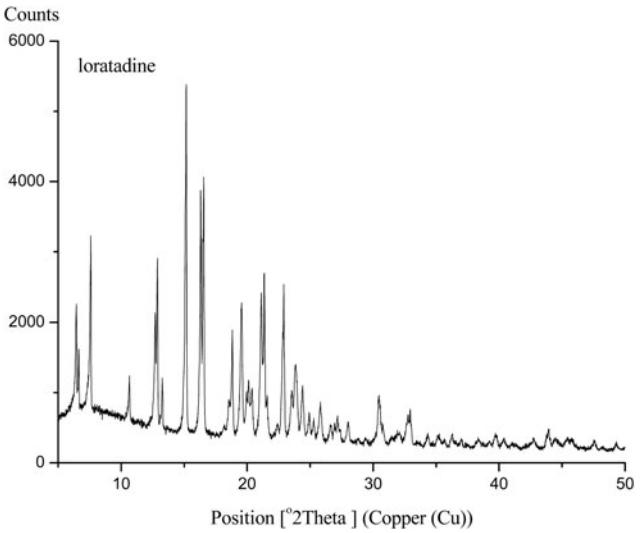

Figure 2. X-ray powder diffraction pattern of the loratadine, using Cu$K\alpha_1$ radiation ($\lambda = 1.54\,056$ Å).

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TABLE I. Indexed X-ray powder diffraction data of loratadine ($C_{22}H_{23}ClN_2O_2$).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
6.4136	13.7697	40	0	0	2	6.4140	13.7689	−0.0003
6.6369	13.3071	28	2	0	0	6.6072	13.3667	0.0296
7.5822	11.6499	59	2	0	−2	7.5491	11.7009	0.0331
10.6678	8.2861	19	2	0	2	10.6218	8.3220	0.0461
12.7030	6.9628	38	4	0	−2	12.6722	6.9797	0.0309
12.8737	6.8709	53	0	0	4	12.8482	6.8845	0.0256
13.2676	6.6677	20	4	0	0	13.2365	6.6833	0.0311
15.1715	5.8350	100	4	0	−4	15.1312	5.8505	0.0403
16.3007	5.4332	60	2	0	4	16.2901	5.4368	0.0106
16.5371	5.3561	68	4	0	2	16.5228	5.3607	0.0143
18.1390	4.8866	7	1	1	−1	18.1401	4.8863	−0.0011
18.5329	4.7836	14	1	1	1	18.5257	4.7854	0.0072
18.8086	4.7141	30	6	0	−2	18.7970	4.7170	0.0117
19.5570	4.5353	41	1	1	2	19.5365	4.5400	0.0205
19.9641	4.4438	15	1	1	−3	19.9658	4.4434	−0.0017
20.0954	4.4150	18	3	1	−1	20.0938	4.4154	0.0016
20.3580	4.3587	16	3	1	0	20.3606	4.3581	−0.0026
21.1064	4.2058	40	3	1	−3	21.0984	4.2073	0.0080
21.3690	4.1547	49	4	0	4	21.3362	4.1610	0.0328
21.5660	4.1172	15	1	1	−4	21.5650	4.1174	0.0010
22.3407	3.9761	7	3	1	2	22.3450	3.9754	−0.0043
22.7871	3.8992	26	6	0	−6	22.7807	3.9003	0.0064
22.9053	3.8794	46	6	0	2	22.8884	3.8822	0.0169
23.5355	3.7769	17	1	1	−5	23.5121	3.7806	0.0234
23.8375	3.7297	24	5	1	−1	23.8394	3.7294	−0.0019
24.3890	3.6466	18	5	1	0	24.3624	3.6505	0.0266
24.9011	3.5728	9	5	1	−4	24.8944	3.5737	0.0067
24.9799	3.5617	9	1	1	5	24.9886	3.5605	−0.0087
25.2556	3.5234	8	8	0	−2	25.2563	3.5233	−0.0007
25.8202	3.4476	14	3	1	−6	25.8105	3.4489	0.0097
26.6080	3.3473	7	5	1	2	26.6055	3.3476	0.0025
26.9231	3.3089	8	4	0	6	26.9127	3.3101	0.0105
27.1726	3.2790	10	6	0	4	27.1537	3.2813	0.0189
27.3696	3.2559	6	8	0	−6	27.3517	3.2580	0.0178
27.9998	3.1840	8	3	1	−7	27.9849	3.1857	0.0149
28.8008	3.0973	4	2	0	8	28.8038	3.0969	−0.0030
29.3916	3.0363	4	5	1	−7	29.3873	3.0368	0.0043
30.3764	2.9401	11	3	1	−8	30.3705	2.9407	0.0059
30.4683	2.9314	15	7	1	1	30.4434	2.9338	0.0250
30.5471	2.9241	13	8	0	−8	30.5346	2.9252	0.0124
30.7572	2.9046	8	4	0	−10	30.7634	2.9040	−0.0062
31.4400	2.8430	4	5	1	−8	31.4449	2.8426	−0.0050
31.6500	2.8246	4	10	0	−4	31.6514	2.8245	−0.0013
31.9126	2.8020	6	10	0	−2	31.9114	2.8021	0.0012
32.0965	2.7864	6	6	0	−10	32.0866	2.7872	0.0099
32.7661	2.7309	10	10	0	−6	32.7405	2.7330	0.0256
32.9368	2.7172	12	3	1	−9	32.9287	2.7178	0.0081
34.3155	2.6111	5	9	1	−1	34.3170	2.6110	−0.0015
35.0377	2.5589	4	9	1	−6	35.0360	2.5590	0.0016
35.6548	2.5160	4	3	1	−10	35.6305	2.5177	0.0243
36.1143	2.4850	3	9	1	−7	36.1015	2.4859	0.0128
36.9941	2.4279	4	4	0	−12	36.9747	2.4292	0.0194
38.3990	2.3423	3	1	1	10	38.3959	2.3425	0.0031
39.2131	2.2955	3	4	0	10	39.2300	2.2946	−0.0169
39.7646	2.2649	4	4	2	2	39.7746	2.2644	−0.0101
40.4079	2.2303	4	11	1	−1	40.3918	2.2312	0.0162
42.7189	2.1149	3	7	1	−12	42.7202	2.1148	−0.0013
43.9269	2.0595	6	6	0	10	43.9260	2.0595	0.0009
44.2682	2.0444	3	2	0	−14	44.2512	2.0451	0.0170
44.3733	2.0398	4	8	2	−3	44.3640	2.0402	0.0092
44.4915	2.0347	4	3	1	11	44.5001	2.0343	−0.0086
45.4106	1.9956	4	13	1	−4	45.4241	1.9950	−0.0135
45.9095	1.9751	3	2	2	−9	45.9314	1.9742	−0.0219
47.5246	1.9116	3	10	0	−14	47.5126	1.9121	0.0120
49.3103	1.8465	3	8	0	10	49.2638	1.8481	0.0464

Only the peaks with I_{rel} of 1 or greater are reported [$a = 28.302(18)$ Å, $b = 4.996(3)$ Å, $c = 29.154(19)$ Å, $\beta = 109.158(2)°$, unit-cell volume $V = 3894.25$ Å^3, $Z = 8$, and space group $C2/c$]. All measured lines were indexed and are consistent with the $C2/c$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54\,056$ Å).

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methods for obtaining an optimal fit between the experimental pattern and calculated pattern. After the Rietveld refinement, the final R_{wp} was 8.94%.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results confirmed that loratadine is monoclinic with space group $C2/c$ and unit-cell parameters after Pawley refinement: $a = 28.302(18)$ Å, $b = 4.996(3)$ Å, $c = 29.154(19)$ Å, $\beta = 109.158(2)°$, unit-cell volume $V = 3894.25$ Å^3, and $Z = 8$ (Table I). A comparison of unit-cell parameters from powder data and single-crystal data (Kaminski *et al.*, 1999) displays a significantly consistency, and the deviations of the two methods were between 0.011 and 0.155%. All lines were indexed and are consistent with the $C2/c$ space group.

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