X-ray powder diffraction data for thiamphenicol, $C_{12}H_{15}Cl_2NO_5S$

Hong Qin Yang, Qing Wang, Pei Xiao Tang, Bin Tang, Ya Ping Li, and Hui Li[a)]
College Chemical Engineering, Sichuan University, Chengdu 610065, China

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X-ray powder diffraction data, unit-cell parameters, and space group for thiamphenicol, $C_{12}H_{15}Cl_2NO_5S$, are reported [$a = 17.346(3)$, $b = 15.341(0)$, $c = 5.790$ (2) Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1540.8(3)$ Å^3, $Z = 4$, and space group $P2_12_12_1$]. All measured lines were indexed and are consistent with the $P2_12_12_1$ space group. No detectable impurities were observed. © *2016 International Centre for Diffraction Data*. [doi:10.1017/S0885715615000834]

Key words: X-ray powder diffraction data, thiamphenicol

I. INTRODUCTION

Thiamphenicol (Figure 1), systematic name 2, 2-dichloro-N-[(1R, 2R)-2-hydroxy -1- (hydroxymethyl)-2-[4-(methylsulfonyl) phenyl] ethyl] acetamide, is a synthetic derivative of chloramphenicol (Shin and Kim, 1983). It has been reported that thiamphenicol possesses a variety of therapeutic effects in respiratory infections, bacterial prostatitis, and venereal diseases (Ma *et al*., 2012). Compared with other chloramphenicol antibiotics, thiamphenicol has a similar antibacterial spectrum, satisfactory pharmacokinetic characteristics and less toxic with high bioavailability. Therefore, thiamphenicol has the potential to be a substitution for other chloramphenicol antibiotics in clinical trials (Kowalski, 2007).

The atomic structure based on single-crystal data of thiamphenicol were reported by Shin and Kim (1983) and Ghosh *et al.* (1987), recorded separately in Powder Diffraction File (PDF)-4/Organic 2015 database with the PDF numbers 02-060-8605 and 02-060-8606. The average values of cell parameters were $a = 5.780(1)$, $b = 15.307(7)$, $c = 17.329(7)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1533.2(7)$ Å^3, $Z = 4$, and space group $P2_12_12_1$. The experimental pattern 00-057-1620 found in PDF™ is unindexed. The detailed X-ray powder diffraction (XRD) data for thiamphenicol have not been reported in the literature so far.

II. EXPERIMENTAL

A. Sample preparation

The title compound (98% purity) was supplied by DaLian MeiLun Biology Technology Co., Ltd. It was recrystallized by slow evaporation of an ethanol solution (analytical grade) at room temperature. The sample was placed in a vacuum drying oven, and the drying temperature was kept under 50 °C . The physical nature of the compound was characterized by melting point at 164 °C, density at 1.596 g cm^{-3}, and microscope measurements. The sample was ground into powder and sieved through 200-mesh screening.

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

B. Diffraction data collection and reduction

The XRD data were recorded using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) equipped with a PIXcel one-dimensional detector and Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å, generator setting: 40 kV and 40 mA). The diffractometer was operated in the angular range from 4 to 60°2θ with a step size of 0.013 13°2θ and a counting time of 90 ms step^{-1}. All data were collected in the constant environments at a controlled relative humidity of 60% and a controlled temperature of 25 °C.

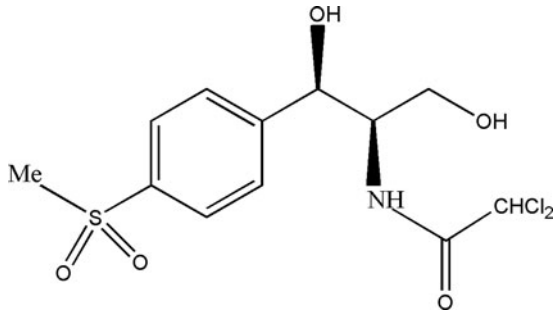

Figure 1. Structural formula of thiamphenicol.

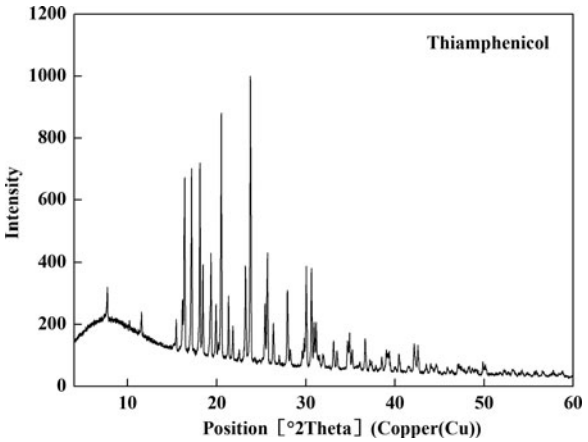

Figure 2. XRD pattern of the thiamphenicol recrystallized in ethanol, using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

TABLE I. XRD data of thiamphenicol recrystallized in ethanol. All measured lines were indexed and are consistent with the $P2_12_12_1$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}/I_o	h	k	l	$2\theta_{cal}$(°)	d_{cal}(Å)	$\Delta 2\theta$
7.7193	11.4433	320	1	1	0	7.6994	11.4729	0.0199
11.5665	7.6443	240	0	2	0	11.5457	7.6580	0.0207
15.4530	5.7293	215	2	2	0	15.4338	5.7364	0.0192
16.1621	5.4796	280	1	0	1	16.1508	5.4833	0.0113
16.3984	5.4011	673M	0	1	1	16.3763	5.4084	0.0221
16.3984	5.4011	M	3	1	0	16.3965	5.4017	0.0019
17.1731	5.1592	702	1	1	1	17.1620	5.1625	0.0111
18.1054	4.8956	719	1	3	0	18.1001	4.8970	0.0053
18.4599	4.8023	393	2	0	1	18.4387	4.8078	0.0212
19.3396	4.5858	430	2	1	1	19.3341	4.5871	0.0055
19.9173	4.4541	264	1	2	1	19.8983	4.4583	0.0190
20.1537	4.4024	142	2	3	0	20.1747	4.3978	−0.0211
20.5082	4.3271	881	4	0	0	20.4966	4.3295	0.0116
21.3091	4.1662	292	4	1	0	21.3089	4.1662	0.0002
21.8081	4.0720	195	2	2	1	21.8089	4.0719	−0.0008
22.5040	3.9476	119	3	1	1	22.5088	3.9468	−0.0048
23.2130	3.8286	388M	0	4	0	23.2108	3.8290	0.0022
23.2130	3.8286	M	0	3	1	23.2253	3.8266	−0.0123
23.2130	3.8286	M	3	3	0	23.2398	3.8243	−0.0267
23.7645	3.7410	1000M	1	4	0	23.7794	3.7387	−0.0149
23.7645	3.7410	1000	1	3	1	23.7936	3.7365	−0.0291
25.4189	3.5012	M	2	4	0	25.4135	3.5019	0.0054
25.4189	3.5012	267	2	3	1	25.4268	3.5001	−0.0079
25.6815	3.4659	431	4	0	1	25.6860	3.4653	−0.0045
26.3512	3.3794	204M	4	1	1	26.3468	3.3799	0.0043
26.3512	3.3794	M	5	1	0	26.3597	3.3783	−0.0085
27.9268	3.1922	311M	0	4	1	27.9263	3.1922	0.0005
27.9268	3.1922	M	3	4	0	27.9385	3.1909	−0.0117
27.9268	3.1922	M	3	3	1	27.9507	3.1895	−0.0239
28.2288	3.1587	119M	4	2	1	28.2431	3.1571	−0.0143
28.2288	3.1587	M	5	2	0	28.2551	3.1558	−0.0263
29.5812	3.0173	114	1	5	0	29.5906	3.0164	−0.0093
29.8044	2.9952	156	2	4	1	29.8048	2.9952	−0.0004
30.0539	2.9709	388	5	0	1	30.0518	2.9711	0.0021
30.6185	2.9174	381	5	1	1	30.6257	2.9167	−0.0071
30.9205	2.8896	207M	0	0	2	30.9119	2.8904	0.0086
30.9205	2.8896	M	2	5	0	30.9400	2.8878	−0.0195
30.9205	2.8896	M	6	0	0	30.9563	2.8863	−0.0358
31.1569	2.8682	M	4	4	0	31.1568	2.8682	0.0000
31.1569	2.8682	M	4	3	1	31.1679	2.8672	−0.0110
31.1569	2.8682	M	5	3	0	31.1789	2.8662	−0.0220
31.4851	2.8391	101M	0	1	2	31.4716	2.8402	0.0135
31.4851	2.8391	M	6	1	0	31.5153	2.8364	−0.0301
33.1133	2.7031	147M	0	5	1	33.0681	2.7067	0.0451
33.1133	2.7031	M	3	5	0	33.0786	2.7058	0.0347
33.1133	2.7031	M	0	2	2	33.0995	2.7042	0.0137
33.1133	2.7031	M	6	2	0	33.1413	2.7009	−0.0280
33.4809	2.6742	113M	1	5	1	33.4813	2.6742	−0.0004
33.4809	2.6742	M	1	2	2	33.5124	2.6718	−0.0315
34.6758	2.5848	147M	3	0	2	34.6795	2.5845	−0.0037
34.6758	2.5848	M	2	5	1	34.6948	2.5834	−0.0190
34.6758	2.5848	M	6	0	1	34.7095	2.5823	−0.0338
34.8990	2.5688	173M	4	4	1	34.8908	2.5693	0.0082
34.8990	2.5688	M	5	4	0	34.9008	2.5686	−0.0018
34.8990	2.5688	M	5	3	1	34.9108	2.5679	−0.0118
35.2010	2.5474	118M	3	1	2	35.1857	2.5485	0.0153
35.2010	2.5474	M	6	1	1	35.2153	2.5464	−0.0144
36.6453	2.4503	153M	3	5	1	36.6389	2.4507	0.0064
36.6453	2.4503	M	3	2	2	36.6676	2.4488	−0.0223
36.6453	2.4503	M	2	6	0	36.6727	2.4485	−0.0274
37.1574	2.4176	86	2	3	2	37.1931	2.4154	−0.0357
37.3544	2.4054	81	4	0	2	37.3777	2.4039	−0.0233
38.4967	2.3366	95M	0	6	1	38.5213	2.3351	−0.0246
38.4967	2.3366	M	3	6	0	38.5304	2.3346	−0.0338
39.0350	2.3056	117M	0	4	2	39.0116	2.3069	0.0235
39.0350	2.3056	M	3	3	2	39.0297	2.3059	0.0053
39.0350	2.3056	M	6	4	0	39.0151	2.3048	0.0199
39.0350	2.3056	M	6	3	1	39.0242	2.3043	0.0109
39.2189	2.2952	107M	4	5	1	39.2208	2.2951	−0.0019
39.2189	2.2952	M	5	5	0	39.2298	2.2946	−0.0109
39.2189	2.2952	M	4	2	2	39.2478	2.2936	−0.0290
40.4268	2.2293	104	2	4	2	40.4306	2.2292	−0.0037
42.1600	2.1416	138M	3	4	2	42.1484	2.1422	0.0116
42.1600	2.1416	M	6	4	1	42.1739	2.1409	−0.0139
42.5671	2.1221	133	2	7	0	42.5830	2.1213	−0.0159
47.0445	1.9300	71	3	7	1	47.0779	1.9287	−0.0334
49.8675	1.8272	80M	3	0	3	49.8497	1.8278	0.0178
49.8675	1.8272	M	2	2	3	49.8835	1.8266	−0.0160
49.8675	1.8272	M	7	5	1	49.8984	1.8261	−0.0309
50.1301	1.8182	71M	0	8	1	50.1535	1.8174	−0.0233
50.1301	1.8182	M	3	8	0	50.1609	1.8172	−0.0308

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 resulting diffractogram was analyzed using various mathematical treatments. To improve the resolution in the calculation processing, the diffractogram should be smoothed before any subsequent processing by removing overlapping peaks and correcting the baseline. Subsequently, the background was subtracted and the $K\alpha_2$ component was eliminated. Automatic indexing of the pretreated experimental XRD pattern was done using DICVOL91 (LoueÈr *et al.*, 1972; Boultif and LoueÈr, 1991). The best result obtained in the indexing of the first 23 peaks of this pattern was an orthorhombic unit cell, which is consistent with the result reported by Shin and Kim (1983) and Ghosh *et al.* (1987). Then the unit-cell parameters were refined using the Pawley method (Pan *et al.*, 2012; Tang *et al.*, 2013) resulting in final R_{wp} of the structure was converged at 4.61%.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. With this pattern, the relative intensity (1000 = maximum) is plotted against 2θ in order to better compare with the PDF. Table I gives the 2θ, d-spacing, relative intensity and hkl for each observed line. The figures of merit are $F_{23} = 67.0$ (0.0067, 51) (Smith and Snyder, 1979) and $M_{23} = 33.8$ (de Wolff, 1968). Indexing results confirmed that thiamphenicol is orthorhombic with space group $P2_12_12_1$ and unit-cell parameters after Pawley refinement are: $a = 17.346(3)$ Å, $b = 15.341(0)$ Å, $c = 5.790$ (2) Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 1540.8(3)$ Å^3, and $Z = 4$. A comparison of the unit-cell parameters from powder data and single-crystal data (Shin and Kim, 1983; Ghosh *et al.*, 1987) displays a significant consistency, and the relative difference in d-spacings of the two patterns compared with the pattern reported here were between 0.096 and 0.493%. The experiment diffraction peaks and d-spacings were consistent with this pattern that provided in PDF 02-060-8605 and 02-060-8606. All lines were indexed and are consistent with the $P2_12_12_1$ space group.

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SUPPLEMENTARY MATERIALS AND METHODS

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