X-ray powder diffraction data for DCT ($C_{12}H_{12}Cl_2N_6O_4S_2 \cdot C_6H_{15}N$)

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X-ray powder diffraction data, unit-cell parameters, and space group for DCT ($C_{12}H_{12}Cl_2N_6O_4S_2 \cdot C_6H_{15}N$) are reported [$a = 18.991(9)$ Å, $b = 19.057(2)$ Å, $c = 7.346(8)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 2659.05$ Å^3, $Z = 4$, and space-group $P2_12_12_1$]. No detectable impurity was observed. © *2015 International Centre for Diffraction Data*. [doi:10.1017/S0885715615000299]

Key words: X-ray powder diffraction, DCT, cefmetazole, intermediate

I. INTRODUCTION

The title compound DCT ($C_{12}H_{12}Cl_2N_6O_4S_2 \cdot C_6H_{15}N$) (Figure 1), systematic name 5-Thia-1-azabicyclo [4.2.0] oct-2-ene-2-carboxylic acid, 7-[(2, 2- dichloroacetyl) amino]-3-[[(1-methyl-1H-tetrazol-5-yl) thio] methyl]-8-oxo-, (6R, 7R)-, compd. with *N*, *N*-diethylethanamine (1:1), is one of the most important intermediates in semi-synthetic *β*-lactam antibiotic cefmetazole sodium (Tang *et al.*, 2014). The CAS register number of DCT is 1597415-32-1.

Presently, the crystal structure of DCT has not been reported in the literature.

II. EXPERIMENTAL

A. Sample preparation

The title compound DCT, with the melting point ranging from 142 to 145°C was obtained from Harbin Pharmaceutical Group Holding Co., Ltd., Harbin, Heilongjiang, China, the

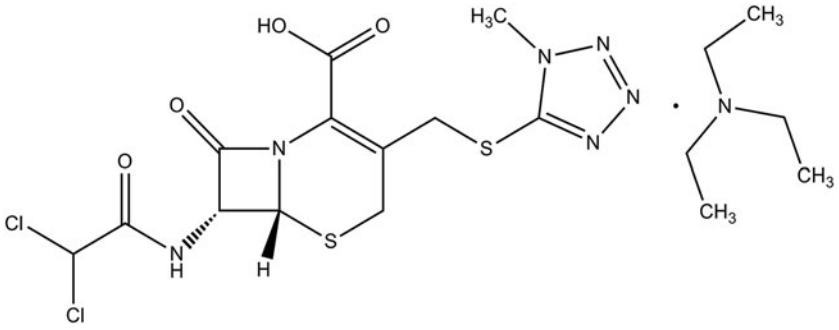


Figure 1. Structural formula of DCT.

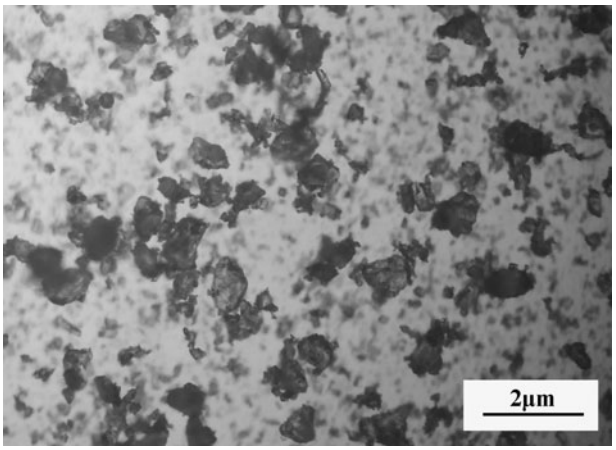


Figure 2. The optical photomicrograph of DCT.

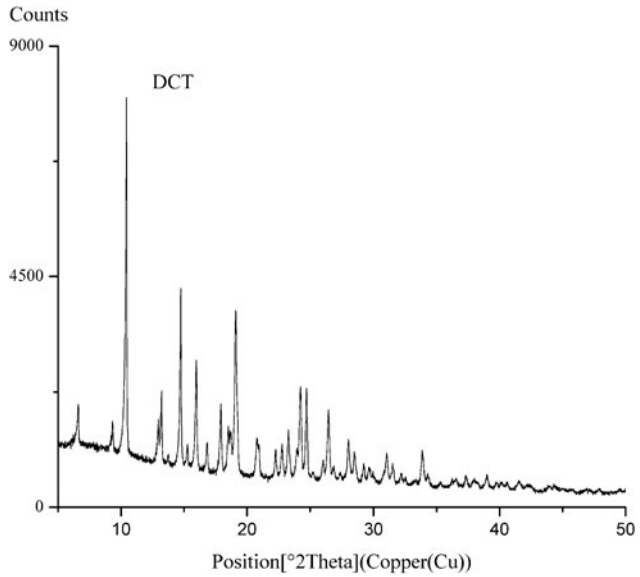


Figure 3. XRD pattern of DCT using Cu*Kα* radiation.

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TABLE I. XRD data of DCT ($C_{12}H_{12}Cl_2N_6O_4S_2 \bullet C_6H_{15}N$). The *d*-values were calculated using Cu$K\alpha_1$ radiation (λ = 1.540 56 Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	*h*	*k*	*l*	$2\theta_{cal}$ (°)	d_{cal} (°)	$\Delta 2\theta$
6.5901	13.4014	25	1	1	0	6.5651	13.4523	0.0250
9.3212	9.4800	21	2	0	0	9.3055	9.4960	0.0157
10.4241	8.4793	100	2	1	0	10.3996	8.4993	0.0245
12.9452	6.8331	22	1	0	1	12.9093	6.8520	0.0358
13.1946	6.7045	27	2	2	0	13.1519	6.7262	0.0428
13.7592	6.4306	13	1	1	1	13.7222	6.4479	0.0371
14.7309	6.0085	53	3	1	0	14.7327	6.0078	−0.0018
15.2692	5.7979	15	2	0	1	15.2353	5.8107	0.0340
15.9520	5.5512	36	2	1	1	15.9321	5.5581	0.0199
16.8317	5.2630	16	3	2	0	16.7998	5.2730	0.0319
17.9084	4.9490	25	2	2	1	17.8644	4.9610	0.0440
18.4993	4.7922	20	3	0	1	18.4853	4.7958	0.0140
18.6174	4.7620	18	0	4	0	18.6086	4.7643	0.0089
19.0639	4.6515	48	3	1	1	19.0669	4.6508	−0.0030
20.7445	4.2783	17	3	2	1	20.7176	4.2838	0.0269
20.8758	4.2517	15	4	2	0	20.8861	4.2496	−0.0103
22.2545	3.9913	14	4	0	1	22.2750	3.9877	−0.0204
22.7666	3.9027	16	4	1	1	22.7638	3.9032	0.0028
23.2656	3.8201	19	3	3	1	23.2200	3.8275	−0.0455
24.2241	3.6711	29	0	0	2	24.2085	3.6734	0.0155
24.7099	3.6000	29	1	0	2	24.6642	3.6066	0.0457
26.4431	3.3678	24	2	1	2	26.4103	3.3719	0.0328
27.3359	3.2598	8	5	3	0	27.3341	3.2600	0.0018
28.0187	3.1819	17	5	2	1	28.0307	3.1806	−0.0120
28.4914	3.1302	14	1	6	0	28.4682	3.1327	0.0232
29.2267	3.0531	11	4	4	1	29.1795	3.0579	0.0472
29.6731	3.0082	10	2	6	0	29.6328	3.0122	0.0403
29.8964	2.9862	8	3	5	1	29.9204	2.9839	−0.0241
31.0518	2.8777	13	1	4	2	31.0753	2.8756	−0.0235
31.5114	2.8368	11	3	6	0	31.4866	2.8389	0.0248
32.2204	2.7759	8	4	2	2	32.1831	2.7791	0.0373
33.8748	2.6440	14	0	5	2	33.8629	2.6449	0.0119
33.9536	2.6381	12	6	4	0	33.9749	2.6365	−0.0213
34.2687	2.6145	8	5	1	2	34.2548	2.6156	0.0139
35.2798	2.5419	6	5	2	2	35.2402	2.5447	0.0396
36.3827	2.4673	7	2	7	1	36.4139	2.4653	−0.0312
37.3544	2.4054	8	0	6	2	37.3985	2.4026	−0.0441
37.9977	2.3661	7	3	7	1	37.9731	2.3676	0.0247
38.9825	2.3086	8	5	4	2	38.9649	2.3096	0.0176
39.6653	2.2704	6	1	3	3	39.6967	2.2687	−0.0314
39.8622	2.2596	6	8	0	1	39.8739	2.2590	−0.0116
40.2036	2.2412	6	6	6	0	40.1878	2.2421	0.0158
40.6238	2.2190	6	3	2	3	40.5839	2.2211	0.0398
41.5429	2.1720	7	1	7	2	41.5252	2.1729	0.0177
42.3570	2.1321	6	2	7	2	42.3712	2.1314	−0.0142
42.4095	2.1296	6	4	8	0	42.4180	2.1292	−0.0085
44.2609	2.0447	5	4	8	1	44.2536	2.0450	0.0073
44.4710	2.0355	5	0	9	1	44.4918	2.0346	−0.0208

purity was more than 98%. Its structure was characterized by IR (Thermo Nicolef 6700) and high-performance liquid chromatography (Agilent 1200). The sample was screened by 200-mesh test sieve to get suitable particle size. The optical photomicrograph was showed in Figure 2.

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$K\alpha$ radiation (generator setting: 40 kV, 40 mA). The diffraction data were collected over the angular range from 5° to 50°2θ with a step size of 0.013 13°2θ and a counting time of 60 ms step^{-1}. Data evaluation was performed using the software package Material Studio 4.2 (Accelrys Co. Ltd., San Diego, CA).

The first stage of structure determination involves pretreatment of the powder diffraction pattern by subtracting the background, smoothing, and eliminating the $K\alpha_2$ component (Li *et al.*, 2014). The task named "indexing" where the X-ray powder diffraction data (XRD) pattern was carried out using peak positions obtained from the powder diffraction profiles by the X-Cell method (Neumann, 2003; Wu *et al.*, 2014) and then the indexing result was refined using Pawley refinement. After Pawley refinement of the unit cell, the final R_{wp} and R_p of the structure were converged at 5.48 and 4.09%, respectively.

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III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 3. Indexing results show that DCT is orthorhombic with the space-group $P2_12_12_1$ and unit-cell parameters: $a =$ 18.991(9) Å, $b = 19.057(2)$ Å, $c = 7.346(8)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 2659.05$ Å^3, and $Z = 4$. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, I_{cal}, and $\Delta 2\theta$ are listed in Table I.

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