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DATA REPORT

X-ray powder diffraction data for ertapenem side chain, $C_{20}H_{19}N_3O_7S$

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X-ray powder diffraction data, unit-cell parameters, and space group for ertapenem side chain, $C_{20}H_{19}N_3O_7S$, are reported [$a = 4.907(6)$ Å, $b = 18.686(3)$ Å, $c = 22.071(1)$ Å, $\alpha = \gamma = 90°$, $\beta = 90.759(5)°$, unit-cell volume $V = 2023.82$ Å^3, $Z = 4$, $\rho_{cal} = 1.462$ g cm^{-3}, and space group $P2_1/c$]. All measured lines were indexed and are consistent with the $P2_1/c$ space group. No detectable impurity was observed. © *2017 International Centre for Diffraction Data*.
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Key words: X-ray powder diffraction, ertapenem side chain, intermediate

I. INTRODUCTION

Ertapenem is an important first-line carbapenem antibiotic. It provides a new, potent, and convenient choice in the treatment of mixed aerobic/anaerobic polymicrobial infections (Shah and Isaacs, 2003). Ertapenem side chain (Figure 1) is the key intermedium for synthesis of ertapenem, and is also the important raw material for a new ertapenem intermediate by reacting with carbapenem derivative (Sheng, 2014).

Presently, the detailed X-ray powder diffraction (XRD) data for ertapenem side chain have not been reported.

II. EXPERIMENTAL

A. Sample preparation

Ertapenem side chain (High Performance Liquid Chromatography [HPLC] ≥ 98%), was obtained from Shandong Xiya chemical industry Co., Ltd., Shandong, China. The melting point and measured density of ertapenem side chain are 258–259 °C and 1.449 g cm^{-3}, respectively. The Fourier transform-infrared (FT-IR) spectrum of ertapenem side chain was presented in Supplementary Figure SI.

B. Diffraction data collection and reduction

The diffraction pattern for ertapenem side chain was collected at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., The Netherlands) with a PIXcel one-dimensional detector and Cu$K\alpha$ radiation (generator setting: 40 kV and 40 mA). The diffraction data were collected over the angular range from 4° to 50°2θ with a step size of 0.01313°2θ and a counting time of 50 ms per step (Figure 2).

Data evaluation was performed using the software package Material Studio 8.0 (Accelrys Co., Ltd., San Diego, California, USA) in the Analytical & Testing Center (Sichuan University, Chengdu, China). The XRD pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha 2$ component. Automatic indexing results were obtained by X-Cell method (Neumann, 2003). The indexing results were then refined using Pawley (Pawley, 1981), which involves assigning the Miller indices (h, k, l) to each observed peak in the experimental XRD pattern.

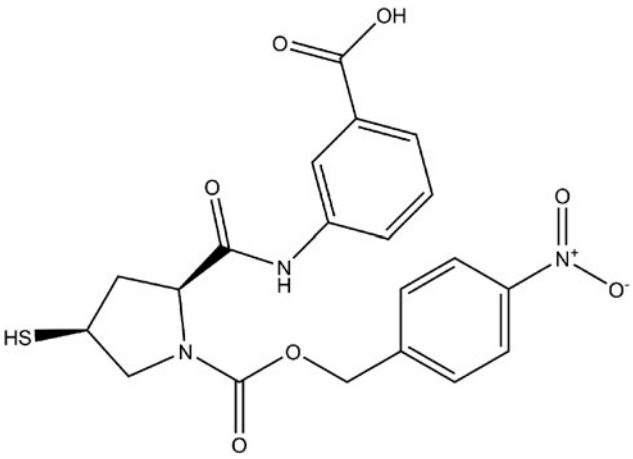


Figure 1. Structural formula of ertapenem side chain.

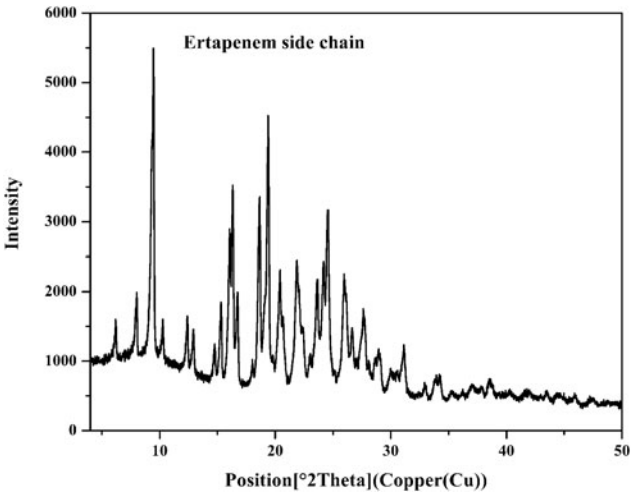


Figure 2. XRD pattern of ertapenem side chain using Cu$K\alpha$ radiation.

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TABLE I. Indexed XRD data for ertapenem side chain. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

$2\theta_{obs}$(°)	d_{obs}(Å)	I_{obs}	h	k	l	$2\theta_{cal}$(°)	d_{cal}(Å)	$\Delta 2\theta$
6.1903	14.2659	29	0	1	1	6.1925	14.2609	0.0022
8.0154	11.0212	36	0	0	2	8.0057	11.0345	−0.0097
9.3153	9.4860	74	0	1	2	9.3000	9.5016	−0.0153
9.4598	9.3415	100	0	2	0	9.4580	9.3432	−0.0017
10.2738	8.6030	29	0	2	1	10.2728	8.6039	−0.0010
12.4009	7.1317	30	0	2	2	12.4032	7.1304	0.0022
12.9130	6.8500	27	0	1	3	12.9225	6.8450	0.0095
14.7644	5.9950	23	0	3	1	14.7654	5.9946	0.0010
15.3027	5.7853	34	0	2	3	15.3172	5.7798	0.0145
16.0512	5.5172	53	0	0	4	16.0509	5.5173	−0.0003
16.3138	5.4289	64	0	3	2	16.3280	5.4243	0.0142
16.7339	5.2936	36	0	1	4	16.7407	5.2914	0.0068
18.0601	4.9077	17	1	0	0	18.0624	4.9071	0.0023
18.6510	4.7536	61	0	3	3	18.6507	4.7536	−0.0003
19.3863	4.5749	82	0	4	1	19.4059	4.5703	0.0196
19.8721	4.4641	18	1	0	2	19.8824	4.4618	0.0103
20.4235	4.3448	42 M	1	2	0	20.4257	4.3444	0.0021
20.4235	4.3448	M	1	1	2	20.4472	4.3398	0.0237
20.6074	4.3065	30	0	4	2	20.6294	4.3019	0.0220
20.8437	4.2582	19	1	2	1	20.8688	4.2531	0.0250
21.8810	4.0586	45	1	2	−2	21.8808	4.0586	−0.0002
22.0386	4.0299	37	1	2	2	22.0588	4.0263	0.0202
22.4062	3.9646	27	1	1	3	22.4037	3.9651	−0.0025
23.0365	3.8576	20	1	3	0	23.0544	3.8546	0.0179
23.3516	3.8062	23	1	3	−1	23.3664	3.8039	0.0148
23.6142	3.7645	40	1	2	−3	23.6420	3.7601	0.0278
24.1919	3.6759	44	0	0	6	24.1766	3.6782	−0.0153
24.5202	3.6274	56 M	1	3	2	24.5213	3.6272	0.0011
24.5202	3.6274	M	1	1	−4	24.5637	3.6211	0.0435
25.9383	3.4322	41	1	2	−4	25.9332	3.4329	−0.0050
26.5948	3.3490	26	1	4	−1	26.5943	3.3490	−0.0005
27.4089	3.2513	24	1	1	−5	27.3942	3.2530	−0.0147
27.6321	3.2256	32	1	4	2	27.6230	3.2266	−0.0091
28.0785	3.1753	18	1	3	−4	28.0774	3.1754	−0.0011
28.6562	3.1126	19	0	6	0	28.6391	3.1144	−0.0172
28.9057	3.0863	21 M	1	4	−3	28.9209	3.0847	0.0152
28.9057	3.0863	M	1	2	5	28.9832	3.0782	0.0775
29.1158	3.0645	19	1	4	3	29.1268	3.0633	0.0110
30.0349	2.9728	16	1	5	0	30.0305	2.9732	−0.0044
30.3238	2.9451	15	1	5	1	30.3413	2.9434	0.0175
30.5339	2.9253	15	1	1	−6	30.5314	2.9255	−0.0025
31.1379	2.8699	22	0	6	3	31.1597	2.8680	0.0219
31.6762	2.8224	10	1	2	−6	31.6624	2.8236	−0.0138
32.5297	2.7502	10	1	5	3	32.5461	2.7489	0.0164
33.0024	2.7119	12	0	6	4	32.9998	2.7121	−0.0026
33.8033	2.6495	14	0	7	1	33.7943	2.6502	−0.0091
33.9346	2.6395	15	1	1	−7	33.9046	2.6418	−0.0300
34.1578	2.6228	14	0	5	6	34.1749	2.6215	0.0171
35.2476	2.5441	10	0	6	5	35.2398	2.5447	−0.0079
36.2193	2.4781	11	1	4	6	36.2069	2.4789	−0.0123
37.0727	2.4230	12	2	1	−1	37.0962	2.4215	0.0234
37.1647	2.4172	12	1	0	−8	37.1450	2.4184	−0.0196
37.7161	2.3831	11	2	1	−2	37.7307	2.3822	0.0145
38.3989	2.3423	12	1	2	−8	38.4164	2.3413	0.0175
39.4099	2.2845	10	0	8	2	39.3981	2.2852	−0.0118
40.2240	2.2401	10	1	7	−3	40.2582	2.2383	0.0342
40.4735	2.2269	10	0	8	3	40.4854	2.2263	0.0119
41.7865	2.1599	11	2	4	1	41.7992	2.1593	0.0126
43.4541	2.0808	10	0	3	10	43.4673	2.0802	0.0132
43.9268	2.0595	9	1	3	9	43.9319	2.0593	0.0051
44.5701	2.0312	10	1	5	−8	44.5900	2.0304	0.0198
44.9640	2.0144	10	2	4	4	44.9928	2.0131	0.0287
47.2618	1.9216	9	2	6	−1	47.2595	1.9217	−0.0024
48.2072	1.8861	7	1	9	−2	48.2193	1.8857	0.0121
49.1395	1.8525	8	1	9	−3	49.1266	1.8530	−0.0129

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III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results show that ertapenem side chain is monoclinic with the space group $P2_1/c$ and unit-cell parameters: $a = 4.907(6)$ Å, $b = 18.686(3)$ Å, $c = 22.071(0)$ Å, $\alpha = \gamma = 90°$, $\beta = 90.759(5)°$, unit-cell volume $V = 2023.82$ Å^3, $Z = 4$ and $\rho_{cal} = 1.462$ g cm^{-3}. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, $\Delta 2\theta$ are listed in Table I.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715617000513.

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

Pawley, G. S. (**1981**). "Unit-cell refinement from powder diffraction scans," J. Appl. Crystallogr. **14**(6), 357–361.

Shah, P. M. and Isaacs, R. D. (**2003**). "Ertapenem, the first of a new group of carbapenems," J. Antimicrob. Chemother. **52**(4), 538–542.

Sheng, L. (**2014**). "New ertapenem intermediate, prepared by subjecting carbapenem derivative and ertapenem side chain to condensation reaction to obtain mixture, reacting with acid, and adding to water," CN patent 103772389-A; 103772389–B.

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