X-ray powder diffraction data for beta-methyl vinyl phosphate ($C_{29}H_{27}N_2O_{10}P$)

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X-ray powder diffraction data, unit-cell parameters, and space group for beta-methyl vinyl phosphate (MAP), $C_{29}H_{27}N_2O_{10}P$, are reported [$a = 17.293(4)$ Å, $b = 6.142(6)$ Å, $c = 14.464(8)$ Å, $\alpha = \gamma = 90°$, $\beta = 112.048(0)°$, unit-cell volume $V = 1424.19$ Å^3, $Z = 2$, and space group $P2_1$]. All measured lines were indexed and are consistent with the $P2_1$ space group. No detectable impurity was observed. © *2015 International Centre for Diffraction Data*. [doi:10.1017/S088571561500007X]

Key words: X-ray powder diffraction, beta-methyl vinyl phosphate, meropenem, intermediate

I. INTRODUCTION

Meropenem is a carbapenem antibiotic with broad antibacterial spectrum, ranging from gram-positive bacteria to gram-negative bacteria, including *Staphylococcus aureus*, *Streptococcus pneumonia*, *Haemophilus influenza*, *Klebsiella oxytoca*, *Enterobacter cloacae*, *Proteus vulgaris*, etc (Edwards *et al.*, 1989; Kitzis *et al.*, 1989). The title compound beta-methyl vinyl phosphate (Figure 1), $C_{29}H_{27}N_2O_{10}P$, systematic name 4-Nitrobenzyl (4R, 5R, 6S)-3-[(diphenylphosphono) oxy]-6-[(R)-1-hydroxyethyl]-4-methyl-7-oxo-1-azabicyclo[3.2.0] hept-2-ene-2-carboxylate, with the CAS register number of 90776-59-3 is an intermediate of meropenem and is also used as the original material in semi-synthesis of meropenem.

Presently, the crystal structure of beta-methyl vinyl phosphate has not been reported in the literature.

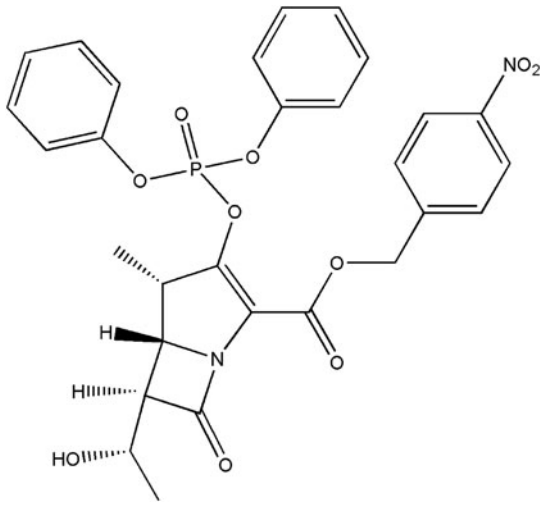


Figure 1. Structural formula of beta-methyl vinyl phosphate.

II. EXPERIMENTAL

A. Sample preparation

The title compound beta-methyl vinyl phosphate, with the purity of more than 98%, was obtained from Qilu Pharmacy Co., Ltd. Jinan, Shandong, China. It was re-crystallized in our Lab. The structure of beta-methyl vinyl phosphate was characterized by infra-red, ultra-violet, and high-performance liquid chromatography.

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) with an X'celerator detector and Cu$K\alpha_1$ radiation ($\lambda = 1.54\,056$ Å, generator setting: 40 kV, 40 mA). The diffraction data (supplementary material for raw 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

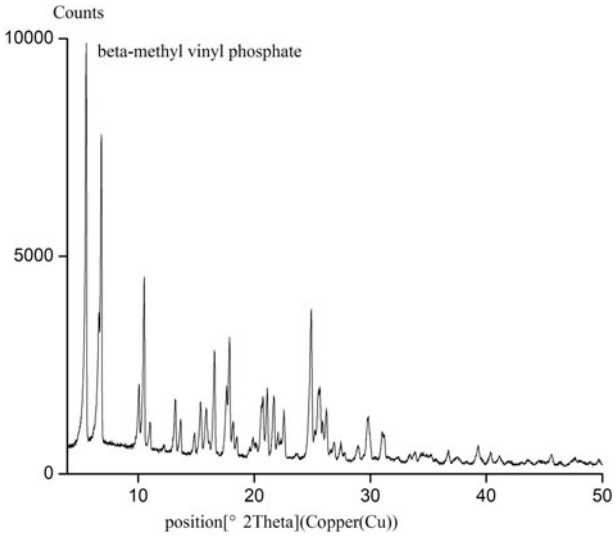


Figure 2. X-ray powder diffraction pattern of beta-methyl vinyl phosphate, using Cu$K\alpha_1$ radiation ($\lambda = 1.54\,056$ Å).

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TABLE I. Indexed X-ray powder diffraction data of beta-methyl vinyl phosphate. Only the peaks with I_{rel} of 1 or greater are presented reported. [a = 17.293(4) Å, b = 6.142(6) Å, c = 14.464(8) Å, $\alpha = \gamma = 90°$, β = 112.048(0)°, unit-cell volume V = 1424.19 Å^3, Z = 2, and space group $P2_1$]. All measured lines were indexed and are consistent with the $P2_1$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation (λ = 1.54 056 Å).

$2\theta_{obs}$(°)	d_{obs}(Å)	I_{obs}	h	k	l	$2\theta_{cal}$(°)	d_{cal}(Å)	$\Delta 2\theta$
5.5228	15.9895	100	1	0	0	5.5093	16.0287	0.0135
6.6126	13.3565	28	0	0	1	6.5877	13.4070	0.0249
6.8227	12.9457	87	1	0	−1	6.8199	12.9510	0.0028
10.0659	8.7808	20	1	0	1	10.0579	8.7878	0.0080
10.5254	8.3984	52	2	0	−1	10.5133	8.4081	0.0121
11.0113	8.0289	8	2	0	0	11.0313	8.0144	−0.0201
12.2061	7.2455	2	1	0	−2	12.2415	7.2247	−0.0354
13.1778	6.7134	19	0	0	2	13.1973	6.7035	−0.0196
13.6636	6.4758	11	2	0	−2	13.6642	6.4755	−0.0006
14.8716	5.9524	7	2	0	1	14.8439	5.9634	0.0277
15.3837	5.7554	13	3	0	−1	15.3640	5.7627	0.0196
15.8563	5.5848	15	0	1	1	15.8576	5.5844	−0.0013
16.5654	5.3474	36	3	0	0	16.5793	5.3429	−0.0139
17.6158	5.0308	23	1	1	1	17.6024	5.0346	0.0134
17.8653	4.9611	41	2	1	−1	17.8694	4.9600	−0.0041
18.1935	4.8723	12	2	1	0	18.1822	4.8753	0.0113
18.4955	4.7934	7	1	0	−3	18.4870	4.7956	0.0086
19.8217	4.4756	5	0	0	3	19.8514	4.4690	−0.0297
19.9005	4.4581	2	2	1	−2	19.9076	4.4565	−0.0071
20.7539	4.2766	22	2	1	1	20.7438	4.2787	0.0102
21.1216	4.2030	24	4	0	−2	21.1165	4.2040	0.0051
21.6862	4.0949	25	1	1	2	21.6858	4.0949	0.0003
22.0538	4.0274	7	3	1	0	22.0324	4.0313	0.0215
22.5528	3.9394	17	1	0	3	22.5540	3.9392	−0.0012
23.6951	3.7520	1	2	1	−3	23.7199	3.7482	−0.0248
24.9162	3.5709	54	2	1	2	24.8960	3.5737	0.0202
25.2182	3.5288	7	4	1	−1	25.2280	3.5274	−0.0097
25.6647	3.4684	23	4	1	−2	25.6578	3.4693	0.0068
25.8879	3.4390	12	5	0	−2	25.8986	3.4376	−0.0107
26.2161	3.3967	20	2	0	3	26.2047	3.3981	0.0114
26.5050	3.3603	1	4	1	0	26.5382	3.3562	−0.0332
26.8726	3.3152	8	1	1	3	26.8662	3.3159	0.0065
27.4766	3.2436	7	5	0	−3	27.4658	3.2449	0.0108
28.9341	3.0835	3	1	1	−4	28.9704	3.0797	−0.0363
29.8401	2.9919	1	5	1	−1	29.8661	2.9893	−0.0260
30.9956	2.8829	7	5	0	1	30.9983	2.8827	−0.0027
31.0218	2.8806	2	6	0	−2	31.0133	2.8813	0.0086
31.1925	2.8652	7	3	0	−5	31.1790	2.8664	0.0135
32.4530	2.7567	2	4	0	−5	32.4404	2.7578	0.0126
33.8580	2.6455	3	3	1	3	33.8774	2.6440	−0.0194
34.3700	2.6072	2	6	1	−2	34.3512	2.6086	0.0188
34.5539	2.5938	1	5	0	−5	34.6030	2.5902	−0.0491
36.7466	2.4439	4	7	0	−3	36.7456	2.4439	0.0010
37.4557	2.3992	2	2	0	−6	37.4784	2.3978	−0.0227
39.3464	2.2882	3	7	0	0	39.3170	2.2898	0.0294
41.1190	2.1935	2	7	0	−5	41.0877	2.1951	0.0313
43.7188	2.0689	1	7	1	−5	43.7594	2.0671	−0.0406
47.6185	1.9082	1	9	0	−2	47.6192	1.9082	−0.0007

30 ms step^{-1}. Data evaluation was performed using the software package Material Studio 4.2(Accelrys Co. Ltd, USA).

The structure determination involves the task named "indexing" the powder XRD pattern, which assigning the Miller indices (h, k, l) to each observed peak in the experimental powder XRD pattern (Harris, 2012). Indexing was carried out using peak positions obtained from the powder diffraction profiles by the X-Cell method and then the indexing result was refined using Pawley-type refinement (Pan *et al.*, 2012). The final R_{wp} and R_p of the structure were 7.78 and 5.75%, respectively.

In order to reduce the difference between the calculated and the measured diffraction data, MC/SA search algorithm in Powder Solve package (Engel *et al.*, 1999) was used to constantly adjust the conformation, position, and orientation of the trial model in the unit cell of beta-methyl vinyl phosphate. Based on the experimental X-ray powder diffraction pattern, the result of Powder Solve was subsequently refined by Rietveld refinement (Young, 1995) techniques. Variables defining the structural model and the powder diffraction profile were adjusted by least-squares methods in order to obtain an optimal fit in the Rietveld refinement.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Indexing results show that beta-methyl vinyl

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phosphate is monoclinic with the space group $P2_1$ and unit-cell parameters: $a = 17.293(4)$ Å, $b = 6.142(6)$ Å, $c = 14.464(8)$ Å, $\alpha = \gamma = 90°$, $\beta = 112.048(0)°$, unit-cell volume $V = 1424.19$ Å^3, $Z = 2$. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, I_{cal}, $\Delta 2\theta$ are listed in Table I.

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SUPPLEMENTARY MATERIALS AND METHODS

The supplementary material for this article can be found at http://www.journals.cambridge.org/PDJ.

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