X-ray powder diffraction data for (*S*)-methyl-2-hexanamido-3-(4-hydroxyphenyl)propanoate, $C_{16}H_{23}NO_4$

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(Received 19 April 2018; accepted 28 August 2018)

X-ray powder diffraction data for (*S*)-methyl-2-hexanamido-3-(4-hydroxyphenyl)propanoate, $C_{16}H_{23}NO_4$, are reported [$a = 17.795(4)$ Å, $b = 15.101(1)$ Å, $c = 12.746(3)$ Å, unit-cell volume $V = 3425.51$ Å^3, $Z = 8$, 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. The single crystallographic data of the compound are also reported [$a = 12.5998(3)$ Å, $b = 17.6856(5)$ Å, $c = 14.6711(5)$ Å, unit-cell volume $V = 3269.25(16)$ Å^3, $Z = 8$, and space group $P2_12_12_1$]. Because of the single-crystal diffraction data were measured at low temperature (110 K), the cell parameters, volume, and calculated density of single-crystal experiment have slight differences with powder diffraction results.
 [doi:10.1017/S088571561800074X]

Key words: tyrosine derivative, X-ray powder diffraction, crystal structure

I. INTRODUCTION

Antimicrobial peptides are natural antimicrobial materials involved in innate immunity mechanisms *in vivo*, and low-molecular weight peptide materials that retain antimicrobial activities against various, microorganisms including bacteria, fungi, and viruses and induce local biophylaxis and systemic immune response. The title compound as a tyrosine derivative has been reported to promote secretion of human antimicrobial peptides *in vivo* (Park *et al*., 2012). Therefore, it is usually used in cosmetics (Commo *et al*., 2014; Jeong *et al*., 2015). So far, the crystal structure of the title compound has not been reported.

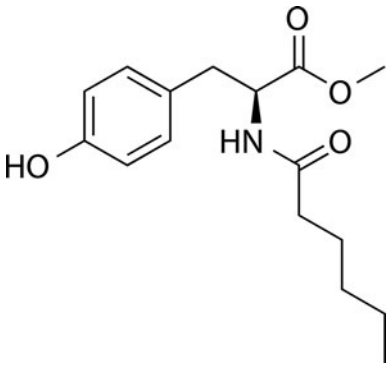

Figure 1. Molecular diagram of the title compound.

II. EXPERIMENTAL

A. Sample preparation

The title compound (Figure 1) was purchased from Shanghai Superlan Chemical Technology Centre. The melting point and measured density of the title compound are 90–91 °C and 1.116 g cm^{-3}, respectively. Crystallization of the title compound at room temperature was successful using methanol as solvent. Then, parts of crystals were dried and ground into powder and mounted on a flat zero background plate.

B. Diffraction data collection and reduction

The X-ray powder diffraction measurement was performed at 298 K using an X'Pert PRO diffractometer (PANalytical Co., Ltd., The Netherlands) with a PIXcel 1D detector and Cu$K\alpha$ radiation (generator setting: 40 kV and 40 mA). The sample was mounted on a flat zero background plate. The diffraction data (Figure 2) were collected over the angular range from 4 to 50° 2θ with a step size of 0.013 13° 2θ and a counting time of 50 ms per step.

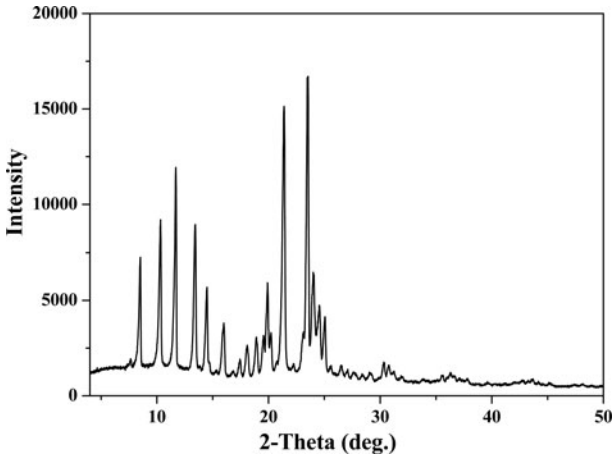

Figure 2. The raw XRD pattern of the title compound.

The software package Material Studio 8.0 (Accelrys Co., Ltd., San Diego, CA, USA) was used to process the data in the

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TABLE I. X-ray powder diffraction data for (*S*)-methyl-2-hexanamido-3-(4-hydroxyphenyl)propanoate, $C_{16}H_{23}NO_4$ at 298 K. The *d*-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	*h*	*k*	*l*	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
7.6740	11.5107	11	1	1	0	7.6717	11.5143	0.0024
8.5144	10.3764	43	1	0	1	8.5258	10.3626	−0.0114
9.9456	8.8862	13	2	0	0	9.9327	8.8977	0.0129
10.3264	8.5594	55	1	1	1	10.3445	8.5444	−0.0181
11.5343	7.6655	34	2	1	0	11.5336	7.6660	0.0008
11.6919	7.5626	71	0	2	0	11.7103	7.5507	−0.0184
13.4382	6.5835	54	2	1	1	13.4670	6.5695	−0.0288
13.6221	6.4950	10	0	2	1	13.6191	6.4965	0.0030
13.8847	6.3728	9	0	0	2	13.8834	6.3734	0.0013
14.4887	6.1084	34	1	2	1	14.5028	6.1025	−0.0141
14.7513	6.0003	9	1	0	2	14.7516	6.0002	−0.0003
15.3815	5.7558	8	2	2	0	15.3780	5.7571	0.0035
15.8805	5.5761	20	1	1	2	15.8803	5.5761	0.0002
16.0118	5.5306	22	3	1	0	16.0395	5.5212	−0.0277
16.8915	5.2445	8	2	2	1	16.8842	5.2468	0.0073
17.4824	5.0686	11	3	1	1	17.4903	5.0663	−0.0079
18.0864	4.9007	16	2	1	2	18.0856	4.9009	0.0007
18.1914	4.8726	13	0	2	2	18.2000	4.8703	−0.0086
18.9136	4.6881	18	1	2	2	18.8753	4.6976	0.0383
18.9136	4.6881	18M	0	3	1	18.9389	4.6819	−0.0253
19.0186	4.6625	14	3	2	0	19.0101	4.6646	0.0085
19.5701	4.5323	19	1	3	1	19.5896	4.5279	−0.0195
19.9246	4.4525	34	4	0	0	19.9410	4.4489	−0.0164
20.2397	4.3839	19	2	3	0	20.2519	4.3813	−0.0121
20.7912	4.2688	11	2	2	2	20.7745	4.2722	0.0167
21.1326	4.2006	30	4	0	1	21.1338	4.2004	−0.0012
21.2770	4.1724	64	3	1	2	21.2737	4.1731	0.0033
21.3952	4.1496	91	2	3	1	21.4281	4.1433	−0.0329
21.4740	4.1346	50	1	0	3	21.4838	4.1327	−0.0098
21.7103	4.0901	9	0	1	3	21.7104	4.0901	−0.0001
22.2749	3.9877	10	1	1	3	22.2837	3.9862	−0.0087
23.0496	3.8554	18	1	3	2	23.0435	3.8564	0.0061
23.1809	3.8339	19	4	2	0	23.1861	3.8330	−0.0052
23.5486	3.7748	100	0	4	0	23.5453	3.7754	0.0033
23.9293	3.7156	32	2	1	3	23.9251	3.7163	0.0042
24.0738	3.6937	38	1	4	0	24.0771	3.6932	−0.0033
24.2313	3.6700	20	4	2	1	24.2269	3.6706	0.0044
24.3889	3.6466	22	4	0	2	24.3796	3.6480	0.0093
24.5333	3.6255	27	1	2	3	24.5350	3.6252	−0.0017
24.6384	3.6103	23	2	3	2	24.6371	3.6105	0.0012
24.9929	3.5599	22	5	0	0	24.9984	3.5591	−0.0055
25.0979	3.5452	20	4	1	2	25.0921	3.5460	0.0058
25.6231	3.4737	9	2	4	0	25.6102	3.4754	0.0130
26.5685	3.3522	9	2	4	1	26.5618	3.3530	0.0067
27.0937	3.2884	8	3	3	2	27.0978	3.2879	−0.0041
27.7109	3.2166	7	5	2	0	27.6862	3.2194	0.0247
27.9078	3.1943	6	1	4	2	27.8978	3.1954	0.0100
28.4462	3.1351	6	1	0	4	28.4303	3.1368	0.0159
29.0633	3.0699	7	1	1	4	29.0503	3.0712	0.0129
29.2340	3.0524	7	2	4	2	29.2446	3.0513	−0.0106
29.9955	2.9766	6	1	5	0	29.9838	2.9777	0.0117
30.2319	2.9538	9	4	3	2	30.2313	2.9539	0.0005
30.3894	2.9389	10	0	5	1	30.3891	2.9389	0.0004
30.4288	2.9352	8	0	2	4	30.4207	2.9359	0.0081
30.6914	2.9106	9	6	1	0	30.6950	2.9103	−0.0036
30.8359	2.8973	9	1	2	4	30.8421	2.8968	−0.0063
31.0459	2.8782	7	4	4	0	31.0420	2.8786	0.0039
31.2560	2.8593	8	2	5	0	31.2486	2.8600	0.0075
31.3611	2.8500	6	3	4	2	31.3719	2.8490	−0.0109
31.8600	2.8065	6	3	0	4	31.8514	2.8072	0.0086
32.0438	2.7908	6	2	5	1	32.0461	2.7906	−0.0023
35.5496	2.5232	6	1	0	5	35.5444	2.5236	0.0053
35.6415	2.5169	6	0	6	0	35.6418	2.5169	−0.0003
36.0092	2.4921	6	1	6	0	36.0086	2.4921	0.0006
36.1930	2.4798	7	3	5	2	36.1986	2.4795	−0.0056

Continued

https://doi.org/10.1017/S088571561800074X Published online by Cambridge University Press

TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
36.3506	2.4694	7	0	6	1	36.3538	2.4692	−0.0032
36.4950	2.4600	6	7	1	1	36.4978	2.4598	−0.0028
36.7182	2.4456	6	1	6	1	36.7144	2.4458	0.0038
37.0990	2.4213	6	2	6	0	37.0902	2.4219	0.0088
37.2041	2.4147	5	0	2	5	37.1935	2.4154	0.0106
37.7161	2.3831	5	4	4	3	37.7155	2.3831	0.0006
37.8868	2.3728	5	2	5	3	37.8898	2.3726	−0.0030
43.2965	2.0880	5	6	5	1	43.3052	2.0876	−0.0087
43.6510	2.0719	5	5	0	5	43.6365	2.0725	0.0145
43.7561	2.0671	5	2	0	6	43.7731	2.0664	−0.0170
45.2529	2.0022	4	7	3	3	45.2654	2.0017	−0.0125
48.1547	1.8881	4	0	8	0	48.1657	1.8877	−0.0110

Analytical & Testing Center (Sichuan University, Chengdu, China). The X-ray powder diffraction pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results were obtained by DICVOL91 method (Boultif and Louër, 1991). The following figures of merit were achieved: $F_{18} = 27.0$ (0.0099, 67) (Smith and Snyder, 1979) and $M_{18} = 13.6$ (de Wolff, 1968). The indexing results were then refined using Pawley ($R_{wp} = 5.45\%$) (Pawley, 1981), which involves assigning the Miller indices (h, k, l) to each observed peak in the experimental powder XRD pattern (Table I).

C. Single-crystal X-ray diffraction

X-ray diffraction data for the title compound were collected on a New Gemini, Dual, Cu at zero, EosS2 diffractometer. The crystal was kept at 110 K during data collection. The structure was solved with Olex2 (Dolomanov *et al.*, 2009), using charge flipping and refined with the SHELXL (Sheldrick, 2008) refinement package using least-squares minimization (Azzam *et al.*, 2017; Park *et al.*, 2017).

TABLE II. Crystal and experimental data of the title compound.

Empirical formula	$C_{16}H_{23}NO_4$
Formula weight	293
Temperature/K	110
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a/Å	12.5998 (3)
b/Å	17.6856 (5)
c/Å	14.6711 (5)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	3269.25 (16)
Z	8
ρ_{calc}/g cm^{-3}	1.192
μ/mm^{-1}	0.692
F(000)	1264.0
Radiation	Cu$K\alpha$ (λ = 1.541 84)
2θ range for data collection/°	8.616–134.108
Index ranges	$-15 \le h \le 15$, $-20 \le k \le 21$, $-17 \le l \le 11$
Reflections collected	11 962
Independent reflections	5848 [R_{int} = 0.0248, R_{sigma} = 0.0311]
Data/restraints/parameters	5848/46/405
Goodness-of-fit on F^2	1.086
Final R indices [$I \ge 2\sigma(I)$]	R_1 = 0.0757, wR_2 = 0.1963
Final R indices [all data]	R_1 = 0.0786, wR_2 = 0.1995
Largest diff. peak/hole/e Å^{-3}	0.75/−0.63

III. RESULTS

Pawley refinement results confirmed that the title compound is orthorhombic with space group $P2_12_12_1$ and unit-cell parameters: $a = 17.795(4)$ Å, $b = 15.101(1)$ Å, $c = 12.746(3)$ Å, unit-cell volume $V = 3425.51$ Å^3, $Z = 8$, $\rho_{cal} = 1.138$ g cm^{-3}. All measured lines (Table I) were indexed and are consistent with the $P2_12_12_1$ space group.

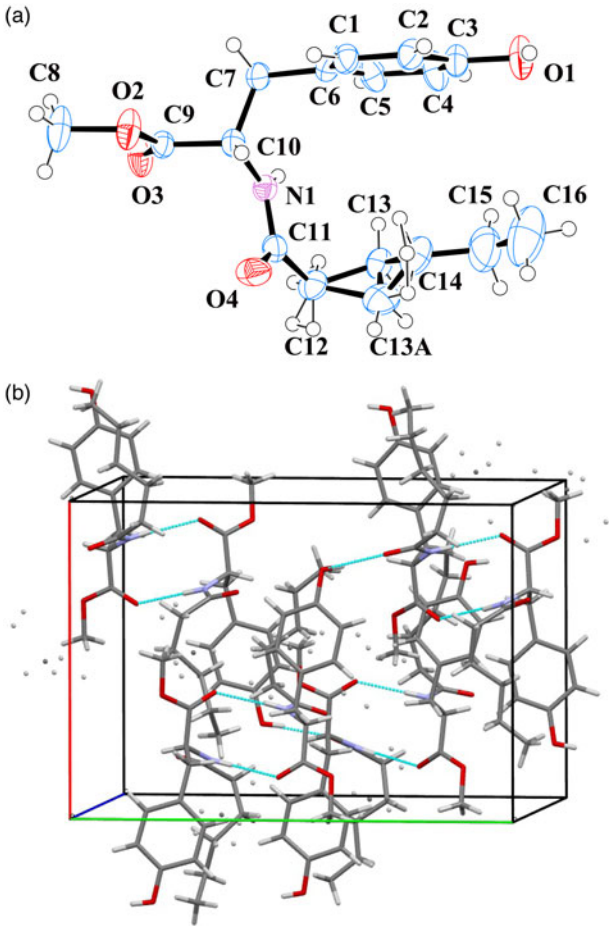

Figure 3. (Color online) (a) ORTEP drawing one of the two independent molecules in the asymmetric unit of the title compound with the labeling of non-H atoms. C13 in hexanoyl group was disordered. (b) Crystal packing of the title compound.

https://doi.org/10.1017/S088571561800074X Published online by Cambridge University Press

The single-crystal experiment was carried out at the temperature of 110 K and the structure solution was obtained [a = 12.5998(3) Å, b = 17.6856(5) Å, c = 14.6711(5) Å, unit-cell volume V = 3269.25(16) Å^3, Z = 8, ρ_{cal} = 1.192 g cm^{-3}, and space group $P2_12_12_1$]. The detailed single-crystal data of title compound and the experimental data were listed in Table II. The figures were drawn with ORTEP-3 (Oak Ridge Thermal Ellipsoid Plot) and Mercury (Figure 3). The title compound as a simple antimicrobial peptide was synthesized by reacting L-tyrosine methyl ester and hexanoyl chloride. The hexanoyl group was rotated by 111.2(4)° (N1–C10–C9) with the methyl propionate. The methyl propionate was rotated by 96.4(6)° (C5–C6–C7–C10) with the plane of benzene ring. C13 in hexanoyl group was disordered. To some degree, the crystal structure was stabilized by some hydrogen bonds. Two strong H bond donors in the title compound were N1–H and O1–H, and two strong acceptors, namely, C9 = O3 and C11 = O4, competed for these H bond donors.

Because of the powder diffraction, data were measured at 298 K and the single-crystal diffraction data were measured at 110 K, the cell parameters, volume, and calculated density of single-crystal experiment have slight differences with powder diffraction results. The deviations of the two methods were between 0.6 and 4.5%.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S088571561800074X.

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