X-ray powder diffraction data for monomenthyl succinate, $C_{14}H_{24}O_4$

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X-ray powder diffraction data, unit-cell parameters, and space group for monomenthyl succinate, $C_{14}H_{24}O_4$, are reported [$a = 19.352(2)$, $b = 30.015(1)$, $c = 5.277(0)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 3065.1(6)$ Å^3, $Z = 8$, and space group *Pba*2]. All measured lines were indexed and are consistent with the *Pba*2 space group. No detectable impurities were observed. © *2016 International Centre for Diffraction Data*. [doi:10.1017/S0885715616000427]

Key words: X-ray powder diffraction data, monomenthyl succinate

I. INTRODUCTION

Monomenthyl succinate, $C_{14}H_{24}O_4$ (Figure 1), systematic name 4-[(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl]oxy-4-oxobutanoic acid, is essentially tasteless and provides a good balance of cooling onset and length of cooling (Erman, 2007). The CAS register number of the compound is 77341-67-4. Monomenthyl succinate is a safe and innocuous additive of mint, so it is widely used in food products, and it has FEMA (Flavor and Extract Manufacture's Association) GRAS (generally recognized as safe) status (Marin and Schippa, 2006).

Presently, the crystal structure of monomenthyl succinate by single-crystal diffraction or powder diffraction has not been reported in the literature.

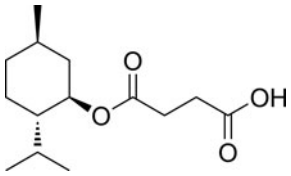

Figure 1. Structural formula of monomenthyl succinate.

II. EXPERIMENTAL

A. Sample preparation

The title compound (purity: 98%) was purchased from J&K Chemical Co., Ltd. (Beijing, China). It was ground into powder ($\rho = 1.06$ g cm^{-3}, $T_{melt} = 61$–63 °C), sieved through a 300-mesh screen, and then mounted on a flat zero background plate. The structure of the compound was characterized by the Fourier transform infrared spectroscopy (FTIR) (Figure S1) and elemental analysis. The elemental analysis showed that the content of C, H, and O were 65.75, 10.39, and 23.86, respectively. The measured FTIR spectrum is consistent with the presumed structure of monomenthyl succinate (Figure 1).

B. Diffraction data collection and reduction

The diffraction pattern for the title compound was collected by an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with an X'celerator detector and Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å, generator setting: 40 kV and 40 mA). The diffractometer was operated in the angular range from 4° to 50°2θ with a step size of 0.01313°2θ and a counting time of 30 ms step^{-1}. The measurement was performed at room temperature and a controlled relative humidity level of 60%. Data evaluation was performed using the Reflex module in the software package Material Studio 4.2 (Accelrys Co., Ltd., USA), which we used to successfully solve the organic crystal structures such as norandrostenedione (Tang *et al.*, 2013), levetiracetam (Xu *et al.*, 2013), and meloxicam (Wu *et al.*, 2014).

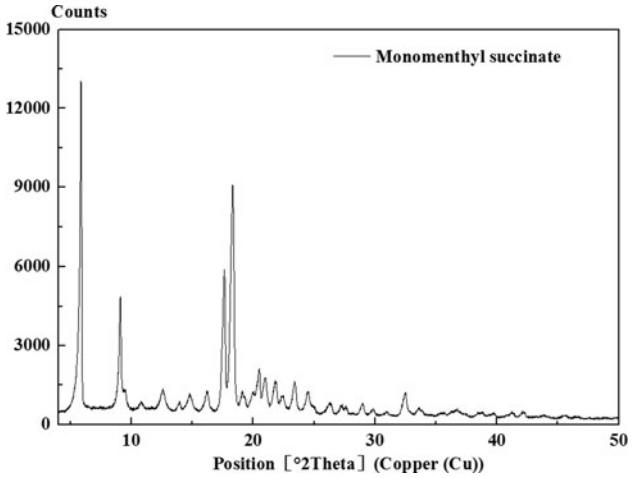

Figure 2. X-ray powder diffraction pattern of monomenthyl succinate, using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

Automatic indexing was carried out using peak positions obtained from the powder diffraction profiles by the X-Cell method (Neumann, 2003). Then the best indexing results 414 for the value of figure of merit were refined using the

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TABLE I. XRD data of monomenthyl succinate.

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
5.8711	15.0409	100	0	2	0	5.8841	15.0076	−0.0131
9.1011	9.7088	37	2	0	0	9.1319	9.6761	−0.0308
9.5082	9.2940	10	2	1	0	9.5957	9.2094	−0.0875
9.9677	8.8665	5	1	3	0	9.9441	8.8875	0.0236
10.8737	8.1297	6	2	2	0	10.8702	8.1323	0.0035
11.7797	7.5064	5	0	4	0	11.7838	7.5038	−0.0041
12.6069	7.0157	11	1	4	0	12.6420	6.9962	−0.0351
12.6988	6.9651	9	2	3	0	12.7166	6.9554	−0.0178
14.0118	6.3152	7	3	1	0	14.0308	6.3067	−0.0190
14.9310	5.9285	8	3	2	0	14.9361	5.9264	−0.0051
16.3096	5.4303	8	3	3	0	16.3362	5.4215	−0.0265
16.7429	5.2907	4	0	0	1	16.7870	5.2770	−0.0440
17.6358	5.0248	45	1	1	1	17.6551	5.0194	−0.0193
17.8459	4.9662	12	0	2	1	17.8024	4.9782	0.0434
18.3054	4.8425	70	1	6	0	18.3023	4.8433	0.0031
19.1327	4.6350	9	2	0	1	19.1417	4.6328	−0.0090
19.2246	4.6130	8	4	2	0	19.2596	4.6047	−0.0351
19.3559	4.5820	8	2	1	1	19.3705	4.5786	−0.0146
19.5660	4.5333	6	1	3	1	19.5479	4.5374	0.0180
19.9599	4.4447	10	2	6	0	19.9641	4.4438	−0.0042
20.0518	4.4245	9	2	2	1	20.0419	4.4267	0.0098
20.1568	4.4017	9	3	5	0	20.1901	4.3945	−0.0332
20.3538	4.3596	13	4	3	0	20.3727	4.3555	−0.0190
20.5376	4.3210	15	0	4	1	20.5591	4.3165	−0.0216
21.0497	4.2170	13	1	4	1	21.0701	4.2129	−0.0204
21.1810	4.1911	8	1	7	0	21.2055	4.1863	−0.0245
21.8244	4.0690	13	4	4	0	21.8398	4.0662	−0.0155
22.4809	3.9516	8	3	6	0	22.4724	3.9531	0.0085
22.5334	3.9425	7	2	4	1	22.5365	3.9420	−0.0031
22.5597	3.9380	7	3	2	1	22.5420	3.9411	0.0177
22.6384	3.9245	6	2	7	0	22.6635	3.9202	−0.0251
22.8485	3.8889	5	1	5	1	22.8851	3.8828	−0.0365
23.1768	3.8345	8	5	1	0	23.1516	3.8387	0.0252
23.4656	3.7880	12	3	3	1	23.5068	3.7815	−0.0411
23.6889	3.7528	5	0	8	0	23.6947	3.7519	−0.0058
24.1353	3.6844	5	1	8	0	24.1424	3.6833	−0.0071
24.2272	3.6706	6	2	5	1	24.2473	3.6676	−0.0201
24.4767	3.6338	10	0	6	1	24.4993	3.6305	−0.0226
24.9100	3.5715	6	4	0	1	24.9485	3.5661	−0.0385
26.3806	3.3757	6	3	5	1	26.3708	3.3769	0.0097
27.0896	3.2889	5	1	9	0	27.1092	3.2866	−0.0196
27.1553	3.2811	6	1	7	1	27.1676	3.2796	−0.0124
27.3916	3.2533	5	5	5	0	27.3950	3.2529	−0.0034
27.6017	3.2290	5	6	0	0	27.6338	3.2254	−0.0321
27.7592	3.2111	4	4	7	0	27.7780	3.2089	−0.0187
28.7571	3.1019	4	5	1	1	28.7350	3.1042	0.0222
29.0591	3.0703	6	6	3	0	29.0642	3.0698	−0.0051
29.9389	2.9821	4	5	3	1	29.9667	2.9794	−0.0279
31.0156	2.8810	4	5	4	1	31.0080	2.8816	0.0075
32.3680	2.7636	8	3	8	1	32.3743	2.7631	−0.0064
32.4861	2.7538	9	7	1	0	32.4968	2.7529	−0.0107
32.5649	2.7473	8	4	9	0	32.5832	2.7458	−0.0183
33.6285	2.6628	5	7	3	0	33.6039	2.6647	0.0246
33.8123	2.6488	4	5	6	1	33.8240	2.6479	−0.0117
34.1143	2.6260	3	2	11	0	34.1117	2.6262	0.0026
34.5607	2.5931	3	7	4	0	34.5468	2.5941	0.0140
34.6658	2.5855	3	1	10	1	34.6644	2.5856	0.0014
35.3485	2.5371	3	2	1	2	35.3583	2.5364	−0.0098
35.6112	2.5190	4	2	10	1	35.6107	2.5190	0.0005
36.7010	2.4467	4	6	8	0	36.7139	2.4458	−0.0129
36.8454	2.4374	4	4	9	1	36.8703	2.4358	−0.0249
37.0817	2.4224	4	2	12	0	37.0936	2.4217	−0.0119
37.4494	2.3995	3	5	8	1	37.4513	2.3993	−0.0019
37.5019	2.3962	3	1	5	2	37.4916	2.3969	0.0103
38.4210	2.3410	4	2	5	2	38.3776	2.3435	0.0435
38.7099	2.3242	4	7	7	0	38.7214	2.3235	−0.0115

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
38.8543	2.3159	4	6	7	1	38.8516	2.3160	0.0027
39.6947	2.2688	3	2	6	2	39.6957	2.2687	−0.0010
41.1127	2.1937	3	3	6	2	41.0964	2.1946	0.0163
41.2178	2.1884	4	2	7	2	41.2079	2.1889	0.0099
41.4279	2.1778	3	8	6	0	41.4277	2.1778	0.0001
42.0450	2.1472	4	8	3	1	42.0347	2.1477	0.0103
42.1500	2.1421	4	0	14	0	42.1123	2.1439	0.0377
43.8176	2.0644	3	8	5	1	43.8079	2.0648	0.0097
44.7367	2.0241	2	9	5	0	44.7314	2.0243	0.0053
45.0518	2.0106	2	3	13	1	45.0683	2.0099	−0.0165
45.3538	1.9980	2	5	6	2	45.3393	1.9986	0.0145
45.5114	1.9914	3	1	15	0	45.5357	1.9904	−0.0243
46.3123	1.9588	2	8	9	0	46.3289	1.9581	−0.0166
46.7194	1.9427	3	5	7	2	46.7028	1.9433	0.0166
47.5335	1.9113	2	3	15	0	47.5369	1.9112	−0.0034
48.0061	1.8936	2	7	2	2	48.0093	1.8935	−0.0031
48.4657	1.8767	2	6	13	0	48.4460	1.8774	0.0197
48.7677	1.8658	2	9	8	0	48.7730	1.8656	−0.0053
49.0303	1.8564	2	5	13	1	49.0374	1.8561	−0.0071
49.0566	1.8555	2	7	12	0	49.0755	1.8548	−0.0190
49.5555	1.8379	2	4	14	1	49.5700	1.8374	−0.0144
49.9494	1.8244	2	5	9	2	49.9367	1.8248	0.0127

All measured lines were indexed and are consistent with the *Pba*2 space group. The *d*-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

Pawley method (Pan *et al*., 2012) resulting in final R_{wp} of the structure was converged at 8.56%.

III. RESULTS

The calculated density is 1.05 g cm^{-3}, which coincides well with the experimental result (1.06 g cm^{-3}). Besides, the elemental compositions calculated for monomenthyl succinate are C: 65.63, H: 9.38, and O: 24.99%, respectively, which are in good agreement with the experimental values and confirmed the high purity.

The experimental X-ray powder diffraction (XRD) pattern is depicted in Figure 2. Indexing results confirmed that monomenthyl succinate is orthorhombic with space group *Pba*2, and unit-cell parameters after Pawley refinement are $a = 19.352(2)$, $b = 30.015(1)$, $c = 5.277(0)$ Å, $\alpha = \beta = \gamma = 90°$, unit-cell volume $V = 3065.1(6)$ Å^3, and $Z = 8$. Table I gives the 2θ, *d*-spacing, relative intensity, and *hkl* for each observed line. All lines were indexed and are consistent with the *Pba*2 space group.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at http://dx.doi.org/10.1017/S0885715616000427

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Erman, M. (**2007**). "New developments in physiological cooling agents," Perfumer Flavorist. **32**(10), 20–35.

Marin, C., Schippa, C. (**2006**). "Identification of monomenthyl succinate in natural mint extracts by LC–ESI–MS–MS and GC–MS," J. Agric. Food Chem. **54**(13), 4814–4819.

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

Pan, Q. P., Guo, J., Duan, Q., Cheng, Q., and Li, H. (**2012**). "Comparative crystal structure determination of griseofulvin: powder X-ray diffraction versus single-crystal X-ray diffraction," Chin. Sci. Bull. **57**, 3867–3871.

Tang, P. X., Wu, X. Q., Pan, Q. Q., Zhang, L. L., Cheng, Q., and Li, H. (**2013**). "X-ray powder diffraction data for norandrostenedione," Powder Diffr. **28**(4), 302–304.

Wu, X. Q., Tang, P. X., Li, S. S., Zhang, L. L., and Li, H. (**2014**). "X-ray powder diffraction data for meloxicam, $C_{14}H_{13}N_3O_4S_2$," Powder Diffr. **29**, 196–198.

Xu, K. L., Liang, B., Wu, X. Q., Zhang, L. L., Tang, P. X., and Li, H. (**2013**). "X-ray powder diffraction data for levetiracetam," Powder Diffr. **29**(1), 51–52.