X-ray powder diffraction data for menthyl lactate, $C_{13}H_{24}O_3$

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X-ray powder diffraction data, unit-cell parameters, and space group for menthyl lactate, $C_{13}H_{24}O_3$, are reported [$a = 5.522(6)$ Å, $b = 11.795(8)$ Å, $c = 17.780(6)$ Å, $\alpha = 50.632(3)°$, $\beta = 90.000(0)°$, $\gamma = 117.632(4)°$, unit-cell volume $V = 716.392(0)$ Å^3, $Z = 2$, and space group $P-1$]. All measured lines were indexed and no detectable impurities were observed. © *2016 International Centre for Diffraction Data*. [doi:10.1017/S0885715616000439]

Key words: X-ray powder diffraction data, menthyl lactate

I. INTRODUCTION

Menthyl lactate is a kind of mint derivatives, and it has advantages in mild and persistent freshening action and cooling on human skin (Erman, 2007). As shown in Figure 1, menthyl lactate ($C_{13}H_{24}O_3$), systematic name (1R,2S,5R)-2-isopropyl-5-methylcyclohexyl(S)-2-hydroxypropionate, with the CAS register number of 61597-98-6. It was tentatively used in personal care products such as shower gel, skin care lotion, and toilet powder freshener (Xie *et al.*, 2009).

Although the compound has been on the market for a long time, the crystal structure of menthyl lactate by single-crystal diffraction or powder diffraction has not been reported in the literature so far.

II. EXPERIMENTAL

A. Sample preparation

The title compound (98% purity) was purchased from Tokyo Chemical Industry Co., Ltd. (Tokyo, Japan) and identified by the Fourier transform infrared spectroscopy (Figure S1) along with an elemental analysis. The elemental analysis showed that the content of C, H, and O were 68.70, 12.09, and 19.21%, respectively. The sample ($\rho = 0.99$ g cm^{-3}, $T_{melt} = 44$–46 °C) was ground into powder and sieved through 300-mesh screening.

B. Diffraction data collection and reduction

The X-ray powder diffraction (XRD) data were recorded at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with a PIXcel 1D detector and Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ 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 0.013 13°2θ and a counting time of 30 ms step^{-1}. The experimental XRD pattern is depicted in Figure 2. The measurement was performed at a controlled relative humidity level of 60%.

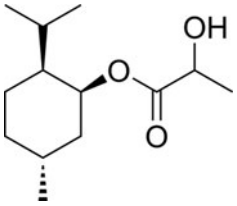

Figure 1. Structural formula of menthyl lactate.

Data evaluation was performed using the Reflex module in the software package Material Studio 4.2 (Accelrys Co., Ltd., USA). The first stage involves the pretreatment of XRD pattern by correcting the baseline, subtracting the background, smoothing, and eliminating the $K\alpha_2$ component before the indexing (Li *et al.*, 2014). Automatic indexing of the pretreated experimental XRD pattern was done using DICVOL91 (LoueÈr and LoueÈr, 1972; Boultif and LoueÈr, 1991) and then the indexing result was refined using the Pawley method (Pawley, 1981). The final R_{wp} of the structure in the Pawley refinement was converged at 9.37%.

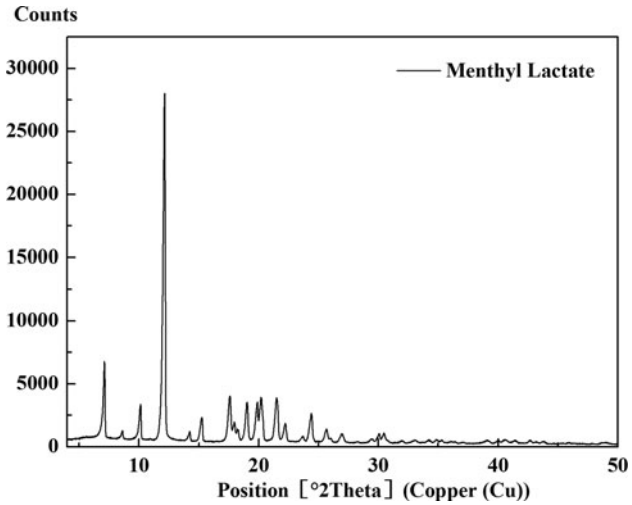

Figure 2. X-ray powder diffraction pattern of menthyl lactate, using Cu$K\alpha_1$ radiation (λ = 1.540 56 Å).

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TABLE I. XRD data of menthyl lactate

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
7.1184	12.4079	24	0	0	1	7.1149	12.4141	0.0035
8.6153	10.2551	2	0	1	1	8.5953	10.2789	0.0200
10.1253	8.7289	12	0	1	2	10.1253	8.7289	0.0000
12.1473	7.2801	100	0	1	0	12.1196	7.2966	0.0277
14.2407	6.2142	4	0	0	2	14.2573	6.2070	−0.0166
15.2330	5.8116	8	0	1	3	15.2729	5.7965	−0.0399
17.5964	5.0360	14	1	−1	1	17.5982	5.0355	−0.0018
17.9509	4.9373	7	0	1	−1	17.9634	4.9339	−0.0125
18.2267	4.8632	5	1	−2	−1	18.2679	4.8524	−0.0412
18.9882	4.6699	12	1	0	2	18.9901	4.6694	−0.0019
19.8548	4.4680	12	0	2	1	19.8373	4.4719	0.0175
20.2225	4.3876	14	1	−2	0	20.2216	4.3878	0.0009
21.5074	4.1283	14	1	−1	2	21.5099	4.1380	−0.0025
22.2051	4.0001	7	1	−2	−3	22.1992	4.0012	0.0059
23.7414	3.7446	2	1	−3	−2	23.7006	3.7510	0.0408
24.3060	3.6589	8	1	−2	1	24.3164	3.6664	−0.0104
24.3848	3.6472	10	0	2	0	24.3775	3.6483	0.0073
25.6584	3.4690	5	0	3	4	25.6720	3.4672	−0.0136
25.9867	3.4259	2	1	1	4	25.9836	3.4263	0.0031
26.9583	3.3046	4	1	−1	3	26.9709	3.3031	−0.0126
27.3392	3.2594	1	0	3	5	27.3446	3.2588	−0.0054
28.2872	3.1523	1	0	3	2	28.2906	3.1598	−0.0034
28.4420	3.1355	1	0	1	5	28.4787	3.1316	−0.0367
29.4531	3.0301	2	1	−3	0	29.4171	3.0338	0.0360
30.0570	2.9706	4	0	2	−1	30.0452	2.9718	0.0118
30.4799	2.9304	3	1	−4	−4	30.4802	2.9303	−0.0003
31.6322	2.8262	1	0	1	−3	31.6355	2.8329	−0.0033
31.9084	2.8024	2	1	−4	−5	31.9017	2.8029	0.0067
31.9741	2.7967	2	0	3	1	32.0079	2.7939	−0.0338
33.0901	2.7049	2	1	−1	−4	33.0916	2.7115	−0.0015
34.0487	2.6309	2	2	−3	−2	34.0433	2.6313	0.0054
34.2456	2.6163	2	1	1	−1	34.2189	2.6247	0.0267
34.8759	2.5704	2	1	−4	−6	34.8444	2.5727	0.0315
35.3223	2.5389	2	0	4	6	35.2785	2.5420	0.0438
36.2020	2.4792	1	1	−4	−1	36.1969	2.4857	0.0051
36.3903	2.4668	1	0	2	−2	36.3883	2.4670	0.0020
38.5392	2.3341	1	2	0	4	38.5284	2.3347	0.0108
38.6574	2.3272	1	2	−4	−4	38.6781	2.3318	−0.0207
39.0119	2.3069	2	1	−5	−6	39.0313	2.3058	−0.0194
39.2482	2.2935	2	2	−3	−4	39.2448	2.2937	0.0034
39.9573	2.2545	2	1	1	−2	39.9897	2.2583	−0.0324
40.3906	2.2313	2	1	3	3	40.4098	2.2303	−0.0192
40.6269	2.2188	2	1	−4	0	40.6067	2.2199	0.0202
41.3885	2.1797	2	1	−5	−7	41.3511	2.1871	0.0374
42.6490	2.1182	2	0	3	−1	42.6564	2.1178	−0.0074
44.5397	2.0326	1	1	3	8	44.5888	2.0304	−0.0491
44.6842	2.0263	1	0	4	1	44.7071	2.0253	−0.0229
45.1700	2.0057	1	2	1	6	45.2057	2.0042	−0.0357
45.3801	1.9969	1	2	0	6	45.3638	1.9975	0.0163
45.4457	1.9941	1	2	−4	−6	45.4100	2.0006	0.0357
45.7609	1.9811	1	1	2	−1	45.7347	1.9822	0.0262
45.8003	1.9795	1	2	−1	−3	45.8207	1.9836	−0.0204
45.9972	1.9715	1	0	5	4	45.9943	1.9716	0.0029
48.5182	1.8748	1	2	−6	−4	48.4991	1.8755	0.0191
48.6233	1.8710	1	1	−6	−8	48.6505	1.8701	−0.0272
48.6758	1.8691	1	2	−5	−7	48.6789	1.8689	−0.0031
48.8859	1.8615	1	1	4	4	48.9105	1.8607	−0.0246
49.1616	1.8517	1	2	−2	−5	49.1842	1.8509	−0.0226

All measured lines were indexed. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.540\,56$ Å).

III. RESULTS

The experimental XRD pattern is depicted in Figure 2. Indexing results confirmed that menthyl lactate is triclinic with space group $P-1$ and unit-cell parameters after the Pawley refinement are [$a = 5.522(6)$ Å, $b = 11.795(8)$ Å, $c = 17.780(6)$ Å, $\alpha = 50.632(3)°$, $\beta = 90.000(0)°$, $\gamma = 117.632(4)°$, unit-cell volume $V = 716.392(0)$ Å^3, and $Z = 2$]. Table I gives the 2θ, d-spacing, relative intensity, and hkl for each observed line. The calculation results of the XRD figure show

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that the merits are $F_{13} = 42.5$ (0.0091, 34) (Smith and Snyder, 1979) and $M_{13} = 23.8$ (de Wolff, 1968). All lines were indexed and are consistent with the $P-1$ space group.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at http://dx.doi.org/10.1017/S0885715616000439.

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