X-ray powder diffraction data for nicotine 2,6-dihydroxybenzoate, $C_{10}H_{15}N_2{\cdot}C_7H_5O_4$

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Nicotine 2,6-dihydroxybenzoate is a nicotine salt that can be used as the nicotine source in tobacco products. X-ray powder diffraction data, unit-cell parameters, and space group for nicotine 2,6-dihydroxybenzoate, $C_{10}H_{15}N_2{\cdot}C_7H_5O_4$, are reported [$a = 7.726(8)$ Å, $b = 11.724(3)$ Å, $c = 9.437(1)$ Å, $\alpha = 90°$, $\beta = 109.081(3)°$, $\gamma = 90°$, unit-cell volume $V = 802.902$ Å^3, $Z = 2$, $\rho_{cal} = 1.309$ g cm^{-3}, and space group $P2_1$] at room temperature. All measured lines were indexed and were consistent with the $P2_1$ space group. © *The Author(s), 2022. Published by Cambridge University Press on behalf of International Centre for Diffraction Data.* [doi:10.1017/S0885715622000070]

Key words: X-ray powder diffraction, nicotine 2,6-dihydroxybenzoate

I. INTRODUCTION

Electronic cigarettes are becoming more and more popular. Compared with conventional cigarettes, electronic cigarettes are relatively less harmful to users and bystanders (Yong *et al.*, 2017). Nicotine salts are important parts of e-liquids and meet the need for more effective and appealing e-cigarette products to provide satisfying alternatives to smoking (Grant *et al.*, 2019). To the best of our knowledge, the structure of nicotine 2,6-dihydroxybenzoate has not been reported in the literature. In this study, nicotine 2,6-dihydroxybenzoate was synthesized. The single-crystal X-ray diffraction (XRD) and detailed powder XRD data of nicotine 2,6-dihydroxybenzoate were supplied at room temperature (25 °C).

II. EXPERIMENTAL

A. Sample preparation

Nicotine and 2,6-dihydroxybenzoic acid each were accurately weighed to achieve a 1:1 molar mixture when combined. Nicotine was added dropwise to the aqueous solution of 2,6-dihydroxybenzoic acid under full stirring. The reaction liquid was transferred to a dark container, and ultrasonic reaction was conducted in a 30–40 °C water bath for 4 h. The reaction solution was freeze-dried to obtain gray solid powder. The solid powder (500 mg) was dissolved in 4 ml ethanol and kept in dark for 1 week to obtain transparent bulk crystal of nicotine 2,6-dihydroxybenzoate. FT-IR and ^{1}H-NMR spectra (provided in the Supplementary Material) confirmed that the obtained compound was nicotine 2,6-dihydroxybenzoate. There was almost no water in nicotine 2,6-dihydroxybenzoate by a Karl Fischer coulometer. Finally, a portion of the crystals were dried at room temperature and ground into powder.

B. Powder diffraction data collection and reduction

The powder XRD measurement was performed at room temperature using an X'Pert PRO diffractometer (PANalytical Co., Ltd., Netherlands) with a PIXcel 1D 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 0.02626° 2θ. The scan speed is 0.01340 °/s. The powder XRD pattern is shown in Figure 1.

The Material Studio 8.0 (Accelrys Co., Ltd., CA, California, USA) software package was used to process the data in the Analytical & Testing Center (Sichuan University, Chengdu, China). The powder XRD pattern was pre-treated by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results obtained by X-Cell algorithm (Neumann, 2003) were refined using the

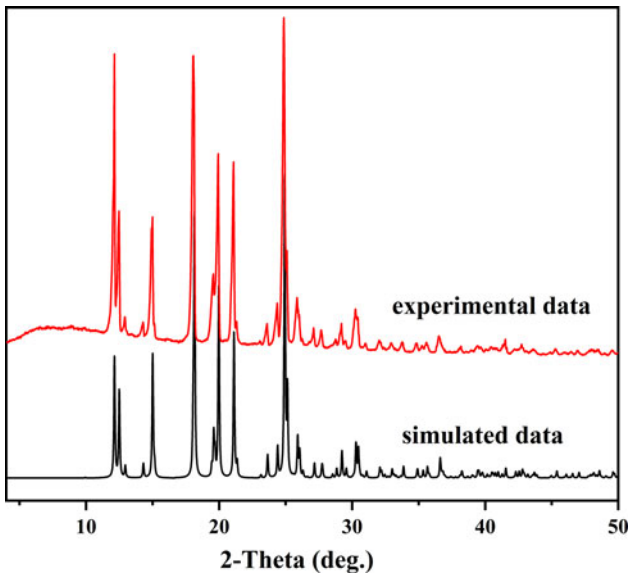


Figure 1. XRD pattern of nicotine 2,6-dihydroxybenzoate using Cu$K\alpha$ radiation (red line) and the simulated pattern of ours (black line).

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TABLE I. Indexed X-ray powder diffraction data for nicotine 2,6-dihydroxybenzoate

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	I_{cal}	$\Delta 2\theta$
12.1276	7.2955	90	1	0	0	12.1103	7.3022	59	−0.0173
12.4690	7.0965	44	0	1	1	12.4600	7.0981	34	−0.0090
12.9155	6.8522	14	1	0	−1	12.9058	6.8539	4	−0.0097
14.2810	6.1999	12	1	1	0	14.2775	6.1983	4	−0.0035
14.9900	5.9082	43	1	1	−1	14.9601	5.9170	36	−0.0300
15.1213	5.8572	12	0	2	0	15.1008	5.8622	4	−0.0206
18.0625	4.9095	89M	1	0	1	18.0269	4.9167	47	−0.0357
18.0625	4.9095	M	0	2	1	18.0939	4.8986	55	0.0314
19.5594	4.5371	26	1	1	1	19.5622	4.5342	15	0.0028
19.6644	4.5131	20	1	0	−2	19.6284	4.5190	5	−0.0361
19.9270	4.4542	61M	0	0	2	19.8949	4.4591	31	−0.0322
19.9270	4.4542	M	1	2	−1	19.9135	4.4549	25	−0.0135
21.0825	4.2126	58	1	1	−2	21.0514	4.2166	50	−0.0311
21.3188	4.1664	12	0	1	2	21.3008	4.1678	6	−0.0180
23.6035	3.7681	12	1	2	1	23.5974	3.7671	6	−0.0061
24.3651	3.6520	18M	2	1	−1	24.3205	3.6567	10	−0.0446
24.3651	3.6520	M	2	0	0	24.3586	3.6511	3	−0.0065
24.8640	3.5798	100M	0	3	1	24.8536	3.5795	100	−0.0105
24.8640	3.5798	M	1	2	−2	24.8569	3.5790	1	−0.0071
25.1004	3.5466	33	0	2	2	25.0704	3.5490	26	−0.0299
25.8619	3.4439	19	1	3	0	25.8353	3.4457	12	−0.0267
25.9407	3.4336	16	2	0	−2	25.9790	3.4269	8	0.0383
26.2296	3.3965	7	1	3	−1	26.2278	3.3950	2	−0.0018
27.0962	3.2898	11	2	1	−2	27.0863	3.2893	5	−0.0099
27.6739	3.2224	10	1	1	2	27.6740	3.2208	5	0.0001
28.7506	3.1041	7	2	2	0	28.7828	3.0992	2	0.0323
29.1970	3.0576	12	1	3	1	29.1654	3.0594	7	−0.0317
29.4596	3.0310	7	1	1	−3	29.4750	3.0279	2	0.0154
30.2474	2.9538	16	1	3	−2	30.2089	2.9560	7	−0.0385
30.3787	2.9413	14	0	3	2	30.3875	2.9391	9	0.0088
30.9827	2.8854	6	0	1	3	31.0092	2.8815	2	0.0265
32.0332	2.7931	7	2	0	−3	31.9857	2.7958	2	−0.0474
32.1645	2.7820	6	0	4	1	32.1179	2.7846	2	−0.0465
32.9260	2.7194	6	2	1	−3	32.9076	2.7195	3	−0.0184
33.7664	2.6536	6	0	2	3	33.7792	2.6513	3	0.0129
34.7643	2.5797	6	2	3	−2	34.7889	2.5766	2	0.0246
34.8430	2.5740	6	3	0	−1	34.8622	2.5714	1	0.0191
35.2369	2.5461	5	1	3	2	35.2598	2.5433	2	0.0229
35.5783	2.5225	6	2	2	−3	35.5460	2.5235	2	−0.0323
36.5237	2.4593	8	1	4	−2	36.5086	2.4591	6	−0.0151
36.6813	2.4491	6	1	3	−3	36.7256	2.4451	2	0.0443
39.6487	2.2724	5	2	3	−3	39.6025	2.2738	1	−0.0462
40.8830	2.2066	5	1	4	2	40.8773	2.2058	2	−0.0057
41.2244	2.1891	5	1	2	−4	41.2236	2.1881	2	−0.0007
41.5132	2.1745	7	3	0	1	41.4920	2.1745	4	−0.0212
42.1960	2.1409	4	1	4	−3	42.1766	2.1408	1	−0.0194
42.4586	2.1283	4	3	2	−3	42.4287	2.1287	1	−0.0299
42.7475	2.1146	6	2	4	1	42.7679	2.1126	1	0.0204
43.0889	2.0986	4	1	3	3	43.1113	2.0965	1	0.0225
43.5878	2.0757	4	0	5	2	43.5727	2.0754	2	−0.0151
45.2947	2.0014	4	3	0	−4	45.2555	2.0021	2	−0.0392
46.4502	1.9543	4	0	6	0	46.4315	1.9541	1	−0.0187
46.9754	1.9336	4	1	1	4	46.9424	1.9340	2	−0.0330
47.8158	1.9016	4	3	3	1	47.8284	1.9002	1	0.0126
47.9733	1.8957	4	1	4	3	47.9640	1.8951	1	−0.0094
48.1309	1.8899	4	4	1	−1	48.0849	1.8907	1	−0.0460
48.2359	1.8860	4	2	4	2	48.2776	1.8836	1	0.0417
48.4197	1.8793	4	1	5	−3	48.4415	1.8776	2	0.0218

The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å), $F_{20} = 43.3$ (0.0145, 32).

Pawley method (Pawley, 1981), which involves the assignment of Miller indices (h, k, l) to each observed peak in the experimental powder XRD pattern. The Pawley refinement was performed on the raw data.

C. Single-crystal XRD

The single-crystal XRD data for nicotine 2,6-dihydroxybenzoate were collected on an Xcalibur, Eos diffractometer. The crystal was kept at 293.15 K during data collection. The

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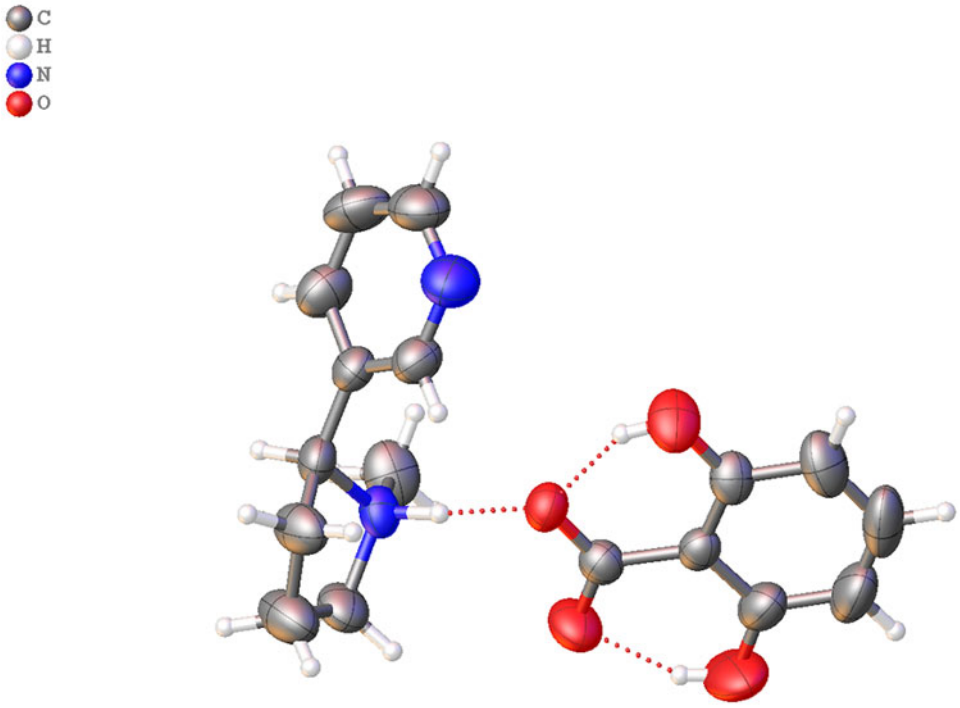


Figure 2. Structural formula of nicotine 2,6-dihydroxybenzoate.

structure was solved with structure solution program Olex2 (Dolomanov *et al.*, 2009) using intrinsic phasing. The structure was refined with the ShelXL (Sheldrick, 2008) refinement package using least squares minimization (Sheldrick, 2015). The single-crystal structure refinement details are provided in the Supplementary Material.

III. RESULTS

The Pawley refinement results confirmed that the title compound was monoclinic with space group $P2_1$ and unit-cell parameters: $a = 7.726(8)$ Å, $b = 11.724(3)$ Å, $c = 9.437(1)$ Å, $\alpha = 90°$, $\beta = 109.081(3)°$, $\gamma = 90°$, unit-cell volume $V = 802.902$ Å^3, $Z = 2$, $\rho_{cal} = 1.309$ 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. The results were in good agreement with single crystal data [$a = 7.7108(7)$ Å, $b = 11.6910(12)$ Å, $c = 9.4114(12)$ Å, $\alpha = 90°$, $\beta = 108.996(12)°$, $\gamma = 90°$, unit-cell volume $V = 802.21(16)$ Å^3, $Z = 2$, $\rho_{cal} = 1.310$ g cm^{-3}]. The principal acquisition parameters and structure refinement values for single-crystal nicotine 2,6-dihydroxybenzoate compound are listed in Supplementary Table SI. The structural formula of 2,6-dihydroxybenzoate is shown in Figure 2. Crystallographic data for nicotine 2,6-dihydroxybenzoate were deposited with the Cambridge Crystallographic Data Center (CCDC) with a supplementary publication number of CCDC-2084167. The comparison of the experimental powder XRD pattern with the simulated pattern of ours is shown in Figure 1. The presented powder pattern for the nicotine 2,6-dihydroxybenzoate compound has been carefully measured and confirmed via a good match to the calculated pattern based on the crystal structure.

IV. DEPOSITED DATA

CIF and RAW data files were deposited with ICDD. You may request this data from ICDD at info@icdd.com.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715622000070.

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