X-ray powder diffraction data for *N,N*-dimethyl-1H-benzo[d]imidazol-2-amine, $C_9H_{11}N_3$

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X-ray powder diffraction data, unit-cell parameters, and space group for *N,N*-dimethyl-1H-benzo[d] imidazol-2-amine, $C_9H_{11}N_3$, are reported [$a = 11.379(3)$ Å, $b = 10.227(5)$ Å, $c = 7.151(1)$ Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$, unit-cell volume $V = 832.318$ Å^3, $Z = 4$, $\rho_{cal} = 1.286$ g cm^{-3}, and space group $P2_12_12$]. All measured lines were indexed and were consistent with the $P2_12_12$ space group. No detectable impurities were observed. © *The Author(s), 2021. Published by Cambridge University Press on behalf of International Centre for Diffraction Data*. [doi:10.1017/S088571562100018X]

Key words: X-ray powder diffraction, *N,N*-dimethyl-1h-benzo[d]imidazol-2-amine, *Cryptotaenia japonica* extractive

I. INTRODUCTION

N,N-Dimethyl-1H-benzo[d]imidazol-2-amine (Figure 1) is an extract of herbal medicine *Cryptotaenia japonica*, which was isolated for the first time by Liu *et al*. (2018). It is one of the special components in this plant, which has not been found in other plants. It is recorded in the pharmacopeia of traditional Chinese medicine that *C. japonica* has many medicinal values, such as anti-inflammatory, detoxification, promoting blood circulation, reducing swelling, treating lung disease, gonorrhea, toothache, anti-infection, and anti-virus (Yang and Xia, 2010). For example, crude flavonoids extracted from this plant have antioxidant activity *in vitro* (Lu *et al*., 2015). Its methanol extract has protective effects against lipopolysaccharide-induced inflammation *in vitro* and *in vivo* (Kang *et al*., 2012). Kaempferol-7-O-β-D-glucuronide and tilianin, isolated from this plant, show anti-melanogenic potential (Seong *et al*., 2016). Hence, it can be speculated that the title compound may play a key role in the pharmacological activity of the designated plant. At present, the detailed X-ray powder diffraction (XRD) data for it have not been reported.

II. EXPERIMENTAL

A. Sample preparation

The sample (Figure 1) was extracted (60% ethanol, heating reflux), isolated, and purified (ethyl acetate extracted, then separated by silica gel column, hydroxypropyl dextran gel column, octadecyl silane bonded silica gel column) from *C. japonica*. The natural sample was analyzed by high-performance liquid chromatography (HPLC). As seen in Figure 2, the chromatogram shows that its purity is satisfactory. The melting point and measured density of it are 325.17 °C (Figure 3) and 1.271 g cm^{-3}, respectively. The crystallization of the title compound at room temperature was successful using chloroform-ethyl acetate (10:1, v/v) as a solvent. Then, the crystals were dried, smashed, and mounted on a flat zero background plate.

B. Diffraction data collection and reduction

XRD measurement was performed at room temperature using a Bruker D8 Advance diffractometer (Bruker, AXS, Germany) with a LynxEye array detector and Cu$K\alpha$ radiation (generator setting: 40 kV and 40 mA). The diffraction data were collected over the angular range from 5 to 50° 2θ with a step size of 0.020464° 2θ (time of diffraction measurement ~10 min) (Figure 4).

The software package Material Studio 8.0 (Accelrys Co., Ltd., CA, USA) was used to process the data in the multi-function computing platform (Guangxi University, Nanning). The XRD pattern was processed by subtracting the background, smoothing, and stripping off the $K\alpha_2$ component. Automatic indexing results were obtained by the DICVOL91 method (Boultif and Louër, 1991). The following figures of merit were achieved: $F_{20} = 19.9$ (0.0157, 46) (Smith

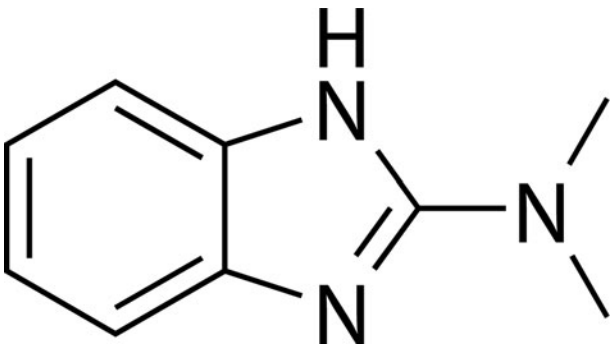

Figure 1. Chemical structure of *N,N*-dimethyl-1H-benzo[d]imidazol-2-amine.

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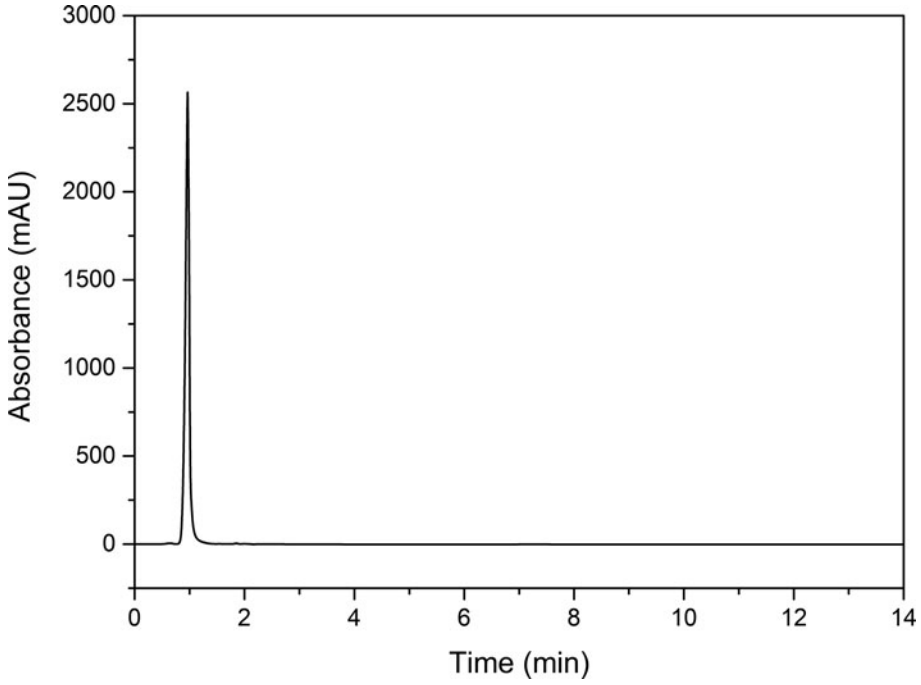

Figure 2. HPLC pattern of *N*,*N*-dimethyl-1H-benzo[d]imidazol-2-amine.

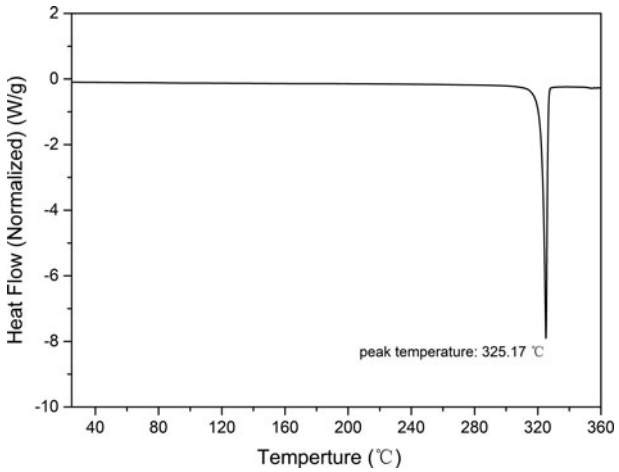

Figure 3. DSC pattern of *N*,*N*-dimethyl-1H-benzo[d]imidazol-2-amine.

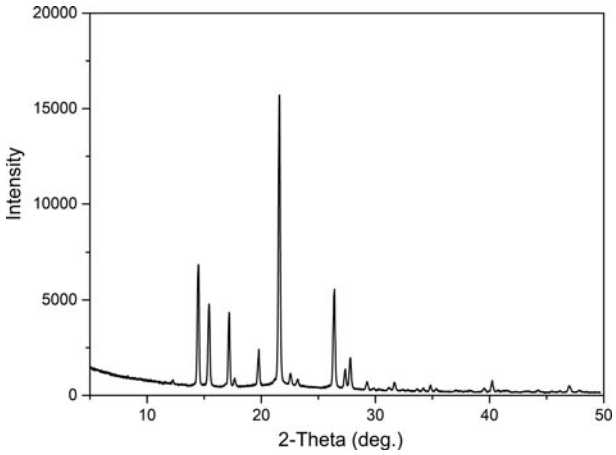

Figure 4. XRD pattern of *N*,*N*-dimethyl-1H-benzo[d]imidazol-2-amine using Cu$K\alpha$ radiation.

TABLE I. Indexed X-ray powder diffraction data for *N*,*N*-dimethyl-1H-benzo[d]imidazol-2-amine.

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
12.3026	7.1887	5	0	0	1	12.2851	7.1987	0.0175
14.5082	6.1004	43	1	0	1	14.5194	6.0956	−0.0112
15.4322	5.7372	30	2	0	0	15.4537	5.7291	−0.0215
16.8629	5.2535	4	1	1	1	16.8886	5.2454	−0.0257
17.1908	5.1540	28	0	2	0	17.2084	5.1487	−0.0176
17.6826	5.0118	6	2	1	0	17.7012	5.0064	−0.0187
19.7839	4.4839	15	2	0	1	19.7888	4.4827	−0.0049
21.1848	4.1905	6	0	2	1	21.1980	4.1878	−0.0132
21.5872	4.1133	100	2	1	1	21.6032	4.1102	−0.0160
22.5857	3.9336	7	1	2	1	22.5869	3.9333	−0.0013
23.1818	3.8338	5	2	2	0	23.2078	3.8295	−0.0261
25.9537	3.4303	4	1	0	2	25.9252	3.4339	0.0286
26.4157	3.3713	35	3	0	1	26.3944	3.3739	0.0213
27.3546	3.2577	9	1	1	2	27.3552	3.2576	−0.0006

Continued

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
27.8017	3.2063	13	3	1	1	27.8021	3.2062	−0.0004
29.2473	3.0511	5	2	0	2	29.2788	3.0478	−0.0315
29.8732	2.9885	3	1	3	1	29.8488	2.9909	0.0244
31.2145	2.8631	3	4	0	0	31.1977	2.8646	0.0167
31.6914	2.8211	4	3	2	1	31.6805	2.8220	0.0109
32.4365	2.7580	2	4	1	0	32.4144	2.7598	0.0222
32.8538	2.7239	2	2	3	1	32.8359	2.7253	0.0179
33.6437	2.6617	2	4	0	1	33.6451	2.6616	−0.0014
34.2100	2.6190	3	3	0	2	34.2023	2.6195	0.0077
34.8359	2.5733	3	0	4	0	34.8209	2.5743	0.0150
35.3277	2.5386	2	3	1	2	35.3268	2.5386	0.0009
37.0416	2.4250	2	0	4	1	37.0565	2.4240	−0.0149
38.2934	2.3486	2	2	4	0	38.2990	2.3482	−0.0056
39.5006	2.2795	2	2	3	2	39.5085	2.2790	−0.0080
40.2308	2.2398	5	4	0	2	40.2006	2.2414	0.0302
40.3202	2.2351	3	5	1	0	40.2841	2.2369	0.0361
40.7524	2.2123	2	2	0	3	40.7333	2.2133	0.0192
41.1697	2.1909	2	4	1	2	41.1841	2.1901	−0.0144
41.4827	2.1751	2	0	2	3	41.4836	2.1750	−0.0010
44.2099	2.0470	2	3	4	1	44.2176	2.0466	−0.0077
45.4916	1.9923	1	3	1	3	45.4628	1.9934	0.0288
46.9819	1.9325	3	5	0	2	46.9652	1.9331	0.0167
47.8314	1.9001	2	5	1	2	47.8363	1.8999	−0.0049

The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5405981$ Å).

and Snyder, 1979) and $M_{20} = 15.6$ (de Wolff, 1968). The indexing results were then refined using Pawley (1981), which involves assigning the Miller indices (h, k, l) to each observed peak in the experimental powder XRD pattern.

III. RESULTS

Pawley refinement results confirmed that the title compound is orthorhombic with space group $P2_12_12$ and unit-cell parameters: $a = 11.379(3)$ Å, $b = 10.227(5)$ Å, $c = 7.151(1)$ Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$, unit-cell volume $V = 832.318$ Å^3, $Z = 4$, $\rho_{cal} = 1.286$ g cm^{-3}. The values of $2\theta_{obs}$, d_{obs}, I_{obs}, h, k, l, $2\theta_{cal}$, d_{cal}, and $\Delta 2\theta$ are listed in Table I.

IV. DEPOSITED DATA

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

ACKNOWLEDGEMENTS

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