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Crystal structure of 2-(piperidin-1-ium-4-yl)-1H-benzo[d]imidazol-3-ium dichloride dihydrate, $C_{12}H_{21}Cl_2N_3O_2$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.21 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.42 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8362, 3043, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2562
$N(\text{param})_{\text{refined}}$:	172
Programs:	SHELX [1], Diamond [2], OLEX2 [3], Mercury [4], PLATON [5]

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Abstract

$C_{12}H_{21}Cl_2N_3O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 7.2704(7)$ Å, $b = 11.4750(12)$ Å, $c = 18.7004(19)$ Å, $\beta = 95.475(2)^\circ$, $V = 1553.0(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0372$, $wR_{\text{ref}}(F^2) = 0.1022$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All of starting materials are commercial. The title compound was synthesized following the reported literature with a slight modification [6]: A mixture of 1,2-diaminobenzene (10.8 g,

0.1 mol), 4-piperidinecarboxylic acid (12.9 g, 0.1 mol) and 6M HCl (100 mL) was dissolved in a flask equipped with a stirrer and reflux condenser, then was refluxed for 4 h at 105 °C. After cooling, the solution was filtered and let evaporate at room temperature in air. Colorless single crystals were obtained after several days, yield: 63% (based on 4-piperidinecarboxylic acid).

Experimental details

All H-atoms from C atoms and N atoms were positioned with idealized geometry and refined isotropically ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.2U_{\text{eq}}(\text{N})$ for all H atoms) using a riding model with C–H = 0.93–0.97 Å and N–H = 0.86–0.90 Å. The water H-atom positions were fixed as found (O–H = 0.85 Å, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$).

Comment

Benzimidazoles that are made of the fusion of benzene rings and imidazole rings, have widely served as ligands in coordination chemistry or functional materials [7–10]. Recently, benzimidazole derivatives have attracted attention because some of them are bioactive and a large number of benzimidazole-based medicines such as albendazole, mebendazole, triclabendazole, have been developed for clinical applications [11]. Encouraged by the above aspects,

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.1664(2)	0.24629(14)	0.11278(9)	0.0381(4)
H1	0.040317	0.242282	0.127083	0.046*
C2	0.2975(3)	0.26465(15)	0.18094(9)	0.0444(4)
H2A	0.282380	0.201499	0.214362	0.053*
H2B	0.424250	0.263669	0.168824	0.053*
C3	0.2583(3)	0.37966(16)	0.21610(10)	0.0501(5)
H3A	0.136045	0.377541	0.232784	0.060*
H3B	0.347406	0.391986	0.257404	0.060*
C4	0.1401(3)	0.46345(17)	0.09884(11)	0.0549(5)
H4A	0.155787	0.527890	0.066410	0.066*
H4B	0.013826	0.464931	0.111534	0.066*
C5	0.1752(3)	0.34989(15)	0.06156(10)	0.0463(4)
H5A	0.296126	0.352400	0.043688	0.056*
H5B	0.083687	0.339790	0.020791	0.056*
C6	0.2046(2)	0.13430(14)	0.07659(9)	0.0362(4)
C7	0.2720(2)	−0.05228(15)	0.06060(9)	0.0383(4)
C8	0.2452(2)	0.00078(15)	−0.00606(9)	0.0373(4)
C9	0.2611(2)	−0.06212(17)	−0.06926(9)	0.0459(4)
H9	0.242361	−0.027420	−0.114306	0.055*
C10	0.3059(2)	−0.17772(17)	−0.06113(10)	0.0492(5)
H10	0.316808	−0.222567	−0.101928	0.059*
C11	0.3356(3)	−0.23040(17)	0.00613(11)	0.0504(5)
H11	0.367145	−0.308920	0.008993	0.060*
C12	0.3194(3)	−0.16872(16)	0.06859(10)	0.0478(4)
H12	0.339188	−0.203501	0.113582	0.057*
Cl1	0.64044(7)	0.51335(5)	0.09740(2)	0.05329(16)
Cl2	0.84727(8)	0.19634(4)	0.25672(3)	0.05816(17)
N1	0.2690(2)	0.47736(13)	0.16458(9)	0.0477(4)
H1A	0.243390	0.544513	0.186429	0.057*
H1B	0.385230	0.482253	0.152009	0.057*
N2	0.2454(2)	0.03434(12)	0.11055(7)	0.0400(3)
H2	0.253903	0.024831	0.156367	0.048*
N3	0.20347(19)	0.11644(12)	0.00618(7)	0.0384(3)
H3	0.180563	0.168544	−0.026510	0.046*
O1W	0.1629(2)	0.25443(13)	0.38418(8)	0.0720(5)
H1WA	0.220673	0.189952	0.386237	0.108*
H1WB	0.067223	0.242652	0.355267	0.108*
O2W	0.7273(3)	0.45430(14)	0.25588(8)	0.0862(6)
H2WA	0.755831	0.382702	0.253326	0.129*
H2WB	0.720261	0.478962	0.212846	0.129*

we report the crystal structure of 2-(piperidin-1-ium-4-yl)-1*H*-benzo[d]imidazol-3-ium dichloride dihydrate.

As shown in the figure, the title compound crystallizes in *P*₂₁/*c* group with two crystal water molecules. The title compound is a 2-substituted benzimidazole derivative with a 4-piperidyl group, which adapts a boat conformation. Both the N atoms from the 4-piperidyl group and the imidazole group are protonated. The protonated title compound as a dication, is countered by two chlorides. There are three

hydrogen bonds: O—H···Cl, N—H···Cl and N—H···O, which link molecules and ions to a three-dimensional supramolecular structure. All bond lengths and angles of the title compound are comparable to its analogues [12, 13].

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