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Synthesis and crystal structure of 6-carboxy-1-(3,5-dicarboxyphenyl)-1*H*-benzo[*d*]imidazol-3-ium-5-carboxylate dihydrate, C₁₈H₁₂N₂O₈

Table 1: Data collection and handling.

Crystal:	Yellow
Size:	0.22 × 0.19 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.14 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7338, 3876, 0.040
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2732
$N(\text{param})_{\text{refined}}$:	287
Programs:	Bruker programs [1], SHELX [2]

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Abstract

C₁₈H₁₂N₂O₈, triclinic, $P\bar{1}$ (no. 2), $a = 8.4592(10)$ Å, $b = 9.8442(11)$ Å, $c = 11.2565(12)$ Å, $\alpha = 66.892(3)^\circ$, $\beta = 86.069(3)^\circ$, $\gamma = 81.658(4)^\circ$, $V = 852.97(17)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0609$, $wR_{\text{ref}}(F^2) = 0.1557$, $T = 153(2)$ K.

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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

Synthesis of the title complex: All solvents and other reagents were of analytical grade and the method of synthesizing the title compound has been improved [3, 4]. The title compound and water as solvent was sealed in a 25 mL Teflon reactor autoclave and heated to 140 °C for 3 days. After cooling down to room temperature, yellow triangular crystals suitable for single crystal X-ray crystallographic analysis were obtained. Elemental analysis-calcd. for C₁₈H₁₂N₂O₈: C,

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.2944(2)	0.4740(2)	0.63143(19)	0.0314(5)
H1	0.2217	0.4207	0.6406	0.047*
O2	0.1421(2)	0.6338(2)	0.46900(19)	0.0339(5)
O3	0.1324(2)	0.9742(2)	0.32567(18)	0.0326(5)
O4	0.2446(2)	0.8542(2)	0.20461(19)	0.0352(5)
O5	0.4598(3)	0.8454(2)	0.93609(18)	0.0362(5)
O6	0.4731(2)	0.6867(2)	1.14270(17)	0.0295(5)
H6	0.4050	0.7525	1.1530	0.044*
O7	0.8244(2)	0.2147(2)	1.27924(16)	0.0273(5)
O8	0.9778(2)	0.1562(2)	1.13317(18)	0.0335(5)
O9	0.1062(3)	0.2800(3)	0.7121(2)	0.0410(6)
H9A	0.1328	0.2107	0.7870	0.061*
O10	0.2198(4)	0.0810(3)	0.9345(2)	0.0610(8)
H10C	0.1349	0.0559	0.9820	0.092*
H10D	0.2868	0.0015	0.9446	0.092*
N1	0.7734(3)	0.9328(2)	0.4379(2)	0.0241(5)
H1A	0.7954	1.0201	0.3822	0.029*
N2	0.7969(3)	0.7171(2)	0.60073(19)	0.0210(5)
C1	0.2646(3)	0.5984(3)	0.5309(2)	0.0229(6)
C2	0.3959(3)	0.6933(3)	0.4995(2)	0.0224(6)
C3	0.5295(3)	0.6441(3)	0.5778(2)	0.0220(6)
H3	0.5387	0.5514	0.6498	0.026*
C4	0.6481(3)	0.7350(3)	0.5468(2)	0.0207(5)
C5	0.6344(3)	0.8724(3)	0.4421(2)	0.0220(6)
C6	0.5032(3)	0.9224(3)	0.3618(2)	0.0240(6)
H6A	0.4953	1.0150	0.2898	0.029*
C7	0.3834(3)	0.8305(3)	0.3915(2)	0.0226(6)
H9B	0.026(3)	0.268(4)	0.680(4)	0.056(12)*
C8	0.2417(3)	0.8867(3)	0.3007(2)	0.0243(6)
H8	1.023(4)	0.088(3)	1.199(3)	0.063(12)*

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C9	0.8674(3)	0.8379(3)	0.5313(2)	0.0236(6)
H9	0.9710	0.8532	0.5472	0.028*
C10	0.8733(3)	0.5830(3)	0.7018(2)	0.0238(6)
H10A	0.9889	0.5904	0.7011	0.029*
H10B	0.8621	0.4962	0.6809	0.029*
C11	0.8077(3)	0.5545(3)	0.8364(2)	0.0187(5)
C12	0.8637(3)	0.4196(3)	0.9342(2)	0.0202(5)
H12	0.9385	0.3496	0.9142	0.024*
C13	0.8107(3)	0.3869(3)	1.0607(2)	0.0194(5)
C14	0.6991(3)	0.4878(3)	1.0903(2)	0.0190(5)
H14	0.6623	0.4649	1.1768	0.023*
C15	0.6413(3)	0.6217(3)	0.9934(2)	0.0199(5)
C16	0.6969(3)	0.6552(3)	0.8665(2)	0.0215(5)
H16	0.6585	0.7476	0.8004	0.026*
C17	0.8726(3)	0.2429(3)	1.1672(2)	0.0217(6)
C18	0.5164(3)	0.7300(3)	1.0211(2)	0.0237(6)

56.74%; H, 3.42%; N, 7.35%. Found: C, 56.26%; H, 3.15%; N, 7.29%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Due to the various geometry configurations of semirigid tetracarboxylic acid ligands and the existence of weak interactions such as hydrogen bonds, their crystal research has been developing rapidly in recent years [5]. The benzimidazole-containing carboxylic acid has both the advantages of one more coordination point, but also the potential for intramolecular reactions. So the benzimidazole-containing caboxylates can be used as a good organic ligands as part of the interest in our department in the synthesis of transition metal complexes [6–10].

The crystal structure of the title compound have been synthesized and structurally characterized by X-ray crystallography analyses. The asymmetric unit contains one organic molecule (*cf.* the figure), and two water molecules. All bond lengths are within the normal ranges. In the formal 1-(3,5-dicarboxyphenyl)-1*H*-benzo[d]imidazole-5,6-dicarboxylic acid the hydrogen atom of one of the carboxy groups is

shifted to the N atom of the benzimidazole moiety to give the typical ylidic structure: 6-carboxy-1-(3,5-dicarboxyphenyl)-1*H*-benzo[d]imidazol-3-ium-5-carboxylate. There is a strong intermolecular O1—H1···N2 hydrogen bond interaction.

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