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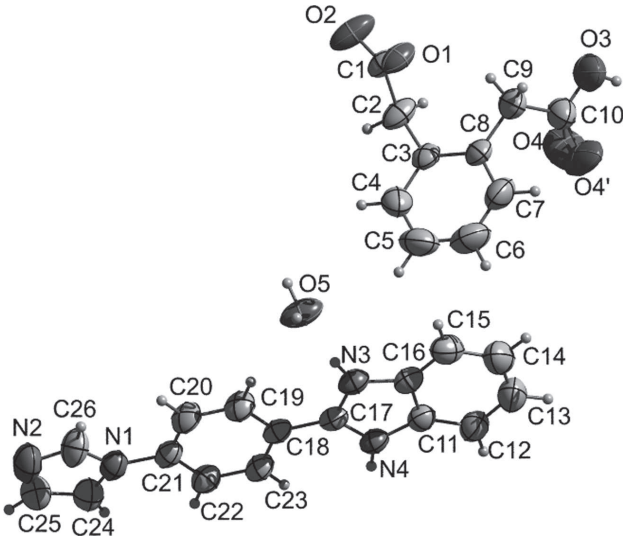
Crystal structure of 2-[4-(1*H*-imidazol-1-yl)phenyl]-1*H*-benzimidazol-3-ium [2-(carboxymethyl)phenyl] acetate monohydrate, $C_{26}H_{24}N_4O_5$



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Abstract

$C_{26}H_{24}N_4O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 8.0382(5)$ Å, $b = 10.0185(6)$ Å, $c = 15.2333(9)$ Å, $\alpha = 81.853(2)°$, $\beta = 76.189(2)°$, $\gamma = 84.700(2)°$, $V = 1177.07(12)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0507$, $wR_{ref}(F^2) = 0.1449$, $T = 296(2)$ K.

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The symmetric unit of the title structure is shown in the figure. Tables 1–3 contain details of the methods used and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless, Block, size 0.21×0.29×0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.94 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω scans
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7891, 4361
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2997
$N(param)_{refined}$:	327
Programs:	Bruker programs [13], SHELX [14]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	2*i*	0.3650	0.1686	0.5964	0.061
H(4N)	2*i*	0.2094	−0.1877	0.6456	0.058
H(3A)	2*i*	1.1472	0.2819	0.9962	0.162
H(2W)	2*i*	0.3903	0.4039	0.5484	0.114
H(1W)	2*i*	0.5498	0.3467	0.5094	0.114
H(2A)	2*i*	1.0089	0.4116	0.5988	0.078
H(2B)	2*i*	1.0998	0.3706	0.6795	0.078
H(4)	2*i*	0.7180	0.4620	0.6266	0.085
H(5)	2*i*	0.4528	0.4901	0.7229	0.109
H(6)	2*i*	0.4322	0.5001	0.8740	0.111
H(7)	2*i*	0.6751	0.4879	0.9292	0.094
H(9A)	2*i*	1.1051	0.4551	0.8017	0.094
H(9B)	2*i*	1.0039	0.5454	0.8751	0.094
H(12)	2*i*	0.2514	−0.2518	0.8265	0.081
H(13)	2*i*	0.3457	−0.1549	0.9338	0.095
H(14)	2*i*	0.4336	0.0609	0.9048	0.095
H(15)	2*i*	0.4378	0.1906	0.7658	0.082
H(19)	2*i*	0.3142	0.2002	0.4615	0.071
H(20)	2*i*	0.2737	0.2232	0.3161	0.072
H(22)	2*i*	0.1384	−0.1592	0.3549	0.065
H(23)	2*i*	0.1790	−0.1822	0.5009	0.061
H(24)	2*i*	0.1339	−0.1386	0.2033	0.086
H(25)	2*i*	0.1144	−0.0254	0.0528	0.095
H(26)	2*i*	0.2011	0.2505	0.1890	0.090

Source of material

Typically, the starting mixture containing o-phenylenediacetic acid (0.1 mmol, 19.4 mg), 2-(4-(imidazol-1-yl))-1*H*-

Table 3: Fractional atomic coordinate and displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(4)[a]	2*i*	0.956(3)	0.2430(8)	0.930(1)	0.180(8)	0.064(4)	0.177(8)	−0.037(4)	−0.099(6)	0.013(4)
O(4′)[b]	2*i*	0.889(1)	0.292(2)	0.971(1)	0.105(5)	0.143(8)	0.131(6)	−0.051(4)	−0.036(4)	0.090(5)
N(1)	2*i*	0.1760(2)	0.0504(2)	0.2306(1)	0.057(1)	0.055(1)	0.051(1)	−0.0007(9)	−0.0093(9)	0.0050(9)
N(2)	2*i*	0.1602(3)	0.1495(3)	0.0938(2)	0.097(2)	0.089(2)	0.063(1)	0.005(1)	−0.026(1)	0.011(1)
N(3)	2*i*	0.3375(2)	0.0903(2)	0.6245(1)	0.051(1)	0.039(1)	0.059(1)	−0.0081(8)	−0.0046(8)	−0.0021(8)
N(4)	2*i*	0.2484(2)	−0.1137(2)	0.6524(1)	0.050(1)	0.0392(9)	0.053(1)	−0.0097(7)	−0.0077(8)	0.0016(8)
O(1)	2*i*	1.0848(2)	0.6680(2)	0.6645(1)	0.075(1)	0.0437(9)	0.065(1)	−0.0200(8)	0.0078(8)	−0.0042(8)
O(2)	2*i*	1.2567(2)	0.5622(2)	0.5578(1)	0.075(1)	0.053(1)	0.105(1)	−0.0092(8)	0.035(1)	0.000(1)
O(3)	2*i*	1.1490(3)	0.3459(3)	0.9560(2)	0.102(2)	0.125(2)	0.094(2)	−0.026(1)	−0.044(1)	0.045(1)
O(5)	2*i*	0.4515(2)	0.3303(2)	0.5433(1)	0.065(1)	0.0404(9)	0.109(1)	−0.0124(7)	0.007(1)	−0.0013(9)
C(1)	2*i*	1.1323(3)	0.5678(2)	0.6237(2)	0.051(1)	0.041(1)	0.051(1)	−0.0054(9)	−0.002(1)	0.007(1)
C(2)	2*i*	1.0297(3)	0.4416(2)	0.6529(2)	0.074(2)	0.045(1)	0.066(2)	−0.018(1)	0.008(1)	−0.004(1)
C(3)	2*i*	0.8608(3)	0.4573(2)	0.7195(1)	0.057(1)	0.038(1)	0.052(1)	−0.0174(9)	−0.007(1)	0.003(1)
C(4)	2*i*	0.7106(4)	0.4669(3)	0.6879(2)	0.083(2)	0.070(2)	0.066(2)	−0.028(1)	−0.026(1)	0.001(1)
C(5)	2*i*	0.5515(4)	0.4835(3)	0.7454(3)	0.059(2)	0.099(2)	0.117(3)	−0.022(2)	−0.031(2)	0.004(2)
C(6)	2*i*	0.5394(4)	0.4901(3)	0.8348(3)	0.057(2)	0.104(2)	0.104(2)	−0.018(2)	0.004(2)	−0.001(2)
C(7)	2*i*	0.6852(4)	0.4821(3)	0.8678(2)	0.079(2)	0.091(2)	0.055(1)	−0.026(2)	0.004(1)	−0.002(1)
C(8)	2*i*	0.8472(3)	0.4656(2)	0.8116(2)	0.059(1)	0.055(1)	0.052(1)	−0.021(1)	−0.012(1)	0.013(1)
C(9)	2*i*	1.0037(4)	0.4609(3)	0.8509(2)	0.082(2)	0.087(2)	0.066(2)	−0.032(2)	−0.029(1)	0.027(1)
C(10)	2*i*	1.0175(4)	0.3483(3)	0.9236(2)	0.088(2)	0.074(2)	0.067(2)	−0.026(2)	−0.030(2)	0.013(1)
C(11)	2*i*	0.2916(3)	−0.0866(2)	0.7305(2)	0.044(1)	0.054(1)	0.053(1)	−0.0040(9)	−0.006(1)	−0.002(1)
C(12)	2*i*	0.2888(3)	−0.1646(3)	0.8141(2)	0.065(2)	0.072(2)	0.060(2)	−0.009(1)	−0.009(1)	0.009(1)
C(13)	2*i*	0.3444(4)	−0.1056(3)	0.8774(2)	0.079(2)	0.100(2)	0.055(2)	−0.008(2)	−0.015(1)	0.004(2)
C(14)	2*i*	0.3984(4)	0.0249(3)	0.8597(2)	0.072(2)	0.105(2)	0.063(2)	−0.012(2)	−0.014(1)	−0.018(2)
C(15)	2*i*	0.4017(3)	0.1030(3)	0.7774(2)	0.061(2)	0.073(2)	0.071(2)	−0.013(1)	−0.007(1)	−0.019(1)
C(16)	2*i*	0.3482(3)	0.0436(2)	0.7132(2)	0.044(1)	0.054(1)	0.053(1)	−0.005(1)	−0.002(1)	−0.005(1)
C(17)	2*i*	0.2774(2)	−0.0060(2)	0.5892(1)	0.037(1)	0.038(1)	0.052(1)	−0.0035(8)	−0.0031(9)	0.001(1)
C(18)	2*i*	0.2508(2)	0.0062(2)	0.4975(1)	0.040(1)	0.036(1)	0.052(1)	−0.0014(8)	−0.0038(9)	0.0014(9)
C(19)	2*i*	0.2786(3)	0.1276(2)	0.4403(2)	0.073(2)	0.039(1)	0.063(1)	−0.012(1)	−0.014(1)	0.003(1)
C(20)	2*i*	0.2542(3)	0.1415(2)	0.3532(2)	0.075(2)	0.039(1)	0.062(1)	−0.011(1)	−0.013(1)	0.011(1)
C(21)	2*i*	0.2007(3)	0.0349(2)	0.3204(1)	0.045(1)	0.046(1)	0.049(1)	0.0014(9)	−0.0045(9)	0.004(1)
C(22)	2*i*	0.1734(3)	−0.0866(2)	0.3764(2)	0.062(1)	0.040(1)	0.059(1)	−0.007(1)	−0.012(1)	0.000(1)
C(23)	2*i*	0.1981(3)	−0.1003(2)	0.4639(2)	0.057(1)	0.037(1)	0.054(1)	−0.0064(9)	−0.009(1)	0.007(1)
C(24)	2*i*	0.1454(3)	−0.0472(3)	0.1827(2)	0.079(2)	0.073(2)	0.063(2)	−0.011(1)	−0.021(1)	0.000(1)
C(25)	2*i*	0.1353(4)	0.0164(3)	0.0995(2)	0.079(2)	0.098(2)	0.062(2)	−0.010(2)	−0.023(1)	−0.003(2)
C(26)	2*i*	0.1827(4)	0.1675(3)	0.1735(2)	0.097(2)	0.063(2)	0.059(2)	0.001(1)	−0.020(1)	0.013(1)

[a]Disordered, occupancy factor: 0.48(3).

[b]Disordered, occupancy factor: 0.52(3).

benzimidazole (0.1 mmol, 26.0 mg), 6 mL of water and NaOH (0.10 mmol, 4.0 mg) was placed in a 23 mL Teflon liner stainless steel reactor. The vessel was heated to 120°C for 4 days, then cooled slowly to the room temperature. Colorless block-shaped crystals were collected by filtration after washing with deionized water, ethanol and acetone, and allowed to dry in air. Yield: 46 %.

Experimental details

The hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX 3, 23, 43 or 147 option) [14]. One of the oxygen atoms is disordered over two sites with occupancy factors of 0.48(3) (O4) and 0.52(3) (O4′) (see the figure).

Discussion

For many years, it has been demonstrated that intermolecular weak interactions including H-bonds, π-π interactions, and even van der Waals forces alone can in concert create stable molecular networks in crystalline solids. They are reproducible, thermally stable, and resilient upon undergoing dynamic processes [1–4]. Thus, the investigation of compounds produced by the spontaneous aggregation among low-dimensional polymers through the intermolecular weak interactions may be a fascinating subject, that is worth studying. Among the reported studies, organic molecules with multi-carboxylate group are particularly interesting for this purpose because the do not only possess more than one donor atom to form an extended structure

[5–8], but they can also act as hydrogen bond acceptors and hydrogen bond donors. o-Phenylenediacetic acid, captures our attention as this molecule contains a flexible $-CH_2COOH$ group which can freely rotate to meet the requirement of the structure [9, 10]. Furthermore dipyridyl-type molecules are widely used coligands, that can pillar polycarboxylate motifs into high dimensional structures [11], while there is relatively little effort to synthesize coordination polymers derived from polydentate aromatic nitrogen heterocyclic molecules, such as pyrazoles, imidazoles, and triazoles, etc. In fact, the introduction of such nitrogen-rich molecules in carboxylate system is beneficial for the formation of hydrogen bonded polymers based on the following considerations: (i) they can act as multi-bridges between hydrogen-bond acceptors and hydrogen-bond donors (ii) the prototropy and conjugation between aromatic nitrogen heterocyclic not only alter the electron density in different parts of the molecules, but also make them flexible [12].

The title salt is a 1:1 crystal of a 1,2-phenylenediacetic acid anion, a 2-(4-(imidazol-1-yl))-1*H*-benzimidazole monovalent cation, and a water molecule. Hydrogen bonds between all ions and the water molecule lead to the formation of a 3D framework π-π stacking interactions (the face to face distance is 4.169 Å) exist between adjacent aryl rings of 2-(4-(imidazol-1-yl))-1*H*-benzimidazole.

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