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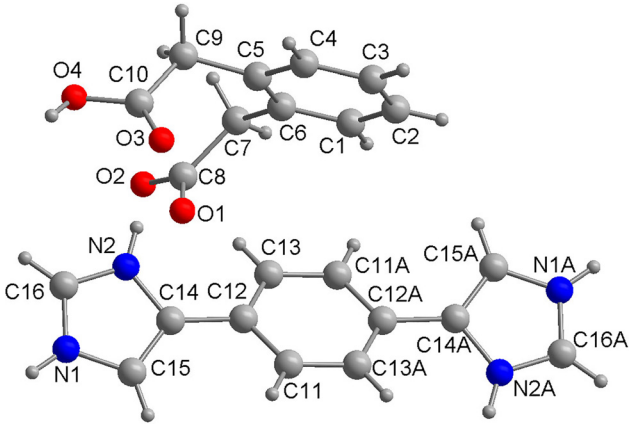
Crystal structure of 5,5′-(1,4-phenylene) bis(1*H*-imidazol-3-ium) bis(2-(2-(carboxymethyl) phenyl)acetate), $C_{32}H_{30}N_4O_8$



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Abstract

$C_{32}H_{30}N_4O_8$, monoclinic, $P2_1/c$ (no. 14), a = 9.4698(10) Å, b = 10.9797(12) Å, c = 14.0366(15) Å, β = 96.807(1)°, V = 1449.2(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0392, $\omega R_{ref}(F^2)$ = 0.1087, T = 293(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All reagents and solvents were used as obtained. The ethanol solution (5 mL) of 1,4-di(1*H*-imidazol-4-yl)benzene (L, 0.10 mmol, 0.021 g) was slowly added to an aqueous solution (25 mL) of 2,2′-(1,2-phenylene)diacetic acid (H_2A 0.1 mmol, 0.0194 g). The mixture was stirred for half an hour at 353 K. The solution was filtered, and the filtrate was kept at the room temperature. After two weeks, colorless crystals of the title salt were obtained with a yield of 50%.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.24 × 0.22 × 0.21 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker Smart Apex, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9861, 2545, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2029
$N(param)_{refined}$:	200
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors U_{iso}(H) = 1.2 times U_{eq}(C, aromatic ring), U_{iso}(H) = 1.2 times U_{eq}(CH_2), U_{iso}(H) = 1.5 times U_{eq}(OH), U_{iso}(H) = 1.2 times U_{eq}(NH). The final refinement by using geometrical restraints, with C—H = 0.93 Å (aromatic ring), C—H = 0.98 Å (CH_2), O—H = 0.82 Å (OH), and N—H = 0.86 Å (NH).

Comment

Due to the fascinating structural topologies and potential applications, the cocrystal is the crucial type of multi-component crystalline supramolecular polymer that has attracted considerable attention [5]. The polyazaheteroaromatic compounds in their neutral and anionic forms have been widely employed for the construction of complicated metal-organic coordination architectures [6–10]. As known, the nitrogen atoms of polyazaheteroaromatic compounds can serve as weak base to accept protons from carboxylic acid or inorganic acid to form acid-base conjugated pairs. In this study, we report the reaction product of the multi-nitrogen compound 1,4-di(1-Himidazol-4-yl)benzene (L) together

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.21822 (13)	0.16393 (12)	0.23258 (9)	0.0447 (3)
O2	0.12210 (15)	0.29655 (14)	0.32479 (9)	0.0606 (4)
O3	0.22075 (13)	−0.13768 (12)	0.22593 (9)	0.0466 (3)
O4	0.03828 (14)	−0.09800 (14)	0.30725 (10)	0.0548 (4)
H4	−0.006344	−0.136267	0.263390	0.082*
C1	0.58165 (19)	0.14413 (19)	0.36385 (13)	0.0450 (5)
H1	0.602236	0.226936	0.363479	0.054*
C2	0.6874 (2)	0.0623 (2)	0.35421 (14)	0.0572 (6)
H2	0.779113	0.089262	0.348477	0.069*
C3	0.6580 (2)	−0.0590 (2)	0.35305 (15)	0.0629 (7)
H3	0.729645	−0.115198	0.346151	0.075*
C4	0.5220 (3)	−0.09884 (18)	0.36209 (14)	0.0549 (6)
H4A	0.502986	−0.181966	0.360818	0.066*
C5	0.41235 (19)	−0.01707 (15)	0.37310 (11)	0.0351 (4)
C6	0.44397 (17)	0.10708 (15)	0.37420 (11)	0.0324 (4)
C7	0.3353 (2)	0.20366 (18)	0.38930 (13)	0.0456 (5)
H7A	0.384636	0.280689	0.400527	0.055*
H7B	0.293377	0.183695	0.447123	0.055*
C8	0.21595 (17)	0.22130 (15)	0.30849 (12)	0.0346 (4)
C9	0.2661 (2)	−0.06243 (19)	0.38690 (13)	0.0508 (5)
H9A	0.276452	−0.130955	0.430785	0.061*
H9B	0.217070	0.001578	0.417459	0.061*
C10	0.17375 (19)	−0.10143 (16)	0.29772 (13)	0.0383 (4)
N1	1.00003 (15)	0.14205 (13)	0.10357 (10)	0.0385 (4)
H1A	1.071742	0.156174	0.145692	0.046*
N2	0.86829 (14)	0.12664 (13)	−0.03169 (10)	0.0347 (3)
H2A	0.841344	0.129337	−0.092354	0.042*
C11	0.54931 (18)	0.04824 (15)	0.08849 (12)	0.0346 (4)
H11	0.582445	0.080688	0.148172	0.042*
C12	0.63930 (17)	0.04427 (14)	0.01652 (11)	0.0298 (4)
C13	0.58789 (18)	−0.00471 (15)	−0.07214 (12)	0.0347 (4)
H13	0.646489	−0.008121	−0.120768	0.042*
C14	0.78590 (17)	0.08888 (14)	0.03739 (11)	0.0305 (4)
C15	0.87034 (18)	0.09888 (16)	0.12181 (12)	0.0364 (4)
H15	0.844638	0.079688	0.181944	0.044*
C16	0.99595 (18)	0.15817 (16)	0.01053 (12)	0.0384 (4)
H16	1.070512	0.187022	−0.020736	0.046*

with 2,2′-(1,2-phenylene)diacetic acid (H_2A) to search for cocrystals.

The asymmetric unit of the title structure contains one half of L^{2+} and one HA^-. As mentioned above, the nitrogen-rich molecule L can serve as weak base to accept protons from carboxylic acid to form L^{2+}, and H_2A are deprotonated to be HA^- anion.

The hydrogen bonding scheme of the title structure creates chains along the *b* direction based on OH···O interactions between a carboxy group and a neighboring carboxylate group of the next anion. The cations are side on attached to the backbone of these chains by NH···O hydrogen bonds. There are some related structures using the cation of this salt compound [11]. All bond lengths and angles are in the expected ranges.

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