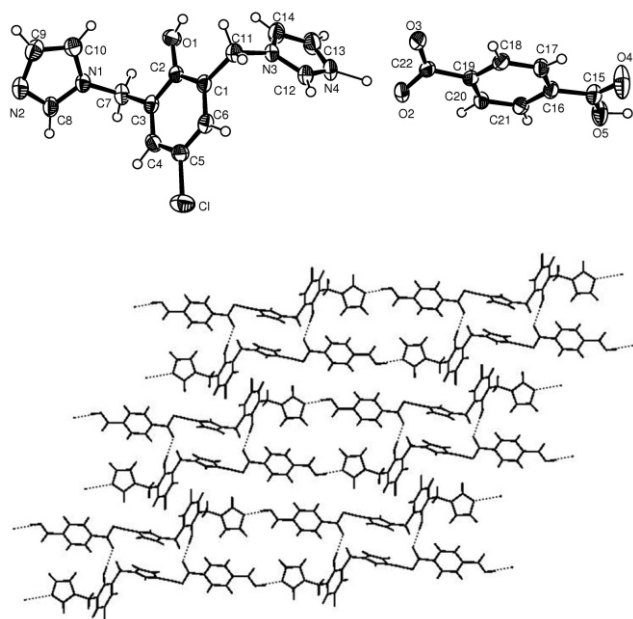


Crystal structure of 2,6-bis[(imidazol-1-yl)methyl]-4-chlorophenol — terephthalic acid (1:1), $C_{14}H_{13}ClN_4O \cdot C_8H_6O_4$

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Abstract

$C_{22}H_{19}ClN_4O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 7.207(2)$ Å, $b = 12.087(7)$ Å, $c = 12.213(4)$ Å, $\alpha = 103.01(4)^\circ$, $\beta = 90.08(3)^\circ$, $\gamma = 102.20(3)^\circ$, $V = 1011.8$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.044$, $wR_{\text{ref}}(F^2) = 0.113$, $T = 290$ K.

Source of material

2,6-bis[(imidazol-1-yl)methyl]-4-chlorophenol (BICP) was prepared according to [1]. The mixture of BICP (0.05 mmol, 14.4 mg) and terephthalic acid (TPA, 0.05 mmol, 8.3 mg) was dissolved in methanol (15 mL) with stirring. Upon slow evaporation of solvent, colorless block single crystals suitable for X-ray analysis were obtained after two weeks (yield 68 %).

Experimental details

All H atoms were located in a difference Fourier map and refined with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ and $1.5 U_{\text{eq}}(\text{O})$.

Discussion

Intermolecular hydrogen bonds have proven to be an ideal and efficient tool in the design and construction of organic crystals because of their strength and directional properties [2]. The architectures assembled from organic ligands and various hydrogen bonding donors has been rapidly developed because of their fascinating structural diversity and potential applications as func-

tional materials [3,4]. Carboxylic acids which can form strong and directional hydrogen bonds are frequently chosen as building blocks for crystal engineering, and a variety of co-crystals have been synthesized by assembling carboxylic acids and organic ligands bearing *N*-donors, such as pyridine-based ligands [5–7]. Imidazoles, which are also *N*-donor compounds, are used to construct organic crystals, but only a few ones composed of carboxylic acids and diimidazole compounds have been reported in the literature to date [8–10].

The asymmetric unit of the title crystal structure contains one TPA and one BICP molecule. Two imidazole rings of the BICP moiety are located at the different sides of the benzene ring with the angles $\angle N1-C7-C3 = 111.3^\circ$ and $\angle N3-C11-C1 = 111.0^\circ$. The carboxyl group of TPA interacts with the imidazol-1-yl nitrogen atom of BICP via $O2-H20 \cdots N2$ and $O5-H5 \cdots N2$ hydrogen bonds, and thus the hydrogen bonds further propagate the acid-base subunits into an infinite one-dimensional zigzag chain. Meanwhile, every two chains are combined into a double chain by $O1-H1 \cdots O3$ hydrogen bonds between the hydroxyl group of BICP and the carboxyl group of TPA. In addition, the C11 and C14 of the BICP molecule and the O3 of the carboxylic group could form $C11-H11A \cdots O3$ and $C14-H14 \cdots O3$ hydrogen bonds, which is another contributor for the combination of two single chains. Further analysis reveals that the weak intermolecular $C-H \cdots \pi$ interaction also contributes to the crystal packing arrangement.

Table 1. Data collection and handling.

Crystal:	colourless block, size $0.18 \times 0.22 \times 0.26$ mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	2.34 cm^{-1}
Diffraction, scan mode:	CAD4, $\omega/2\theta$
$2\theta_{\text{max}}$:	50.08°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	3539, 3539
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3539
$N(\text{param})_{\text{refined}}$:	365
Programs:	SHELXS-97, SHELXL-97 [11]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}
H(4)	2i	0.716(4)	1.149(3)	−0.071(2)	0.047
H(6)	2i	0.790(4)	0.949(2)	0.143(2)	0.051
H(7B)	2i	0.630(4)	1.058(3)	−0.269(2)	0.044
H(7A)	2i	0.622(4)	0.926(2)	−0.321(2)	0.039
H(8)	2i	0.911(4)	1.209(2)	−0.296(2)	0.047
H(9)	2i	1.278(5)	1.015(3)	−0.410(3)	0.060
H(10)	2i	0.974(4)	0.883(2)	−0.361(2)	0.046

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Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(11B)	2i	0.844(4)	0.758(2)	0.075(2)	0.038
H(11A)	2i	0.817(4)	0.694(2)	−0.057(2)	0.035
H(12)	2i	0.555(5)	0.725(3)	0.193(3)	0.070
H(13)	2i	0.167(4)	0.528(3)	−0.048(2)	0.054
H(14)	2i	0.484(5)	0.602(3)	−0.138(3)	0.080
H(18)	2i	0.123(4)	0.343(2)	0.407(2)	0.041

Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(17)	2i	−0.100(4)	0.265(2)	0.523(2)	0.045
H(21)	2i	−0.493(4)	0.414(2)	0.419(2)	0.031
H(20)	2i	−0.269(4)	0.503(2)	0.310(2)	0.039
H(5)	2i	−0.711(6)	0.243(4)	0.591(3)	0.110
H(1)	2i	0.745(5)	0.726(3)	−0.220(3)	0.070
H(4A)	2i	0.191(6)	0.592(4)	0.171(4)	0.130

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	2i	0.7568(3)	0.8600(2)	−0.0233(2)	0.025(1)	0.033(2)	0.035(1)	0.004(1)	0.007(1)	0.012(1)
C(2)	2i	0.7322(3)	0.8723(2)	−0.1332(2)	0.026(1)	0.029(1)	0.032(1)	0.001(1)	0.005(1)	0.007(1)
C(3)	2i	0.7175(3)	0.9811(2)	−0.1509(2)	0.024(1)	0.038(2)	0.034(1)	0.005(1)	0.005(1)	0.014(1)
C(4)	2i	0.7306(4)	1.0743(2)	−0.0608(2)	0.030(2)	0.028(2)	0.047(2)	0.005(1)	0.006(1)	0.013(1)
C(5)	2i	0.7575(4)	1.0620(2)	0.0471(2)	0.029(2)	0.030(2)	0.043(2)	0.003(1)	0.006(1)	0.005(1)
C(6)	2i	0.7711(4)	0.9558(2)	0.0659(2)	0.028(2)	0.040(2)	0.032(2)	0.003(1)	0.004(1)	0.011(1)
Cl	2i	0.7776(1)	1.18079(7)	0.16060(6)	0.0610(5)	0.0395(4)	0.0522(5)	0.0103(4)	−0.0009(4)	−0.0061(3)
C(7)	2i	0.6968(4)	0.9968(3)	−0.2694(2)	0.031(2)	0.044(2)	0.042(2)	0.004(1)	0.003(1)	0.022(1)
N(1)	2i	0.8827(3)	1.0328(2)	−0.3143(2)	0.034(1)	0.037(1)	0.033(1)	0.007(1)	0.005(1)	0.016(1)
C(8)	2i	0.9717(4)	1.1423(3)	−0.3157(2)	0.039(2)	0.041(2)	0.040(2)	0.008(1)	0.010(1)	0.021(1)
N(2)	2i	1.1430(3)	1.1458(2)	−0.3539(2)	0.040(2)	0.049(2)	0.044(1)	0.005(1)	0.008(1)	0.023(1)
C(9)	2i	1.1644(5)	1.0335(3)	−0.3777(2)	0.043(2)	0.058(2)	0.042(2)	0.018(2)	0.011(1)	0.022(2)
C(10)	2i	1.0064(4)	0.9628(3)	−0.3537(2)	0.048(2)	0.039(2)	0.040(2)	0.011(2)	0.008(1)	0.014(1)
O(1)	2i	0.7169(3)	0.7858(2)	−0.2270(2)	0.056(1)	0.032(1)	0.033(1)	0.012(1)	0.0043(9)	0.0075(9)
C(11)	2i	0.7648(4)	0.7453(2)	0.0033(2)	0.040(2)	0.037(2)	0.033(2)	0.008(1)	0.006(1)	0.013(1)
N(3)	2i	0.5724(3)	0.6802(2)	0.0195(2)	0.040(1)	0.030(1)	0.031(1)	0.005(1)	0.008(1)	0.013(1)
C(12)	2i	0.4860(5)	0.6840(3)	0.1174(3)	0.060(2)	0.040(2)	0.037(2)	0.012(2)	0.016(2)	0.019(1)
N(4)	2i	0.3101(4)	0.6223(2)	0.0991(2)	0.054(2)	0.043(2)	0.053(2)	0.008(1)	0.022(1)	0.026(1)
C(13)	2i	0.2848(5)	0.5764(3)	−0.0144(3)	0.049(2)	0.047(2)	0.052(2)	−0.001(2)	0.006(2)	0.017(2)
C(14)	2i	0.4452(4)	0.6119(3)	−0.0639(3)	0.050(2)	0.048(2)	0.033(2)	0.002(2)	0.007(1)	0.013(1)
C(19)	2i	−0.0516(4)	0.4280(2)	0.3465(2)	0.038(2)	0.025(1)	0.025(1)	0.001(1)	0.005(1)	0.001(1)
C(22)	2i	0.0906(4)	0.4752(2)	0.2698(2)	0.037(2)	0.028(2)	0.033(1)	0.000(1)	0.010(1)	0.001(1)
O(2)	2i	0.0597(3)	0.5624(2)	0.2347(2)	0.056(1)	0.041(1)	0.057(1)	0.014(1)	0.025(1)	0.027(1)
O(3)	2i	0.2287(3)	0.4313(2)	0.2438(2)	0.054(1)	0.043(1)	0.063(1)	0.019(1)	0.034(1)	0.022(1)
C(18)	2i	−0.0037(4)	0.3559(2)	0.4104(2)	0.032(2)	0.035(2)	0.030(1)	0.007(1)	0.006(1)	0.005(1)
C(17)	2i	−0.1342(4)	0.3070(2)	0.4771(2)	0.038(2)	0.037(2)	0.032(2)	0.008(1)	0.005(1)	0.015(1)
C(16)	2i	−0.3173(4)	0.3270(2)	0.4803(2)	0.033(2)	0.034(2)	0.029(1)	0.002(1)	0.006(1)	0.008(1)
C(15)	2i	−0.4551(4)	0.2690(2)	0.5516(2)	0.035(2)	0.045(2)	0.039(2)	0.006(1)	0.008(1)	0.017(1)
O(4)	2i	−0.4117(3)	0.2098(2)	0.6107(2)	0.043(1)	0.112(2)	0.096(2)	0.021(1)	0.022(1)	0.080(2)
O(5)	2i	−0.6258(3)	0.2881(2)	0.5475(2)	0.038(1)	0.070(2)	0.070(2)	0.017(1)	0.022(1)	0.044(1)
C(21)	2i	−0.3660(4)	0.3994(2)	0.4168(2)	0.030(2)	0.036(2)	0.036(2)	0.007(1)	0.006(1)	0.009(1)
C(20)	2i	−0.2316(4)	0.4506(2)	0.3515(2)	0.040(2)	0.032(2)	0.029(1)	0.005(1)	0.004(1)	0.010(1)

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