Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bisimidazolium terephthalate

Zhi-gao Tian

Department of Chemistry and Biology Science Engineering, Xiangfan University, Hubei 441053, People's Republic of China
Correspondence e-mail: zhiguo_tian@126.com

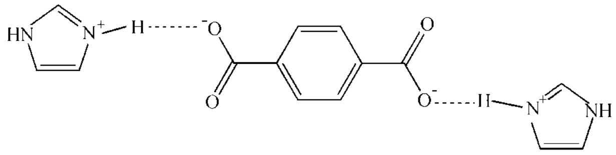
Received 2 September 2007; accepted 10 September 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.137; data-to-parameter ratio = 15.7.

The formula unit of the title compound, $2C_3H_5N_2^+\cdot C_8H_4O_4^{2-}$, consists of two imidazole cations which are N—H···O hydrogen-bonded to a terephthalate anion which lies on a crystallographic inversion center. In the crystal structure, two N—H···O and two C—H···O hydrogen bonds link the components into a three-dimensional network.

Related literature

For related literature, see: Bernstein *et al.* (1995); Batten & Robson (1998)



Experimental

Crystal data

$2C_3H_5N_2^+\cdot C_8H_4O_4^{2-}$
M_r = 302.29
Monoclinic, $P2_1/n$
a = 9.6288 (6) Å
b = 8.3351 (6) Å
c = 9.8244 (6) Å
β = 113.854 (1)°
V = 721.12 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.966, T_{max} = 0.979
7596 measured reflections
1696 independent reflections
1368 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.137
S = 1.12
1696 reflections
108 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2	1.010 (18)	1.665 (19)	2.6307 (14)	158.6 (15)
C6—H6A···O1^{i}	0.93	2.22	3.1046 (18)	159
C5—H5A···O2^{ii}	0.93	2.50	3.3601 (17)	154
N1—H1···O1^{iii}	1.04 (2)	1.61 (2)	2.6336 (14)	167.1 (17)

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

This work was financially supported mostly by a Key Fundamental Project.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2499).

References

Batten, S. R. & Robson, R. (1998). *Angew. Chem. Int. Ed.* **37**, 1460–1494.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2001). *SAINT-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SADABS* (Version 2.10), *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

doi:10.1107/S1600536807044236

supplementary materials

Acta Cryst. (2007). E**63**, o4067 [**doi:10.1107/S1600536807044236**]

Bisimidazolium terephthalate

Z. Tian

Comment

With the aim of researching hydrogen-bonded actions in new classes of organic adducts, we have investigated the reactions of terephthalic acid with imidazole. The 1:2 organic salt is formed as a consequence of protons being transferred from the carboxyl oxygen atoms to the imine N atoms. We report here the molecular and supramolecular structure of the title compound.

The asymmetric unit consists of half of a terephthalate anion and one imidazolium cation, the formula unit being generated by a crystallogrphic inversion center (Fig.1). The supramolecular structure can be readily analysed in terms of simple substructures listed below. Firstly, a combination of the N1–H1···O1^{iii} [N–O = 2.6336 (14) Å, N–H···O = 167.1 (17)°, symmetry code: (iii) $-x + 1/2$, $y + 1/2$, $-z + 3/2$] and N2–H2···O2 hydrogen bonds (Table 1) links the terephthalate anion and imidazolium cations into a (30–1) sheet (Fig.2) in the form of a (6,6) net (Batten & Robson, 1998) which is built from $R_8^8(22)$ rings (Bernstein *et al.*, 1995). Three networks of this type pass through the unit cell by translation. Secondly, the imidazolium C5 and C6 atoms at (*x*,*y*,*z*) act as soft hydrogen-bond donor, *via*. H5A and H6A, to the carboxyl O2 and O1 atoms at (1 − *x*, −*y*, 1 − *z*) and (−*x*, −*y*, 1 − *z*), respectively, linking the adjacent networks into a simple three-dimensional network (Fig.3). Although aryl and imidazole rings exist, no π–π stacking and C–H···π interactions are observed in the supramolecular structure.

Experimental

All reagents and solvents were used as obtained without further purification. 1:2 molar amount of imidazole (0.4 mmol, 27 mg) and terephthalic acid (0.2 mmol, 33 mg) were dissolved in 95% methanol (10 ml). The mixture was stirred for ten minutes at ambient temperature. The resulting colorless solution was kept in air for several days. Crystals suitable for single-crystal X-ray diffraction analysis were grown by slow evaporation of the solution at the bottom of the vessel.

Refinement

H atoms bonded to carbon atoms were located at the geometrical positions with C—H=0.93 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$. H1 and H2A were located from the difference maps with the N–H distances refined freely and their U_{iso} values being set 1.2 times of their carrier atoms.

Figures

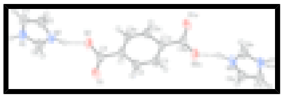


Fig. 1. Molecular structure, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. Atoms marked with 'a' are symmetry position (−*x*, −*y*, −*z*).

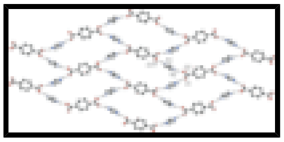


Fig. 2. Part of the crystal structure, showing the formation of the two-dimensional $(30\bar{1})$ network fromed by N–H···O hydrogen bonds. Hydrogen bonds are shown as dashed lines. Atom marked with (iii) is at symmetry position ($-x + 1/2$, $y + 1/2$, $-z + 3/2$).

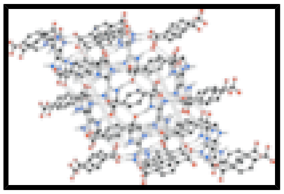


Fig. 3. Part of the crystal structure, showing the formation of the three-dimensional network formed by C–H···O and N–H···O hydrogen bonds. Hydrogen bonds are shown as dashed lines.

Bisimidazolium terephthalate

Crystal data

$2C_3H_5N_2^+\cdot C_8H_4O_4^{2-}$

$M_r = 302.29$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 9.6288$ (6) Å

$b = 8.3351$ (6) Å

$c = 9.8244$ (6) Å

$\beta = 113.854$ (1)°

$V = 721.12$ (8) Å^3

$Z = 2$

$F_{000} = 316$

$D_x = 1.392$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 3264 reflections

$\theta = 2.3$–28.1°

$\mu = 0.11$ mm^{-1}

$T = 298$ (2) K

Block, colorless

0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine focus sealed Siemens Mo tube

Monochromator: graphite

$T = 298(2)$ K

0.3° wide ω exposures scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1997)

$T_{min} = 0.966$, $T_{max} = 0.979$

7596 measured reflections

1696 independent reflections

1368 reflections with $I > 2\sigma(I)$

$R_{int} = 0.041$

$\theta_{max} = 28.0$°

$\theta_{min} = 2.5$°

$h = -12 \rightarrow 12$

$k = -10 \rightarrow 10$

$l = -11 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.137$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of
independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0835P)^2 + 0.0143P]$

where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: none

$S = 1.12$
1696 reflections
108 parameters
Primary atom site location: structure-invariant direct methods

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.05078 (13)	−0.03576 (14)	0.15160 (12)	0.0333 (3)
C2	−0.05484 (15)	−0.13358 (15)	0.04568 (13)	0.0382 (3)
H2	−0.0920	−0.2237	0.0757	0.046*
C3	−0.10550 (14)	−0.09803 (15)	−0.10470 (13)	0.0372 (3)
H3	−0.1766	−0.1643	−0.1746	0.045*
C4	0.10569 (15)	−0.07248 (15)	0.31591 (13)	0.0370 (3)
O1	0.02858 (11)	−0.17027 (15)	0.35300 (10)	0.0590 (4)
O2	0.22390 (11)	−0.00486 (13)	0.40159 (9)	0.0511 (3)
C5	0.52371 (16)	0.05999 (17)	0.75057 (15)	0.0464 (4)
H5A	0.5859	0.0091	0.7120	0.056*
C6	0.32334 (17)	0.15446 (19)	0.77332 (15)	0.0505 (4)
H6A	0.2231	0.1806	0.7535	0.061*
C7	0.56890 (16)	0.13368 (18)	0.88368 (15)	0.0484 (4)
H7A	0.6682	0.1432	0.9542	0.058*
N1	0.44244 (13)	0.19155 (15)	0.89584 (12)	0.0483 (3)
H1	0.438 (2)	0.251 (2)	0.988 (2)	0.084 (6)*
N2	0.36978 (14)	0.07411 (14)	0.68328 (12)	0.0452 (3)
H2A	0.3003 (19)	0.027 (2)	0.584 (2)	0.068 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0375 (6)	0.0407 (6)	0.0203 (6)	0.0038 (5)	0.0103 (5)	0.0013 (4)
C2	0.0470 (7)	0.0409 (6)	0.0259 (6)	−0.0047 (5)	0.0140 (6)	0.0027 (5)
C3	0.0432 (7)	0.0422 (7)	0.0220 (6)	−0.0057 (5)	0.0089 (5)	−0.0036 (5)
C4	0.0412 (7)	0.0474 (7)	0.0218 (6)	0.0086 (5)	0.0120 (5)	0.0031 (5)

O1	0.0588 (7)	0.0867 (8)	0.0293 (6)	−0.0078 (5)	0.0154 (5)	0.0186 (5)
O2	0.0526 (6)	0.0717 (7)	0.0207 (5)	−0.0033 (5)	0.0062 (4)	−0.0042 (4)
C5	0.0489 (8)	0.0550 (8)	0.0367 (8)	−0.0012 (6)	0.0189 (6)	−0.0082 (6)
C6	0.0468 (8)	0.0713 (10)	0.0318 (7)	0.0022 (7)	0.0141 (6)	−0.0073 (6)
C7	0.0454 (8)	0.0611 (8)	0.0340 (8)	−0.0049 (6)	0.0112 (6)	−0.0068 (6)
N1	0.0541 (7)	0.0623 (7)	0.0271 (6)	−0.0010 (6)	0.0149 (5)	−0.0097 (5)
N2	0.0517 (7)	0.0557 (7)	0.0258 (6)	−0.0048 (5)	0.0132 (5)	−0.0074 (5)

Geometric parameters (Å, °)

C1—C2	1.3878 (17)	C5—N2	1.3621 (19)
C1—C3^{i}	1.3892 (17)	C5—H5A	0.9300
C1—C4	1.5128 (16)	C6—N1	1.3203 (17)
C2—C3	1.3877 (16)	C6—N2	1.3232 (17)
C2—H2	0.9300	C6—H6A	0.9300
C3—C1^{i}	1.3892 (17)	C7—N1	1.3592 (17)
C3—H3	0.9300	C7—H7A	0.9300
C4—O2	1.2437 (16)	N1—H1	1.04 (2)
C4—O1	1.2518 (16)	N2—H2A	1.010 (18)
C5—C7	1.3478 (19)		
C2—C1—C3^{i}	118.96 (11)	C7—C5—H5A	126.5
C2—C1—C4	121.01 (11)	N2—C5—H5A	126.5
C3^{i}—C1—C4	120.03 (11)	N1—C6—N2	108.92 (13)
C3—C2—C1	120.56 (12)	N1—C6—H6A	125.5
C3—C2—H2	119.7	N2—C6—H6A	125.5
C1—C2—H2	119.7	C5—C7—N1	107.33 (12)
C2—C3—C1^{i}	120.49 (11)	C5—C7—H7A	126.3
C2—C3—H3	119.8	N1—C7—H7A	126.3
C1^{i}—C3—H3	119.8	C6—N1—C7	108.39 (12)
O2—C4—O1	125.89 (12)	C6—N1—H1	125.2 (11)
O2—C4—C1	117.37 (12)	C7—N1—H1	126.3 (11)
O1—C4—C1	116.73 (11)	C6—N2—C5	108.35 (12)
C4—O2—H2A	133.4 (6)	C6—N2—H2A	124.6 (9)
C7—C5—N2	107.01 (12)	C5—N2—H2A	126.9 (9)
C3^{i}—C1—C2—C3	−0.2 (2)	O1—C4—O2—H2A	−18.7 (8)
C4—C1—C2—C3	179.41 (10)	C1—C4—O2—H2A	162.3 (8)
C1—C2—C3—C1^{i}	0.3 (2)	N2—C5—C7—N1	0.01 (16)
C2—C1—C4—O2	163.41 (12)	N2—C6—N1—C7	0.20 (18)
C3^{i}—C1—C4—O2	−16.93 (18)	C5—C7—N1—C6	−0.13 (18)
C2—C1—C4—O1	−15.68 (18)	N1—C6—N2—C5	−0.19 (17)
C3^{i}—C1—C4—O1	163.98 (12)	C7—C5—N2—C6	0.11 (16)

Symmetry codes: (i) −*x*, −*y*, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O2	1.010 (18)	1.665 (19)	2.6307 (14)	158.6 (15)

C6—H6A···O1$^{\text{ii}}$	0.93	2.22	3.1046 (18)	159
C5—H5A···O2$^{\text{iii}}$	0.93	2.50	3.3601 (17)	154
N1—H1···O1$^{\text{iv}}$	1.04 (2)	1.61 (2)	2.6336 (14)	167.1 (17)

Symmetry codes: (ii) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*, −*z*+1; (iv) −*x*+1/2, *y*+1/2, −*z*+3/2.

Fig. 1

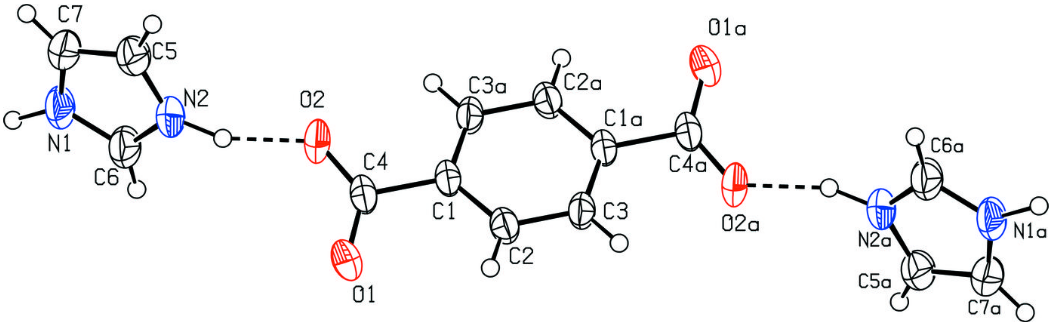

C7
C5
N2
O2
N1
C6
C4
C1
C3a
C2a
C1a
O1a
C4a
C3
C2
O1
O2a
C6a
N2a
N1a
C5a
C7a

Fig. 2

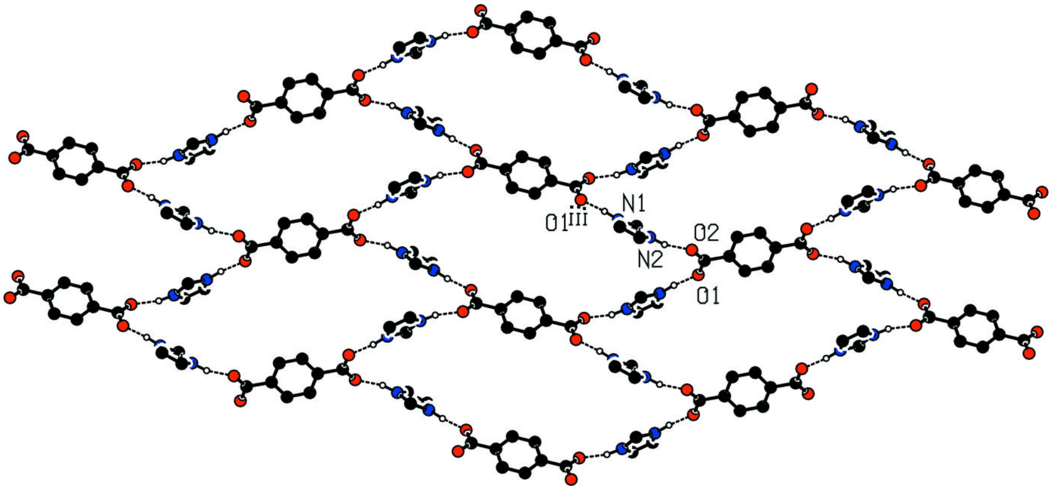


Fig. 3