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1,1′-Biphenyl-4,4′-diammonium bis(2-nitrobenzoate) dihydrate

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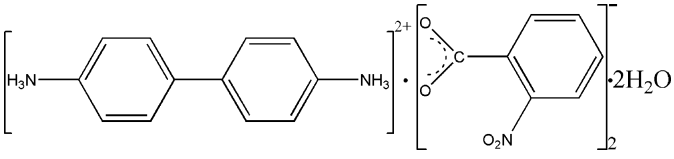
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.121; data-to-parameter ratio = 11.5.

In the title hydrated molecular salt, $C_{12}H_{14}N_2^{+}\cdot 2C_7H_4NO_4^{-}\cdot 2H_2O$, the complete cation is generated by a crystallographic twofold axis running perpendicular to the central C—C bond. The components give rise to an infinite three-dimensional framework *via* intermolecular N—H···O and O—H···O hydrogen bonds.

Related literature

For related literature, see: Wei (2007).



Experimental

Crystal data

$C_{12}H_{14}N_2^{2+}\cdot 2C_7H_4NO_4^{-}\cdot 2H_2O$
M_r = 554.51
Monoclinic, $C2/c$
a = 29.411 (10) Å
b = 6.731 (6) Å
c = 13.511 (6) Å
β = 110.91 (2)°
V = 2499 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.15 × 0.10 × 0.08 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.983, T_{max} = 0.991
5946 measured reflections
2185 independent reflections
1386 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.121
S = 0.96
2185 reflections
190 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.89	1.86	2.744 (3)	176
N2—H2B···O2	0.89	1.88	2.772 (3)	176
N2—H2C···O5^{ii}	0.89	1.90	2.786 (2)	178
O5—H5A···O2	0.819 (10)	2.034 (10)	2.852 (3)	177 (3)
O5—H5B···O1^{iii}	0.825 (10)	1.980 (14)	2.722 (3)	149 (2)

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2569).

References

Bruker (2001). *SAINT-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
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supplementary materials

Acta Cryst. (2007). E**63**, o4305 [**doi:10.1107/S1600536807048994**]

1,1'-Biphenyl-4,4'-diammonium bis(2-nitrobenzoate) dihydrate

L. Wei

Comment

This work continues our previous synthetic and structural studies of supramolecular salts (Wei, 2007). Herein we present the crystal structure of the title salt, (I).

The title salt contains one (1,1'-biphenyl)-4,4'-diammonium dication (with the complete molecule generated from the asymmetry atoms by 2-fold symmetry), one 2-nitrobenzoate anion and one crystallization water molecule (Fig. 1). Interestingly, these components are organized into an infinite two-dimensional network in (100) *via* intermolecular N—H···O and O—H···O hydrogen bonds (Table 1). Further hydrogen bonds link the planes into an infinite three-dimensional framework (Fig. 2).

Experimental

A 5-ml ethanol soloution of (1,1'-biphenyl)-4,4'-diamine (0.5 mmol, 0.090 g) was added to an aqueous solution (20 ml) of 2-nitrobenzoic acid (1.0 mmol, 0.210 g). The mixture was stirred for 10 minutes at 373 K. The solution was filtered, and the filtrate was kept at the room temperature. After 3 d, colourless blocks of (I) were obtained.

Refinement

The water H atoms were located in a difference map and refined isotropically with O—H = 0.82 (1)Å and H···H = 1.34 (1) Å. All remaining H atoms were positioned geometrically with C—H = 0.93Å and N—H = 0.89 Å, and refined as riding with U_{iso}(H) = 1.2U_{eq}(C, N).

Figures

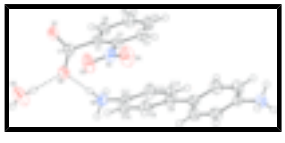

Fig. 1. The molecular structure unit of (I). Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. Unlabeled atoms in the N2 cation are related to labeled atoms by (−*x*, *y*, 1/2 − *z*).

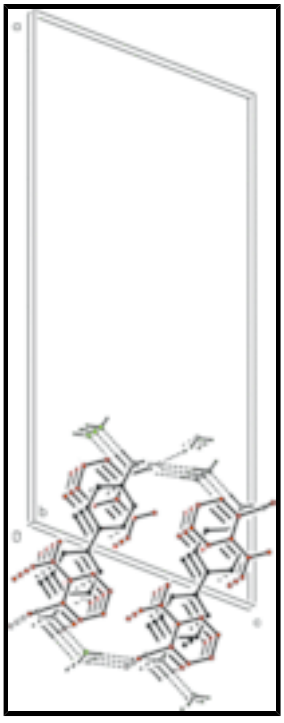

Fig. 2. Crystal packing of (I). Hydrogen bonds are shown as dashed lines. For clarity, H atoms not involved in hydrogen bonds are omitted.

1,1'-Biphenyl-4,4'-diammonium bis(2-nitrobenzoate) dihydrate

Crystal data

$C_{12}H_{14}N_2^{2+}\cdot 2C_7H_4NO_4^-\cdot 2H_2O$	F_{000} = 1160
M_r = 554.51	D_x = 1.474 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2540 reflections
a = 29.411 (10) Å	θ = 2.3–27.8º
b = 6.731 (6) Å	μ = 0.12 mm^{-1}
c = 13.511 (6) Å	*T* = 298 (2) K
β = 110.91 (2)º	Block, colorless
V = 2499 (3) Å^3	0.15 × 0.10 × 0.08 mm
Z = 4	

Data collection

Bruker SMART APEX CCD diffractometer	2185 independent reflections
Radiation source: fine-focus sealed tube	1386 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.062
T = 298(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	*h* = −34→34

T_{min} = 0.983, T_{max} = 0.991
5946 measured reflections

k = −8→4
l = −15→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.121
S = 0.96
2185 reflections
190 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difmap and geom
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0029 (5)

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 $F^2 > 2$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}
N2	0.17925 (6)	0.6702 (3)	0.43129 (13)	0.0362 (5)
H2A	0.1883	0.7790	0.4709	0.043*
H2B	0.1879	0.5631	0.4724	0.043*
H2C	0.1937	0.6678	0.3836	0.043*
C8	0.02670 (7)	0.6736 (3)	0.27712 (15)	0.0317 (5)
C9	0.04800 (8)	0.6479 (3)	0.38501 (16)	0.0395 (6)
H9	0.0282	0.6307	0.4249	0.047*
C10	0.09734 (7)	0.6471 (3)	0.43505 (17)	0.0399 (6)
H10	0.1108	0.6308	0.5081	0.048*
C11	0.12677 (7)	0.6702 (3)	0.37748 (15)	0.0301 (5)
C12	0.10755 (8)	0.6939 (3)	0.27140 (16)	0.0380 (5)
H12	0.1278	0.7076	0.2324	0.046*
C13	0.05784 (7)	0.6975 (3)	0.22153 (17)	0.0391 (6)
H13	0.0448	0.7165	0.1486	0.047*

O1	0.20236 (5)	0.0065 (2)	0.55407 (10)	0.0438 (4)
O2	0.20105 (5)	0.3356 (2)	0.55937 (11)	0.0422 (4)
O3	0.10450 (6)	0.1729 (3)	0.54010 (12)	0.0624 (5)
O4	0.03547 (6)	0.1668 (3)	0.41844 (13)	0.0690 (6)
N1	0.07935 (6)	0.1705 (3)	0.44818 (14)	0.0384 (5)
C1	0.15299 (7)	0.1746 (3)	0.40264 (15)	0.0290 (5)
C2	0.10279 (7)	0.1757 (3)	0.36982 (15)	0.0298 (5)
C3	0.07362 (7)	0.1805 (3)	0.26614 (15)	0.0354 (5)
H3	0.0399	0.1823	0.2468	0.042*
C4	0.09457 (8)	0.1827 (3)	0.19041 (16)	0.0425 (6)
H4	0.0751	0.1864	0.1190	0.051*
C5	0.14405 (8)	0.1796 (3)	0.21997 (17)	0.0455 (6)
H5	0.1582	0.1794	0.1684	0.055*
C6	0.17296 (7)	0.1768 (3)	0.32460 (16)	0.0384 (5)
H6	0.2066	0.1763	0.3435	0.046*
C7	0.18748 (7)	0.1723 (3)	0.51510 (15)	0.0327 (5)
O5	0.22537 (7)	0.3304 (3)	0.78346 (15)	0.0677 (6)
H5A	0.2185 (10)	0.337 (5)	0.7192 (8)	0.105 (13)*
H5B	0.2483 (8)	0.406 (4)	0.8133 (17)	0.099 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0350 (10)	0.0391 (10)	0.0321 (10)	0.0017 (8)	0.0091 (8)	0.0001 (8)
C8	0.0355 (11)	0.0287 (11)	0.0308 (11)	0.0001 (9)	0.0118 (9)	−0.0007 (10)
C9	0.0386 (12)	0.0512 (14)	0.0318 (12)	0.0013 (11)	0.0164 (10)	−0.0006 (11)
C10	0.0397 (13)	0.0501 (15)	0.0270 (11)	0.0038 (11)	0.0084 (10)	−0.0017 (10)
C11	0.0308 (11)	0.0244 (11)	0.0313 (11)	0.0010 (9)	0.0066 (9)	0.0002 (9)
C12	0.0351 (12)	0.0457 (14)	0.0351 (12)	0.0000 (10)	0.0146 (10)	0.0062 (11)
C13	0.0382 (13)	0.0470 (14)	0.0295 (11)	−0.0006 (11)	0.0087 (10)	0.0030 (10)
O1	0.0495 (9)	0.0399 (10)	0.0303 (8)	0.0061 (8)	0.0000 (7)	0.0033 (7)
O2	0.0475 (9)	0.0372 (9)	0.0318 (8)	−0.0027 (7)	0.0016 (7)	−0.0023 (7)
O3	0.0518 (10)	0.1070 (16)	0.0290 (9)	0.0001 (10)	0.0155 (8)	0.0046 (10)
O4	0.0327 (10)	0.1222 (17)	0.0561 (11)	−0.0020 (10)	0.0209 (8)	0.0071 (11)
N1	0.0352 (11)	0.0479 (12)	0.0340 (11)	0.0011 (9)	0.0144 (9)	0.0027 (9)
C1	0.0319 (11)	0.0280 (11)	0.0249 (10)	0.0011 (9)	0.0076 (9)	0.0001 (9)
C2	0.0322 (11)	0.0307 (11)	0.0272 (11)	0.0012 (9)	0.0116 (9)	−0.0007 (9)
C3	0.0298 (11)	0.0387 (12)	0.0327 (12)	0.0026 (10)	0.0050 (9)	0.0000 (10)
C4	0.0427 (13)	0.0547 (15)	0.0252 (11)	0.0080 (12)	0.0061 (10)	0.0020 (11)
C5	0.0473 (14)	0.0613 (16)	0.0320 (12)	0.0053 (12)	0.0190 (11)	0.0011 (12)
C6	0.0294 (11)	0.0512 (14)	0.0345 (12)	0.0038 (11)	0.0114 (10)	0.0015 (11)
C7	0.0313 (11)	0.0376 (13)	0.0276 (11)	0.0002 (11)	0.0088 (9)	−0.0001 (11)
O5	0.0542 (12)	0.1079 (17)	0.0425 (11)	−0.0330 (12)	0.0192 (9)	−0.0181 (11)

Geometric parameters (Å, °)

N2—C11	1.453 (2)	O3—N1	1.198 (2)
N2—H2A	0.8900	O4—N1	1.207 (2)
N2—H2B	0.8900	N1—C2	1.455 (2)

N2—H2C	0.8902	C1—C6	1.378 (3)
C8—C9	1.377 (3)	C1—C2	1.382 (3)
C8—C13	1.386 (3)	C1—C7	1.496 (3)
C8—C8^{i}	1.477 (4)	C2—C3	1.357 (3)
C9—C10	1.365 (3)	C3—C4	1.370 (3)
C9—H9	0.9300	C3—H3	0.9300
C10—C11	1.363 (3)	C4—C5	1.365 (3)
C10—H10	0.9300	C4—H4	0.9300
C11—C12	1.349 (3)	C5—C6	1.365 (3)
C12—C13	1.374 (3)	C5—H5	0.9300
C12—H12	0.9300	C6—H6	0.9300
C13—H13	0.9300	O5—H5A	0.819 (10)
O1—C7	1.245 (2)	O5—H5B	0.825 (10)
O2—C7	1.247 (2)		
C11—N2—H2A	109.7	O3—N1—C2	118.44 (17)
C11—N2—H2B	109.1	O4—N1—C2	119.08 (18)
H2A—N2—H2B	109.5	C6—C1—C2	116.92 (18)
C11—N2—H2C	109.6	C6—C1—C7	117.24 (17)
H2A—N2—H2C	109.5	C2—C1—C7	125.84 (17)
H2B—N2—H2C	109.5	C3—C2—C1	122.73 (18)
C9—C8—C13	116.70 (19)	C3—C2—N1	117.55 (18)
C9—C8—C8^{i}	121.8 (2)	C1—C2—N1	119.72 (17)
C13—C8—C8^{i}	121.5 (2)	C2—C3—C4	118.96 (19)
C10—C9—C8	121.8 (2)	C2—C3—H3	120.5
C10—C9—H9	119.1	C4—C3—H3	120.5
C8—C9—H9	119.1	C5—C4—C3	119.88 (19)
C11—C10—C9	119.75 (19)	C5—C4—H4	120.1
C11—C10—H10	120.1	C3—C4—H4	120.1
C9—C10—H10	120.1	C4—C5—C6	120.53 (19)
C12—C11—C10	120.59 (18)	C4—C5—H5	119.7
C12—C11—N2	119.94 (17)	C6—C5—H5	119.7
C10—C11—N2	119.47 (17)	C5—C6—C1	120.97 (19)
C11—C12—C13	119.47 (19)	C5—C6—H6	119.5
C11—C12—H12	120.3	C1—C6—H6	119.5
C13—C12—H12	120.3	O1—C7—O2	125.68 (19)
C12—C13—C8	121.70 (19)	O1—C7—C1	116.56 (18)
C12—C13—H13	119.2	O2—C7—C1	117.60 (18)
C8—C13—H13	119.2	H5A—O5—H5B	109.5 (16)
O3—N1—O4	122.47 (18)		

Symmetry codes: (i) −*x*, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1^{ii}	0.89	1.86	2.744 (3)	176
N2—H2B···O2	0.89	1.88	2.772 (3)	176
N2—H2C···O5^{iii}	0.89	1.90	2.786 (2)	178
O5—H5A···O2	0.819 (10)	2.034 (10)	2.852 (3)	177 (3)

O5—H5B···O1^{iv}	0.825 (10)	1.980 (14)	2.722 (3)	149 (2)

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

Fig. 1

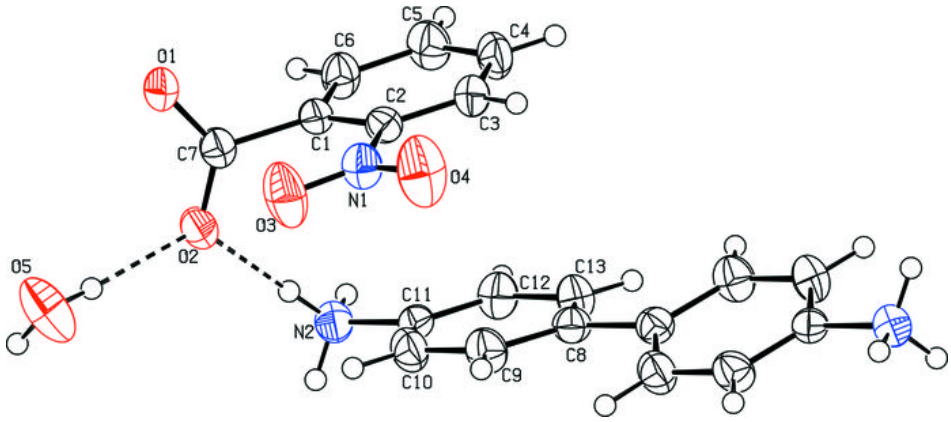

Fig. 2