Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Biphenyl-3,3′,4,4′-tetraamine

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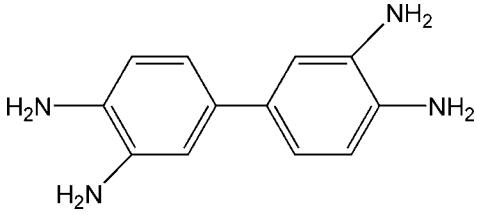
Received 14 January 2010; accepted 3 April 2010

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.051; wR factor = 0.156; data-to-parameter ratio = 13.4.

The title compound, $C_{12}H_{14}N_4$, has a crystallographically imposed centre of symmetry. Intermolecular N—H···N hydrogen bonds between amino groups link adjacent molecules into a three-dimensional network where ten-membered hydrogen-bonded rings are observed.

Related literature

For a related compound, see: Dobrzycki & Wozniak (2007).



Experimental

Crystal data

$C_{12}H_{14}N_4$
M_r = 214.27
Monoclinic, $P2_1/c$
a = 9.646 (4) Å
b = 7.476 (3) Å
c = 7.751 (3) Å
β = 95.773 (5)°
V = 556.1 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 291 K
0.14 × 0.12 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.989, T_{max} = 0.992
2698 measured reflections
979 independent reflections
724 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.156
S = 1.09
979 reflections
73 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N2^{i}	0.90	2.39	3.224 (2)	154
N2—H2A···N1^{ii}	0.90	2.35	3.124 (2)	145

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

WH acknowledges the National Natural Science Foundation of China (grant No. 20871065) and the Jiangsu Province Department of Science and Technology (grant No. BK2009226) for financial aid.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2140).

References

Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dobrzycki, L. & Wozniak, K. (2007). *CrystEngComm*, **9**, 1029–1040.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2010). E**66**, o1060 [**doi:10.1107/S1600536810012511**]

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Comment

The crystal structure of 3,3',4,4'-tetrammoniobiphenyl tetrachloride dihydrate (Dobrzycki & Wozniak, 2007) has been reported in literature. In this paper, we report the X-ray single-crystal structure of 3,3',4,4'-tetrammoniobiphenyl (I).

The molecular structure of (I) is illustrated in Fig. 1. Two amino groups in the 3-position lie in the opposite sides of the molecular plane. The dihedral angle between phenyl rings of adjacent molecules is 86.3 (2)°. Intermolecular N—H···N hydrogen bonds between amino groups link adjacent molecules into a three-dimensional network, where ten-membered hydrogen-bonded rings are observed (Fig. 2).

Experimental

The title compound was purchased directly from TCI. Single crystals suitable for X-ray diffraction were grown from a methanol solution by slow evaporation in air at room temperature for one week.

Refinement

H atoms were placed in geometrically idealized positions and refined as riding, with C—H = 0.93 Å and N—H = 0.86–0.90 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N).

Figures

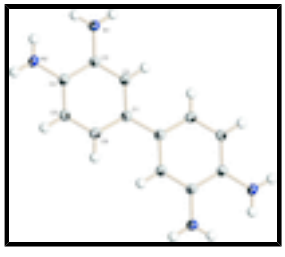

Fig. 1. The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

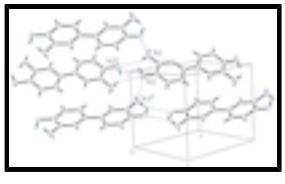

Fig. 2. Perspective view of the hydrogen bonding interactions in the crystal packing of (I), where the hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) -*x*, -*y* + 2, -*z*; (ii) -*x*, *y* - 1/2, -*z* + 1/2.]

Biphenyl-3,3',4,4'-tetraamine

Crystal data

$C_{12}H_{14}N_4$	$F(000) = 228$
$M_r = 214.27$	$D_x = 1.280$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 931 reflections
$a = 9.646$ (4) Å	$\theta = 2.5$–$27.0°$
$b = 7.476$ (3) Å	$\mu = 0.08$ mm^{-1}
$c = 7.751$ (3) Å	$T = 291$ K
$\beta = 95.773$ (5)°	Block, colourless
$V = 556.1$ (4) Å^3	0.14 × 0.12 × 0.10 mm
$Z = 2$	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	979 independent reflections
Radiation source: fine-focus sealed tube	724 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.075$
ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -9 \rightarrow 11$
$T_{min} = 0.989$, $T_{max} = 0.992$	$k = -6 \rightarrow 8$
2698 measured reflections	$l = -8 \rightarrow 9$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.051$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.156$	H-atom parameters constrained
$S = 1.09$	$w = 1/[\sigma^2(F_o^2) + (0.0926P)^2 + 0.0016P]$ where $P = (F_o^2 + 2F_c^2)/3$
979 reflections	$(\Delta/\sigma)_{max} < 0.001$
73 parameters	$\Delta\rho_{max} = 0.18$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Experimental. The structure was solved by direct methods (Bruker, 2000) and successive difference Fourier syntheses.

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 $F^2 > \sigma(F^2)$ 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	0.42719 (17)	0.9895 (2)	0.4590 (2)	0.0335 (5)
C2	0.37707 (18)	1.0872 (2)	0.3125 (2)	0.0356 (5)
H2	0.4378	1.1639	0.2629	0.043*
C3	0.24074 (18)	1.0749 (2)	0.2378 (2)	0.0336 (5)
C4	0.14684 (18)	0.9615 (2)	0.3120 (2)	0.0341 (5)
C5	0.1965 (2)	0.8586 (2)	0.4523 (2)	0.0391 (6)
H5	0.1367	0.7785	0.4991	0.047*
C6	0.3330 (2)	0.8714 (3)	0.5255 (2)	0.0421 (6)
H6	0.3629	0.8003	0.6205	0.051*
N1	0.18955 (16)	1.1838 (2)	0.0986 (2)	0.0442 (5)
H1A	0.1515	1.1130	0.0127	0.053*
H1B	0.2437	1.2600	0.0562	0.053*
N2	0.00747 (15)	0.9522 (2)	0.23637 (19)	0.0418 (5)
H2A	−0.0484	0.9167	0.3161	0.050*
H2B	−0.0130	1.0651	0.2025	0.050*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0327 (11)	0.0338 (10)	0.0336 (10)	0.0017 (8)	0.0013 (8)	−0.0006 (8)
C2	0.0326 (11)	0.0381 (11)	0.0362 (10)	−0.0008 (8)	0.0043 (8)	0.0023 (8)
C3	0.0355 (11)	0.0348 (10)	0.0300 (9)	0.0026 (8)	0.0004 (8)	−0.0012 (7)
C4	0.0327 (11)	0.0353 (10)	0.0337 (10)	−0.0013 (8)	0.0007 (8)	−0.0053 (8)
C5	0.0376 (12)	0.0392 (11)	0.0397 (11)	−0.0082 (8)	−0.0003 (9)	0.0049 (8)
C6	0.0422 (12)	0.0420 (11)	0.0404 (11)	−0.0036 (9)	−0.0046 (9)	0.0092 (8)
N1	0.0434 (11)	0.0480 (10)	0.0396 (10)	−0.0045 (7)	−0.0033 (8)	0.0113 (7)
N2	0.0324 (10)	0.0493 (11)	0.0424 (10)	−0.0036 (7)	−0.0026 (7)	0.0017 (7)

Geometric parameters (Å, °)

C1—C2	1.395 (3)	C4—N2	1.413 (2)
C1—C6	1.401 (3)	C5—C6	1.384 (3)
C1—C1^{i}	1.491 (3)	C5—H5	0.9300
C2—C3	1.386 (2)	C6—H6	0.9300
C2—H2	0.9300	N1—H1A	0.8999
C3—N1	1.401 (2)	N1—H1B	0.8600
C3—C4	1.405 (2)	N2—H2A	0.9000

C4—C5	1.379 (3)	N2—H2B	0.9000
C2—C1—C6	116.41 (17)	C4—C5—C6	121.72 (17)
C2—C1—C1^{i}	121.8 (2)	C4—C5—H5	119.1
C6—C1—C1^{i}	121.8 (2)	C6—C5—H5	119.1
C3—C2—C1	122.83 (17)	C5—C6—C1	121.21 (18)
C3—C2—H2	118.6	C5—C6—H6	119.4
C1—C2—H2	118.6	C1—C6—H6	119.4
C2—C3—N1	121.97 (16)	C3—N1—H1A	108.3
C2—C3—C4	119.50 (16)	C3—N1—H1B	119.9
N1—C3—C4	118.29 (16)	H1A—N1—H1B	108.9
C5—C4—C3	118.20 (17)	C4—N2—H2A	109.9
C5—C4—N2	122.70 (16)	C4—N2—H2B	104.2
C3—C4—N2	119.05 (16)	H2A—N2—H2B	110.4
C6—C1—C2—C3	2.1 (3)	N1—C3—C4—N2	4.4 (2)
C1^{i}—C1—C2—C3	−177.55 (18)	C3—C4—C5—C6	3.2 (3)
C1—C2—C3—N1	175.14 (17)	N2—C4—C5—C6	−179.28 (17)
C1—C2—C3—C4	0.8 (3)	C4—C5—C6—C1	−0.3 (3)
C2—C3—C4—C5	−3.4 (3)	C2—C1—C6—C5	−2.3 (3)
N1—C3—C4—C5	−177.99 (15)	C1^{i}—C1—C6—C5	177.30 (19)
C2—C3—C4—N2	178.99 (15)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···N2^{ii}	0.90	2.39	3.224 (2)	154.
N2—H2A···N1^{iii}	0.90	2.35	3.124 (2)	145.

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

Fig. 1

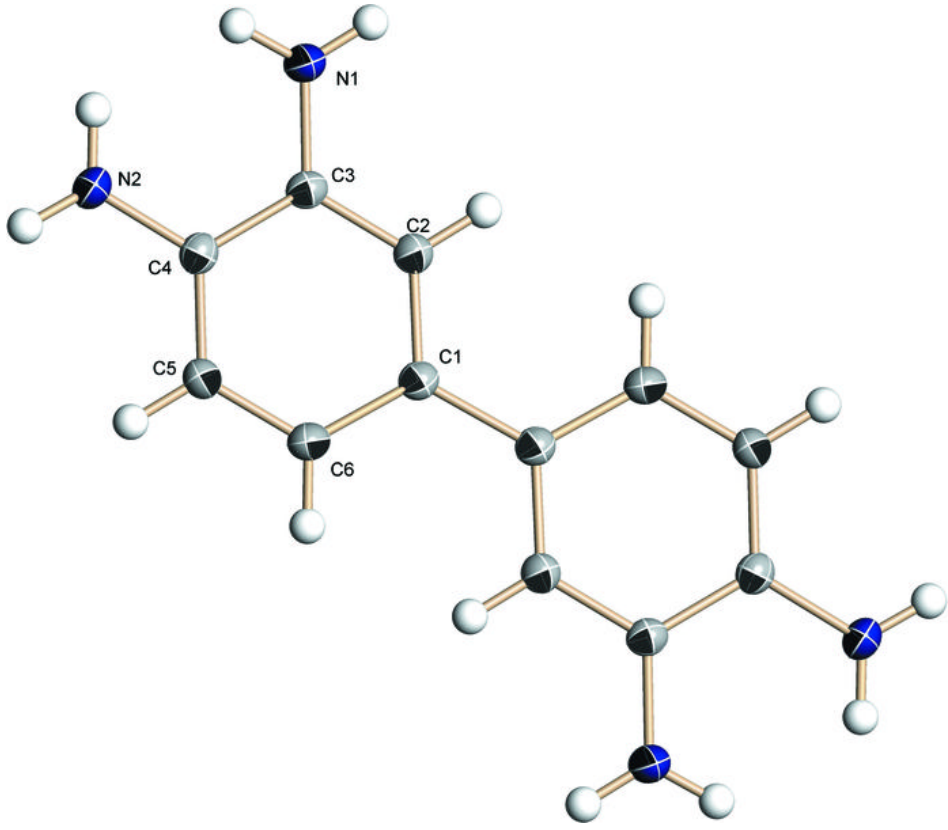

Fig. 2