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## Structure Reports

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

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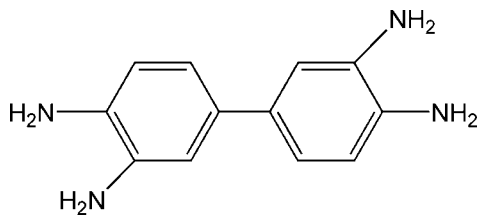
Received 14 January 2010; accepted 3 April 2010

Key indicators: single-crystal X-ray study;  $T = 291$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.051;  $wR$  factor = 0.156; data-to-parameter ratio = 13.4.

The title compound,  $\text{C}_{12}\text{H}_{14}\text{N}_4$ , has a crystallographically imposed centre of symmetry. Intermolecular  $\text{N}-\text{H}\cdots\text{N}$  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

$\text{C}_{12}\text{H}_{14}\text{N}_4$   
 $M_r = 214.27$   
Monoclinic,  $P2_1/c$   
 $a = 9.646$  (4) Å  
 $b = 7.476$  (3) Å  
 $c = 7.751$  (3) Å  
 $\beta = 95.773$  (5)°  
 $V = 556.1$  (4) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 291$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.075$

## 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<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.30$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                                     | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{N1}-\text{H1A}\cdots\text{N2}^{\text{i}}$  | 0.90  | 2.39        | 3.224 (2)   | 154           |
| $\text{N2}-\text{H2A}\cdots\text{N1}^{\text{ii}}$ | 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.

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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.* **A64**, 112–122.

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# supporting information

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

Hui-Fen Qian and Wei Huang

### S1. Comment

The crystal structure of 3,3',4,4'-tetrammonibiphenyl 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'-tetrammonibiphenyl (I).

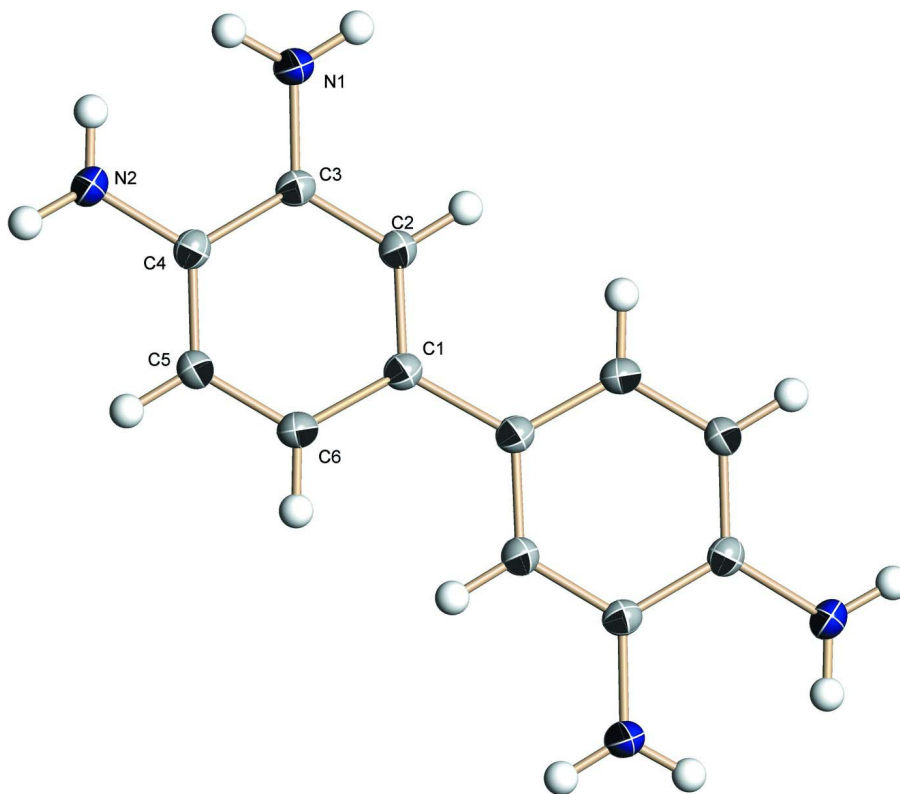
The molecular structure of (I) is illustrated in Fig. 1. Two amino groups in the 3-position lie in the opposite sides of the molecular plane. The dihedral angle between phenyl rings of adjacent molecules is 86.3 (2)°. Intermolecular N—H⋯N hydrogen bonds between amino groups link adjacent molecules into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

### S2. Experimental

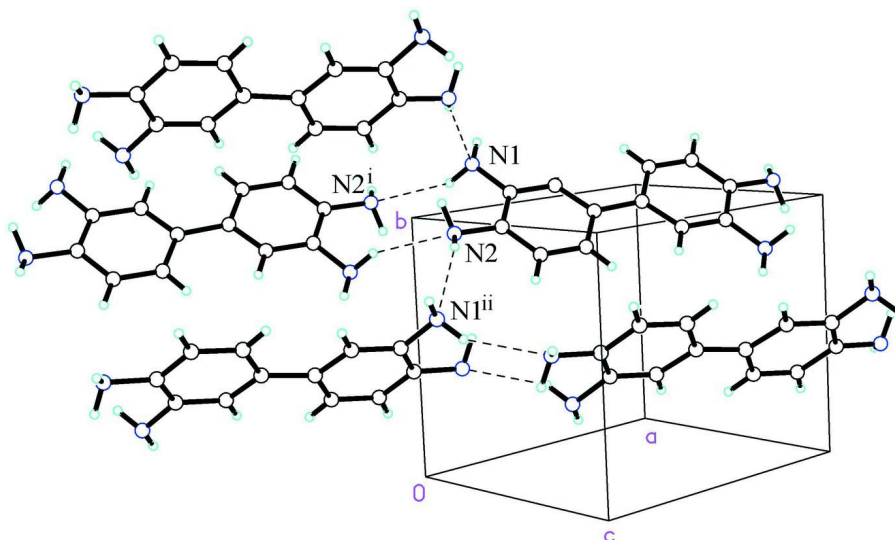
The title compound was purchased directly from TCI. Single crystals suitable for X-ray diffraction were grown from a methanol solution by slow evaporation in air at room temperature for one week.

### S3. 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  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ .

**Figure 1**

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

**Figure 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<sub>12</sub>H<sub>14</sub>N<sub>4</sub> $M_r = 214.27$ Monoclinic,  $P2_1/c$ 

Hall symbol: -P 2ybc

 $a = 9.646$  (4) Å $b = 7.476$  (3) Å $c = 7.751$  (3) Å $\beta = 95.773$  (5)° $V = 556.1$  (4) Å<sup>3</sup> $Z = 2$  $F(000) = 228$  $D_x = 1.280$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 931 reflections

 $\theta = 2.5$ – $27.0$ ° $\mu = 0.08$  mm<sup>-1</sup> $T = 291$  K

Block, colourless

 $0.14 \times 0.12 \times 0.10$  mm

## Data collection

Bruker SMART 1K CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega$  scansAbsorption correction: multi-scan  
(SADABS; Bruker, 2000) $T_{\min} = 0.989$ ,  $T_{\max} = 0.992$ 

2698 measured reflections

979 independent reflections

724 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.075$  $\theta_{\text{max}} = 25.0$ °,  $\theta_{\text{min}} = 2.1$ ° $h = -9 \rightarrow 11$  $k = -6 \rightarrow 8$  $l = -8 \rightarrow 9$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.051$  $wR(F^2) = 0.156$  $S = 1.09$ 

979 reflections

73 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0926P)^2 + 0.0016P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} < 0.001$  $\Delta\rho_{\text{max}} = 0.18$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.30$  e Å<sup>-3</sup>

## 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<sup>2</sup>)

|    | $x$          | $y$        | $z$        | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|--------------|------------|------------|----------------------------------|
| 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<sup>2</sup>)*

|    | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|----|-------------|-------------|-------------|-------------|-------------|-------------|
| 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 <sup>i</sup>    | 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 <sup>i</sup> | 121.8 (2)   | C4—C5—H5   | 119.1       |
| C6—C1—C1 <sup>i</sup> | 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 <sup>i</sup> —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 <sup>i</sup> —C1—C6—C5 | 177.30 (19)  |
| C2—C3—C4—N2               | 178.99 (15)  |                           |              |

Symmetry code: (i)  $-x+1, -y+2, -z+1$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

| <i>D</i> —H $\cdots$ <i>A</i>     | <i>D</i> —H | H $\cdots$ <i>A</i> | <i>D</i> $\cdots$ <i>A</i> | <i>D</i> —H $\cdots$ <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N1—H1A $\cdots$ N2 <sup>ii</sup>  | 0.90        | 2.39                | 3.224 (2)                  | 154                           |
| N2—H2A $\cdots$ N1 <sup>iii</sup> | 0.90        | 2.35                | 3.124 (2)                  | 145                           |

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