Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,4′-(Phenylimino)dibenzaldehyde

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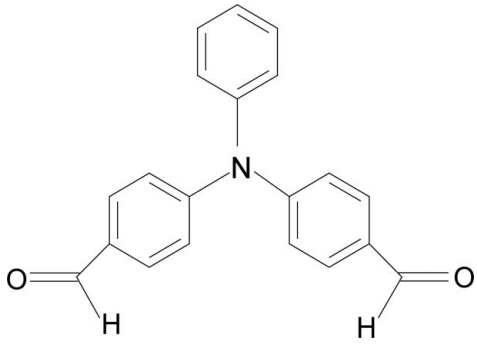
Received 15 July 2009; accepted 12 August 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.187; data-to-parameter ratio = 10.4.

The asymmetric unit of the title compound, $C_{20}H_{15}NO_2$, contains one half-molecule with the central N atom and two C atoms of the benzene moiety lying on a twofold rotation axis. Weak C—H···O interactions join the molecules together into an infinite three-dimensional network.

Related literature

The title compound was obtained unintentionally as the product of an attempted purification of tris(4-formylphenyl)amine, which is used as a building block in materials chemistry (Thomas *et al.*, 2005). For hydrogen bonding, see: Krishnamohan Sharma & Desiraju (1994). =



Experimental

Crystal data

$C_{20}H_{15}NO_2$ M_r = 301.33
Orthorhombic, *Pbcn*
a = 8.836 (2) Å
b = 9.710 (2) Å
c = 18.621 (4) Å
V = 1597.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 K
0.32 × 0.18 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.980, T_{max} = 0.992
7399 measured reflections
1412 independent reflections
1087 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.187
S = 1.07
1412 reflections
136 parameters
All H-atom parameters refinemed
$\Delta\rho_{max}$ = 0.29 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
C5—H3···O1^{i}	0.96 (3)	2.48 (3)	3.396 (4)	159 (3)
C9—H7···O1^{ii}	0.95 (3)	2.55 (4)	3.495 (4)	173 (3)

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank the Shandong Distinguished Middle-aged and Young Scientist Encouragement and Reward Scheme (No. 2006BS04006) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2093).

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supporting information

Acta Cryst. (2009). E**65**, o2192 [doi:10.1107/S1600536809031924]

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S1. Comment

The popularity of tris(4-formylphenyl)amine as a building block is rapidly growing in materials chemistry (Thomas *et al.*, 2005). The title compound, (I) (Fig. 1), [$C_{20}H_{15}NO_2$], was obtained unintentionally as the product of an attempted purification of tris(4-formylphenyl)amine.

The molecule of (I) has three phenyl rings, but the asymmetric unit contains only one half of (I). The ring (C7 to C10) makes a dihedral angle of 70.36 (8)° with ring (C1 to C6), and a dihedral angle of 70.22 (8)° with ring ($C1^i$ to $C6^i$) (symmetry code: (i) -*x*, +*y*, 0.5 - *z*). The dihedral angle of the latter two is 66.66 (8)°.

The *PLATON* program (Spek, 2009) suggests that there are no classic hydrogen bonds, but there are weak C—H···O hydrogen bonds (Table 2, Krishnamohan Sharma & Desiraju, 1994) between carbonyl oxygen and H atoms on the adjacent molecules, which link them into infinite three-dimensional network[Fig. 2].

S2. Experimental

Phosphorus oxychloride ($POCl_3$) and *N*,*N*-dimethylformamide (DMF) were analytical reagent and used after the process of removing oxygen and water. Other organic solvents and common materials used for synthesis were used without further purification. The compound (I) was prepared by mixing 5.0 g triphenylamine and an ice-cooled mixture of $POCl_3$(47.5 mL) and DMF(36.3 mL) under N_2. The resulting mixture was stirred at 95°C for 4 h under N_2. After cooling to room temperature,the mixture was poured into ice-water(1*L*),and basified with 1*M* NaOH. After filtration, the crude product was purified by column chromatography with petroleum ether/ethyl acetate (8/1,in volume ratio) to yield I(yellow transparent crystal). Elemental analysis Calcd: C 79.72, H 5.02, N 4.65%. Found: C 79.81, H 5.16, N 4.57%.

S3. Refinement

All the H atoms were located in the difference Fourier map and all parameters are refined independently.

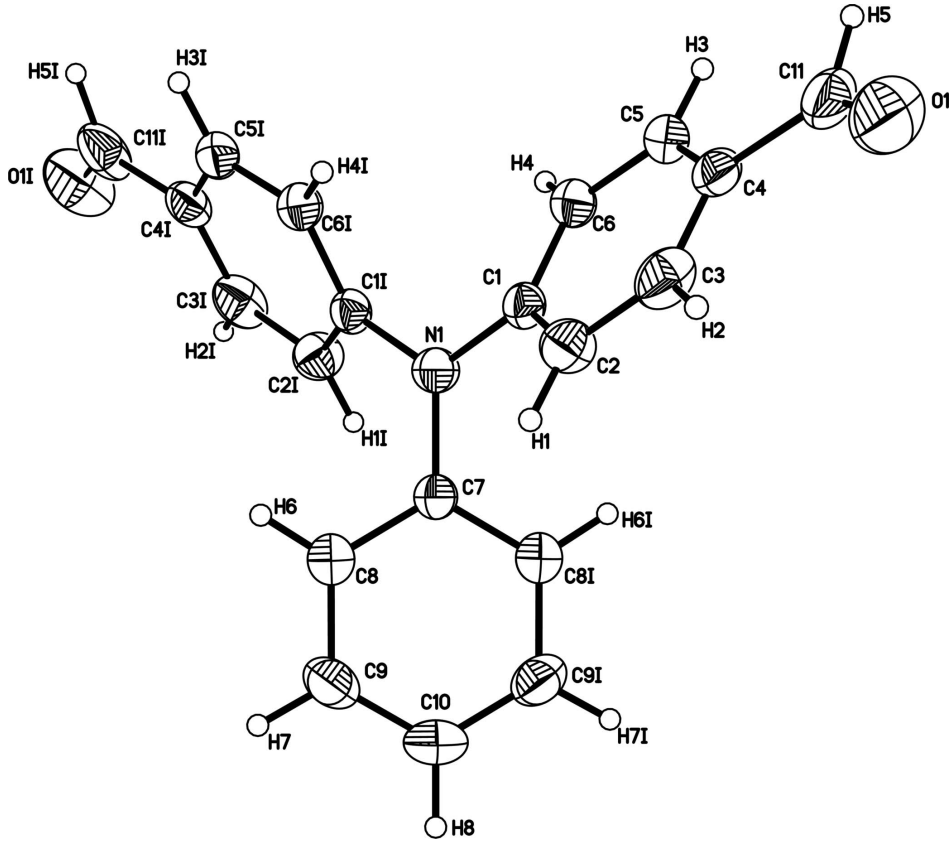

Figure 1

The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms. C1I to C6I,C8I,C9I,C11I,O1I and H1I to H7I were created by GROW and the symmetry code of "I" is -*x*, +*y*, 0.5 - *z*.

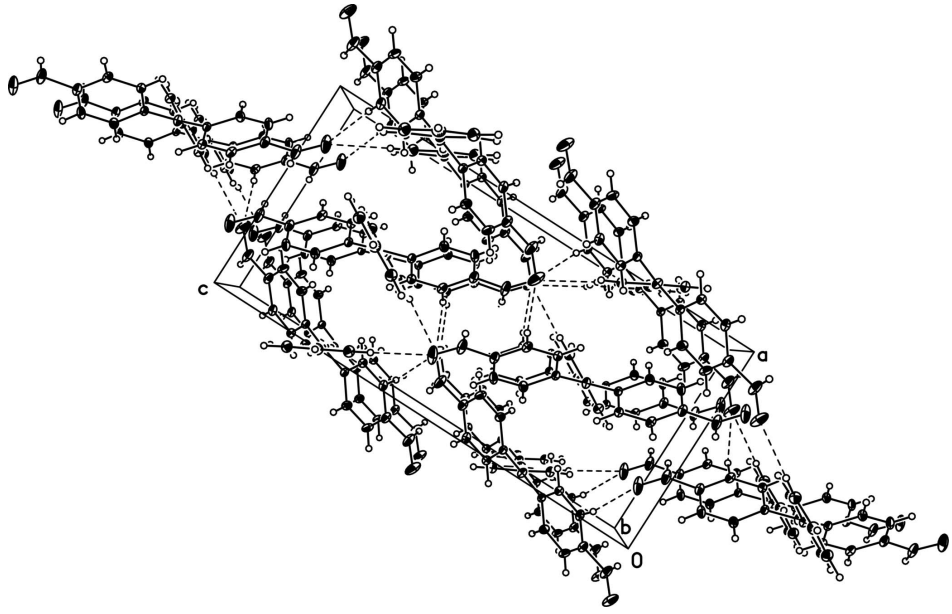

Figure 2

The packing of (I), viewed down the *b* axis.

4,4′-(Phenylimino)dibenzaldehyde

Crystal data

$C_{20}H_{15}NO_2$
M_r = 301.33
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
a = 8.836 (2) Å
b = 9.710 (2) Å
c = 18.621 (4) Å
V = 1597.6 (6) Å^3
Z = 4
F(000) = 632
D_x = 1.253 Mg m^{-3}
Melting point = 417–419 K
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 378 reflections
θ = 1.7–25.0°
μ = 0.08 mm^{-1}
T = 298 K
Block, yellow
0.32 × 0.18 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
π and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.980, T_{max} = 0.992
7399 measured reflections
1412 independent reflections
1087 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −10→10
k = −11→8
l = −22→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.187
S = 1.07
1412 reflections
136 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0996P)^2 + 0.4228P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.0438 (3)	0.5517 (3)	0.31252 (11)	0.0446 (6)

C2	0.1601 (3)	0.6013 (3)	0.35553 (15)	0.0587 (8)
C3	0.1977 (4)	0.5311 (3)	0.41719 (16)	0.0670 (9)
C4	0.1216 (3)	0.4133 (3)	0.43801 (13)	0.0593 (8)
C5	0.0081 (3)	0.3648 (3)	0.39479 (14)	0.0573 (7)
C6	−0.0305 (3)	0.4314 (3)	0.33246 (13)	0.0499 (7)
C7	0.0000	0.7700 (3)	0.2500	0.0458 (8)
C8	0.0651 (3)	0.8417 (3)	0.19405 (15)	0.0581 (8)
C9	0.0634 (4)	0.9841 (3)	0.19400 (18)	0.0697 (9)
C10	0.0000	1.0547 (5)	0.2500	0.0712 (12)
C11	0.1559 (5)	0.3415 (4)	0.50518 (16)	0.0873 (12)
N1	0.0000	0.6232 (3)	0.2500	0.0528 (8)
O1	0.2531 (4)	0.3709 (3)	0.54591 (13)	0.1245 (12)
H1	0.212 (3)	0.682 (3)	0.3407 (14)	0.067 (8)*
H2	0.271 (4)	0.566 (4)	0.4436 (17)	0.089 (10)*
H3	−0.041 (3)	0.282 (4)	0.4107 (16)	0.090 (10)*
H4	−0.111 (3)	0.399 (3)	0.3032 (13)	0.059 (8)*
H5	0.101 (4)	0.248 (5)	0.5133 (19)	0.113 (13)*
H6	0.106 (3)	0.790 (3)	0.1563 (16)	0.071 (8)*
H7	0.110 (3)	1.031 (3)	0.1554 (18)	0.088 (10)*
H8	0.0000	1.153 (6)	0.2500	0.092 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0513 (14)	0.0464 (14)	0.0362 (12)	0.0041 (11)	−0.0018 (10)	−0.0007 (10)
C2	0.0626 (17)	0.0572 (17)	0.0563 (16)	−0.0078 (14)	−0.0105 (13)	−0.0025 (14)
C3	0.0714 (19)	0.073 (2)	0.0566 (17)	0.0136 (16)	−0.0257 (15)	−0.0160 (16)
C4	0.086 (2)	0.0492 (15)	0.0429 (14)	0.0234 (14)	−0.0042 (14)	−0.0046 (12)
C5	0.0769 (19)	0.0508 (16)	0.0441 (14)	0.0065 (14)	0.0073 (14)	0.0005 (12)
C6	0.0540 (15)	0.0510 (15)	0.0447 (13)	−0.0005 (12)	−0.0015 (12)	−0.0007 (12)
C7	0.0535 (19)	0.0444 (19)	0.0396 (17)	0.000	−0.0017 (15)	0.000
C8	0.0658 (17)	0.0573 (18)	0.0513 (15)	0.0000 (13)	0.0066 (13)	0.0003 (13)
C9	0.081 (2)	0.0585 (19)	0.0696 (19)	−0.0096 (15)	0.0021 (16)	0.0158 (16)
C10	0.080 (3)	0.044 (2)	0.090 (3)	0.000	−0.005 (2)	0.000
C11	0.138 (3)	0.077 (2)	0.0460 (17)	0.045 (2)	−0.017 (2)	−0.0119 (17)
N1	0.072 (2)	0.0447 (17)	0.0419 (15)	0.000	−0.0079 (14)	0.000
O1	0.181 (3)	0.117 (2)	0.0762 (16)	0.059 (2)	−0.0570 (19)	−0.0115 (15)

Geometric parameters (Å, º)

C1—C2	1.389 (4)	C7—C8^{i}	1.379 (3)
C1—C6	1.391 (4)	C7—C8	1.379 (3)
C1—N1	1.410 (3)	C7—N1	1.425 (4)
C2—C3	1.376 (4)	C8—C9	1.383 (4)
C2—H1	0.95 (3)	C8—H6	0.93 (3)
C3—C4	1.383 (4)	C9—C10	1.368 (4)
C3—H2	0.88 (3)	C9—H7	0.95 (3)
C4—C5	1.369 (4)	C10—C9^{i}	1.368 (4)

C4—C11	1.464 (4)	C10—H8	0.95 (5)
C5—C6	1.372 (4)	C11—O1	1.181 (4)
C5—H3	0.96 (3)	C11—H5	1.04 (4)
C6—H4	0.95 (3)	N1—C1^{i}	1.410 (3)
C2—C1—C6	119.1 (2)	C8^{i}—C7—C8	119.4 (4)
C2—C1—N1	120.6 (2)	C8^{i}—C7—N1	120.32 (18)
C6—C1—N1	120.3 (2)	C8—C7—N1	120.32 (18)
C3—C2—C1	119.2 (3)	C7—C8—C9	120.1 (3)
C3—C2—H1	122.3 (16)	C7—C8—H6	117.3 (17)
C1—C2—H1	118.5 (16)	C9—C8—H6	122.6 (17)
C2—C3—C4	121.7 (3)	C10—C9—C8	120.3 (3)
C2—C3—H2	117 (2)	C10—C9—H7	121 (2)
C4—C3—H2	121 (2)	C8—C9—H7	119 (2)
C5—C4—C3	118.4 (3)	C9^{i}—C10—C9	119.9 (4)
C5—C4—C11	119.3 (3)	C9^{i}—C10—H8	120.1 (2)
C3—C4—C11	122.2 (3)	C9—C10—H8	120.1 (2)
C4—C5—C6	121.1 (3)	O1—C11—C4	125.8 (4)
C4—C5—H3	115.9 (19)	O1—C11—H5	117 (2)
C6—C5—H3	122.9 (19)	C4—C11—H5	116 (2)
C5—C6—C1	120.3 (3)	C1—N1—C1^{i}	121.0 (3)
C5—C6—H4	121.0 (16)	C1—N1—C7	119.50 (14)
C1—C6—H4	118.6 (16)	C1^{i}—N1—C7	119.50 (14)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H3···O1^{ii}	0.96 (3)	2.48 (3)	3.396 (4)	159 (3)
C9—H7···O1^{iii}	0.95 (3)	2.55 (4)	3.495 (4)	173 (3)

Symmetry codes: (ii) $x-1/2, -y+1/2, -z+1$; (iii) $-x+1/2, -y+3/2, z-1/2$.