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4,4′-(Phenylimino)dibenzaldehyde

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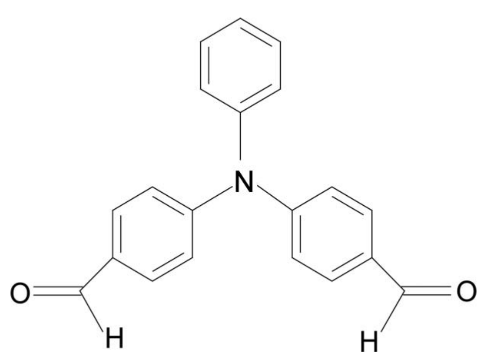
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.187; data-to-parameter ratio = 10.4.

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).

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

References

Bruker (2000). *SMART*, *SAINT-Plus* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Krishnamohan Sharma, C. V. & Desiraju, G. R. (1994). *J. Chem. Soc. Perkin Trans. 2*, pp. 2345–2352.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Thomas, M., Said, G., Mohamed, A., Mireille, B. & Olivier, M. (2005). *Synthesis*, pp. 1771–1774.

supplementary materials

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4,4'-(Phenylimino)dibenzaldehyde

Y.-G. Wang, X.-J. Tan and D.-X. Xing

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].

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%.

Refinement

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

Figures

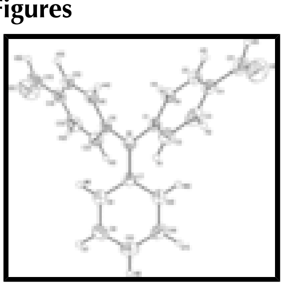

Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms. C1I to C6I,C8I,C9I,C11I,O1I and H1I to H7I were created by GROW and the symmetry code of "I" is -*x*, +*y*, 0.5 - *z*.

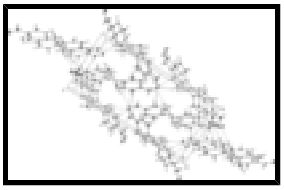

Fig. 2. The packing of (I), viewed down the *b* axis.

4,4'-(Phenylimino)dibenzaldehyde

Crystal data

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

Data collection

Bruker SMART CCD area-detector diffractometer	1412 independent reflections
Radiation source: fine-focus sealed tube	1087 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.043
T = 298 K	θ_{max} = 25.0°
π and ω scans	θ_{min} = 2.2°
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -10 \rightarrow 10$
T_{min} = 0.980, T_{max} = 0.992	$k = -11 \rightarrow 8$
7399 measured reflections	$l = -22 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.064	All H-atom parameters refined
$wR(F^2)$ = 0.187	$w = 1/[\sigma^2(F_o^2) + (0.0996P)^2 + 0.4228P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max}$ < 0.001
1412 reflections	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
136 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	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, °)

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

Hydrogen-bond geometry (Å, °)

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) $-x+1/2, -y+3/2, z-1/2$.

Fig. 1

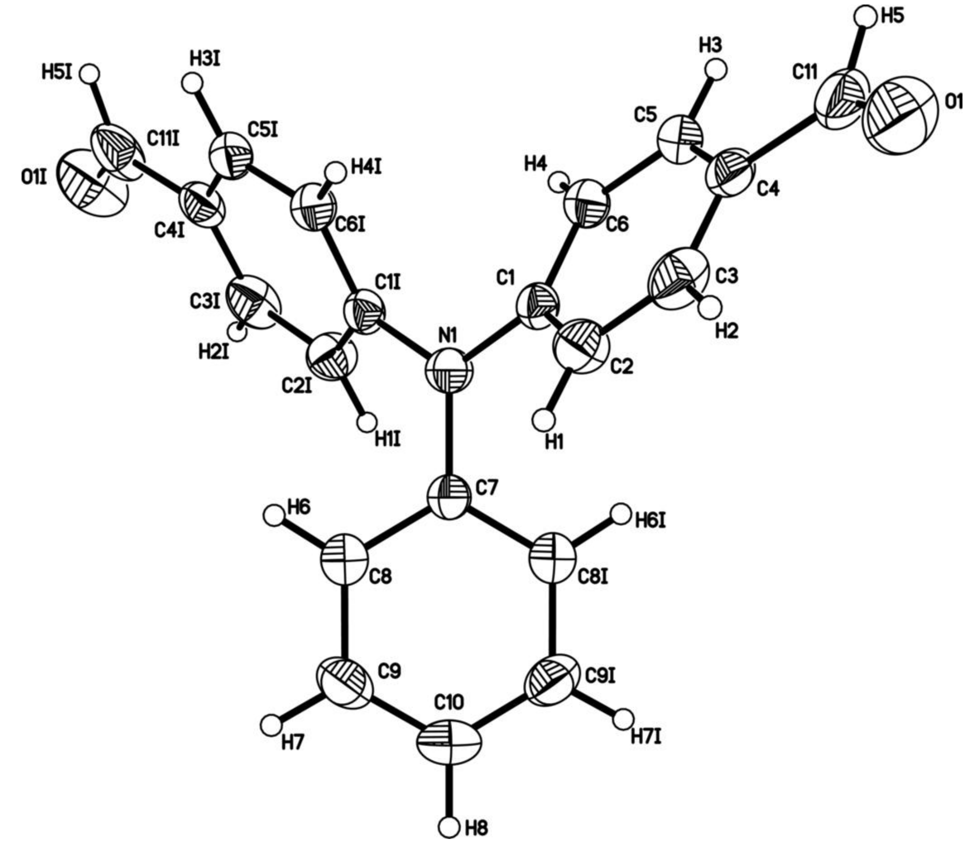
H5I
H3I
H3
H5
C11
O1
C5
C11I
C5I
O1I
H4I
H4
C4
C4I
C6I
C6
C3
C1I
C1
C3I
H2
N1
H2I
C2
C2I
H1I
H1
C7
H6
H6I
C8
C8I
C9
C9I
C10
H7
H7I
H8

Fig. 2