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2,5-Dibenzoylbenzene-1,4-diaminium dichloride

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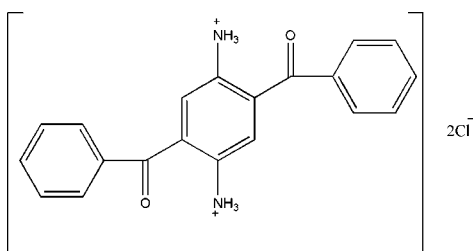
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.055; wR factor = 0.163; data-to-parameter ratio = 13.5.

The asymmetric unit of the title compound, $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_2^{2+} \cdot 2\text{Cl}^-$, is composed of one-half of the 2,5-dibenzoylbenzene-1,4-diaminium dication, located on a centre of inversion, and one Cl^- ion. The dihedral angle between the central benzene ring and the benzoyl phenyl ring is $53.3(2)^\circ$. In the crystal structure, ions are linked to form a two-dimensional network parallel to the $(10\bar{1})$ plane by $\text{N}-\text{H} \cdots \text{Cl}$ hydrogen bonds.

Related literature

For bond-length data, see: Allen *et al.* (1987). For general background, see: Antoniadis *et al.* (1994); Imai *et al.* (1975); Kolosov *et al.* (2002); Tonzola *et al.* (2003).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_2^{2+} \cdot 2\text{Cl}^-$
 $M_r = 389.26$
 Monoclinic, $P2_1/n$
 $a = 12.373(3)$ Å
 $b = 5.195(1)$ Å
 $c = 14.315(3)$ Å
 $\beta = 104.46(3)^\circ$

$V = 891.0(4)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.38$ mm⁻¹
 $T = 298(2)$ K
 $0.40 \times 0.10 \times 0.10$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.862$, $T_{\max} = 0.963$
 1754 measured reflections

1754 independent reflections
 1232 reflections with $I > 2\sigma(I)$
 3 standard reflections every 200 reflections
 intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.162$
 $S = 1.08$
 1754 reflections
 130 parameters
 3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N1}-\text{H1N} \cdots \text{Cl1}^{\text{i}}$	0.87 (4)	2.29 (4)	3.155 (3)	172 (4)
$\text{N1}-\text{H2N} \cdots \text{Cl1}$	0.87 (4)	2.33 (4)	3.187 (3)	174 (4)
$\text{N1}-\text{H3N} \cdots \text{Cl1}^{\text{ii}}$	0.87 (3)	2.29 (3)	3.159 (4)	175 (2)

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

Data collection: *CAD-4 Software* (Enraf-Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Bruker, 2000); 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: CI2546).

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supplementary materials

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2,5-Dibenzoylbenzene-1,4-diaminium dichloride

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Comment

2,5-Dibenzoyl-1,4-phenylenediamine (DBPDA) is one of the important monomers, being utilized to synthesize organic semiconductors and conjugated polymers containing anthrazoline unit (Tonzola *et al.*, 2003), which are of wide current interest for applications in electronic and optoelectronic devices including light-emitting diodes (Kolosov *et al.*, 2002), thin film transistors, and photovoltaic cells (Antoniadis *et al.*, 1994). We report here the crystal structure of the title compound.

The asymmetric unit is composed of one-half of the 2,5-dibenzoyl-1,4-phenylenediaminium dication located on a centre of inversion, and one chloride ion (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The dihedral angle between the C1—C6 and C8—C10/C8A—C10A rings is 53.3 (2)°.

In the crystal structure, molecules are connected together by N—H...Cl hydrogen bonds (Table 1) to form a two-dimensional network parallel to the (1 0 $\bar{1}$) plane (Fig. 2).

Experimental

2,5-Dibenzoyl-1,4-phenylenediamine was synthesized as reported elsewhere (Imai *et al.*, 1975). Single crystals suitable for X-ray diffraction were obtained by dissolving the compound (2.0 g, 6.3 mmol) in hydrochloric acid (50 ml, 1.0 mol/l) and allowing the solution to evaporate at room temperature for about 25 d.

Refinement

N-bound H atoms were located in a difference map and refined with the N—H distances restrained to be equal. C-bound H atoms were positioned geometrically (C—H = 0.93 Å) and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

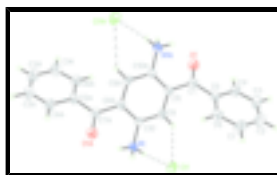


Fig. 1. The molecular structure of the title compound, showing 40% probability displacement ellipsoids. Atoms labelled with the suffix a are generated by the symmetry operations (1 - x, 1 - y, -z). Hydrogen bonds are shown as dashed lines.

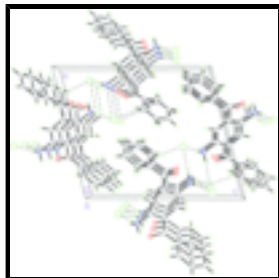


Fig. 2. Crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

2,5-Dibenzoylbenzene-1,4-diaminium dichloride

Crystal data

$C_{20}H_{18}N_2O_2^{2+} \cdot 2Cl^-$

$M_r = 389.26$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 12.373$ (3) Å

$b = 5.195$ (1) Å

$c = 14.315$ (3) Å

$\beta = 104.46$ (3)°

$V = 891.0$ (4) Å³

$Z = 2$

$F_{000} = 404$

$D_x = 1.451$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9\text{--}13^\circ$

$\mu = 0.38$ mm⁻¹

$T = 298$ (2) K

Block, colourless

$0.40 \times 0.10 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.862$, $T_{\max} = 0.963$

1754 measured reflections

1754 independent reflections

1232 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.0000$

$\theta_{\max} = 26.0^\circ$

$\theta_{\min} = 2.0^\circ$

$h = -15 \rightarrow 14$

$k = 0 \rightarrow 6$

$l = 0 \rightarrow 17$

3 standard reflections

every 200 reflections

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.162$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0704P)^2 + 0.5685P]$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.08$ $(\Delta/\sigma)_{\max} = 0.001$
 1754 reflections $\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$
 130 parameters $\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$
 3 restraints Extinction correction: none
 Primary atom site location: structure-invariant direct methods

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.2971 (2)	0.0151 (5)	-0.11097 (19)	0.0386 (7)
N1	0.6436 (3)	0.5247 (6)	0.1879 (2)	0.0265 (7)
H1N	0.635 (4)	0.655 (7)	0.224 (3)	0.073 (17)*
H2N	0.633 (3)	0.394 (7)	0.222 (3)	0.045 (12)*
H3N	0.714 (2)	0.517 (8)	0.188 (3)	0.040 (12)*
C1	0.1700 (4)	0.2318 (8)	0.1666 (3)	0.0438 (10)
H1	0.1688	0.3459	0.2164	0.053*
C2	0.1003 (3)	0.0237 (9)	0.1510 (3)	0.0450 (10)
H2	0.0508	-0.0008	0.1896	0.054*
C3	0.1022 (3)	-0.1489 (8)	0.0793 (3)	0.0442 (10)
H3	0.0558	-0.2922	0.0706	0.053*
C4	0.1732 (3)	-0.1104 (7)	0.0200 (3)	0.0372 (9)
H4	0.1731	-0.2260	-0.0296	0.045*
C5	0.2448 (3)	0.1002 (7)	0.0339 (3)	0.0274 (8)
C6	0.2430 (3)	0.2733 (7)	0.1081 (3)	0.0352 (9)
H6	0.2902	0.4154	0.1184	0.042*
C7	0.3141 (3)	0.1395 (6)	-0.0361 (2)	0.0262 (8)
C8	0.4074 (3)	0.3311 (6)	-0.0166 (2)	0.0230 (7)
C9	0.4812 (3)	0.3497 (7)	0.0737 (2)	0.0250 (7)
H9	0.4697	0.2484	0.1239	0.030*
C10	0.5707 (3)	0.5142 (6)	0.0907 (2)	0.0230 (7)
C11	0.59773 (8)	0.02097 (17)	0.29914 (7)	0.0350 (3)

Atomic displacement parameters (\AA^2)

U^{11} U^{22} U^{33} U^{12} U^{13} U^{23}

supplementary materials

O1	0.0478 (16)	0.0345 (15)	0.0357 (14)	-0.0127 (13)	0.0148 (12)	-0.0065 (12)
N1	0.0268 (15)	0.0226 (16)	0.0272 (15)	-0.0029 (14)	0.0015 (12)	0.0026 (14)
C1	0.054 (3)	0.037 (2)	0.047 (2)	-0.004 (2)	0.025 (2)	-0.008 (2)
C2	0.039 (2)	0.051 (3)	0.050 (2)	0.002 (2)	0.0205 (19)	0.010 (2)
C3	0.041 (2)	0.035 (2)	0.060 (3)	-0.0108 (19)	0.019 (2)	0.004 (2)
C4	0.048 (2)	0.026 (2)	0.040 (2)	-0.0095 (18)	0.0165 (18)	-0.0018 (17)
C5	0.0274 (18)	0.0209 (17)	0.0343 (19)	-0.0024 (14)	0.0082 (15)	0.0009 (15)
C6	0.042 (2)	0.0258 (19)	0.039 (2)	-0.0010 (16)	0.0108 (17)	0.0012 (16)
C7	0.0291 (18)	0.0184 (16)	0.0309 (18)	0.0025 (14)	0.0070 (15)	0.0041 (14)
C8	0.0242 (16)	0.0171 (16)	0.0276 (17)	0.0007 (13)	0.0059 (13)	0.0017 (14)
C9	0.0307 (18)	0.0199 (17)	0.0254 (17)	-0.0008 (14)	0.0091 (14)	0.0050 (14)
C10	0.0284 (17)	0.0182 (16)	0.0214 (15)	0.0012 (14)	0.0046 (13)	-0.0003 (14)
C11	0.0418 (5)	0.0276 (5)	0.0393 (5)	0.0038 (4)	0.0168 (4)	0.0054 (4)

Geometric parameters (\AA , $^\circ$)

O1—C7	1.223 (4)	C4—C5	1.390 (5)
N1—C10	1.458 (4)	C4—H4	0.93
N1—H1N	0.87 (3)	C5—C6	1.396 (5)
N1—H2N	0.87 (3)	C5—C7	1.487 (5)
N1—H3N	0.87 (2)	C6—H6	0.93
C1—C2	1.366 (6)	C7—C8	1.497 (5)
C1—C6	1.393 (5)	C8—C9	1.388 (5)
C1—H1	0.93	C8—C10 ⁱ	1.409 (4)
C2—C3	1.367 (6)	C9—C10	1.372 (5)
C2—H2	0.93	C9—H9	0.93
C3—C4	1.381 (5)	C10—C8 ⁱ	1.409 (4)
C3—H3	0.93		
C10—N1—H1N	117 (3)	C4—C5—C6	119.0 (3)
C10—N1—H2N	111 (3)	C4—C5—C7	117.7 (3)
H1N—N1—H2N	102 (4)	C6—C5—C7	123.1 (3)
C10—N1—H3N	112 (3)	C1—C6—C5	119.6 (4)
H1N—N1—H3N	108 (4)	C1—C6—H6	120.2
H2N—N1—H3N	105 (4)	C5—C6—H6	120.2
C2—C1—C6	120.1 (4)	O1—C7—C5	121.1 (3)
C2—C1—H1	119.9	O1—C7—C8	118.1 (3)
C6—C1—H1	119.9	C5—C7—C8	120.9 (3)
C1—C2—C3	120.8 (4)	C9—C8—C10 ⁱ	117.1 (3)
C1—C2—H2	119.6	C9—C8—C7	121.2 (3)
C3—C2—H2	119.6	C10 ⁱ —C8—C7	121.6 (3)
C2—C3—C4	120.0 (4)	C10—C9—C8	121.5 (3)
C2—C3—H3	120.0	C10—C9—H9	119.2
C4—C3—H3	120.0	C8—C9—H9	119.2
C3—C4—C5	120.4 (4)	C9—C10—C8 ⁱ	121.4 (3)
C3—C4—H4	119.8	C9—C10—N1	118.1 (3)
C5—C4—H4	119.8	C8 ⁱ —C10—N1	120.5 (3)

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···C11 ⁱⁱ	0.87 (4)	2.29 (4)	3.155 (3)	172 (4)
N1—H2N···C11	0.87 (4)	2.33 (4)	3.187 (3)	174 (4)
N1—H3N···C11 ⁱⁱⁱ	0.87 (3)	2.29 (3)	3.159 (4)	175 (2)

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

Fig. 1

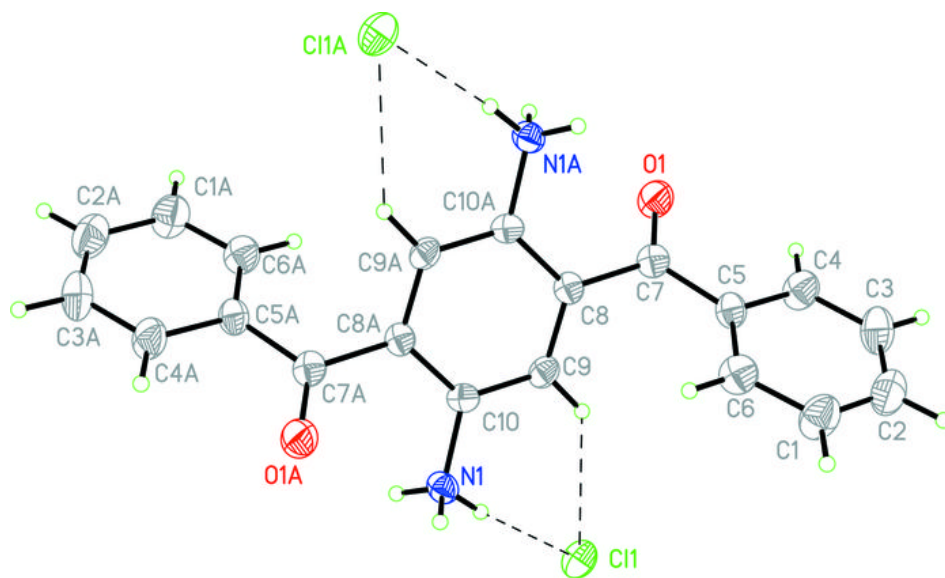


Fig. 2

