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Acetone (2,6-dichlorobenzoyl)hydrazone: chains of π-stacked hydrogen-bonded dimers

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.096
Data-to-parameter ratio = 18.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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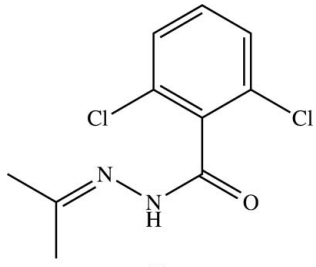
In the title compound, $C_{10}H_{10}Cl_2N_2O$, the aryl ring is almost orthogonal to the rest of the molecule. Molecules are linked into centrosymmetric dimers by N—H···O hydrogen bonds, and these dimers are linked into chains by a single π–π stacking interaction.

Comment

We report here the molecular and supramolecular structure of the title compound, (I) (Fig. 1). Apart from the dichlorophenyl ring, the non-H atoms are nearly coplanar, as shown by the leading torsion angles (Table 1). The aryl ring is almost orthogonal to the rest of the molecule, with a dihedral angle of 82.5 (2)° between the aryl ring and the mean plane through the rest of the non-H atoms. This is a consequence of the repulsive interactions between the lone pairs of electrons on the two Cl atoms and those on atoms N2 and O7.



(I)

The molecules are linked by paired N—H···O hydrogen bonds (Table 2) into cyclic centrosymmetric $R_2^2(8)$ (Bernstein *et al.*, 1995) dimers (Fig. 2), and these dimers are linked into chains by a single aromatic π–π stacking interaction. The aryl rings of the molecules at (x, y, z) and ($1-x$, $2-y$, $-z$) are strictly parallel, with an interplanar spacing of 3.593 (2) Å. The ring-centroid separation is 3.695 (2) Å, corresponding to a ring offset of 0.862 (2) Å. Propagation by inversion of this interaction then links the hydrogen-bonded dimers into a π-stacked chain running parallel to the [01$\bar{1}$] direction (Fig. 3), but there are no direction-specific interactions between adjacent chains.

Experimental

2,6-Dichlorobenzoylhydrazine (3 mmol) was dissolved in acetone (30 ml) and the solution was heated under reflux for 1 h. The solution was then cooled and the excess solvent was removed under reduced pressure. The resulting solid product, (I), was crystallized from ethanol.

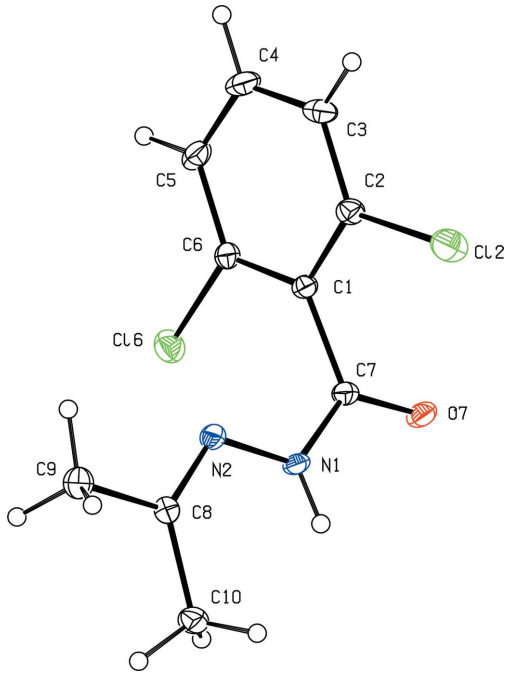


Figure 1
A molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

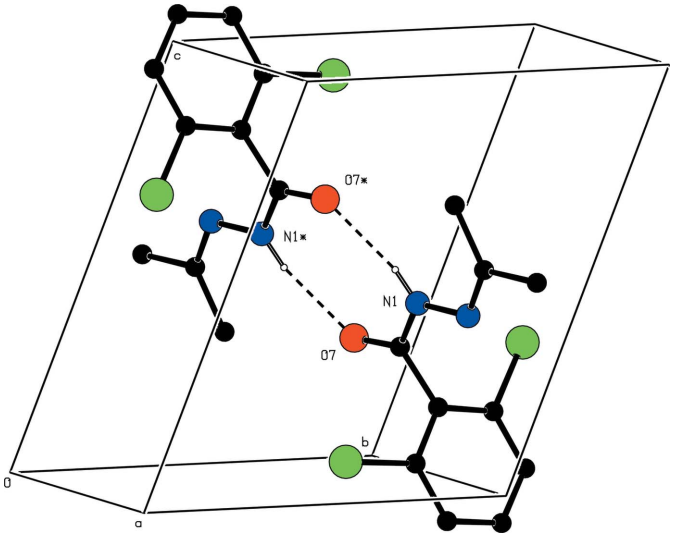


Figure 2
The molecular structure of compound (I), showing the formation of a hydrogen-bonded (dashed lines) $R_2^2(8)$ dimer. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1-x, 1-y, 1-z)$.

Crystal data

$C_{10}H_{10}Cl_2N_2O$	V = 558.03 (3) Å^3
M_r = 245.10	Z = 2
Triclinic, $P\overline{1}$	D_x = 1.459 Mg m^{-3}
a = 7.4980 (3) Å	Mo $K\alpha$ radiation
b = 8.1320 (2) Å	μ = 0.56 mm^{-1}
c = 9.7759 (3) Å	T = 120 (2) K
α = 71.609 (2)°	Lath, colourless
β = 80.822 (2)°	0.42 × 0.10 × 0.08 mm
γ = 89.033 (2)°	

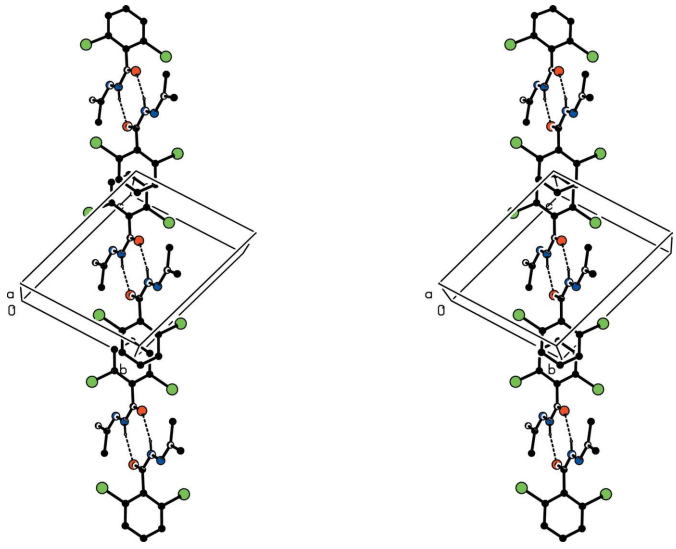


Figure 3
A stereoscopic view of part of the crystal structure of compound (I), showing the formation of a π-stacked chain of hydrogen-bonded (dashed lines) dimers along $[01\overline{1}]$. For the sake of clarity, H atoms bonded to C atoms have been omitted.

Data collection

Bruker Nonius KappaCCD area-detector diffractometer	13458 measured reflections
φ and ω scans	2568 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	1970 reflections with $I > 2\sigma(I)$
T_{min} = 0.822, T_{max} = 0.957	R_{int} = 0.045
	θ_{max} = 27.6°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.2175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.096	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
2568 reflections	
138 parameters	
H-atom parameters constrained	

Table 1
Selected torsion angles (°).

C2—C1—C7—N1	101.4 (2)	C7—N1—N2—C8	175.32 (16)
C1—C7—N1—N2	−2.8 (2)	N1—N2—C8—C9	178.37 (15)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O7^{i}	0.85	2.09	2.9232 (18)	169

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

All atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 (aromatic) or 0.98 Å (methyl) and N—H = 0.85 Å, and with U_{iso}(H) = kU_{eq}(C,N), where k = 1.5 for the methyl groups and k = 1.2 for all other H atoms.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

The X-ray data were collected at the EPSRC X-Ray Crystallographic Service, University of Southampton; the authors thank the staff of the Service for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

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supporting information

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$\gamma = 89.033$ (2)°
$V = 558.03$ (3) Å^3
$Z = 2$
$F(000) = 252$
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Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2540 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.56$ mm^{-1}
$T = 120$ K
Lath, colourless
0.42 × 0.10 × 0.08 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.822$, $T_{max} = 0.957$
13458 measured reflections
2568 independent reflections
1970 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.8°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.096$
$S = 1.04$
2568 reflections
138 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.2175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6756 (2)	0.8506 (2)	0.18118 (17)	0.0197 (4)
C2	0.7648 (2)	0.8102 (2)	0.06043 (19)	0.0225 (4)
Cl2	0.80670 (8)	0.59432 (6)	0.07718 (5)	0.03773 (16)
C3	0.8217 (3)	0.9350 (3)	−0.07220 (19)	0.0276 (4)
C4	0.7856 (3)	1.1063 (3)	−0.0867 (2)	0.0306 (5)
C5	0.6953 (3)	1.1521 (2)	0.0295 (2)	0.0273 (4)
C6	0.6420 (2)	1.0247 (2)	0.16195 (19)	0.0213 (4)
Cl6	0.52761 (7)	1.08133 (6)	0.30856 (5)	0.03135 (15)
N1	0.6983 (2)	0.67279 (18)	0.43117 (15)	0.0213 (3)
N2	0.8638 (2)	0.75866 (19)	0.41427 (16)	0.0221 (3)
C7	0.6030 (2)	0.7097 (2)	0.32012 (18)	0.0213 (4)
O7	0.46049 (18)	0.63126 (16)	0.33026 (13)	0.0288 (3)
C8	0.9384 (2)	0.7238 (2)	0.52866 (19)	0.0217 (4)
C9	1.1187 (3)	0.8105 (3)	0.5128 (2)	0.0304 (4)
C10	0.8629 (3)	0.6040 (2)	0.6766 (2)	0.0284 (4)
H3	0.8847	0.9037	−0.1522	0.036*
H4	0.8234	1.1935	−0.1777	0.040*
H5	0.6699	1.2699	0.0187	0.035*
H1	0.6567	0.5918	0.5083	0.026*
H9A	1.1539	0.8857	0.4118	0.039*
H9B	1.2092	0.7222	0.5374	0.039*
H9C	1.1109	0.8802	0.5789	0.039*
H10A	0.7368	0.6313	0.7031	0.037*
H10B	0.9338	0.6181	0.7484	0.037*
H10C	0.8683	0.4840	0.6753	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0177 (9)	0.0220 (9)	0.0171 (9)	−0.0042 (7)	−0.0063 (7)	−0.0011 (7)
C2	0.0221 (9)	0.0238 (9)	0.0205 (9)	0.0013 (7)	−0.0077 (7)	−0.0035 (7)
Cl2	0.0544 (4)	0.0293 (3)	0.0310 (3)	0.0131 (2)	−0.0103 (2)	−0.0106 (2)
C3	0.0254 (10)	0.0374 (11)	0.0163 (9)	−0.0007 (8)	−0.0030 (7)	−0.0032 (8)
C4	0.0299 (11)	0.0314 (10)	0.0212 (9)	−0.0071 (8)	−0.0045 (8)	0.0053 (8)
C5	0.0280 (10)	0.0203 (9)	0.0290 (10)	−0.0041 (8)	−0.0096 (8)	0.0014 (8)
C6	0.0194 (9)	0.0233 (9)	0.0211 (9)	−0.0035 (7)	−0.0052 (7)	−0.0055 (7)
Cl6	0.0327 (3)	0.0336 (3)	0.0308 (3)	0.0005 (2)	−0.0033 (2)	−0.0155 (2)
N1	0.0221 (8)	0.0220 (7)	0.0151 (7)	−0.0069 (6)	−0.0035 (6)	0.0015 (6)
N2	0.0204 (8)	0.0244 (8)	0.0199 (8)	−0.0060 (6)	−0.0028 (6)	−0.0045 (6)
C7	0.0231 (9)	0.0224 (9)	0.0164 (8)	−0.0028 (7)	−0.0039 (7)	−0.0027 (7)
O7	0.0276 (7)	0.0296 (7)	0.0225 (7)	−0.0123 (6)	−0.0085 (6)	0.0038 (5)
C8	0.0237 (9)	0.0212 (9)	0.0211 (9)	−0.0002 (7)	−0.0043 (7)	−0.0076 (7)
C9	0.0245 (10)	0.0362 (11)	0.0303 (10)	−0.0058 (8)	−0.0063 (8)	−0.0090 (9)
C10	0.0303 (11)	0.0317 (10)	0.0214 (9)	−0.0040 (8)	−0.0101 (8)	−0.0031 (8)

Geometric parameters (Å, º)

C1—C2	1.391 (2)	N1—N2	1.395 (2)
C1—C6	1.392 (2)	N1—H1	0.8475
C1—C7	1.502 (2)	N2—C8	1.280 (2)
C2—C3	1.379 (2)	C7—O7	1.228 (2)
C2—Cl2	1.7387 (18)	C8—C10	1.493 (2)
C3—C4	1.382 (3)	C8—C9	1.498 (2)
C3—H3	0.95	C9—H9A	0.98
C4—C5	1.379 (3)	C9—H9B	0.98
C4—H4	0.95	C9—H9C	0.98
C5—C6	1.383 (2)	C10—H10A	0.98
C5—H5	0.95	C10—H10B	0.98
C6—Cl6	1.7360 (18)	C10—H10C	0.98
N1—C7	1.347 (2)		
C2—C1—C6	116.86 (15)	N2—N1—H1	122.3
C2—C1—C7	120.72 (15)	C8—N2—N1	115.92 (14)
C6—C1—C7	122.13 (16)	O7—C7—N1	121.67 (15)
C3—C2—C1	122.47 (17)	O7—C7—C1	120.05 (15)
C3—C2—Cl2	118.67 (15)	N1—C7—C1	118.27 (15)
C1—C2—Cl2	118.86 (13)	N2—C8—C10	126.04 (16)
C2—C3—C4	118.84 (18)	N2—C8—C9	117.10 (16)
C2—C3—H3	120.6	C10—C8—C9	116.85 (16)
C4—C3—H3	120.6	C8—C9—H9A	109.5
C5—C4—C3	120.68 (17)	C8—C9—H9B	109.5
C5—C4—H4	119.7	H9A—C9—H9B	109.5
C3—C4—H4	119.7	C8—C9—H9C	109.5
C4—C5—C6	119.33 (17)	H9A—C9—H9C	109.5
C4—C5—H5	120.3	H9B—C9—H9C	109.5
C6—C5—H5	120.3	C8—C10—H10A	109.5
C5—C6—C1	121.81 (17)	C8—C10—H10B	109.5
C5—C6—Cl6	119.56 (14)	H10A—C10—H10B	109.5
C1—C6—Cl6	118.62 (13)	C8—C10—H10C	109.5
C7—N1—N2	120.40 (14)	H10A—C10—H10C	109.5
C7—N1—H1	117.2	H10B—C10—H10C	109.5
C2—C1—C7—N1	101.4 (2)	C4—C5—C6—C1	−0.6 (3)
C1—C7—N1—N2	−2.8 (2)	C4—C5—C6—Cl6	−179.81 (14)
C7—N1—N2—C8	175.32 (16)	C2—C1—C6—C5	−0.3 (3)
N1—N2—C8—C9	178.37 (15)	C7—C1—C6—C5	−174.12 (16)
C6—C1—C2—C3	1.3 (3)	C2—C1—C6—Cl6	178.92 (13)
C7—C1—C2—C3	175.21 (17)	C7—C1—C6—Cl6	5.1 (2)
C6—C1—C2—Cl2	−178.85 (13)	N2—N1—C7—O7	177.13 (16)
C7—C1—C2—Cl2	−5.0 (2)	C2—C1—C7—O7	−78.5 (2)
C1—C2—C3—C4	−1.4 (3)	C6—C1—C7—O7	95.0 (2)
Cl2—C2—C3—C4	178.78 (14)	C6—C1—C7—N1	−85.1 (2)
C2—C3—C4—C5	0.4 (3)	N1—N2—C8—C10	−1.3 (3)

C3—C4—C5—C6	0.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O7^{i}	0.85	2.09	2.9232 (18)	169

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