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(*E*,*E*)-2,5-Bis(5-chloro-2-methoxyphenyl)-3,4-diazahexa-2,4-diene

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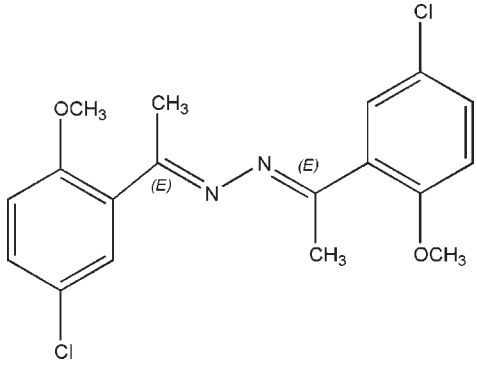
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.129; data-to-parameter ratio = 14.1.

The title compound, $C_{18}H_{18}Cl_2N_2O_2$, was synthesized by the reaction of 1-(5-chloro-2-methoxyphenyl)ethanone with hydrazine hydrate. The molecule lies on a crystallographic twofold axis passing through the mid-point of the N—N bond with one half-molecule in the asymmetric unit. The dihedral angle between the two aromatic rings is 44.33 (4)°. In the crystal, intermolecular C—H···O interactions link the molecules into columns along the *c* axis

Related literature

For azine compounds containing both a diimine linkage and N—N bonding, see: Kesslen *et al.* (1999); Kundu *et al.* (2005). For related structures, see: Glaser *et al.* (1995); Hunig *et al.* (2000).



Experimental

Crystal data

$C_{18}H_{18}Cl_2N_2O_2$
M_r = 365.24
Orthorhombic, $P2_12_12$
a = 7.9030 (19) Å
b = 27.862 (7) Å
c = 3.9819 (10) Å
V = 876.8 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 295 K
0.22 × 0.16 × 0.12 mm

Data collection

Bruker APEXII CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.921, T_{max} = 0.956
4469 measured reflections
1566 independent reflections
1417 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.129
S = 1.01
1566 reflections
111 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Flack (1983), 592 Friedel pairs
Flack parameter: 0.08 (12)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O1^{i}	0.96	2.68	3.521 (3)	146

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: FL2273).

References

Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Glaser, R., Chen, G. S., Anthamatten, M. & Barnes, C. L. (1995). *J. Chem. Soc. Perkin Trans. 2*, pp. 1449–1458.
Hunig, S., Kemmer, M. & Wenner, H. (2000). *Chem. Eur. J.* **6**, 2618–2632.
Kesslen, E. C., Euler, W. B. & Foxman, B. M. (1999). *Chem. Mater.* **11**, 336–340.
Kundu, N., Chatterjee, P. B., Chaudhury, M. & Tiekink, E. R. T. (2005). *Acta Cryst.* E**61**, m1583–m1585.
Sheldrick, G. M. (2003). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2009). E**65**, o3185 [**doi:10.1107/S1600536809048351**]

(*E*,*E*)-2,5-Bis(5-chloro-2-methoxyphenyl)-3,4-diazahexa-2,4-diene

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Comment

Recently, a number of azine compounds containing both a diimine linkage and N—N bonding have been investigated in terms of their crystallography and coordination chemistry (Kundu *et al.*, 2005; Kesslen *et al.*, 1999). As an extension of the work on the structural characterization of azine derivatives, the title compound, (I),was synthesized and its crystal structure is reported here.

The molecule lies on a crystallogrpahic 2-fold axis passing through the mid-point of the N-N bond to give 1/2 molecule per asymmetric unit. (Fig. 1). The dihedral angle between the two aromatic rings is 44.33 (4)°. The N atom and the phenyl ring liie on opposite side of the C8=N1 bond to give an (E, E) conformation with respect to the C8=N1 bond (and its symmetry related C8*a*=N1a double bond (Fig. 1.). This configuration agrees with those commonly found in similar compounds (Glaser *et al.*, 1995; Hunig *et al.*, 2000). Intermolecular C—H···O interactions link the molecules into columns along the c axis (Table 1, Fig. 2).

Experimental

An ethanol solution (30 ml) of hydrazine (0.02 mol) and 1-(5-chloro-2-methoxyphenyl)ethanone (0.04 mol) was refluxed and stirred for 6 h; the mixture was cooled and the resulting solid product, (I), was collected by filtration. Colourless crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in acetone.

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms,with *C*—*H*(methyl) = 0.96 Å, C—H(aromatic) = 0.93 Å, and with U_{iso}(H) =1.5U_{eq}(C_{methyl}) and 1.2U_{eq}($C_{aromatic}$).

Figures

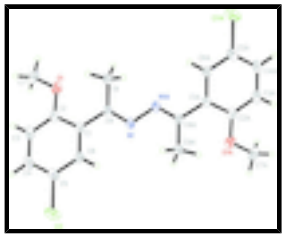

Fig. 1. The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

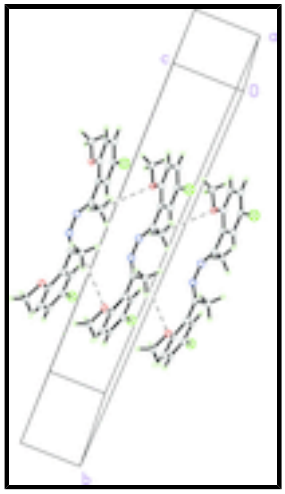

Fig. 2. The crystal packing of (I) , Dashed lines show intermolecular C—H···O interactions .

(*E*,*E*)-2,5-Bis(5-chloro-2-methoxyphenyl)-3,4-diazahexa-2,4-diene

Crystal data

$C_{18}H_{18}Cl_2N_2O_2$	$F(000) = 380$
$M_r = 365.24$	$D_x = 1.383$ Mg m^{-3}
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2 2ab	Cell parameters from 2313 reflections
$a = 7.9030$ (19) Å	$\theta = 2.7$–$27.8°$
$b = 27.862$ (7) Å	$\mu = 0.38$ mm^{-1}
$c = 3.9819$ (10) Å	$T = 295$ K
$V = 876.8$ (4) Å^3	Block, colourless
$Z = 2$	0.22 × 0.16 × 0.12 mm

Data collection

Bruker APEXII CCD area detector diffractometer	1566 independent reflections
Radiation source: fine-focus sealed tube	1417 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.019$
phi and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$h = -9 \rightarrow 8$
$T_{min} = 0.921$, $T_{max} = 0.956$	$k = -33 \rightarrow 32$
4469 measured reflections	$l = -4 \rightarrow 4$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained

$wR(F^2) = 0.129$	$w = 1/[\sigma^2(F_o^2) + (0.1019P)^2 + 0.021P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
1566 reflections	$\Delta\rho_{max} = 0.12$ e Å^{-3}
111 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
0 restraints	Absolute structure: Flack (1983), 592 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.08 (12)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.35358 (10)	0.29469 (2)	0.1116 (2)	0.0643 (3)
O1	0.9306 (2)	0.40920 (6)	0.5876 (6)	0.0520 (5)
N1	0.5044 (3)	0.47464 (6)	0.4965 (5)	0.0367 (5)
C1	0.6541 (3)	0.40440 (7)	0.3671 (6)	0.0333 (5)
C2	0.8000 (3)	0.38071 (8)	0.4798 (6)	0.0373 (6)
C3	0.8050 (4)	0.33068 (10)	0.4871 (7)	0.0487 (7)
H3	0.9006	0.3150	0.5674	0.058*
C4	0.6670 (3)	0.30434 (8)	0.3743 (7)	0.0468 (7)
H4	0.6702	0.2710	0.3770	0.056*
C5	0.5261 (4)	0.32784 (9)	0.2588 (7)	0.0429 (6)
C6	0.5175 (3)	0.37744 (8)	0.2532 (6)	0.0381 (6)
H6	0.4208	0.3927	0.1736	0.046*
C7	1.0770 (4)	0.38654 (12)	0.7221 (9)	0.0611 (8)
H7A	1.0453	0.3671	0.9111	0.092*
H7B	1.1565	0.4106	0.7929	0.092*
H7C	1.1279	0.3667	0.5532	0.092*
C8	0.6407 (3)	0.45789 (8)	0.3653 (6)	0.0330 (5)
C9	0.7746 (3)	0.48801 (9)	0.2056 (7)	0.0413 (6)
H9A	0.7244	0.5081	0.0369	0.062*
H9B	0.8580	0.4676	0.1039	0.062*
H9C	0.8274	0.5077	0.3734	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0758 (5)	0.0434 (4)	0.0738 (5)	−0.0196 (3)	−0.0095 (5)	−0.0004 (4)
O1	0.0404 (9)	0.0499 (10)	0.0656 (13)	0.0104 (8)	−0.0170 (10)	−0.0051 (10)
N1	0.0331 (10)	0.0273 (9)	0.0497 (12)	0.0040 (8)	−0.0013 (9)	0.0009 (9)
C1	0.0368 (12)	0.0316 (12)	0.0315 (11)	0.0055 (9)	0.0014 (11)	0.0012 (9)
C2	0.0404 (13)	0.0386 (12)	0.0329 (12)	0.0102 (10)	−0.0014 (10)	−0.0024 (10)
C3	0.0568 (16)	0.0408 (13)	0.0485 (15)	0.0213 (12)	−0.0006 (13)	0.0047 (12)
C4	0.0618 (17)	0.0286 (11)	0.0500 (15)	0.0080 (11)	0.0084 (15)	0.0008 (11)
C5	0.0567 (16)	0.0327 (12)	0.0392 (13)	−0.0037 (12)	0.0022 (12)	0.0010 (10)
C6	0.0382 (13)	0.0343 (12)	0.0420 (13)	0.0049 (10)	−0.0002 (11)	0.0035 (10)
C7	0.0409 (14)	0.082 (2)	0.0605 (19)	0.0158 (15)	−0.0150 (13)	−0.0022 (17)
C8	0.0340 (11)	0.0311 (11)	0.0337 (11)	0.0024 (9)	−0.0040 (11)	−0.0019 (9)
C9	0.0400 (12)	0.0362 (13)	0.0475 (15)	−0.0018 (11)	0.0057 (11)	−0.0039 (11)

Geometric parameters (Å, °)

Cl1—C5	1.748 (3)	C4—C5	1.371 (4)
O1—C2	1.371 (3)	C4—H4	0.9300
O1—C7	1.422 (3)	C5—C6	1.384 (3)
N1—C8	1.285 (3)	C6—H6	0.9300
N1—N1^{i}	1.415 (3)	C7—H7A	0.9600
C1—C6	1.391 (3)	C7—H7B	0.9600
C1—C2	1.403 (3)	C7—H7C	0.9600
C1—C8	1.494 (3)	C8—C9	1.493 (3)
C2—C3	1.395 (4)	C9—H9A	0.9600
C3—C4	1.389 (4)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C2—O1—C7	118.2 (2)	C5—C6—H6	120.1
C8—N1—N1^{i}	113.8 (2)	C1—C6—H6	120.1
C6—C1—C2	119.2 (2)	O1—C7—H7A	109.5
C6—C1—C8	118.9 (2)	O1—C7—H7B	109.5
C2—C1—C8	121.9 (2)	H7A—C7—H7B	109.5
O1—C2—C3	123.4 (2)	O1—C7—H7C	109.5
O1—C2—C1	116.53 (19)	H7A—C7—H7C	109.5
C3—C2—C1	120.0 (2)	H7B—C7—H7C	109.5
C4—C3—C2	119.9 (2)	N1—C8—C9	124.3 (2)
C4—C3—H3	120.0	N1—C8—C1	114.8 (2)
C2—C3—H3	120.0	C9—C8—C1	120.9 (2)
C5—C4—C3	119.6 (2)	C8—C9—H9A	109.5
C5—C4—H4	120.2	C8—C9—H9B	109.5
C3—C4—H4	120.2	H9A—C9—H9B	109.5
C4—C5—C6	121.5 (3)	C8—C9—H9C	109.5
C4—C5—Cl1	119.57 (19)	H9A—C9—H9C	109.5
C6—C5—Cl1	118.9 (2)	H9B—C9—H9C	109.5
C5—C6—C1	119.7 (2)		

C7—O1—C2—C3	−1.7 (4)	C4—C5—C6—C1	−0.1 (4)
C7—O1—C2—C1	176.4 (2)	Cl1—C5—C6—C1	−179.54 (19)
C6—C1—C2—O1	179.7 (2)	C2—C1—C6—C5	1.3 (4)
C8—C1—C2—O1	−0.2 (3)	C8—C1—C6—C5	−178.8 (2)
C6—C1—C2—C3	−2.1 (4)	N1^{i}—N1—C8—C9	−3.8 (3)
C8—C1—C2—C3	178.0 (2)	N1^{i}—N1—C8—C1	179.13 (16)
O1—C2—C3—C4	179.8 (2)	C6—C1—C8—N1	48.8 (3)
C1—C2—C3—C4	1.7 (4)	C2—C1—C8—N1	−131.3 (2)
C2—C3—C4—C5	−0.5 (4)	C6—C1—C8—C9	−128.4 (2)
C3—C4—C5—C6	−0.3 (4)	C2—C1—C8—C9	51.5 (3)
C3—C4—C5—Cl1	179.2 (2)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9B···O1^{ii}	0.96	2.68	3.521 (3)	146.

Symmetry codes: (ii) *x*, *y*, *z*−1.

Fig. 1

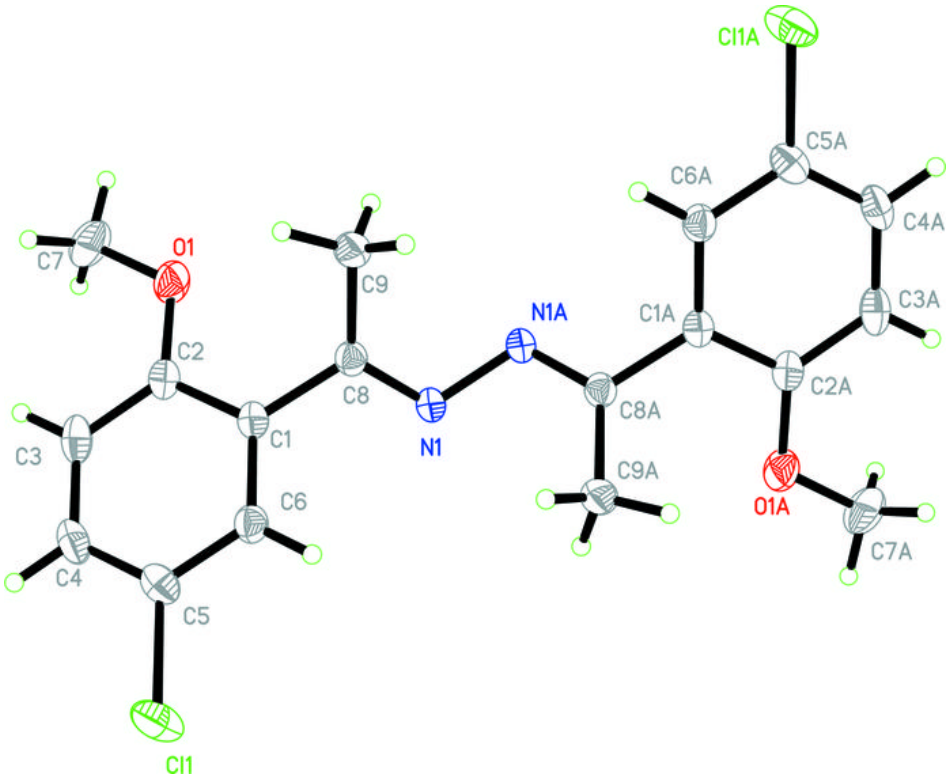
Cl1A
C5A
C6A
C4A
C7
O1
C9
N1A
C1A
C3A
C2
C8
C2A
C8A
C1
C3
N1
C9A
C6
O1A
C4
C7A
C5
Cl1

Fig. 2