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Diethyl 2-[(4-chlorophenylamino)(2,6-dichlorophenyl)methyl]malonate

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The dihedral angle between the two benzene rings of the title compound, $C_{20}H_{20}Cl_3NO_4$, is 80.67 (6)°. In the solid state, the molecules are joined into dimers by a pair of hydrogen bonds between the amino N atom and the carbonyl O atom of the carboxylate substituent [$N\cdots O = 2.986$ (2) Å and $N-H\cdots O =$ 174 (2)°].

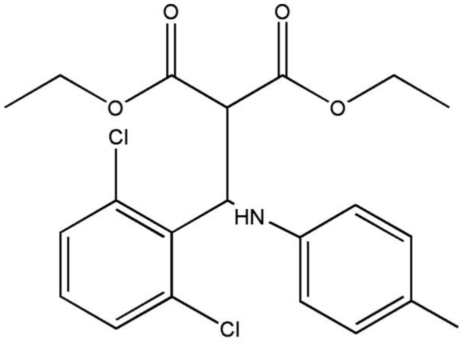
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.122
Data-to-parameter ratio = 16.9

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

Comment

β-Amino acids are important structural units of several biologically active compounds, such as dolastins, astins, onchidin, jasplakinolide and motuporin (Cardillo *et al.*, 1996), and useful building blocks for β-lactams (Hart *et al.*, 1989) and β-peptides (Krauthäuser *et al.*, 1997) that are present in a variety of drugs (Rosenblum *et al.*, 1998). Hence, there is much interest in developing convenient methods for the synthesis of β-amino esters. The title compound, (I), was synthesized through a Mannich-type reaction between *N*-(2,6-dichlorobenzylidene)-4-chlorobenzenamine and diethyl malonate. An X-ray crystal structure determination of (I) was undertaken in order to elucidate the conformation.



(I)

The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles (Table 1) are in good agreement with those observed for a closely related structure, diethyl 2-[(4-bromophenylamino)(2-chlorophenyl)methyl]malonate (Shou *et al.*, 2006). The dihedral angle between the two benzene rings is 80.67 (6)° in (I). Intermolecular $N-H\cdots O$ hydrogen bonding is observed in the crystal structure of (I) (Table 2 and Fig. 1).

Experimental

The starting materials were purchased from Acros and used without purification. The title compound was synthesized according to the method described previously by Shou *et al.* (2006) and identified additionally by IR, ^{1}H NMR, ^{13}C NMR, MS, melting point and

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elemental analyses; full details are given in the CIF. Analysis calculated for $C_{20}H_{20}Cl_3NO_4$: C 54.01, H 4.53, N 3.15%; found: C 54.00, H 4.52, N 3.18%. The crystal used for the data collection was obtained by slow evaporation of a saturated hexane–dichloromethane (1:1 *v*/*v*) solution of (I) at room temperature.

Crystal data

$C_{20}H_{20}Cl_3NO_4$
$M_r = 444.72$
Triclinic, $P\bar{1}$
$a = 8.4561\ (9)$ Å
$b = 11.6301\ (12)$ Å
$c = 11.9854\ (12)$ Å
$\alpha = 65.401\ (2)^\circ$
$\beta = 84.735\ (2)^\circ$
$\gamma = 86.205\ (2)^\circ$
$V = 1066.70\ (19)$ Å^3
$Z = 2$
$D_x = 1.385$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.46$ mm^{-1}
$T = 293\ (2)$ K
Plate, colorless
$0.50 \times 0.33 \times 0.13$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.756$, $T_{\max} = 0.942$
6304 measured reflections
4524 independent reflections
3340 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.063$
$\theta_{\max} = 27.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.122$
$S = 0.95$
4524 reflections
267 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C3	1.189 (2)	O4—C4	1.321 (2)
O2—C3	1.326 (2)	O4—C5	1.454 (2)
O2—C7	1.462 (2)	N1—C9	1.376 (2)
O3—C4	1.187 (2)	N1—C1	1.450 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H3···O1$^{\rm i}$	0.80 (2)	2.19 (2)	2.986 (2)	174 (2)

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

Methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$, but were allowed to rotate freely about the C—C bonds. Aromatic H atoms were included using a riding model, with C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. Other H atoms, located in a difference Fourier map, were refined freely. C—H distances are in the range 0.90 (2)–0.94 (2) Å and the N—H distance is 0.80 (2) Å.

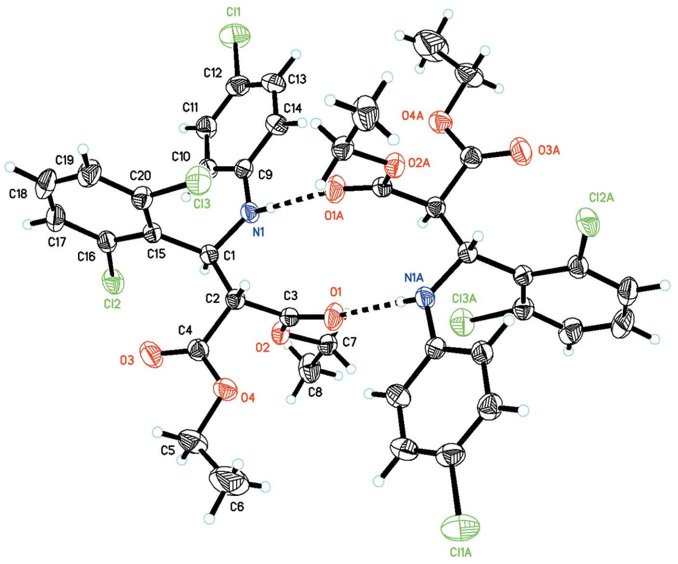

Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Dashed lines indicate hydrogen bonds [symmetry code: (A) $-x$, $1 - y$, $-z$.]

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL*.

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