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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.064
 wR factor = 0.189
Data-to-parameter ratio = 14.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Diethyl 2-[(3-chlorophenylamino)(3-nitrophenyl)methyl]malonate

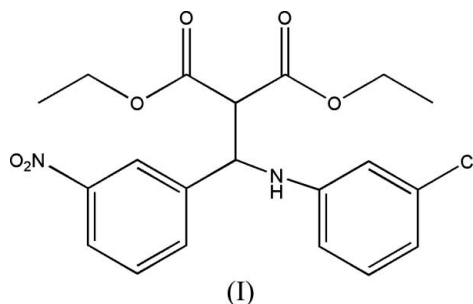
The principal structural feature of the title compound, $\text{C}_{20}\text{H}_{21}\text{ClN}_2\text{O}_6$, is the dihedral angle of $89.15(8)^\circ$ between the two benzene rings of the molecule. The $\text{N}-\text{Csp}^2$ bond distance of $1.387(3)$ Å is significantly shorter than the $\text{N}-\text{Csp}^3$ bond distance of $1.461(3)$ Å. Classical $\text{N}-\text{H}\cdots\text{O}$ and weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding occurs in the crystal structure.

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Comment

In recent years, there has been increasing interest in the synthesis of proteinogenic and non-proteinogenic amino acids. This is due to the wide utility of such compounds as components of proteins and peptides and as starting materials for the synthesis of naturally occurring biologically active compounds (Cardillo & Tomasini, 1996). Hence there is great interest in developing convenient methods for the synthesis of β -amino esters. The title compound, (I), a β -amino ester, was synthesized through a Mannich-type reaction between *m*-nitrobenzaldehyde, *m*-chlorobenzeneamine and diethyl malonate. The X-ray crystal structure determination of (I) was undertaken in order to elucidate the conformation of the molecule.



The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles of (I) are in agreement with those found in a closely related compound, diethyl 2-[(4-bromophenylamino)(2-chlorophenyl)methyl]malonate (Shou *et al.*, 2006). The dihedral angle between the two benzene rings is $89.15(8)^\circ$. The average $\text{C}-\text{O}$ and $\text{C}=\text{C}$ bond distances are $1.389(3)$ and $1.194(3)$ Å, respectively. The $\text{N1}-\text{C2}$ bond distance is significantly longer than that of $\text{N1}-\text{C9}$ (Table 1).

While a classical hydrogen bond occurs between the imino N atom and the carbonyl O atom, weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding is also observed in the crystal structure (Table 2).

Experimental

Compound (I) was synthesized according to the method described previously by Shou *et al.* (2006) and was identified by IR, ^1H NMR,

^{13}C NMR, MS, melting point and elemental analysis. Single crystals of (I) were obtained by slow evaporation of a hexane–dichloromethane (1:1 v/v) solution of the compound at room temperature.

Crystal data

$\text{C}_{20}\text{H}_{21}\text{ClN}_2\text{O}_6$

$M_r = 420.84$

Triclinic, $P\bar{1}$

$a = 9.0765$ (11) Å

$b = 11.0454$ (13) Å

$c = 12.0174$ (14) Å

$\alpha = 101.010$ (2)°

$\beta = 111.271$ (2)°

$\gamma = 102.040$ (2)°

$V = 1050.0$ (2) Å³

$Z = 2$

$D_x = 1.331$ Mg m⁻³

Mo $K\alpha$ radiation

$\mu = 0.22$ mm⁻¹

$T = 293$ (2) K

Block, green

$0.51 \times 0.50 \times 0.32$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.885$, $T_{\max} = 0.930$

5463 measured reflections

3748 independent reflections

2717 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.115$

$\theta_{\text{max}} = 25.2^\circ$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.189$

$S = 1.02$

3748 reflections

264 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1023P)^2]$

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

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 0.35$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

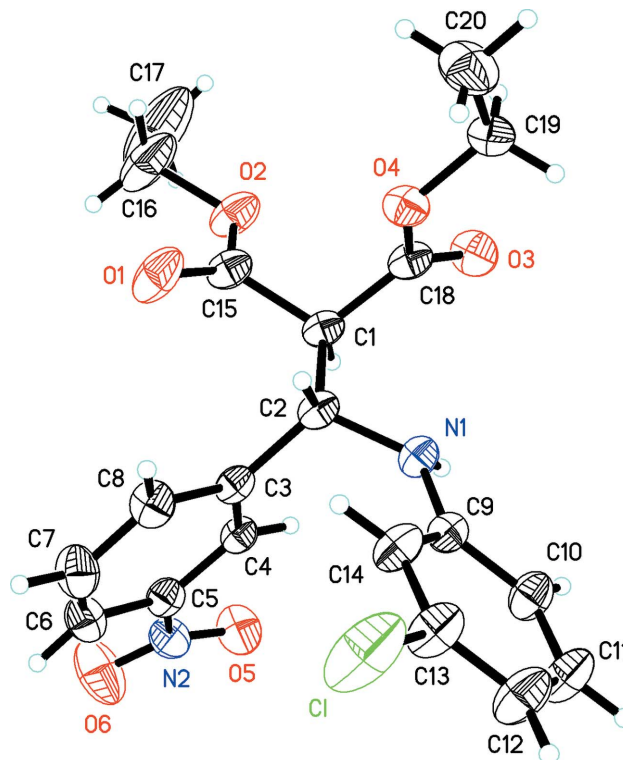


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

Table 1

Selected bond lengths (Å).

N1—C2	1.461 (3)	N1—C9	1.387 (3)
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Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O3 ⁱ	0.88	2.24	3.088 (3)	162
C1—H1 \cdots O6 ⁱⁱ	0.98	2.49	3.363 (4)	149
C4—H4 \cdots O3 ⁱ	0.93	2.47	3.353 (3)	159
C8—H8 \cdots O1 ⁱⁱⁱ	0.93	2.42	3.258 (3)	151

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

The imino H atom was located in a difference Fourier map and refined as riding in its as-found relative position, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{N})$. Methyl H atoms were constrained to an ideal geometry ($\text{C—H} = 0.96$ Å) and torsion angles were refined to fit the electron density; $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. Other H atoms were placed in calculated positions with $\text{C—H} = 0.93\text{--}0.98$ Å, and refined in a riding mode, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; 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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