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Key indicators

Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.056
 wR factor = 0.162
Data-to-parameter ratio = 15.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Ethyl 2-methoxycarbonylamino-3-phenylpropionate

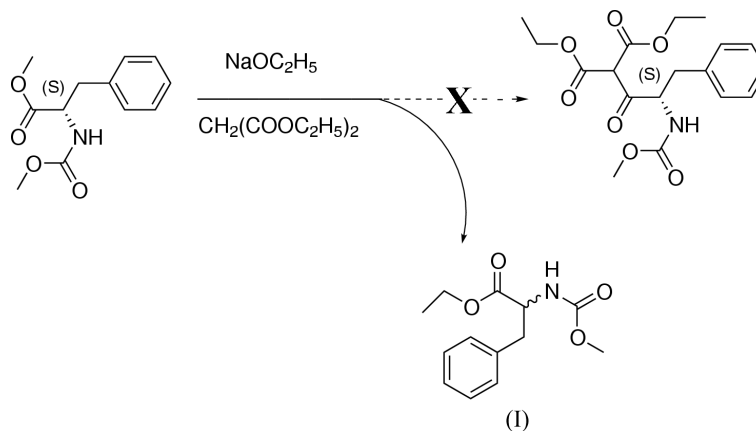
The title compound, $\text{C}_{13}\text{H}_{17}\text{NO}_4$, has been obtained as an unexpected product when attempting to prepare diethyl (*S*)-2-(2-methoxycarbonylamino-3-phenylpropionyl)malonate. The crystal structure involves intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

In our studies on the total synthesis of an anti-AIDS drug (saquinavir), we attempted to use methyl (*S*)-2-methoxycarbonylamino-3-phenylpropionate and diethyl malonate in sodium ethoxide to prepare diethyl (*S*)-2-(2-methoxycarbonylamino-3-phenyl-propionyl)malonate (Yutaka *et al.*, 2004). During this experiment, the title compound, (I), was isolated unexpectedly. Fortunately, ethyl 2-methoxycarbonylamino-3-phenyl-propionate is an interesting target molecule, since it can be regarded as an important chiral agent if it could be obtained as a chirally pure isomer (Yamada & Takeuchi, 1974). We have obtained it here as a racemateThe crystal structure involves intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2).

Experimental

To a solution of sodium (69 mg, 3 mmol) in anhydrous ethanol (5 ml), diethyl malonate (480 mg, 3 mmol) was added and the temperature kept below 273 K (Ikan *et al.*, 1971). The mixture was stirred for 30 min at 258 K, and a solution of methyl (*S*)-2-methoxycarbonylamino-3-phenylpropionate (237 mg, 1 mmol) in ethanol (2 ml) was then added dropwise over a period of 30 min at 258 K. The resulting mixture was stirred at 258 K for 120 min. After completion of the reaction, the solvent was removed to give the crude product, which was purified by recrystallization from ethyl acetate.

Crystal data

$C_{13}H_{17}NO_4$
 $M_r = 251.28$
 Triclinic, $P\bar{1}$
 $a = 8.000$ (4) Å
 $b = 8.753$ (4) Å
 $c = 9.752$ (5) Å
 $\alpha = 102.716$ (9)°
 $\beta = 94.939$ (8)°
 $\gamma = 94.976$ (9)°
 $V = 659.7$ (5) Å³

$Z = 2$
 $D_x = 1.265$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 1615 reflections
 $\theta = 2.4$ – 26.8 °
 $\mu = 0.09$ mm⁻¹
 $T = 273$ (2) K
 Block, colorless
 $0.60 \times 0.43 \times 0.21$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ scans
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.946$, $T_{\max} = 0.981$
 3549 measured reflections

2514 independent reflections
 1767 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$
 $\theta_{\text{max}} = 26.0$ °
 $h = -9 \rightarrow 9$
 $k = -5 \rightarrow 10$
 $l = -12 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.162$
 $S = 1.01$
 2514 reflections
 164 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0875P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.008$
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.023 (8)

Table 1

Selected geometric parameters (Å, °).

O4–C12	1.336 (2)	C2–C3	1.528 (3)
O4–C13	1.423 (2)	C5–C6	1.369 (3)
N1–C12	1.332 (2)	C5–C4	1.388 (2)
N1–C2	1.443 (2)	C4–C9	1.375 (3)
O2–C1	1.315 (2)	C4–C3	1.499 (3)
O2–C10	1.456 (3)	C9–C8	1.377 (3)
C12–O3	1.194 (2)	C6–C7	1.357 (3)
O1–C1	1.186 (2)	C7–C8	1.364 (3)
C2–C1	1.517 (3)	C10–C11	1.484 (3)
C12–O4–C13	116.41 (15)	O1–C1–C2	124.67 (16)
C12–N1–C2	120.93 (14)	O2–C1–C2	110.95 (16)
C1–O2–C10	116.79 (16)	C9–C4–C5	117.57 (18)
O3–C12–N1	124.72 (17)	C9–C4–C3	121.02 (17)
O3–C12–O4	124.27 (18)	C5–C4–C3	121.40 (18)
N1–C12–O4	111.01 (15)	C4–C9–C8	121.42 (19)
N1–C2–C1	111.13 (15)	C7–C6–C5	120.81 (19)
N1–C2–C3	110.80 (15)	C4–C3–C2	113.97 (16)
C1–C2–C3	108.40 (15)	C6–C7–C8	119.8 (2)
C6–C5–C4	120.63 (19)	C7–C8–C9	119.8 (2)
O1–C1–O2	124.33 (18)	O2–C10–C11	107.18 (19)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots O1^i$	0.86	2.21	3.045 (2)	163

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

The H atoms were positioned geometrically ($C-H = 0.93, 0.98, 0.97$ or 0.96 Å for phenyl, tertiary, methylene or methyl H atoms, respectively, and $N-H = 0.86$ Å) and were included in the refinement in the riding-model approximation. $U_{\text{iso}}(H)$ for methyl H atoms were set equal to $1.5U_{\text{eq}}(\text{carrier atom})$; for other H atoms $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(\text{carrier atom})$.

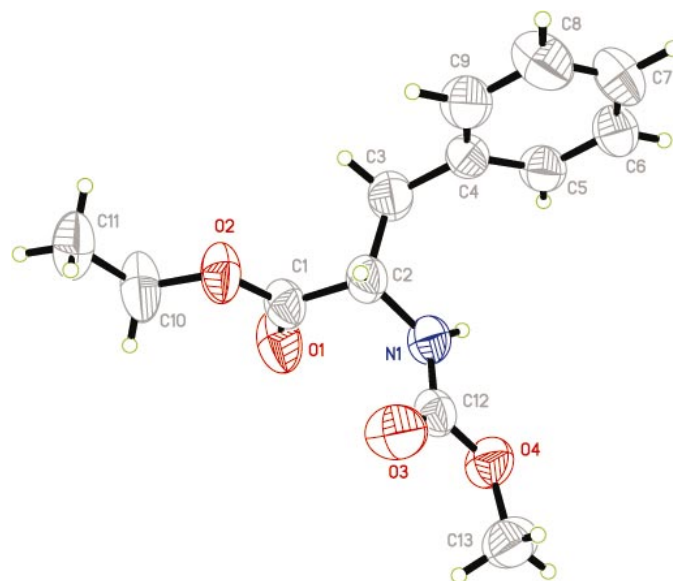


Figure 1

ORTEP-3 (Farrugia, 1997) plot of (I) with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

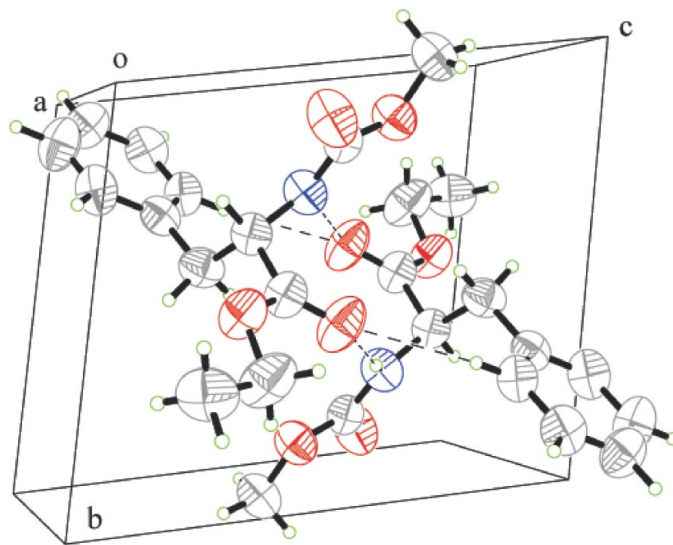


Figure 2

Diagram of the title compound indicating the hydrogen-bonding interactions (dashed lines). $[N1\cdots O1^i]$ 2.891 (3) Å; symmetry code: (i) $-x, -y + 1, -z + 1$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and ViewerPro (Accelrys, 2001); software used to prepare material for publication: SHELXL97.

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