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(1*R*,5*R*,6*R*)-Ethyl 3-benzyl-2,4-dioxo-3-aza-bicyclo[3.1.0]hexane-6-carboxylate

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Key indicators

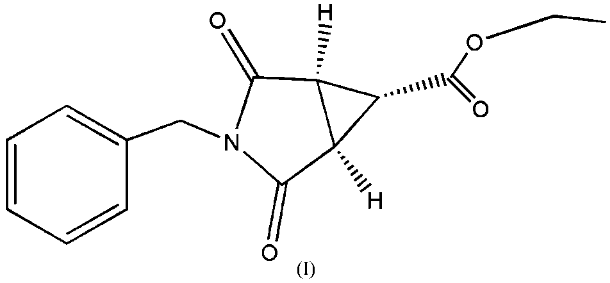
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.046
wR factor = 0.157
Data-to-parameter ratio = 12.7

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

The title compound, $C_{15}H_{15}NO_4$, contains a five-membered heterocyclic ring, in an envelope conformation, fused to a three-membered cyclopropane ring. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds formed between centrosymmetric pairs.

Comment

The title compound, (I), first reported in 2004 (Anquetin *et al.*, 2004; Srivastava *et al.*, 2004), customarily serves as an important intermediate in the synthesis of fluoroquinolones.



(I)

The molecular structure of (I) (Fig. 1) contains a five-membered heterocyclic ring, in an envelope conformation, fused to a three-membered cyclopropane ring. The dihedral angel between five- and three-membered ring is 106°

The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds formed between centrosymmetric pairs. Thus, C12—H12*A*···O2^{i} is 2.37 Å, C12···O2^{i} is 3.227 (3) Å and the angle at H12*A* is 146° [symmetry code: (i) $-x, -\frac{1}{2}+y, \frac{1}{2}-z$].

Experimental

The title compound was prepared from *N*-benzylmaleimide (173 mg, 1 mmol) and diazoacetic acid ethyl ester (114 mg, 1 mmol) in solution in diethyl ether (15 ml) by refluxing for 2 h. The solution was then

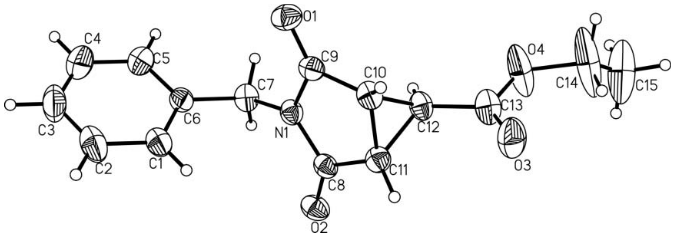


Figure 1
A view of (I), showing the atom-labelling scheme. Only the major component of the disordered ethyl group is shown. Displacement ellipsoids are drawn at the 30% probability level.

placed under computer-controlled cooling crystallization. The crystals obtained were refined by batch cooling recrystallization in diethyl ether several times. The melting point of the crystalline product was determined as 378 K, which agrees with the literature value of 378 K (Srivastava *et al.*, 2004). White single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a diethyl ether solution at room temperature.

Crystal data

$C_{15}H_{15}NO_4$
$M_r = 273.28$
Monoclinic, $P2_1/c$
$a = 14.185$ (3) Å
$b = 5.2973$ (11) Å
$c = 18.661$ (4) Å
$\beta = 101.26$ (3)°
$V = 1375.3$ (5) Å^3

$Z = 4$
$D_x = 1.320$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 296$ (2) K
Column, white
0.53 × 0.33 × 0.16 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.951$, $T_{\max} = 0.985$

10595 measured reflections
2546 independent reflections
1862 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 25.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.157$
$S = 1.08$
2546 reflections
201 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.102P)^2 + 0.0027P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.009$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.009 (3)

The ester ethyl group was found to be disordered. The occupancies of the disordered positions C14/C14′, C15/C15′and O4/O4′ were refined to be 0.533 (13) and 0.467 (13), respectively. The C—C distances involving the disordered atoms were restrained to 1.52 (1) Å, and the U^{ij} components of these atoms were approximated to isotropic behaviour. The H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å, and with $U_{\rm iso}(\rm H) = 1.5U_{\rm eq}(\rm C)$ for methyl H or $1.2U_{\rm eq}(\rm C)$ for the remaining H atoms.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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