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Ethyl 7-chloro-2,3-dihydro-2-oxo-5-phenyl-1*H*-1,4-benzodiazepine-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.075
wR factor = 0.214
Data-to-parameter ratio = 15.1

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

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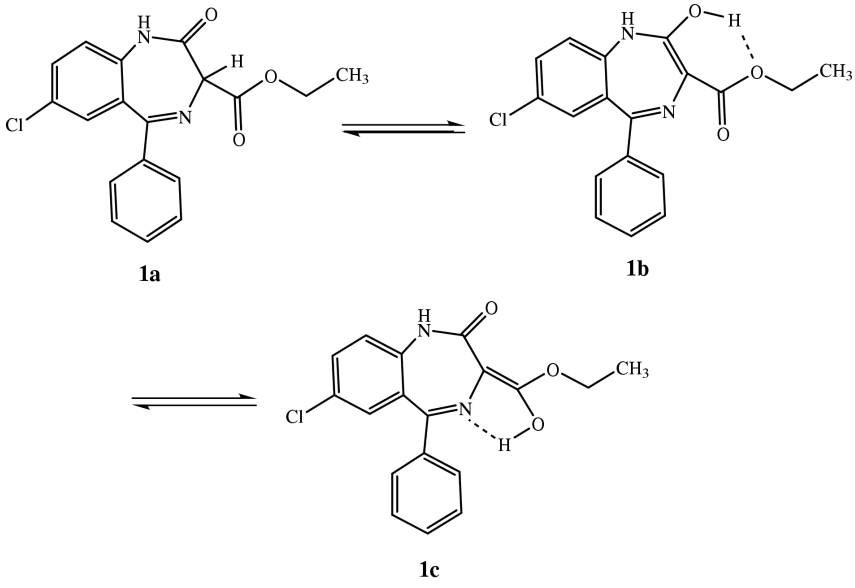
The benzodiazepine ring in the racemic title compound, $C_{18}H_{15}ClN_2O_3$, adopts a boat conformation.

Comment

The configurational stability of chiral 1,4-benzodiazepin-2-ones has been studied in some depth in connection with the difference in pharmacological activity of their enantiomers (Jira *et al.*, 1993; Šunjić *et al.*, 1976). Recently, it was found that the enantiomeric separation of (1) on a chiral HPLC column had been followed by a fast on-column enantiomerization (Abatangelo *et al.*, 2001).



1a **1b** **1c**

The configurational instability of (1) is due to three double bonds flanking the C3 stereogenic center. Such an arrangement induces a high acidity in the C3—H atom which facilitates the two tautomeric forms shown in the scheme above. However, the X-ray structure analysis of (1) reveals form (1*a*) exclusively (Fig. 1). The molecular structure is characterized by a puckered 1,4-benzodiazepin-2-one ring in the usual boat conformation where C3 deviates significantly from the plane defined by N4, C5, N1 and C2. Bond distances and angles in the benzodiazepine ring are in accordance with average values of analogous structures. No classic hydrogen bonds are found in the crystal structure.

Experimental

Compound (1) was obtained from CRC, Compagnia di Ricercha Chimica, Italy. Crystals of (1) were grown by evaporation from an acetonitrile solution of the compound at 277 K.

Crystal data

$C_{18}H_{15}ClN_2O_3$
$M_r = 342.77$
Orthorhombic, *Pbca*
$a = 15.428$ (1) Å
$b = 9.346$ (2) Å
$c = 22.506$ (5) Å
$V = 3245$ (2) Å^3
$Z = 8$
$D_x = 1.403$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 4.4$–$11.8°$
$\mu = 0.25$ mm^{-1}
$T = 293$ (2) K
Plate, colourless
0.20 × 0.09 × 0.03 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.821$, $T_{max} = 0.943$
3730 measured reflections
3286 independent reflections
837 reflections with $I > 2\sigma(I)$
$R_{int} = 0.081$
$\theta_{max} = 26.3°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 28$
3 standard reflections
frequency: 120 min
intensity decay: none

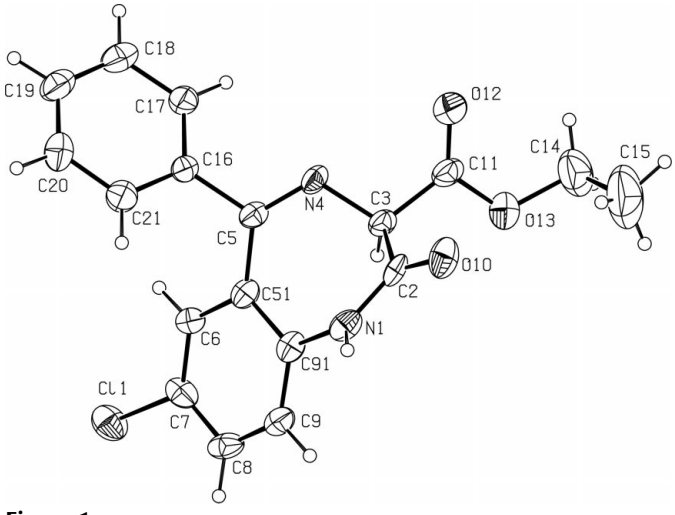


Figure 1
An *ORTEP*II (Johnson, 1976) drawing of (1) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.214$
$S = 0.84$
3286 reflections
218 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1031P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

All H atoms were placed in calculated positions and included in the refinement in the riding model approximation. Some disorder in the ethyl group is evident but multiple sites for these atoms were not resolved.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SET*4 and *CELDIM* in *CAD*-4 *Software* (Enraf–Nonius, 1989); data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *PLATON* (Spek, 1990).

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