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2,2-Dichloro-1-(4-ethoxyphenyl)cyclopropanyl 2,3-dimethylpiperidin-1-yl ketone

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In the title compound, $C_{19}H_{25}Cl_2NO_2$, the piperidine ring shows a normal chair conformation and the cyclopropane ring forms a dihedral angle of 55.68 (17)° with the benzene ring.

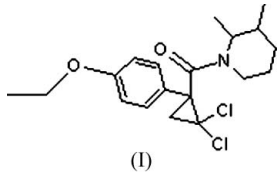
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.085
Data-to-parameter ratio = 18.9

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

Comment

Cycloprothrin derivatives have a high potential for biological activity. They are commonly characterized by low toxicity and good environmental compatibility. As part of our ongoing study on the structure–activity relationships for cycloprothrin derivatives and related compounds, we have recently isolated the title compound, (I), and determined its crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The piperidine ring shows a normal chair conformation. The cyclopropane ring forms a dihedral angle of 55.68 (17)° with the benzene ring. Weak C—H···O hydrogen bonding is observed between neighboring molecules (Table 1).

Experimental

2,3-Dimethylpiperdine (1.0 g, 8.8 mmol) and triethylamine (1.2 g, 11.9 mmol) were dissolved in dichloromethane (15 ml) with stirring. Then 2,2-dichloro-1-(4-ethoxyphenyl)cyclopropanecarbonyl chloride (2.94 g, 10 mmol) was added dropwise to the mixture at room temperature. The mixture was stirred at room temperature for 15 h, washed three times with water and then dried, yielding 2.93 g of a solid product (yield 90.0%). This was recrystallized from ethanol to give single crystals of (I).

Crystal data

$C_{19}H_{25}Cl_2NO_2$
M_r = 370.32
Monoclinic, Cc
a = 11.662 (5) Å
b = 16.242 (8) Å
c = 11.398 (5) Å
β = 115.078 (19)°
V = 1955.3 (15) Å^3
Z = 4
D_x = 1.258 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 298 (1) K
Chunk, colorless
0.33 × 0.30 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.888, T_{max} = 0.934
9469 measured reflections
4133 independent reflections
3347 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.024
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.085$
$S = 1.02$
4133 reflections
219 parameters
H-atom parameters constrained
$w = 1/[0.0004F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 145 (16)
Absolute structure: Flack (1983), 1889 Friedel Pairs
Flack parameter: 0.009 (4)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O1^{i}	0.93	2.40	3.322 (3)	170

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$.

Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angles were refined to fit the electron density. Other H atoms were placed in calculated positions with C—H = 0.93 (aromatic), 0.98 (methine) and 0.97 Å (methylene), and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 1996); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

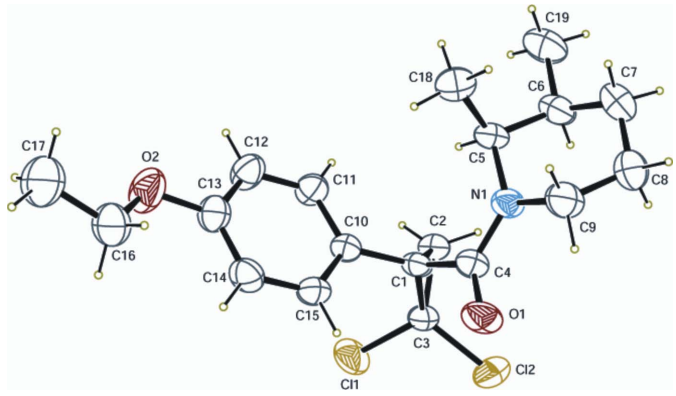


Figure 1
The structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

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