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(5*S*,6*R*)-4,5-Dimethyl-6-phenylmorpholine-2,3-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.087
Data-to-parameter ratio = 9.9

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

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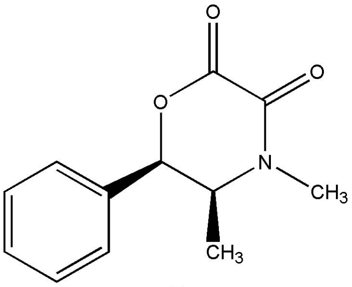
In the title compound, $C_{12}H_{13}NO_3$, the angles at the Csp^3 atoms in the vicinity of the morpholine ring are slightly strained, as indicated by the values of 113.08 (13) and 115.18 (14)° for C—C—C angles and 106.98 (13)° for the N—C—C angle.

Comment

The title compound, (I), was prepared by the reaction of ephedrine hydrochloride and oxalyl chloride (Pansare *et al.*, 2002). The present crystal structure determination of the compound has been carried out in order to elucidate the molecular conformation.



(I)

In the molecular structure of (I), the angles at atoms C3 and C4 indicate strain, compared with the expected value of 109.5° for sp^3 hydridization (Table 1). Similarly, the angles at the sp^2 hybridized atom C2 are distorted from the ideal value of 120°. In the six-membered morpholine ring, the atoms C1/C2/N1/C3 form the best least-squares plane, while atoms C4 and O1 deviate by 0.6838 (2) and 0.1179 (2) Å, respectively, from this plane. The conformation of the heterocyclic ring is twist–boat.

Experimental

To a stirred suspension of ephedrine hydrochloride (2 g, 9.9 mmol) and DMAP (dimethylpyridin-4-ylamine) (60 mg, 0.49 mmol) in dichloromethane (200 ml) at 273 K, triethylamine (5.5 ml, 39.6 mmol) was added. The mixture was stirred for 10 min and a solution of oxalyl chloride (1.3 ml, 14.9 mmol) in dichloromethane (100 ml) was added dropwise over a period of 4 h at 273 K. The mixture was further stirred at 273 K for 1 h and ice was added. The mixture was warmed to ambient temperature and the two phases were separated. The dichloromethane layer was washed with water (70 ml), dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography on silica gel to furnish (I) as a white solid (1.42 g, 65%). The title compound was crystallized from ethyl acetate (m.p.

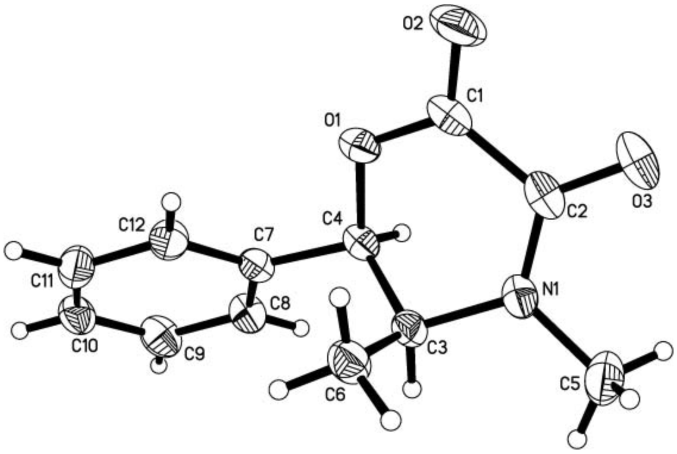


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

455 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3018, 1771, 1693, 1406, 1292, 1215, 1186, 1009; ^{1}H NMR (CDCl$_3$, δ, p.p.m.): 7.50–7.28 (*m*, 5H), 5.90 (*d*, 1H), 3.77–3.66 (*dp*, 1H), 1.12 (*d*, 3H).

Crystal data

$C_{12}H_{13}NO_3$
$M_r = 219.23$
Monoclinic, $P2_1$
$a = 7.3829$ (11) Å
$b = 6.7658$ (10) Å
$c = 11.1074$ (17) Å
$\beta = 93.826$ (2)°
$V = 553.59$ (14) Å^3
$Z = 2$
$D_x = 1.315$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1600 reflections
$\theta = 3.4$–26.6°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.976$, $T_{\max} = 0.981$
3758 measured reflections
1454 independent reflections
1263 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$
$\theta_{\max} = 28.1$°
$h = -9 \rightarrow 7$
$k = -8 \rightarrow 8$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.087$
$S = 1.10$
1454 reflections
147 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.0289P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.335 (2)	C3—C4	1.514 (3)
O1—C4	1.4564 (19)	C3—C6	1.519 (3)
C1—C2	1.539 (3)	C4—C7	1.508 (2)
O3—C2—N1	124.82 (19)	C4—C3—C6	115.18 (14)
O3—C2—C1	118.51 (16)	O1—C4—C7	108.27 (12)
N1—C2—C1	116.65 (14)	O1—C4—C3	109.22 (14)
N1—C3—C4	106.98 (13)	C7—C4—C3	113.08 (13)
N1—C3—C6	110.76 (15)		

All H atoms were positioned geometrically and refined in a riding-model approximation (C—H = 0.93–0.98 Å) with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(carrier atom), or 1.5$U_{\rm eq}$(carrier atom) for methyl H. In the absence of anomalous dispersion effects, the Friedel pairs were merged. The absolute configuration was assigned on the basis of the known configuration of ephedrine hydrochloride.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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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o2168 Yan *et al.* • $C_{12}H_{13}NO_3$ *Acta Cryst.* (2005). E**61**, o2167–o2168