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tert-Butyl 3a-chloroperhydro-2,6a-epoxyoxireno[e]isoindole-5-carboxylate

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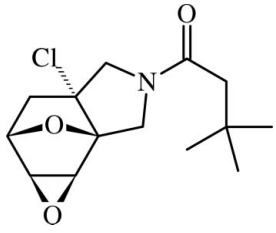
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.112; data-to-parameter ratio = 16.3.

In the title compound, $C_{13}H_{18}ClNO_4$, the molecules are linked only by weak van der Waals interactions. The boat form of the six-membered ring is almost symmetric with respect to the epoxy bridge. The three five-membered rings adopt envelope conformations.

Related literature

For related literature, see: Brickwood *et al.* (1999); Christoffers & Baro (2005); Cremer & Pople (1975); Koşar *et al.* (2007); Prajapati *et al.* (1993).



Experimental

Crystal data

$C_{13}H_{18}ClNO_4$
M_r = 287.73
Monoclinic, $P2_1/c$
a = 18.7133 (15) Å
b = 8.3800 (8) Å
c = 9.2123 (7) Å
β = 90.134 (7)°
V = 1444.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 293 (2) K
0.50 × 0.32 × 0.03 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED*; Stoe & Cie, 2002)
T_{min} = 0.911, T_{max} = 0.992
11238 measured reflections
3390 independent reflections
1847 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.112
S = 0.88
3390 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2388).

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supplementary materials

Acta Cryst. (2007). E**63**, o3323 [**doi:10.1107/S1600536807030346**]

tert-Butyl 3a-chloroperhydro-2,6a-epoxyoxireno[*e*]isoindole-5-carboxylate

B. Kosar, M. Karaarslan, I. Demir and O. Büyükgüngör

Comment

Intramolecular Diels-Alder (IMDA) cycloadducts derived from hetero-atoms in tether of furans have recently been of great interest since they involve a ring formation for natural product synthesis such as Azadirachtin (Prajapati *et al.*, 1993) and Fraquinocin E (Christoffers *et al.*, 2005). The utility of an IMDA cycloadduct is another part of research concept in which usually the fragmentation of an oxa bridge in the cyclohexene part is required (Brickwood *et al.*, 1999).

Figure 1 shows the molecular structure of the title compound. The pyrrolidine (N1/C7/C6/C1/C8), tetrahydrofuran (O1/C6—C3) and chloro-attached tetrahydrofuran (O1/C6/C1/C2/C3) rings adopt envelope conformations, and the total puckering parameter Q_T values are 0.331 (3), 0.525 (3) and 0.569 (3) A°, respectively (Cremer & Pople, 1975).

For a closely related compound, *tert*-Butyl 3a-chloro-2-methylperhydro-2,6a- epoxyoxireno[*e*]isoindole-5-carboxylate, see Koşar *et al.* (2007).

Experimental

To a solution of *meta*-chloroperbenzoic acid (*m*-CPBA) (120 mg, 0.66 mmol), which had previously been purified and re-crystallized from dry diethyl ether, in dichloromethane (10 ml), cooled to 273 K, was added dropwise a solution of *tert*-butyl 7a-chloro-1,6,7,7a-tetrahydro-3a,6-epoxyisoindole- 2-carboxylate (0.66 mmol) in dichloromethane (10 ml) over a period of 3 min. The reaction mixture was stirred at room temperature for 4 h and then diluted with cold 4% sodium bicarbonate solution (4 ml). The organic layer was separated, washed with water (20 ml) and concentrated *in vacuo*. The residue was subjected to flash column chromatography and yielded colourless crystals (110 mg, 56%). m.p: 410–412 K, t.l.c., (Hexane: Ethyl acetate (7:3)): Rf: 0.26.

Refinement

The methyl H atoms were positioned geometrically and refined using a riding model with C—H = 0.96 Å, and U_{iso}=1.5 U_{eq}(C). Other H atoms were located in a difference map and refined freely.

Figures

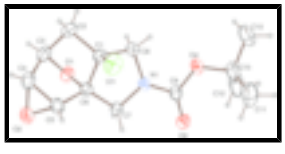

Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

tert-Butyl 3a-chloroperhydro-2,6a-epoxyoxireno[e]isoindole-5-carboxylate

Crystal data

$C_{13}H_{18}ClNO_4$	F_{000} = 608
M_r = 287.73	D_x = 1.323 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1048 reflections
a = 18.7133 (15) Å	θ = 2.2–27.9º
b = 8.3800 (8) Å	μ = 0.27 mm^{-1}
c = 9.2123 (7) Å	T = 293 (2) K
β = 90.134 (7)º	Plate, colourless
V = 1444.6 (2) Å^3	0.50 × 0.32 × 0.03 mm
Z = 4	

Data collection

Stoe IPDSII diffractometer	3390 independent reflections
Radiation source: fine-focus sealed tube	1847 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.045
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 27.9º
T = 293(2) K	θ_{min} = 2.2º
ω scans	h = −24→24
Absorption correction: integration (X-RED; Stoe & Cie, 2002)	k = −10→10
T_{min} = 0.911, T_{max} = 0.992	l = −11→11
11238 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.112	$w = 1/[\sigma^2(F_o^2) + (0.1002P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.88	$(\Delta/\sigma)_{max}$ = 0.001
3390 reflections	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
208 parameters	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
H7B	0.8797 (12)	−0.369 (3)	0.287 (2)	0.051 (6)*
H2B	0.8158 (14)	0.132 (3)	0.432 (3)	0.069 (8)*
H5	0.9326 (13)	−0.042 (3)	0.043 (3)	0.063 (7)*
H8B	0.7149 (14)	−0.099 (3)	0.293 (3)	0.057 (6)*
H7A	0.8627 (12)	−0.324 (3)	0.120 (3)	0.051 (6)*
H4	0.9735 (13)	0.188 (3)	0.195 (3)	0.061 (7)*
H8A	0.7649 (12)	−0.123 (3)	0.423 (3)	0.059 (7)*
H2A	0.8411 (12)	0.225 (3)	0.290 (3)	0.062 (7)*
H3	0.9437 (14)	0.108 (3)	0.459 (3)	0.071 (8)*
Cl1	0.78679 (4)	0.04055 (8)	0.07373 (8)	0.0707 (2)
O3	0.75689 (9)	−0.53162 (17)	0.1873 (2)	0.0633 (5)
O1	0.91221 (8)	−0.09924 (16)	0.38532 (16)	0.0494 (4)
N1	0.78055 (9)	−0.28137 (18)	0.2605 (2)	0.0446 (4)
O2	1.00890 (8)	−0.04342 (19)	0.2061 (2)	0.0653 (5)
C9	0.73723 (11)	−0.4063 (2)	0.2387 (2)	0.0462 (5)
C1	0.81622 (11)	−0.0154 (2)	0.2528 (2)	0.0438 (5)
O4	0.67030 (8)	−0.37214 (19)	0.28057 (19)	0.0612 (4)
C7	0.85606 (11)	−0.2919 (2)	0.2225 (3)	0.0443 (5)
C3	0.92012 (13)	0.0720 (3)	0.3739 (3)	0.0581 (6)
C5	0.93750 (12)	−0.0444 (3)	0.1468 (3)	0.0510 (5)
C6	0.88277 (10)	−0.1245 (2)	0.2436 (2)	0.0392 (4)
C8	0.76129 (13)	−0.1262 (3)	0.3189 (3)	0.0490 (5)
C10	0.61231 (13)	−0.4860 (3)	0.2554 (3)	0.0697 (7)
C2	0.84309 (14)	0.1263 (3)	0.3439 (3)	0.0589 (6)
C4	0.96174 (13)	0.0886 (3)	0.2346 (3)	0.0604 (7)
C13	0.54841 (16)	−0.3970 (5)	0.3134 (5)	0.1233 (15)
H13A	0.5409	−0.3020	0.2570	0.185*
H13B	0.5568	−0.3684	0.4129	0.185*
H13C	0.5068	−0.4637	0.3071	0.185*
C12	0.62559 (18)	−0.6360 (4)	0.3405 (5)	0.1143 (14)
H12A	0.6664	−0.6905	0.3015	0.171*
H12B	0.5844	−0.7040	0.3342	0.171*
H12C	0.6345	−0.6094	0.4403	0.171*

C11	0.60394 (19)	−0.5166 (5)	0.0956 (4)	0.1048 (12)
H11A	0.6448	−0.5742	0.0606	0.157*
H11B	0.6002	−0.4167	0.0451	0.157*
H11C	0.5615	−0.5784	0.0789	0.157*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0830 (5)	0.0629 (4)	0.0661 (4)	0.0032 (3)	−0.0240 (3)	0.0150 (3)
O3	0.0566 (9)	0.0427 (8)	0.0907 (13)	−0.0066 (7)	0.0049 (9)	−0.0068 (8)
O1	0.0569 (9)	0.0499 (8)	0.0413 (9)	−0.0035 (7)	−0.0076 (7)	0.0012 (7)
N1	0.0385 (9)	0.0375 (9)	0.0577 (12)	−0.0029 (7)	0.0077 (8)	−0.0018 (8)
O2	0.0477 (9)	0.0570 (9)	0.0914 (14)	−0.0100 (7)	0.0072 (9)	0.0031 (9)
C9	0.0430 (12)	0.0449 (11)	0.0507 (14)	−0.0051 (9)	−0.0008 (9)	0.0059 (10)
C1	0.0473 (11)	0.0397 (10)	0.0444 (12)	0.0016 (8)	−0.0030 (9)	−0.0036 (9)
O4	0.0399 (8)	0.0628 (10)	0.0811 (13)	−0.0099 (7)	0.0080 (8)	−0.0060 (9)
C7	0.0409 (11)	0.0390 (10)	0.0531 (15)	−0.0017 (9)	0.0063 (10)	−0.0037 (10)
C3	0.0672 (16)	0.0457 (12)	0.0613 (17)	−0.0072 (10)	−0.0137 (12)	−0.0120 (11)
C5	0.0491 (13)	0.0518 (12)	0.0521 (15)	−0.0077 (10)	0.0061 (10)	0.0051 (11)
C6	0.0434 (11)	0.0384 (10)	0.0357 (12)	−0.0026 (8)	0.0008 (8)	0.0003 (8)
C8	0.0439 (13)	0.0470 (12)	0.0562 (17)	0.0017 (9)	0.0062 (11)	−0.0077 (11)
C10	0.0452 (13)	0.0734 (16)	0.090 (2)	−0.0162 (12)	−0.0038 (13)	0.0052 (14)
C2	0.0667 (16)	0.0422 (13)	0.0680 (19)	0.0003 (11)	0.0000 (13)	−0.0143 (12)
C4	0.0563 (14)	0.0439 (12)	0.0811 (19)	−0.0134 (11)	−0.0030 (13)	0.0080 (12)
C13	0.0484 (18)	0.130 (3)	0.192 (5)	−0.0154 (18)	0.020 (2)	−0.023 (3)
C12	0.081 (2)	0.110 (3)	0.152 (4)	−0.0485 (19)	−0.022 (2)	0.058 (2)
C11	0.085 (2)	0.131 (3)	0.098 (3)	−0.030 (2)	−0.0279 (19)	−0.002 (2)

Geometric parameters (Å, °)

Cl1—C1	1.800 (2)	C5—C4	1.450 (3)
O3—C9	1.210 (3)	C5—C6	1.516 (3)
O1—C6	1.432 (2)	C5—H5	0.96 (3)
O1—C3	1.446 (3)	C8—H8B	0.93 (2)
N1—C9	1.339 (3)	C8—H8A	0.96 (3)
N1—C8	1.453 (3)	C10—C12	1.502 (4)
N1—C7	1.459 (3)	C10—C11	1.502 (4)
O2—C4	1.440 (3)	C10—C13	1.508 (4)
O2—C5	1.442 (3)	C2—H2B	0.96 (3)
C9—O4	1.342 (3)	C2—H2A	0.96 (3)
C1—C8	1.514 (3)	C4—H4	0.93 (2)
C1—C2	1.538 (3)	C13—H13A	0.9600
C1—C6	1.548 (3)	C13—H13B	0.9600
O4—C10	1.463 (3)	C13—H13C	0.9600
C7—C6	1.501 (3)	C12—H12A	0.9600
C7—H7B	0.98 (2)	C12—H12B	0.9600
C7—H7A	0.99 (3)	C12—H12C	0.9600
C3—C4	1.509 (4)	C11—H11A	0.9600
C3—C2	1.536 (4)	C11—H11B	0.9600

C3—H3	0.95 (3)	C11—H11C	0.9600
C6—O1—C3	96.88 (15)	C1—C8—H8B	112.2 (15)
C9—N1—C8	127.19 (17)	N1—C8—H8A	112.0 (14)
C9—N1—C7	120.21 (17)	C1—C8—H8A	109.9 (14)
C8—N1—C7	112.59 (16)	H8B—C8—H8A	108 (2)
C4—O2—C5	60.40 (15)	O4—C10—C12	110.0 (2)
O3—C9—N1	123.6 (2)	O4—C10—C11	110.0 (2)
O3—C9—O4	125.62 (19)	C12—C10—C11	112.7 (3)
N1—C9—O4	110.82 (18)	O4—C10—C13	102.1 (2)
C8—C1—C2	118.4 (2)	C12—C10—C13	111.1 (3)
C8—C1—C6	101.95 (16)	C11—C10—C13	110.5 (3)
C2—C1—C6	102.96 (17)	C3—C2—C1	100.05 (18)
C8—C1—Cl1	108.77 (16)	C3—C2—H2B	111.4 (16)
C2—C1—Cl1	113.45 (17)	C1—C2—H2B	109.0 (16)
C6—C1—Cl1	110.36 (14)	C3—C2—H2A	112.4 (14)
C9—O4—C10	120.50 (18)	C1—C2—H2A	111.5 (15)
N1—C7—C6	103.57 (16)	H2B—C2—H2A	112 (2)
N1—C7—H7B	109.3 (13)	O2—C4—C5	59.88 (15)
C6—C7—H7B	112.7 (13)	O2—C4—C3	113.7 (2)
N1—C7—H7A	111.6 (13)	C5—C4—C3	104.00 (19)
C6—C7—H7A	109.4 (13)	O2—C4—H4	118.0 (15)
H7B—C7—H7A	110.1 (19)	C5—C4—H4	122.9 (15)
O1—C3—C4	101.94 (19)	C3—C4—H4	122.2 (15)
O1—C3—C2	102.19 (18)	C10—C13—H13A	109.5
C4—C3—C2	107.8 (2)	C10—C13—H13B	109.5
O1—C3—H3	107.8 (16)	H13A—C13—H13B	109.5
C4—C3—H3	115.5 (16)	C10—C13—H13C	109.5
C2—C3—H3	119.3 (16)	H13A—C13—H13C	109.5
O2—C5—C4	59.71 (15)	H13B—C13—H13C	109.5
O2—C5—C6	114.0 (2)	C10—C12—H12A	109.5
C4—C5—C6	102.9 (2)	C10—C12—H12B	109.5
O2—C5—H5	117.6 (15)	H12A—C12—H12B	109.5
C4—C5—H5	124.9 (15)	C10—C12—H12C	109.5
C6—C5—H5	122.0 (14)	H12A—C12—H12C	109.5
O1—C6—C7	112.54 (17)	H12B—C12—H12C	109.5
O1—C6—C5	102.24 (16)	C10—C11—H11A	109.5
C7—C6—C5	124.24 (19)	C10—C11—H11B	109.5
O1—C6—C1	99.80 (15)	H11A—C11—H11B	109.5
C7—C6—C1	106.95 (16)	C10—C11—H11C	109.5
C5—C6—C1	108.39 (16)	H11A—C11—H11C	109.5
N1—C8—C1	103.32 (17)	H11B—C11—H11C	109.5
N1—C8—H8B	111.1 (14)		

C8—N1—C9—O3	178.8 (2)	C2—C1—C6—C7	153.6 (2)
C7—N1—C9—O3	−0.4 (3)	Cl1—C1—C6—C7	−85.05 (19)
C8—N1—C9—O4	−0.8 (3)	C8—C1—C6—C5	166.53 (18)
C7—N1—C9—O4	179.98 (19)	C2—C1—C6—C5	−70.3 (2)
O3—C9—O4—C10	−4.9 (4)	Cl1—C1—C6—C5	51.11 (19)
N1—C9—O4—C10	174.7 (2)	C9—N1—C8—C1	−153.1 (2)

C9—N1—C7—C6	172.53 (19)	C7—N1—C8—C1	26.1 (3)
C8—N1—C7—C6	−6.8 (3)	C2—C1—C8—N1	−145.2 (2)
C6—O1—C3—C4	−52.72 (19)	C6—C1—C8—N1	−33.1 (2)
C6—O1—C3—C2	58.7 (2)	Cl1—C1—C8—N1	83.45 (19)
C4—O2—C5—C6	−91.3 (2)	C9—O4—C10—C12	64.0 (3)
C3—O1—C6—C7	−170.96 (18)	C9—O4—C10—C11	−60.7 (3)
C3—O1—C6—C5	53.53 (19)	C9—O4—C10—C13	−178.0 (3)
C3—O1—C6—C1	−57.89 (17)	O1—C3—C2—C1	−34.1 (3)
N1—C7—C6—O1	93.4 (2)	C4—C3—C2—C1	72.8 (2)
N1—C7—C6—C5	−142.5 (2)	C8—C1—C2—C3	110.3 (2)
N1—C7—C6—C1	−15.2 (2)	C6—C1—C2—C3	−1.2 (2)
O2—C5—C6—O1	28.3 (2)	Cl1—C1—C2—C3	−120.42 (19)
C4—C5—C6—O1	−34.1 (2)	C5—O2—C4—C3	92.9 (2)
O2—C5—C6—C7	−100.2 (2)	C6—C5—C4—O2	110.41 (19)
C4—C5—C6—C7	−162.5 (2)	O2—C5—C4—C3	−109.5 (2)
O2—C5—C6—C1	133.09 (19)	C6—C5—C4—C3	0.9 (2)
C4—C5—C6—C1	70.8 (2)	O1—C3—C4—O2	−30.8 (2)
C8—C1—C6—O1	−86.96 (18)	C2—C3—C4—O2	−137.96 (19)
C2—C1—C6—O1	36.2 (2)	O1—C3—C4—C5	32.1 (2)
Cl1—C1—C6—O1	157.62 (12)	C2—C3—C4—C5	−75.0 (2)
C8—C1—C6—C7	30.4 (2)		

Fig. 1