organic papers

5-*tert*-Butylperoxy-5-(oxiran-2-yl)-1-(prop-2-ynyl)-pyrrolidin-2-one

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Key indicators

Single-crystal X-ray study

$T = 150$ K

Mean σ(C–C) = 0.003 Å

R factor = 0.040

wR factor = 0.096

Data-to-parameter ratio = 12.3

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

The title compound, $C_{13}H_{19}NO_4$, contains the 5-*tert*-butylperoxy-, 5-oxiranyl- and 1-prop-2-ynyl-substituted pyrrolidin-2-one ring system in an envelope conformation, with one CH_2 C atom out of the plane by 0.272 (3) Å.

Comment

The action of *tert*-butyl hydroperoxide and tin(IV) chloride upon allylic alcohols containing a lactam ring leads mainly to epoxy alkyl peroxides with high diastereoselection. The structure of the title compound, (I), is the *anti* diastereoisomer of one such peroxide. The core of the structure is a pyrrolidinone ring in an envelope conformation (Cremer & Pople, 1975), with C6 out of the ring plane by 0.272 (3) Å. The substituents consist of a prop-2-ynyl chain attached to the ring N atom and both *tert*-butylperoxy and oxiranyl moieties attached to C7 in the 5-position, making it an *S* configuration. All bond lengths and angles were found to be within expected ranges. No classic hydrogen bonds were found; however, a number of C—H···O interactions with separations less than the sum of the van der Waals radii (Bondi, 1964) were found to form a three-dimensional network. These interactions are detailed in Table 2.

(I)
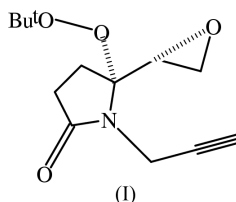
Experimental

The reaction of a diperoxide and an epoxy peroxide in the presence of $SnCl_4$ produced a mixture of diastereoisomers. Crystals of (I) were obtained from an ethyl acetate/petroleum ether (313–333 K) mixture (Hursthouse *et al.*, 1995; Marson *et al.*, 2001).

Crystal data

$C_{13}H_{19}NO_4$	$D_x = 1.257$ Mg m^{-3}
$M_r = 253.29$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/a$	Cell parameters from 5318
$a = 10.2870$ (14) Å	reflections
$b = 10.729$ (2) Å	$\theta = 2.5$–24.9°
$c = 13.1233$ (13) Å	$\mu = 0.09$ mm^{-1}
$\beta = 112.423$ (13)°	$T = 150$ (2) K
$V = 1338.9$ (4) Å^3	Block, colourless
$Z = 4$	$0.20 \times 0.20 \times 0.15$ mm
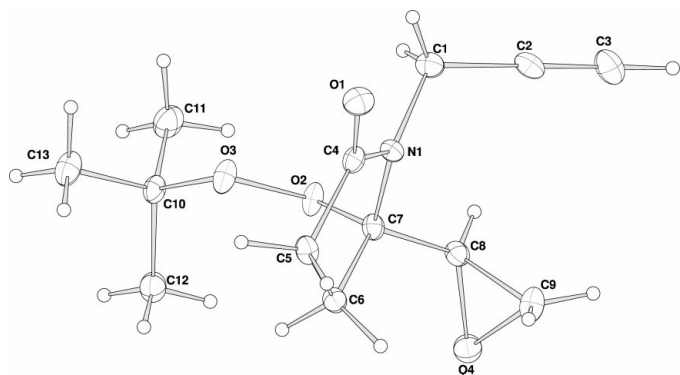
Figure 1
View of (I) shown with 50% probability displacement ellipsoids.

Data collection

FAST TV area-detector diffractometer	2041 independent reflections
φ and ω scans	1516 reflections with $I > 2\sigma(I)$
Absorption correction: refined from ΔF (Walker & Stuart, 1983)	$R_{int} = 0.073$
$T_{min} = 0.985$, $T_{max} = 0.989$	$\theta_{max} = 24.9°$
5318 measured reflections	$h = -11 \rightarrow 12$
	$k = -9 \rightarrow 12$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	H-atom parameters constrained
$R(F) = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2]$
$wR(F^2) = 0.096$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.91$	$(\Delta/\sigma)_{max} = 0.003$
2041 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
166 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C4	1.361 (2)	O4—C8	1.431 (2)
N1—C7	1.460 (2)	O4—C9	1.445 (2)
O2—C7	1.431 (2)	C4—C5	1.496 (2)
O2—O3	1.479 (2)	C5—C6	1.529 (2)
O3—C10	1.448 (2)	C6—C7	1.530 (2)
C4—N1—C7	114.00 (13)	N1—C4—C5	108.76 (14)
C7—O2—O3	106.66 (11)	C4—C5—C6	105.05 (13)
C10—O3—O2	106.92 (11)	C7—C6—C5	105.71 (14)
C8—O4—C9	60.63 (11)	O4—C8—C9	60.15 (11)
N1—C1—C2	113.8 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C12—H12$B\cdots$O1[i]	0.96	2.59	3.387 (2)	140
C3—H3$\cdots$O1[ii]	0.93	2.42	3.275 (3)	153
C9—H9$B\cdots$O1[iii]	0.97	2.46	3.420 (2)	169
C5—H5$A\cdots$O4[iv]	0.97	2.42	3.348 (2)	160

Symmetry codes: (i) $x - \frac{1}{2}, \frac{3}{2} - y, z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $\frac{1}{2} + x, \frac{3}{2} - y, z$.

Details of the data collection method are described by Darr *et al.* (1993). H atoms were treated as riding atoms (C—H = 0.93 and 0.97 Å).

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1989); cell refinement: *MADNES*; data reduction: *ABSMAD* (Karaulov, 1992); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *CAMERON* (Watkin *et al.*, 1993).

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