

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

**(±)-2'-Phenylcyclohexanespiro-4'-(azepano[1,2-*b*]isoxazolidine)****Daryl Crimmins, Ka Wai Choi, Peter D. W. Boyd and Margaret A. Brimble\***

Department of Chemistry, The University of Auckland, Private Bag 92019, Auckland, New Zealand

Correspondence e-mail: m.brimble@auckland.ac.nz

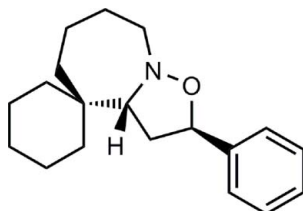
Received 2 July 2008; accepted 14 July 2008

Key indicators: single-crystal X-ray study;  $T = 85$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.084;  $wR$  factor = 0.195; data-to-parameter ratio = 17.0.

In the crystal structure of the racemic title isoxazolidine,  $\text{C}_{19}\text{H}_{27}\text{NO}$ , the relative stereochemistry between the phenyl group and the bridgehead H atom is shown to be *syn*. There are two molecules in the asymmetric unit, one of which is the  $7R^*,13R^*$  enantiomer, and one of which is the  $7S^*,13S^*$  enantiomer. These enantiomers adopt different orientations of the phenyl ring with respect to the isoxazolidine ring, with C—C—C—C torsion angles of 63.6 (4) and 86.8 (4)°, respectively. In both enantiomers, the six-membered ring adopts a chair conformation, while the seven-membered ring adopts a twist-chair conformation.

**Related literature**

For related literature regarding the synthesis towards the spiroimine unit of the spiroalides, see: Brimble & Trzoss (2004); Brimble *et al.* (2005); O'Connor *et al.* (2008). For the crystal structure of the related 7,6-spirolactam unit, see: Guéret *et al.* (2008). For isolation of spiroalides from natural resources, see: Hu *et al.* (2001); MacKinnon *et al.* (2006); Ciminiello *et al.* (2007).

**Experimental***Crystal data*

$\text{C}_{19}\text{H}_{27}\text{NO}$	$\gamma = 96.527$ (1)°
$M_r = 285.42$	$V = 1612.34$ (2) Å <sup>3</sup>
Triclinic, $P\bar{1}$	$Z = 4$
$a = 9.8516$ (1) Å	Mo $K\alpha$ radiation
$b = 10.4560$ (1) Å	$\mu = 0.07$ mm <sup>-1</sup>
$c = 16.0957$ (1) Å	$T = 85$ (2) K
$\alpha = 101.058$ (1)°	$0.32 \times 0.24 \times 0.22$ mm
$\beta = 92.833$ (1)°	

*Data collection*

Siemens SMART CCD diffractometer	15138 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	6437 independent reflections
$T_{\min} = 0.978$ , $T_{\max} = 0.985$	4959 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.026$

*Refinement*

$R[F^2 > 2\sigma(F^2)] = 0.084$	379 parameters
$wR(F^2) = 0.194$	H-atom parameters constrained
$S = 1.06$	$\Delta\rho_{\text{max}} = 1.21$ e Å <sup>-3</sup>
6437 reflections	$\Delta\rho_{\text{min}} = -0.32$ e Å <sup>-3</sup>

Data collection: SMART (Siemens, 1995); cell refinement: SAINT (Siemens, 1995); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEPIII (Burnett & Johnson, 1996) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXTL (Sheldrick, 2008) and publCIF (Westrip, 2008).

The authors thank Tania Groutso for help with the data collection and the Royal Society of New Zealand for the award of a Marsden Funds Grant (to DC).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2294).

**References**

- Brimble, M. A., Crimmins, D. & Trzoss, M. (2005). *ARKIVOC*, i, 39–52.  
 Brimble, M. A. & Trzoss, M. (2004). *Tetrahedron*, **60**, 5613–5622.  
 Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.  
 Ciminiello, P., Dell'Aversano, C., Fattorusso, E., Forino, M., Grauso, L., Tartaglione, L., Guerrini, F. & Pistocchi, R. (2007). *J. Nat. Prod.* **70**, 1878–1883.  
 Guéret, S. M., Choi, K. W., O'Connor, P. D., Boyd, P. D. W. & Brimble, M. A. (2008). *Acta Cryst.* **E64**, o1151.  
 Hu, T. M., Burton, I. W., Cembella, A. D., Curtis, J. M., Quilliam, M. A., Walter, J. A. & Wright, J. L. C. (2001). *J. Nat. Prod.* **64**, 308–312.  
 MacKinnon, S. L., Walter, J. A., Quilliam, M. A., Cembella, A. D., Leblanc, P., Burton, I. W., Hardstaff, W. R. & Lewis, N. I. (2006). *J. Nat. Prod.* **69**, 983–987.  
 Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.  
 O'Connor, P. D., Körber, K. & Brimble, M. A. (2008). *Synlett*, pp. 1036–1038.  
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Siemens (1995). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Westrip, S. P. (2008). *publCIF*. In preparation.

**supplementary materials**

*Acta Cryst.* (2008). E64, o1535 [ doi:10.1107/S1600536808021867 ]

**(±)-2'-Phenylcyclohexanespiro-4'-(azepano[1,2-*b*]isoxazolidine)**

**D. Crimmins, K. W. Choi, P. D. W. Boyd and M. A. Brimble**

**Comment**

The title isoxazolidine was prepared as part of a synthetic program (Brimble & Trzoss, 2004; Brimble *et al.* 2005; O'Connor *et al.*, 2008 and Guéret *et al.*, 2008) directed towards the synthesis of heterocycles related to the spiroimine unit of the spiroside family of shellfish toxins that were isolated from the culture of a toxic clone of the dinoflagellate *Alexandrium ostenfeldii* (Hu *et al.*, 2001; MacKinnon *et al.*, 2006 and Ciminiello *et al.*, 2007). With this idea in mind, a study of the thermal-promoted 1,3-dipolar cycloaddition of a novel 7,6-spirotrione to a range of alkenes was investigated. In particular, the addition of the 7,6-spirotrione to styrene proceeded regioselectively to afford the title racemic isoxazolidine. However, establishment of the *exo* or *endo* selectivity of this 1,3-dipolar cycloaddition was required and this could not be deduced from detailed NMR studies. The crystal structure of the cycloadduct (Fig. 1) clearly indicates the *syn* stereochemistry between the phenyl group at C13 and the bridgehead proton at C7, thereby confirming that the 1,3-dipolar cycloaddition took place with *exo* selectivity.

The relative stereochemistry of the racemic isoxazolidine is shown to be  $7R^*,13R^*$  and  $7S^*,13S^*$  from the crystal structure. Apart from the opposite stereochemistry, the two molecules in the asymmetric unit also differ from each other in the orientation adopted by the phenyl ring relative to the adjacent isoxazolidine ring. This is shown by the different torsion angle adopted at C12–C13–C14–C15: for one enantiomer, the torsion angle is  $63.6(4)^\circ$  while the torsion angle for the opposite enantiomer is  $86.8(4)^\circ$ . In both molecules, the six-membered ring adopts a chair conformation while the seven-membered ring adopts a twist-chair conformation. A view of the molecular packing is depicted in Fig. 2.

**Experimental**

8-Azoniaspiro[5.6]dodec-7-en-8-olate (550 mg, 1.96 mmol) and styrene (905  $\mu$ l, 7.9 mmol) were dissolved in toluene (10 ml) under an argon atmosphere and the solution was heated under reflux for 12 h. The reaction mixture was cooled to room temperature and the solvent was concentrated *in vacuo*. The residue was purified directly by flash chromatography (hexane-ethyl acetate 10:1) to give the title compound as a white solid (353 mg, 63%). Recrystallization from diethyl ether afforded white needles.

**Refinement**

H atoms were placed in calculated positions and were refined using a riding model (C–H = 0.93 or 0.97 Å), with  $U_{\text{iso}}(\text{H}) = 1.2$  or  $1.5$  times  $U_{\text{eq}}(\text{C})$ . Two peaks are present in the final difference density:  $1.21 \text{ e}\text{\AA}^{-3}$  at a distance of  $1.11 \text{ \AA}$  from O, and  $1.02 \text{ e}\text{\AA}^{-3}$  at a distance of  $1.20 \text{ \AA}$  from C14.

## Figures

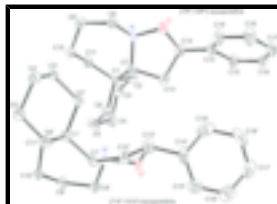


Fig. 1. The molecular structure and atom numbering scheme of (7*R*\*,13*R*\* and 7*S*\*,13*S*\*)-isoxazolidine with displacement ellipsoids drawn at the 50% probability level. H atoms are omitted.



Fig. 2. Molecular packing of racemic isoxazolidine. H atoms are omitted. The origin of the unit cell is labelled as *O* while cell axes are labelled as *a* (red), *b* (green) and *c* (blue), respectively. [Symmetry code: (ii) 1 - *x*, 1 - *y*, 1 - *z*.]

## (±)-2'-Phenylcyclohexanespiro-4'-(azepano[1,2-*b*]isoxazolidine)

### Crystal data

$C_{19}H_{27}NO$	$Z = 4$
$M_r = 285.42$	$F_{000} = 624$
Triclinic, <i>PT</i>	$D_x = 1.176 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Melting point: 338.8(7) K
$a = 9.8516 (1) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 10.4560 (1) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$c = 16.0957 (1) \text{ \AA}$	Cell parameters from 6437 reflections
$\alpha = 101.058 (1)^\circ$	$\theta = 1.3\text{--}26.4^\circ$
$\beta = 92.833 (1)^\circ$	$\mu = 0.07 \text{ mm}^{-1}$
$\gamma = 96.527 (1)^\circ$	$T = 85 (2) \text{ K}$
$V = 1612.34 (2) \text{ \AA}^3$	Needles, white
	$0.32 \times 0.24 \times 0.22 \text{ mm}$

### Data collection

Siemens SMART CCD diffractometer	6437 independent reflections
Radiation source: fine-focus sealed tube	4959 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.026$
$T = 85(2) \text{ K}$	$\theta_{\text{max}} = 26.4^\circ$
$\omega$ scans	$\theta_{\text{min}} = 1.3^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.978$ , $T_{\text{max}} = 0.985$	$k = -13 \rightarrow 12$
15138 measured reflections	$l = 0 \rightarrow 20$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.084$	H-atom parameters constrained
$wR(F^2) = 0.194$	$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 4.1372P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
6437 reflections	$(\Delta/\sigma)_{\max} < 0.001$
379 parameters	$\Delta\rho_{\max} = 1.21 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$
	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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O	0.6455 (2)	0.6154 (2)	0.33169 (13)	0.0268 (5)
N	0.6590 (3)	0.5526 (3)	0.24237 (15)	0.0226 (6)
C1	0.8405 (3)	0.4282 (3)	0.17312 (17)	0.0161 (6)
C2	0.9653 (3)	0.5340 (3)	0.1897 (2)	0.0243 (7)
H2A	0.9346	0.6189	0.1894	0.029*
H2B	1.0085	0.5368	0.2456	0.029*
C3	1.0710 (3)	0.5099 (3)	0.1238 (2)	0.0303 (7)
H3A	1.0313	0.5166	0.0687	0.036*
H3B	1.1496	0.5768	0.1390	0.036*
C4	1.1179 (3)	0.3742 (3)	0.1186 (2)	0.0347 (8)
H4A	1.1678	0.3714	0.1715	0.042*
H4B	1.1796	0.3592	0.0735	0.042*
C5	0.9964 (3)	0.2656 (3)	0.1013 (2)	0.0319 (8)
H5A	1.0288	0.1820	0.1033	0.038*
H5B	0.9542	0.2603	0.0448	0.038*
C6	0.8892 (3)	0.2914 (3)	0.1667 (2)	0.0252 (7)
H6A	0.9280	0.2842	0.2219	0.030*
H6B	0.8107	0.2245	0.1511	0.030*

## supplementary materials

---

C7	0.7425 (3)	0.4433 (3)	0.24678 (18)	0.0219 (6)
H7A	0.6798	0.3616	0.2397	0.026*
C8	0.5176 (3)	0.5018 (3)	0.20965 (18)	0.0213 (6)
H8A	0.4585	0.5690	0.2264	0.026*
H8B	0.4865	0.4267	0.2341	0.026*
C9	0.5073 (3)	0.4610 (3)	0.11317 (18)	0.0229 (6)
H9A	0.4157	0.4169	0.0949	0.028*
H9B	0.5186	0.5399	0.0896	0.028*
C10	0.6094 (3)	0.3717 (3)	0.07546 (19)	0.0238 (7)
H10A	0.5858	0.3437	0.0150	0.029*
H10B	0.6010	0.2939	0.1003	0.029*
C11	0.7593 (3)	0.4353 (3)	0.08922 (18)	0.0228 (6)
H11A	0.7609	0.5272	0.0861	0.027*
H11B	0.8080	0.3946	0.0423	0.027*
C12	0.8092 (3)	0.4744 (3)	0.33864 (19)	0.0249 (7)
H12A	0.7685	0.4139	0.3719	0.030*
H12B	0.9071	0.4705	0.3393	0.030*
C13	0.7780 (3)	0.6141 (3)	0.37195 (19)	0.0239 (7)
H13A	0.8452	0.6775	0.3535	0.029*
C14	0.7717 (3)	0.6482 (3)	0.46735 (19)	0.0254 (7)
C15	0.8925 (3)	0.6960 (3)	0.51785 (19)	0.0275 (7)
H15A	0.9748	0.7075	0.4926	0.033*
C16	0.8910 (3)	0.7266 (3)	0.60540 (19)	0.0271 (7)
H16A	0.9722	0.7578	0.6387	0.033*
C17	0.7693 (4)	0.7109 (3)	0.6432 (2)	0.0324 (8)
H17A	0.7683	0.7323	0.7020	0.039*
C18	0.6486 (4)	0.6633 (4)	0.5938 (2)	0.0414 (9)
H18A	0.5666	0.6529	0.6194	0.050*
C19	0.6496 (3)	0.6311 (4)	0.5058 (2)	0.0333 (8)
H19A	0.5685	0.5981	0.4728	0.040*
O'	0.8141 (2)	0.0639 (2)	0.33601 (12)	0.0231 (5)
N'	0.7585 (2)	0.0078 (2)	0.24822 (14)	0.0186 (5)
C1'	0.5197 (3)	-0.0548 (3)	0.18622 (17)	0.0159 (6)
C2'	0.4734 (3)	0.0835 (3)	0.20648 (17)	0.0172 (6)
H2'A	0.4426	0.0989	0.2634	0.021*
H2'B	0.5512	0.1487	0.2051	0.021*
C3'	0.3578 (3)	0.1009 (3)	0.14386 (18)	0.0210 (6)
H3'A	0.3322	0.1888	0.1592	0.025*
H3'B	0.3892	0.0898	0.0870	0.025*
C4'	0.2331 (3)	-0.0004 (3)	0.1454 (2)	0.0250 (7)
H4'A	0.1980	0.0148	0.2012	0.030*
H4'B	0.1614	0.0096	0.1045	0.030*
C5'	0.2713 (3)	-0.1402 (3)	0.1242 (2)	0.0234 (6)
H5'A	0.2940	-0.1593	0.0655	0.028*
H5'B	0.1930	-0.2019	0.1304	0.028*
C6'	0.3936 (3)	-0.1592 (3)	0.18186 (19)	0.0190 (6)
H6'A	0.4205	-0.2454	0.1615	0.023*
H6'B	0.3644	-0.1569	0.2387	0.023*
C7'	0.6255 (3)	-0.0735 (3)	0.25610 (17)	0.0161 (6)

H7'A	0.6404	-0.1661	0.2461	0.019*
C8'	0.8619 (3)	-0.0742 (3)	0.21325 (19)	0.0227 (6)
H8'A	0.8582	-0.1516	0.2384	0.027*
H8'B	0.9525	-0.0253	0.2272	0.027*
C9'	0.8360 (3)	-0.1158 (3)	0.11659 (18)	0.0221 (6)
H9'A	0.8963	-0.1805	0.0965	0.027*
H9'B	0.8611	-0.0398	0.0916	0.027*
C10'	0.6885 (3)	-0.1737 (3)	0.08409 (18)	0.0196 (6)
H10C	0.6601	-0.2454	0.1122	0.024*
H10D	0.6863	-0.2095	0.0237	0.024*
C11'	0.5856 (3)	-0.0725 (3)	0.09918 (17)	0.0181 (6)
H11C	0.5124	-0.0976	0.0544	0.022*
H11D	0.6319	0.0121	0.0936	0.022*
C12'	0.5939 (3)	-0.0310 (3)	0.34989 (18)	0.0207 (6)
H12C	0.5004	-0.0109	0.3543	0.025*
H12D	0.6083	-0.0987	0.3818	0.025*
C13'	0.6976 (4)	0.0927 (4)	0.3809 (2)	0.0321 (8)
H13B	0.6620	0.1689	0.3646	0.039*
C14'	0.7431 (3)	0.1240 (3)	0.47534 (19)	0.0287 (7)
C15'	0.8144 (4)	0.0435 (4)	0.5145 (2)	0.0370 (8)
H15B	0.8305	-0.0371	0.4832	0.044*
C16'	0.8617 (4)	0.0779 (4)	0.5967 (3)	0.0420 (9)
H16B	0.9115	0.0222	0.6209	0.050*
C17'	0.8369 (4)	0.1928 (4)	0.6440 (2)	0.0367 (9)
H17B	0.8691	0.2142	0.7009	0.044*
C18'	0.7657 (4)	0.2789 (4)	0.6110 (2)	0.0399 (9)
H18B	0.7493	0.3575	0.6449	0.048*
C19'	0.7168 (4)	0.2452 (4)	0.5227 (2)	0.0374 (9)
H19B	0.6693	0.3018	0.4979	0.045*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O	0.0293 (12)	0.0325 (12)	0.0177 (11)	0.0086 (10)	0.0025 (9)	-0.0005 (9)
N	0.0224 (13)	0.0262 (14)	0.0178 (12)	0.0041 (10)	0.0022 (10)	0.0001 (10)
C1	0.0192 (14)	0.0138 (13)	0.0145 (13)	0.0037 (11)	0.0001 (11)	0.0005 (10)
C2	0.0219 (15)	0.0196 (15)	0.0301 (17)	-0.0010 (12)	-0.0013 (13)	0.0046 (12)
C3	0.0240 (16)	0.0380 (19)	0.0284 (17)	-0.0063 (14)	-0.0009 (13)	0.0120 (14)
C4	0.0202 (16)	0.042 (2)	0.0372 (19)	0.0034 (14)	0.0091 (14)	-0.0053 (16)
C5	0.0310 (18)	0.0240 (17)	0.040 (2)	0.0096 (14)	0.0065 (15)	0.0000 (14)
C6	0.0241 (16)	0.0220 (16)	0.0305 (17)	0.0053 (12)	0.0016 (13)	0.0066 (13)
C7	0.0270 (16)	0.0177 (14)	0.0201 (15)	0.0011 (12)	0.0006 (12)	0.0024 (11)
C8	0.0174 (14)	0.0245 (15)	0.0220 (15)	0.0021 (12)	0.0010 (12)	0.0048 (12)
C9	0.0191 (15)	0.0273 (16)	0.0207 (15)	-0.0011 (12)	-0.0024 (12)	0.0041 (12)
C10	0.0263 (16)	0.0249 (16)	0.0183 (15)	0.0028 (13)	-0.0030 (12)	0.0007 (12)
C11	0.0240 (16)	0.0260 (16)	0.0176 (14)	0.0020 (12)	0.0030 (12)	0.0024 (12)
C12	0.0262 (16)	0.0267 (16)	0.0215 (15)	0.0017 (13)	-0.0001 (12)	0.0057 (12)
C13	0.0226 (15)	0.0272 (16)	0.0211 (15)	0.0014 (13)	0.0016 (12)	0.0037 (12)

## supplementary materials

---

C14	0.0248 (16)	0.0300 (17)	0.0189 (15)	0.0024 (13)	0.0021 (12)	-0.0007 (13)
C15	0.0239 (16)	0.0377 (19)	0.0204 (15)	0.0019 (14)	0.0027 (12)	0.0052 (13)
C16	0.0330 (18)	0.0259 (16)	0.0204 (15)	0.0001 (13)	-0.0059 (13)	0.0034 (12)
C17	0.043 (2)	0.0352 (19)	0.0162 (15)	0.0062 (15)	0.0028 (14)	-0.0023 (13)
C18	0.0274 (18)	0.068 (3)	0.0247 (18)	0.0012 (17)	0.0085 (14)	0.0000 (17)
C19	0.0243 (17)	0.050 (2)	0.0194 (16)	-0.0016 (15)	0.0021 (13)	-0.0039 (15)
O'	0.0212 (11)	0.0300 (12)	0.0135 (10)	-0.0037 (9)	-0.0021 (8)	-0.0023 (8)
N'	0.0180 (12)	0.0218 (13)	0.0128 (11)	-0.0035 (10)	-0.0024 (9)	0.0001 (9)
C1'	0.0177 (14)	0.0144 (13)	0.0136 (13)	-0.0011 (11)	0.0004 (11)	0.0002 (10)
C2'	0.0204 (14)	0.0150 (14)	0.0152 (13)	0.0010 (11)	0.0024 (11)	0.0013 (10)
C3'	0.0253 (15)	0.0190 (15)	0.0193 (14)	0.0073 (12)	0.0020 (12)	0.0027 (11)
C4'	0.0186 (15)	0.0265 (16)	0.0295 (17)	0.0065 (12)	-0.0008 (12)	0.0030 (13)
C5'	0.0167 (14)	0.0229 (16)	0.0276 (16)	-0.0021 (12)	-0.0034 (12)	0.0018 (12)
C6'	0.0171 (14)	0.0162 (14)	0.0227 (15)	-0.0008 (11)	0.0004 (11)	0.0032 (11)
C7'	0.0164 (14)	0.0135 (13)	0.0179 (14)	-0.0001 (10)	0.0021 (11)	0.0030 (10)
C8'	0.0166 (14)	0.0283 (16)	0.0217 (15)	0.0027 (12)	0.0007 (12)	0.0018 (12)
C9'	0.0187 (15)	0.0252 (16)	0.0213 (15)	0.0013 (12)	0.0026 (12)	0.0021 (12)
C10'	0.0199 (14)	0.0213 (15)	0.0160 (14)	0.0035 (12)	0.0017 (11)	-0.0009 (11)
C11'	0.0174 (14)	0.0209 (15)	0.0149 (13)	0.0030 (11)	0.0007 (11)	0.0009 (11)
C12'	0.0222 (15)	0.0231 (15)	0.0154 (14)	0.0000 (12)	0.0004 (11)	0.0026 (11)
C13'	0.0323 (18)	0.0367 (19)	0.0263 (17)	0.0035 (15)	0.0017 (14)	0.0043 (14)
C14'	0.0202 (15)	0.044 (2)	0.0161 (15)	-0.0059 (14)	-0.0006 (12)	-0.0018 (13)
C15'	0.035 (2)	0.0290 (18)	0.047 (2)	0.0034 (15)	-0.0019 (16)	0.0073 (16)
C16'	0.041 (2)	0.043 (2)	0.043 (2)	-0.0023 (17)	-0.0005 (17)	0.0170 (18)
C17'	0.0293 (18)	0.058 (2)	0.0209 (17)	-0.0085 (17)	-0.0013 (14)	0.0122 (16)
C18'	0.035 (2)	0.0308 (19)	0.045 (2)	-0.0044 (15)	0.0185 (17)	-0.0152 (16)
C19'	0.0246 (17)	0.045 (2)	0.050 (2)	0.0079 (15)	0.0074 (16)	0.0244 (18)

### *Geometric parameters (Å, °)*

O—C13	1.431 (4)	O'—C13'	1.417 (4)
O—N	1.480 (3)	O'—N'	1.474 (3)
N—C8	1.466 (4)	N'—C8'	1.469 (4)
N—C7	1.493 (4)	N'—C7'	1.503 (3)
C1—C2	1.533 (4)	C1'—C2'	1.546 (4)
C1—C6	1.545 (4)	C1'—C6'	1.547 (4)
C1—C11	1.554 (4)	C1'—C7'	1.551 (4)
C1—C7	1.561 (4)	C1'—C11'	1.561 (4)
C2—C3	1.529 (4)	C2'—C3'	1.533 (4)
C2—H2A	0.970	C2'—H2'A	0.970
C2—H2B	0.970	C2'—H2'B	0.970
C3—C4	1.529 (5)	C3'—C4'	1.532 (4)
C3—H3A	0.970	C3'—H3'A	0.970
C3—H3B	0.970	C3'—H3'B	0.970
C4—C5	1.529 (5)	C4'—C5'	1.530 (4)
C4—H4A	0.970	C4'—H4'A	0.970
C4—H4B	0.970	C4'—H4'B	0.970
C5—C6	1.537 (4)	C5'—C6'	1.538 (4)
C5—H5A	0.970	C5'—H5'A	0.970



C5—H5B	0.970	C5'—H5'B	0.970
C6—H6A	0.970	C6'—H6'A	0.970
C6—H6B	0.970	C6'—H6'B	0.970
C7—C12	1.547 (4)	C7'—C12'	1.547 (4)
C7—H7A	0.980	C7'—H7'A	0.980
C8—C9	1.525 (4)	C8'—C9'	1.533 (4)
C8—H8A	0.970	C8'—H8'A	0.970
C8—H8B	0.970	C8'—H8'B	0.970
C9—C10	1.519 (4)	C9'—C10'	1.535 (4)
C9—H9A	0.970	C9'—H9'A	0.970
C9—H9B	0.970	C9'—H9'B	0.970
C10—C11	1.536 (4)	C10'—C11'	1.542 (4)
C10—H10A	0.970	C10'—H10C	0.970
C10—H10B	0.970	C10'—H10D	0.970
C11—H11A	0.970	C11'—H11C	0.970
C11—H11B	0.970	C11'—H11D	0.970
C12—C13	1.527 (4)	C12'—C13'	1.539 (4)
C12—H12A	0.970	C12'—H12C	0.970
C12—H12B	0.970	C12'—H12D	0.970
C13—C14	1.514 (4)	C13'—C14'	1.525 (4)
C13—H13A	0.980	C13'—H13B	0.980
C14—C19	1.390 (4)	C14'—C15'	1.376 (5)
C14—C15	1.393 (4)	C14'—C19'	1.407 (5)
C15—C16	1.385 (4)	C15'—C16'	1.348 (5)
C15—H15A	0.930	C15'—H15B	0.930
C16—C17	1.379 (5)	C16'—C17'	1.348 (5)
C16—H16A	0.930	C16'—H16B	0.930
C17—C18	1.383 (5)	C17'—C18'	1.370 (6)
C17—H17A	0.930	C17'—H17B	0.930
C18—C19	1.392 (5)	C18'—C19'	1.441 (5)
C18—H18A	0.930	C18'—H18B	0.930
C19—H19A	0.930	C19'—H19B	0.930
C13—O—N	103.2 (2)	C13'—O'—N'	104.6 (2)
C8—N—O	103.8 (2)	C8'—N'—O'	103.8 (2)
C8—N—C7	111.2 (2)	C8'—N'—C7'	111.8 (2)
O—N—C7	105.4 (2)	O'—N'—C7'	105.51 (19)
C2—C1—C6	109.4 (2)	C2'—C1'—C6'	109.1 (2)
C2—C1—C11	109.5 (2)	C2'—C1'—C7'	111.1 (2)
C6—C1—C11	110.1 (2)	C6'—C1'—C7'	108.3 (2)
C2—C1—C7	112.5 (2)	C2'—C1'—C11'	108.8 (2)
C6—C1—C7	106.4 (2)	C6'—C1'—C11'	110.1 (2)
C11—C1—C7	108.9 (2)	C7'—C1'—C11'	109.4 (2)
C3—C2—C1	112.9 (3)	C3'—C2'—C1'	112.7 (2)
C3—C2—H2A	109.0	C3'—C2'—H2'A	109.1
C1—C2—H2A	109.0	C1'—C2'—H2'A	109.1
C3—C2—H2B	109.0	C3'—C2'—H2'B	109.1
C1—C2—H2B	109.0	C1'—C2'—H2'B	109.1
H2A—C2—H2B	107.8	H2'A—C2'—H2'B	107.8
C4—C3—C2	111.2 (3)	C4'—C3'—C2'	110.0 (2)

## supplementary materials

---

C4—C3—H3A	109.4	C4'—C3'—H3'A	109.7
C2—C3—H3A	109.4	C2'—C3'—H3'A	109.7
C4—C3—H3B	109.4	C4'—C3'—H3'B	109.7
C2—C3—H3B	109.4	C2'—C3'—H3'B	109.7
H3A—C3—H3B	108.0	H3'A—C3'—H3'B	108.2
C3—C4—C5	111.5 (3)	C5'—C4'—C3'	110.9 (2)
C3—C4—H4A	109.3	C5'—C4'—H4'A	109.5
C5—C4—H4A	109.3	C3'—C4'—H4'A	109.5
C3—C4—H4B	109.3	C5'—C4'—H4'B	109.5
C5—C4—H4B	109.3	C3'—C4'—H4'B	109.5
H4A—C4—H4B	108.0	H4'A—C4'—H4'B	108.1
C4—C5—C6	111.4 (3)	C4'—C5'—C6'	112.1 (2)
C4—C5—H5A	109.3	C4'—C5'—H5'A	109.2
C6—C5—H5A	109.3	C6'—C5'—H5'A	109.2
C4—C5—H5B	109.3	C4'—C5'—H5'B	109.2
C6—C5—H5B	109.3	C6'—C5'—H5'B	109.2
H5A—C5—H5B	108.0	H5'A—C5'—H5'B	107.9
C5—C6—C1	112.9 (3)	C5'—C6'—C1'	114.1 (2)
C5—C6—H6A	109.0	C5'—C6'—H6'A	108.7
C1—C6—H6A	109.0	C1'—C6'—H6'A	108.7
C5—C6—H6B	109.0	C5'—C6'—H6'B	108.7
C1—C6—H6B	109.0	C1'—C6'—H6'B	108.7
H6A—C6—H6B	107.8	H6'A—C6'—H6'B	107.6
N—C7—C12	104.7 (2)	N'—C7'—C12'	104.8 (2)
N—C7—C1	110.6 (2)	N'—C7'—C1'	108.3 (2)
C12—C7—C1	117.3 (2)	C12'—C7'—C1'	118.0 (2)
N—C7—H7A	108.0	N'—C7'—H7'A	108.5
C12—C7—H7A	108.0	C12'—C7'—H7'A	108.5
C1—C7—H7A	108.0	C1'—C7'—H7'A	108.5
N—C8—C9	111.0 (2)	N'—C8'—C9'	110.4 (2)
N—C8—H8A	109.4	N'—C8'—H8'A	109.6
C9—C8—H8A	109.4	C9'—C8'—H8'A	109.6
N—C8—H8B	109.4	N'—C8'—H8'B	109.6
C9—C8—H8B	109.4	C9'—C8'—H8'B	109.6
H8A—C8—H8B	108.0	H8'A—C8'—H8'B	108.1
C10—C9—C8	116.2 (3)	C8'—C9'—C10'	115.6 (2)
C10—C9—H9A	108.2	C8'—C9'—H9'A	108.4
C8—C9—H9A	108.2	C10'—C9'—H9'A	108.4
C10—C9—H9B	108.2	C8'—C9'—H9'B	108.4
C8—C9—H9B	108.2	C10'—C9'—H9'B	108.4
H9A—C9—H9B	107.4	H9'A—C9'—H9'B	107.5
C9—C10—C11	114.3 (2)	C9'—C10'—C11'	113.3 (2)
C9—C10—H10A	108.7	C9'—C10'—H10C	108.9
C11—C10—H10A	108.7	C11'—C10'—H10C	108.9
C9—C10—H10B	108.7	C9'—C10'—H10D	108.9
C11—C10—H10B	108.7	C11'—C10'—H10D	108.9
H10A—C10—H10B	107.6	H10C—C10'—H10D	107.7
C10—C11—C1	117.6 (2)	C10'—C11'—C1'	116.6 (2)
C10—C11—H11A	107.9	C10'—C11'—H11C	108.1

C1—C11—H11A	107.9	C1'—C11'—H11C	108.1
C10—C11—H11B	107.9	C10'—C11'—H11D	108.1
C1—C11—H11B	107.9	C1'—C11'—H11D	108.1
H11A—C11—H11B	107.2	H11C—C11'—H11D	107.3
C13—C12—C7	102.9 (2)	C13'—C12'—C7'	102.6 (2)
C13—C12—H12A	111.2	C13'—C12'—H12C	111.3
C7—C12—H12A	111.2	C7'—C12'—H12C	111.3
C13—C12—H12B	111.2	C13'—C12'—H12D	111.3
C7—C12—H12B	111.2	C7'—C12'—H12D	111.3
H12A—C12—H12B	109.1	H12C—C12'—H12D	109.2
O—C13—C14	109.4 (2)	O'—C13'—C14'	107.4 (3)
O—C13—C12	103.1 (2)	O'—C13'—C12'	103.4 (3)
C14—C13—C12	114.1 (3)	C14'—C13'—C12'	116.3 (3)
O—C13—H13A	110.0	O'—C13'—H13B	109.8
C14—C13—H13A	110.0	C14'—C13'—H13B	109.8
C12—C13—H13A	110.0	C12'—C13'—H13B	109.8
C19—C14—C15	119.2 (3)	C15'—C14'—C19'	119.1 (3)
C19—C14—C13	121.9 (3)	C15'—C14'—C13'	123.3 (3)
C15—C14—C13	118.8 (3)	C19'—C14'—C13'	117.4 (3)
C16—C15—C14	120.4 (3)	C16'—C15'—C14'	122.0 (4)
C16—C15—H15A	119.8	C16'—C15'—H15B	119.0
C14—C15—H15A	119.8	C14'—C15'—H15B	119.0
C17—C16—C15	120.1 (3)	C17'—C16'—C15'	120.2 (4)
C17—C16—H16A	119.9	C17'—C16'—H16B	119.9
C15—C16—H16A	119.9	C15'—C16'—H16B	119.9
C16—C17—C18	120.0 (3)	C16'—C17'—C18'	122.1 (3)
C16—C17—H17A	120.0	C16'—C17'—H17B	118.9
C18—C17—H17A	120.0	C18'—C17'—H17B	118.9
C17—C18—C19	120.2 (3)	C17'—C18'—C19'	118.6 (3)
C17—C18—H18A	119.9	C17'—C18'—H18B	120.7
C19—C18—H18A	119.9	C19'—C18'—H18B	120.7
C14—C19—C18	120.0 (3)	C14'—C19'—C18'	117.9 (3)
C14—C19—H19A	120.0	C14'—C19'—H19B	121.0
C18—C19—H19A	120.0	C18'—C19'—H19B	121.0

Fig. 1

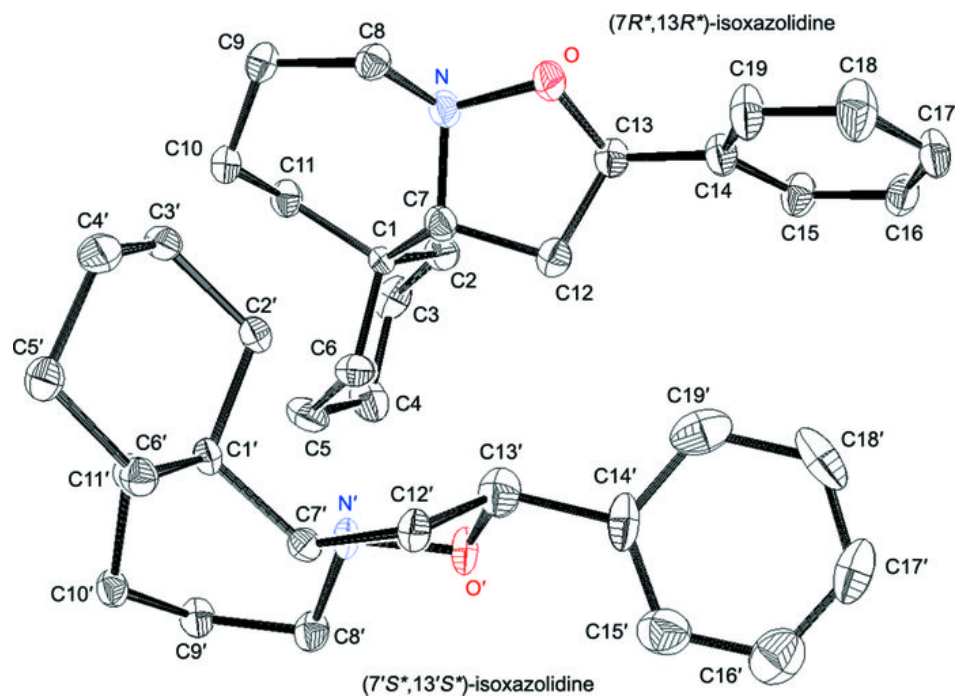


Fig. 2

