

## References

- Blessing, R. H. (1986). *Acta Cryst.* B42, 613–621.  
 Boullé, A. (1938). *C. R. Acad. Sci.* 206, 517–519.  
 Brown, I. D. (1976). *Acta Cryst.* A32, 24–31.  
 Durif, A. (1995). In *Crystal Chemistry of Condensed Phosphates*. New York: Plenum.  
 Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.  
 Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.  
 Gharbi, A., Jouini, A. & Durif, A. (1995). *J. Solid State Chem.* 114, 42–51.  
 Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

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## A Dihydrooxazinone

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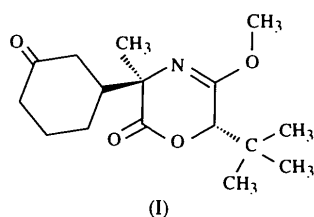
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## Abstract

(3*RS*,6*SR*,1'*RS*)-6-*tert*-Butyl-3,6-dihydro-5-methoxy-3-methyl-3-(3-oxocyclohexyl)-2*H*-1,4-oxazin-2-one, C<sub>16</sub>H<sub>25</sub>NO<sub>4</sub>, is an intermediate in the stereoselective synthesis of  $\alpha$ -methylated amino acids. Its structure agrees well with those of other dihydrooxazinones. The heterocyclic ring is almost planar while the side chains exhibit the expected staggered conformations.

## Comment

The stereospecific synthesis of dihydrooxazinones is a versatile synthetic route to enantiomerically pure  $\alpha$ -methylated amino acids formed upon hydrolysis (Maywald, 1987). In order to study the influence of the size and nature of the substituents on the conformation of the heterocyclic ring, several dihydrooxazinones with an isopropyl group at C(6) have been investigated (Bolte, 1995). We have determined the structure of a racemic derivative, (I), with a tertiary butyl group replacing the isopropyl moiety.



The bond lengths and angles agree well with the other dihydrooxazinones studied so far (Bolte, 1995). In contrast to lactides (1,4-dioxane-2,5-diones) in which N(4) is replaced by an O atom (Bolte, Beck, Nieger & Egert, 1994), the heterocyclic ring is almost planar ( $\sigma = 0.04 \text{ \AA}$ ). The cyclohexanone ring adopts a chair conformation and is oriented such that the five-atom chain from C(2) to C(5') is fully extended.

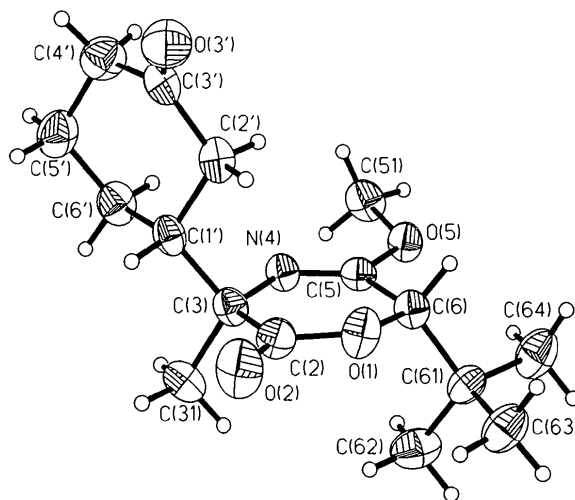


Fig. 1. Molecular structure of (I) showing 40% probability displacement ellipsoids.

## Experimental

The compound was provided by Professor U. Schöllkopf (University of Göttingen) and recrystallized from cyclohexane.

## Crystal data

C<sub>16</sub>H<sub>25</sub>NO<sub>4</sub>  
*M<sub>r</sub>* = 295.4  
 Triclinic  
*P* $\bar{1}$   
*a* = 6.806 (1) Å  
*b* = 8.166 (1) Å  
*c* = 15.568 (2) Å  
 $\alpha$  = 100.69 (1)°  
 $\beta$  = 94.99 (2)°  
 $\gamma$  = 103.29 (1)°  
*V* = 819.8 Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.197 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K* $\alpha$  radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 50 reflections  
 $\theta$  = 10–12.5°  
 $\mu$  = 0.08 mm<sup>-1</sup>  
*T* = 293 K  
 Block  
 0.6 × 0.4 × 0.1 mm  
 Colourless

## Data collection

Stoe–Siemens four-circle diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 3763 measured reflections  
 2882 independent reflections  
 1947 observed reflections  
 $[F > 3\sigma(F)]$

*R*<sub>int</sub> = 0.025  
 $\theta_{\text{max}}$  = 25°  
*h* = -8 → 7  
*k* = -9 → 9  
*l* = -4 → 18  
 3 standard reflections monitored every 100 reflections  
 intensity decay: none

## Refinement

Refinement on <i>F</i>	$(\Delta/\sigma)_{\max} = 0.008$
<i>R</i> = 0.067	$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$
<i>wR</i> = 0.057	$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$
<i>S</i> = 1.58	Extinction correction: none
1941 reflections	Atomic scattering factors
206 parameters	from <i>International Tables</i>
H atoms riding, C—H	for <i>X-ray Crystallography</i>
0.96 Å	(1974, Vol. IV)
$w = 1/[\sigma^2(F) + 0.0003F^2]$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
O(1)	0.7960 (2)	0.8109 (2)	0.7747 (1)	0.064 (1)
C(2)	0.6957 (4)	0.9153 (3)	0.7439 (2)	0.056 (1)
C(3)	0.4657 (4)	0.8623 (3)	0.7239 (2)	0.050 (1)
N(4)	0.3689 (3)	0.6942 (2)	0.7397 (1)	0.047 (1)
C(5)	0.4747 (4)	0.6086 (3)	0.7731 (2)	0.044 (1)
C(6)	0.7004 (4)	0.6564 (3)	0.8023 (2)	0.049 (1)
O(2)	0.7968 (3)	1.0483 (2)	0.7317 (1)	0.089 (1)
C(31)	0.3859 (4)	0.9994 (3)	0.7819 (2)	0.069 (1)
O(5)	0.3907 (3)	0.4502 (2)	0.7867 (1)	0.055 (1)
C(51)	0.1792 (4)	0.3774 (3)	0.7530 (2)	0.066 (1)
C(61)	0.7673 (4)	0.6789 (3)	0.9021 (2)	0.055 (1)
C(62)	0.6516 (5)	0.7914 (4)	0.9551 (2)	0.083 (1)
C(63)	0.9962 (4)	0.7644 (4)	0.9226 (2)	0.078 (1)
C(64)	0.7302 (5)	0.5020 (4)	0.9269 (2)	0.082 (1)
C(1')	0.4108 (3)	0.8588 (3)	0.6247 (2)	0.049 (1)
C(2')	0.5015 (4)	0.7292 (3)	0.5680 (2)	0.058 (1)
C(3')	0.4483 (4)	0.7120 (3)	0.4711 (2)	0.059 (1)
O(3')	0.5752 (3)	0.7141 (3)	0.4213 (1)	0.085 (1)
C(4')	0.2298 (4)	0.6912 (4)	0.4397 (2)	0.075 (1)
C(5')	0.1394 (4)	0.8192 (4)	0.4968 (2)	0.074 (1)
C(6')	0.1836 (4)	0.8210 (3)	0.5941 (2)	0.063 (1)

Table 2. Selected geometric parameters (Å, °)

O(1)—C(2)	1.336 (3)	O(1)—C(6)	1.439 (3)
C(2)—C(3)	1.513 (3)	C(3)—N(4)	1.455 (3)
N(4)—C(5)	1.256 (3)	C(5)—C(6)	1.502 (3)
C(2)—O(1)—C(6)	124.5 (2)	O(1)—C(2)—C(3)	120.2 (2)
C(2)—C(3)—N(4)	115.4 (2)	C(3)—N(4)—C(5)	120.0 (2)
N(4)—C(5)—C(6)	128.5 (2)	O(1)—C(6)—C(5)	110.3 (2)
C(6)—O(1)—C(2)—C(3)	-8.7 (3)	C(2)—O(1)—C(6)—C(5)	12.6 (3)
O(1)—C(2)—C(3)—N(4)	-0.5 (3)	C(2)—C(3)—N(4)—C(5)	4.3 (3)
C(3)—N(4)—C(5)—C(6)	0.8 (3)	N(4)—C(5)—C(6)—O(1)	-8.9 (3)

Six low-angle reflections suffered from extinction and were thus omitted. The methyl groups were refined as rigid groups in order to allow for internal rotation.

Data collection: profile fitting (Clegg, 1981). Cell refinement: program in Clegg (1981). Data reduction: *SHELXTL* (Sheldrick, 1983). Program(s) used to solve structure: *SHELXTL* using direct methods. Program(s) used to refine structure: *SHELXTL* using blocked-cascade least squares. Molecular graphics: *XP (SHELXTL)*. Software used to prepare material for publication: *SHELXTL*.

We thank Professor U. Schöllkopf (University of Göttingen, Germany) for kindly providing the sample.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1117). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Bolte, M. (1995). *Acta Cryst.* **C51**, 2587–2593.  
 Bolte, M., Beck, H., Nieger, M. & Egert, E. (1994). *Acta Cryst.* **C50**, 1717–1721.  
 Clegg, W. (1981). *Acta Cryst.* **A37**, 22–28.  
 Maywald, V. (1987). Dissertation, University of Göttingen, Germany.  
 Sheldrick, G. M. (1983). *SHELXTL Users Manual*. Revision 4. Nicolet XRD Corporation, Madison, Wisconsin, USA.

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## A Fully Cyclo-Substituted Cyclohexane

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## Abstract

1*r*,9*t*,16*t*-Trioxahexaspiro[2.0.3.0.2.0.3.0.2.0.3.0]heneicosane, C<sub>18</sub>H<sub>24</sub>O<sub>3</sub>, is mirror-symmetric with alternating three- and four-membered rings. Its structure agrees well with that of the parent hydrocarbon. Notable features are the strongly alternating bond angles within the cyclohexane ring and the differing bond lengths within the cyclobutane rings.

## Comment

The structure and dynamics of cyclohexane derivatives depend on the number and the size of the substituents. Some highly substituted compounds show unusually high barriers of ring inversion (Fitjer *et al.*, 1988). For example, hexaspiro[2.0.3.0.2.0.3.0.2.0.3.0]heneicosane, a fully cycloalkylated cyclohexane with alternating three- and four-membered rings, adopts a chair conformation in the crystal but shows considerable populations of twist-boat conformations in solution (Fitjer *et al.*, 1984). We have determined the structure of a derivative, (I), in which a methylene group is replaced by an O atom in each of the three-membered rings.

