# organic papers

# (4S)-tert-Butyl 4-(hydroxydiphenylmethyl)-2,2-dimethyloxazolidine-3-carboxylate

Yu-Xing Gao,[a] Wan-Yun Liu,[a] Yong-Gang Bai,[a] Ting-Ting Guo[b] and Yu-Fen Zhao[a]*

[a]Department of Chemistry, The Key Laboratory for Chemical Biology of Fujian Province, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen 361005, People's Republic of China, and [b]Department of Chemistry, College of Life Sciences, Xiamen University, Xiamen 361005, People's Republic of China

Correspondence e-mail: yfzhao@xmu.edu.cn

The title compound, $C_{23}H_{29}NO_4$, has been obtained by a Grignard reaction of 3-tert-butyl 4-methyl (4S)-2,2-dimethyl-oxazolidine-3,4-dicarboxylate with phenylmagnesium bromide. An intramolecular hydrogen bond occurs between the oxazole and hydroxy groups.

## Comment

The title compound, (I), is an important intermediate in the synthesis of non-protein $\alpha$-amino acids, which is widely used in pharmaceutical applications as well as in chiral starting materials and auxiliaries (Koskinen et al., 1995).



(I)
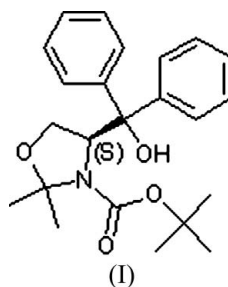
The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles in (I) are in agreement with those reported for similar compounds (Avenoza et al., 2003; Chen et al., 2006). The dihedral angle between the C12-phenyl and C18-phenyl planes is 65.6 (3)°. The oxazole displays an envelope configuration, with C5 atom lying at the flap position. Intramolecular hydrogen bonding is observed between the oxazole and adjacent hydroxy group; O4—H1D = 0.82 Å, O1···H1D = 2.10 Å, O1···O4 = 2.845 (4) Å and O4—H1D···O1 = 150°.
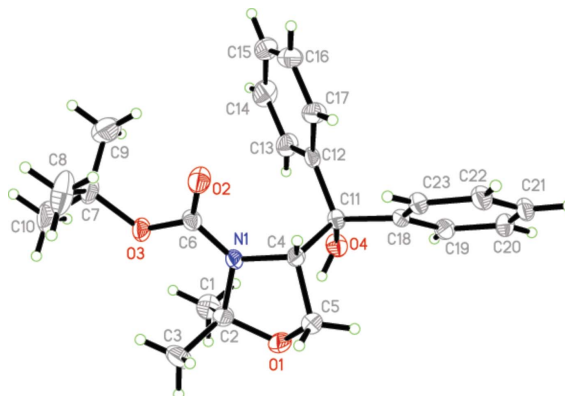


**Figure 1**
The molecular structure of (I), shown with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

## Experimental

To a precooled solution of 3-*tert*-butyl 4-methyl (4*S*)-2,2-dimethyl-oxazolidine-3,4-dicarboxylate (0.52 g, 2 mmol) in dry tetrahydrofuran (20 ml) at 273 K was added dropwise a 3 *M* solution of phenyl-magnesium bromide (4.7 ml, 14 mmol) in tetrahydrofuran. The cooling bath was removed and the mixture warmed to 308 K for 12 h. The reaction was quenched with a saturated $NH_4Cl$ solution (12 ml) and extracted with ethyl acetate. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, concentrated under vacuum and the crude product was purified by column chromatography (petro-leum ether–ethyl acetate, 20:1) to give the title compound as a white solid in 78% yield. Single crystals of (I) were obtained by slow evaporation of a petroleum ether/ethyl acetate solution (10:1 *v/v*).

### Crystal data

| | |
|---|---|
| $C_{23}H_{29}NO_4$ | $Z = 4$ |
| $M_r = 383.47$ | $D_x = 1.163$ Mg m$^{-3}$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation |
| $a = 6.167$ (2) Å | $\mu = 0.08$ mm$^{-1}$ |
| $b = 17.497$ (6) Å | $T = 273$ (2) K |
| $c = 20.288$ (7) Å | Chunk, colorless |
| $V = 2189.2$ (13) Å$^3$ | $0.48 \times 0.25 \times 0.11$ mm |

### Data collection

| | |
|---|---|
| Bruker APEX area-detector diffractometer | 11521 measured reflections |
| $\varphi$ and $\omega$ scans | 2480 independent reflections |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002) | 2254 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.955$, $T_{max} = 0.989$ | $R_{int} = 0.052$ |
| | $\theta_{max} = 26.0°$ |

### Refinement

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0764P)^2 + 0.0673P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.064$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.157$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.31$ | $\Delta\rho_{max} = 0.27$ e Å$^{-3}$ |
| 2480 reflections | $\Delta\rho_{min} = -0.24$ e Å$^{-3}$ |
| 253 parameters | |
| H-atom parameters constrained | |

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were placed in idealized positions, with C—H = 0.93 (aromatic), 0.98 (methine) or 0.97 Å (methylene) and O—H = 0.82 Å, and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration of (I) was assigned assuming that the absolute config-uration of the starting materials was retained during the synthesis.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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: *SHELXL97*.

## References

Avenoza, A., Busto, J. H., Cativiela, C., Peregrina, J. M., Sucunza, D. & Zurbano, M. M. (2003). *Tetrahedron Asymmetry*, **14**, 399–405.
Bruker (2001). *SAINT* (Version 6.22) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, W.-Z., Zeng, Q.-L., Fang, H., Gao, Y.-X. & Zhao, Y.-F. (2006). *Acta Cryst.* E**62**, o869–o870.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Koskinen, A. M. P., Haslla, H., Myllymäki, M. T. & Rissanen, K. (1995). *Tetrahedron Lett.* **36**, 5619–5622.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2002). *SADABS*. Version 2.03. University of Göttingen, Germany.