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tert-Butyl 2,2-dimethyl-4-(3-phenyl-2,5-dihydrofuran-2-yl)oxazolidine-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.057
Data-to-parameter ratio = 9.6

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

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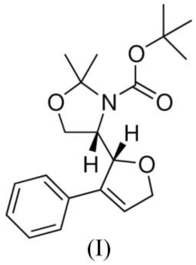
The title compound, $C_{20}H_{27}NO_4$, is a synthetic intermediate towards the preparation of novel furanomycin derivatives. Its structure is consistent with the assumed *anti* selectivity of the allenylation step.

Comment

The title compound, (I), is a synthetic intermediate in the construction of novel furanomycin derivatives using the gold-catalysed cyclization of α-hydroxyallenes (Hoffmann-Röder & Krause, 2001). The allene precursor is obtained by diastereoselective allenylation of Garner's Aldehyde (Garner & Park, 1987) with γ-phenylpropargyl bromide mediated by indium (Erdsack & Krause, 2007). A crystal structure determination of (I) has now been carried out to establish the relative configurations of the stereogenic centers at the dihydrofuranyl ring to prove the *anti* selectivity of the allenylation step. Fig. 1 shows that the relative configuration of C10 and C11 is as expected. The torsion angle C7—C10—C11—N1 is −178.30 (14)°. The configurations of the chiral C atoms in (I) (C10 *R* and C11 *S*) were assigned based on those of the equivalent atoms in the known starting materals.



(I)

Experimental

In a Schlenk tube with a magnetic stirrer bar was dissolved 420 mg (1.22 mmol) of *tert*-butyl 4-(1-hydroxy-2-phenyl-buta-2,3-dienyl)-2,2-dimethyloxazolidine-3-carboxylate (the preparation of this allene will be described elsewhere; Erdsack & Krause, 2007) in dry dichloromethane (6 ml) under argon. With vigorous stirring, $AuCl_3$ (370 µl) in acetonitrile (0.1645 *M* stock solution, 5 mol%) was added *via* a syringe at room temperature. After 15 min with stirring, the mixture was diluted with diethyl ether (35 ml) and an aqueous saturated sodium bicarbonate solution (50 ml). The organic phase was separated and the residue was extracted with ethyl acetate (3 × 30 ml). The combined organic extracts were washed with brine, dried ($MgSO_4$), filtered and the solvent was evaporated. Purification of the residue by column chromatography (isohexane/ethyl acetate 90:10 → 70:30) furnished 188 mg (45%) of the title compound as a colorless solid, along with 10% of a side product after acetal cleavage as an unstable oil. Single crystals of (I) were obtained by recrystallization from ethanol to yield colorless cubes: m.p. 436–438 K (EtOH), R_F = 0.54 (isohexane/ethyl acetate 85/15). IR (KBr pellet), cm^{-1}: 3012 (*w*), 2979 (*m*), 2952 (*m*), 2879 (*m*), 2837 (*m*), 1690 (*s*), 1393 (*s*), 1362 (*s*), 1083 (*s*), $[\alpha]_D^{22}$: +127.5 (*c* 0.2, CHCl$_3$). HRMS calculated for $C_{20}H_{27}NO_4$: $[M + H]^+$ = 346.2018; found 346.2043 $[M + H]^+$.

Crystal data

$C_{20}H_{27}NO_4$
$M_r = 345.43$
Monoclinic, $P2_1$
$a = 9.7530$ (6) Å
$b = 9.3216$ (8) Å
$c = 10.792$ (7) Å
$\beta = 110.462$ (4)°
$V = 919.2$ (6) Å^3
$Z = 2$
$D_x = 1.248$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 173$ (1) K
Block, colorless
0.50 × 0.50 × 0.45 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: none
11172 measured reflections
2232 independent reflections
1472 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\rm max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.057$
$S = 0.90$
2232 reflections
232 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0224P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.12$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.038 (2)

Due to negligible anomalous dispersion effects, Friedel pairs were merged prior to refinement. H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and were refined as riding, with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C) for methyl and $1.2U_{\rm eq}$(C) for all other H atoms; the methyl groups were allowed to rotate but not to tip.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

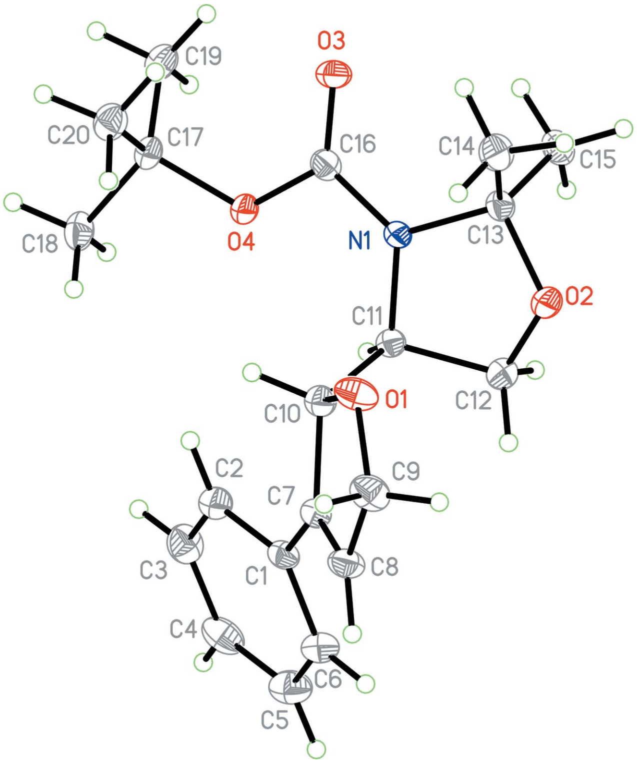


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level (arbitrary spheres for the H atoms).

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