A chiral oxazolidin-2-one-derived Reissert compound

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Key indicators

Single-crystal X-ray study

$T = 298$ K

Mean σ(C–C) = 0.004 Å

R factor = 0.035

wR factor = 0.085

Data-to-parameter ratio = 9.1

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

The title compound, (2S)-1-{[(4S)-4-benzyl-2-oxo-1,3-oxazolidin-3-yl]carbonyl}-1,2-dihydroquinoline-2-carbonitrile, $C_{21}H_{17}N_3O_3$, is the result of a diastereoselective Reissert-type reaction performed on a quinoline ring bearing a 4-benzyl oxazolidinone ring as chiral auxiliary. By means of X-ray analysis and according to the known S configuration at the C atom to which the benzyl group is attached, the S configuration at the C atom to which the nitrile group is attached was determined.

Comment

The Reissert reaction (Reissert, 1905) consists of the 1,2-addition reaction of a cyano group to an *N*-acyl quinolinium salt (Duarte & Popp, 1991; Akiba *et al.*, 1979; Abushanab & Lee, 1975). Recently, the first enantioselective Reissert reaction has been described by Takamura *et al.* (2001) and applied to a synthesis of the potent NMDA receptor antagonist (−)-L-689,560 implicated in several neurodegenerative disorders (Leeson *et al.*, 1992; Carling *et al.*, 1993).

(I)
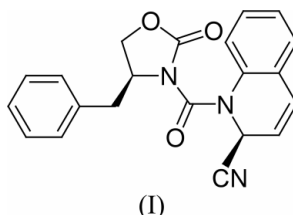
In the course of an ongoing project directed towards the asymmetric synthesis of alkaloids of biological interest, we have developed a new diastereoselective Reissert reaction using chiral oxazolidinones as chiral auxiliaries. Herein, we report the structure of a Reissert adduct, (I), isolated after reaction of trimethylsilyl cyanide with the *in situ* generated (4S)-1-(4-benzyl-2-oxo-oxazolidine-3-carbonyl)-quinolinium chloride. The absolute configuration of the newly created stereogenic carbon C1 could be deduced from the relative configurations of C atoms C1 and C14.

The two molecules molecules in the asymmetric unit are similar, as indicated by the following dihedral angles: 52.5 (3) and 50.6 (3)° for N1—C11—N2—C12, −15.3 (3) and −13.1 (4)° for C11—N2—C12—O3 and 13.5 (3) and 18.8 (3)° for C1—N1—C11—O1 (values for molecules *a* and *b*, respectively).

Experimental

To a solution of quinoline (1.00 mmol) and (4S)-4-benzyl-2-oxo-oxazolidine-3-carbonyl chloride (1.10 mmol) in anhydrous CH_2Cl_2 (2 ml) was added trimethylsilyl cyanide (2.00 mmol). The reaction

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mixture was then stirred at room temperature for 16 h. Water (3 ml) was added and the aqueous phase was extracted with CH_2Cl_2 (3 × 5 ml). The CH_2Cl_2 extracts were combined, washed with brine (10 ml), dried over anhydrous $MgSO_4$, filtered and concentrated. The crude mixture was flash-chromatographed on silica (eluent: CH_2Cl_2) to afford (I) (175 mg, 49%), along with its diastereomer (I*a*) (140 mg, 39%). Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation from CH_2Cl_2 at room temperature.

Crystal data

$C_{21}H_{17}N_3O_3$	$D_x = 1.294$ Mg m^{-3}
$M_r = 359.4$	Mo Kα radiation
Monoclinic, $P2_1$	Cell parameters from 70335
$a = 10.7330$ (5) Å	reflections
$b = 12.6853$ (5) Å	$\theta = 2.9$–32.0°
$c = 14.0824$ (5) Å	$\mu = 0.09$ mm^{-1}
$\beta = 105.875$ (3)°	$T = 298$ K
$V = 1844.21$ (13) Å^3	Block, colourless
$Z = 4$	0.46 × 0.26 × 0.23 mm

Data collection

|	
Nonius KappaCCD diffractometer	$R_{int} = 0.054$
φ and ω scans	$\theta_{max} = 32.0°$
68 549 measured reflections	$h = -16 \rightarrow 16$
4422 independent reflections	$k = -18 \rightarrow 18$
3918 reflections with $I > 2\sigma(I)$	$l = -20 \rightarrow 21$

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.14$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{min} = -0.12$ e Å^{-3}
$wR(F^2) = 0.085$	Extinction correction: B–C type 1
$S = 1.57$	Lorentzian isotropic (Becker &
4422 reflections	Coppens, 1974)
487 parameters	Extinction coefficient: 5.5 (5)
H-atom parameters constrained	Absolute structure: from known S
$w = 1/[\sigma^2(I) + 0.001296I^2]$	configuration at C14
$(\Delta/\sigma)_{max} = 0.001$	

As no atoms heavier than Si are present in the structure, Friedel-pair reflections were merged. All non-H atoms were refined with anisotropic atomic displacement parameters. H atoms were found initially in difference Fourier maps and then fixed at calculated positions. A riding isotropic displacement parameter was used for all H atoms.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995);
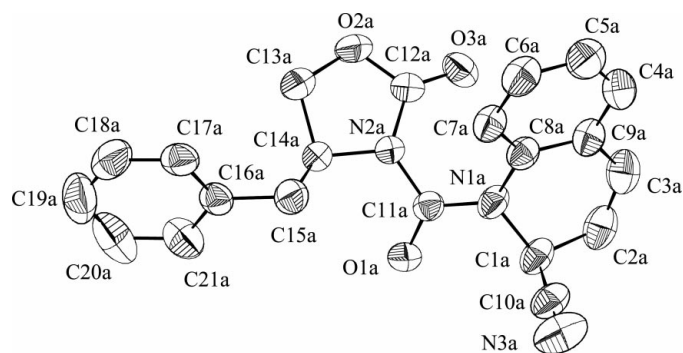
Figure 1
Molecular structure (molecule *a*), showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

program(s) used to refine structure: *JANA*2000 (Petricek & Dusek, 2000); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *JANA*2000.

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