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# (1*R*,2*R*)-1,3-Bis(*tert*-butyldimethylsilyloxy)-2-(4-hydroxybutanamido)-1-(4-nitrophenyl)-propane

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**Key indicators**

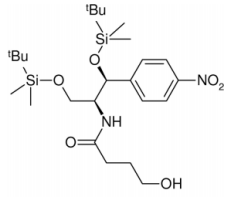
Single-crystal X-ray study
*T* = 180 K
Mean σ(C–C) = 0.012 Å
Disorder in main residue
*R* factor = 0.056
*wR* factor = 0.148
Data-to-parameter ratio = 6.3

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

The title compound, $C_{25}H_{46}N_2O_6Si_2$, is a new derivative of chloramphenicol. At 180 K, the structure has $Z' = 2$ in space group $P2_1$.

## Comment

Chloramphenicol is produced naturally by the soil bacterium *Streptomyces venezuealae* and is a widely used antibiotic in the treatment of eye infections (Long & Porse, 2003); it blocks the peptidyl transferase activity by hampering the binding of transfer RNA to the A-site in the ribosome (Schlünzen *et al.*, 2001). The synthesis of biologically active analogues of chloramphenicol has attracted chemists since preparation of the first examples in 1951 (Huebner & Scholtz, 1951). The title compound, (I), was synthesized as part of our effort to produce new chloramphenicol analogues. It is made in two steps from (1*R*,2*R*)-2-amino-1-(4-nitrophenyl)-1,3-propanediol in 66% yield. This is the first structural report of a chloroamphenicol derivative bearing a hydroxybutanoyl substituent on the amine N atom.




(I)

In space group $P2_1$, there are two independent molecules in the asymmetric unit. The conformations of these two molecules are largely comparable, but differ in the orientation of the hydroxybutanoyl substituents. In molecule *A*, the hydroxyl group O6*A* is brought into a position effectively *cis* to the carbonyl oxygen O5*A*. In molecule *B*, O6*B* lies effectively *trans* to O5*B*. The *tert*-butyldimethylsilane (TBDMS) groups containing Si2*A*/*B* also differ in their orientation: the torsion angles C5*A*—O3*A*—Si2*A*—C23*A* and C5*B*—O3*B*—Si2*B*—C23*B* are 174.7 (6) and 107.2 (6)°, respectively. Viewed in projection on to the plane of the benzene ring, the C4*A*—C5*A* bond in molecule *A* lies approximately parallel to one Si—$CH_3$ bond (Si2*A*—C18*A*) of the TBDMS group, while in molecule *B*, the C4*B*—C5*B* bond lies approximately parallel to the bisector of the C18*B*—Si2*B*—C19*B* angle. In both molecules, the *tert*-butyl group is staggered with respect to the $Si(CH_3)_2$ moiety.

## Experimental

Compound (I) was synthesized by reaction of γ-butyrolactone with (1*R*,2*R*)-amino-1-(4-nitrophenyl)-1,3-bis(*tert*-butyldimethylsilyloxy)-

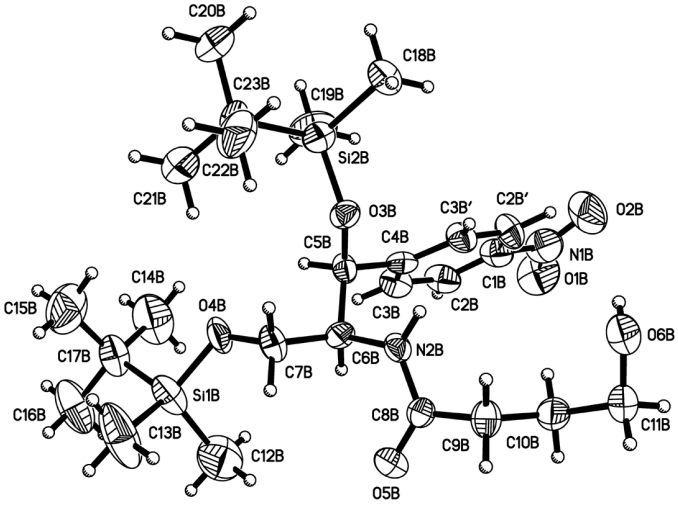



**Figure 1**
One symmetry-independent molecule in (I), showing displacement ellipsoids at the 50% probability level and H atoms drawn as spheres of arbitrary radius.

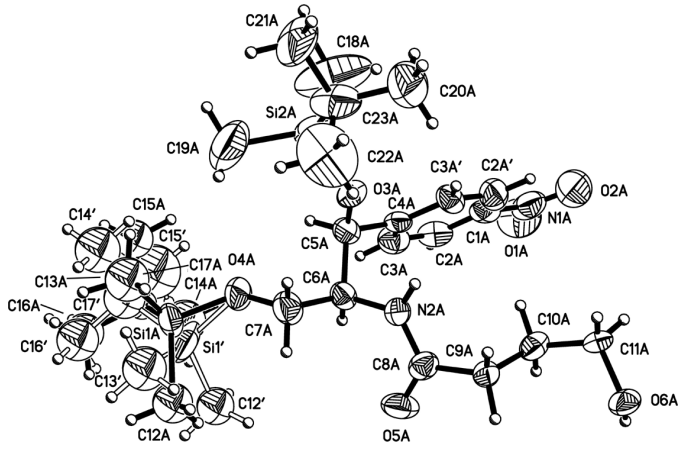



**Figure 2**
The second symmetry-independent molecule in (I), displaying disorder of one *tert*-butyldimethylsilane (TBDMS) group. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

propane. Colourless crystals suitable for X-ray analysis were obtained by slow evaporation of a hexane solution. The melting point of (I) is 359–361 K.

*Crystal data*

| | |
|---|---|
| $C_{25}H_{46}N_2O_6Si_2$ | $D_x$ = 1.135 Mg m$^{-3}$ |
| $M_r$ = 526.82 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1$ | Cell parameters from 2489 reflections |
| $a$ = 14.9014 (12) Å | $\theta$ = 2.4–18.4° |
| $b$ = 12.0241 (10) Å | $\mu$ = 0.15 mm$^{-1}$ |
| $c$ = 17.2103 (14) Å | $T$ = 180 (2) K |
| $\beta$ = 91.288 (2)° | Needle, colourless |
| $V$ = 3082.9 (4) Å$^3$ | 0.50 × 0.10 × 0.10 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker SMART 1000 CCD diffractometer | 3985 independent reflections |
| Thin-slice $\omega$ scans | 2691 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002 | $R_{int}$ = 0.076 |
| $T_{min}$ = 0.881, $T_{max}$ = 0.985 | $\theta_{max}$ = 22.0° |
| 12 674 measured reflections | $h = -15 \rightarrow 15$ |
| | $k = -12 \rightarrow 7$ |
| | $l = -18 \rightarrow 18$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0794P)^2 + 0.6178P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.056 | $(\Delta/\sigma)_{max} < 0.001$ |
| $wR(F^2)$ = 0.148 | $\Delta\rho_{max}$ = 0.33 e Å$^{-3}$ |
| $S$ = 1.03 | $\Delta\rho_{min}$ = −0.36 e Å$^{-3}$ |
| 3985 reflections | |
| 637 parameters | |
| H-atom parameters constrained | |

The diffraction data were relatively weak and were truncated to $\theta$ = 22° (0.95 Å resolution). As a result, the refined structure is of relatively low precision. The absolute configuration cannot be determined reliably and was assigned on the basis of the known chirality of the starting materials. Friedel opposites (1809 pairs) were merged in the final cycles of refinement. One *tert*-butyldimethylsilane (TBDMS) group is disordered and was modelled in two orientations. The geometries of these groups were restrained and the C atoms were refined with isotropic displacement parameters. H atoms bound to C atoms were positioned geometrically and allowed to ride during subsequent refinement, with $U_{iso}$(H) = 1.2$U_{eq}$(C) and distances of 0.95 Å for H atoms bound to benzene rings, 1.00 Å for H atoms bound to tertiary C atoms, 0.99 Å for H atoms of $CH_2$ groups, 0.88 Å for H atoms bound to N atoms, 0.84 Å for the H atom of the OH group and 0.98 Å for the H atoms of the methyl groups. In the last two cases, $U_{iso}$(H) = 1.5$U_{eq}$(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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