Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

3-*tert*-Butyl-5-{[5-(2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyl-tetrahydrofuro[2,3-*d*][1,3]-dioxol-6-yloxy]methyl}-2-hydroxybenzaldehyde

Shan-Shan Zhao, Ji-Quan Zhao* and Dong-Min Zhao

School of Chemical Engineering, Hebei University of Technology, Tianjin 300130, People's Republic of China

Correspondence e-mail:
zhaojiquan@jsmail.hebut.edu.cn

Received 17 April 2006
Accepted 23 May 2006

Key indicators

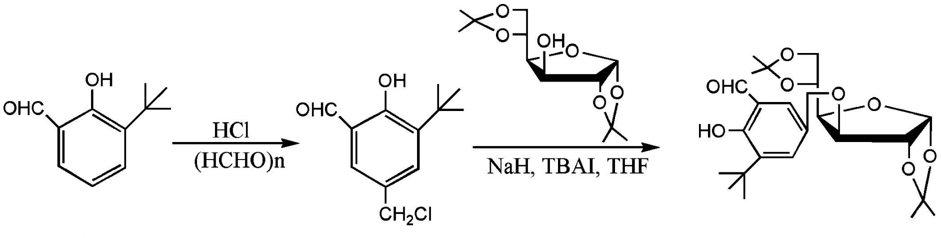
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.006 Å
R factor = 0.045
wR factor = 0.126
Data-to-parameter ratio = 8.8

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

In the title compound, $C_{24}H_{34}O_8$, a strong intramolecular O—H···O hydrogen bond is formed between the OH substituent of salicyaldehyde and the aldehyde O atom.

Comment

It is common practice to introduce chiral units into the building block of complexes as asymmetric catalysts (Yan & Klemm, 2002). Recently, we have focused our attention on the synthesis of a series of chiral Mn–salen complexes [salen is bis(salicylidene)ethylenediamine] with carbohydrates in order to test their application in the asymmetric epoxidation of unfunctionalized olefins. Thus, the title compound, (3), as one of the intermediates, was synthesized and its structure is presented here.



(1) (2) (3)

Compound (3) (Fig. 1) was obtained by the condensation of 3-*tert*-butyl-5-(chloromethyl)-2-hydroxybenzaldehyde and 1,2:5,6-di-*O*-isopropylidene-α-D-glucofuranose. The distance between the newly bonded atoms (C12 and O3) is 1.437 (4) Å. The other bond distances and angles are normal within experimental error (Allen *et al.*, 1987). There is a strong intramolecular hydrogen bond involving the OH group and the aldehyde O atom (Table 1), forming a nearly planar six-membered ring [maximum deviation 0.017 (4) Å for atom C11]. The crystal packing is mainly determined by van der Waals interactions.

Experimental

3-*tert*-Butyl-5-(chloromethyl)-2-hydroxybenzaldehyde, (2), was prepared in 87% yield by chloromethylation of 3-*tert*-butyl-2-hydroxybenzaldehyde, (1) (Kureshy *et al.*, 2002). Oil-free NaH (0.10 g, 4 mmol) was suspended in dry THF (10 ml) under nitrogen at 273 K (Tripathi *et al.*, 2005). A solution of 1,2:5,6-di-*O*-isopropylidene-α-D-glucofuranose (0.52 g, 2 mmol) in THF (10 ml) was added dropwise using a dropping funnel and the mixture was stirred for 3 h at room temperature. A solution of compound (2) (0.57 g, 2.5 mmol) in THF (10 ml) was then added very slowly at 273 K followed by addition of tetrabutylammonium iodide (TBAI) (0.02 g, 0.06 mmol) (Karche *et al.*, 2003). The reaction mixture was heated under reflux for 11 h. The solvent was evaporated *in vacuo* after destroying excess NaH by adding a few drops of water at 273 K. The resulting mass was extracted with $CHCl_3$ after removal of THF. The organic layer was washed with brine, dried over Na_2SO_4 and concentrated to afford a crude residue. The product (3) was purified by silica-gel column chromatography, eluting with a 1:3 EtOAc–petroleum ether mixture to afford a white solid (0.6 g, 67%). Colourless single crystals of (3) were grown by slow evaporation of a solution in petroleum ether and EtOAc (5:1 *v*/*v*). The structure of (3) was confirmed by melting point, optical rotation, elemental analysis, NMR and IR (m.p. 369–371 K). $[\alpha]_D^{20} = -34.1^\circ$ (EtOH). Analysis calculated for $C_{24}H_{34}O_8$: C 63.98, H 7.61%; found: C 63.71, H 7.83%.

Crystal data

$C_{24}H_{34}O_8$
$M_r = 450.51$
Monoclinic, $P2_1$
$a = 10.288$ (4) Å
$b = 9.992$ (3) Å
$c = 11.983$ (4) Å
$\beta = 101.552$ (6)°
$V = 1206.9$ (7) Å^3
$Z = 2$
$D_x = 1.240$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 294$ (2) K
Block, colourless
0.26 × 0.22 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.976$, $T_{max} = 0.985$
6896 measured reflections
2644 independent reflections
1831 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 26.6^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.126$
$S = 1.02$
2644 reflections
300 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 0.1782P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.92 (4)	1.77 (5)	2.609 (4)	150 (6)

The H atom of the hydroxy group was located in a difference Fourier map and refined freely, with an O—H distance restraint of 0.92 Å. All other H atoms were included in calculated positions, with C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$ for methyl H atoms. In the absence of significant anomalous scattering, Friedel pairs were merged prior to the final refinement; the absolute configuration is known from that of the glucofuranose reagent.

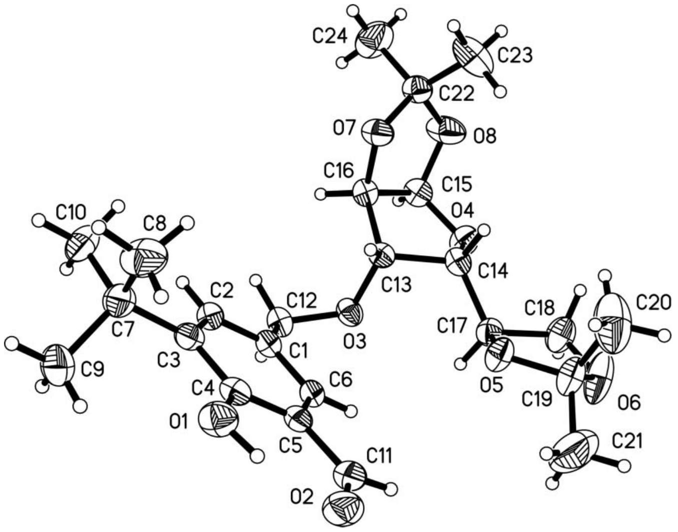


Figure 1
A view of the molecular structure of (3). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

This work was financially supported by the National Natural Science Foundation, China (grant No. 20376017).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Karche, N. P., Jachak, S. M. & Dhavale, D. D. (2003). *J. Org. Chem.* **68**, 4531–4534.
Kureshy, R. I., Khan, N. H., Abdi, S. H. R., Patel, S. T., Iyer, P. K., Subramanian, P. S. & Jasra, R. V. (2002). *J. Catal.* **209**, 99–104.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tripathi, S., Maity, J. K., Achari, B. & Mandal, S. B. (2005). *Carbohydr. Res.* **340**, 1081–1087.
Yan, S. & Klemm, D. (2002). *Tetrahedron*, **58**, 10065–10071.

o2538 Zhao *et al.* • $C_{24}H_{34}O_8$ *Acta Cryst.* (2006). E**62**, o2537–o2538