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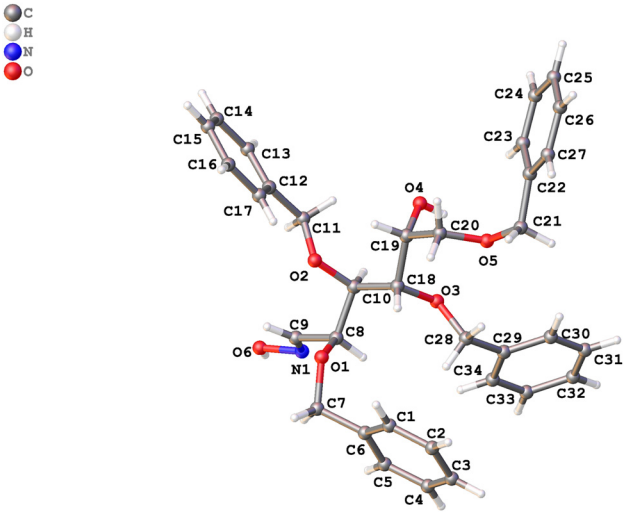
Crystal structure of (2*S*,3*S*,4*S*,5*S*, *Z*)-2,3,5,6-tetrakis(benzyloxy)-4-hydroxyhexanal oxime, $C_{34}H_{37}NO_6$



https://doi.org/10.1515/ncrs-2023-0174
Received April 10, 2023; accepted June 26, 2023;
published online September 15, 2023

Abstract

$C_{34}H_{37}NO_6$, triclinic, $P\bar{1}$ (no. 2), a = 6.398(6) Å, b = 10.779(10) Å, c = 12.030(11) Å, α = 71.103(10)°, β = 89.519(11)°, γ = 74.154(11)°, V = 752.2(12) Å^3, Z = 1, R_{gt}(F) = 0.0599, wR_{ref}(F^2) = 0.1614, T = 296 K.

CCDC no.: 2254652

Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

Dissolve 5.54 g of (2*R*,3*S*,4*S*,5*R*)-3,4,5-tris(benzyloxy)-2-((benzyloxy)methyl)-6-methoxytetrahydro-2*H*-pyran using 30 mL of acetonitrile [4–6], add 168 mL of 80 % acetic acid and 68 mL of 2N hydrochloric acid, react at 3535 K for 8 h. Concentrate, extract with dichloromethane and then combine to obtain 4.05 g of colorless crystals.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.19 × 0.18 × 0.16 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.084 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6937, 6172, 0.0598
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3228
$N(param)_{refined}$:	380
Programs:	Bruker programs [1], Olex2 [2] SHELX [3]

2 Experimental details

All hydrogen atoms were placed in the calculated positions.

3 Discussion

Rhodioloside, which is condensed from one molecule of glucose and one molecule of 2-(4-hydroxy)phenethyl alcohol, is the most representative active ingredient with anti-cancer, anti-aging, anti-inflammatory, neuroprotective and metabolic regulating effects [7–9], and has also been proven to play a therapeutic role in pulmonary fibrosis in recent years [10]. Altering the glycocyclic ring of rhodiroside may have an improving effect on its biological activity, so it is devoted to the synthesis of the drug intermediate cyclic nitrone, and the compound studied in the title is an important synthetic substrate for cyclic nitrone.

The molecule forming the title crystal structure is shown in the figure. Several important bond angle data are involved as follows C18–C10–C19 = 111.6 Å, O3–C8–C10 = 105.0 Å, O6–N1–C9 = 110.98 Å. The bond lengths and angles are in the expected ranges.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2400 (4)	0.7825 (3)	0.2749 (2)	0.0526 (6)
O2	0.3487 (4)	0.5132 (2)	0.2953 (2)	0.0472 (6)
O3	0.2824 (4)	0.5542 (3)	0.5791 (2)	0.0547 (6)
O4	0.5092 (6)	0.2994 (3)	0.5676 (3)	0.0771 (8)
H4A	0.443616	0.310082	0.624177	0.116*
O5	0.7415 (5)	0.3946 (3)	0.6990 (3)	0.0738 (9)
O6	−0.3843 (4)	0.7253 (4)	0.1683 (3)	0.0741 (9)
H6	−0.509452	0.729386	0.186082	0.111*
N1	−0.2597 (5)	0.7239 (4)	0.2618 (3)	0.0593 (8)
C1	0.3670 (7)	0.9574 (4)	0.4010 (4)	0.0610 (10)
H1	0.491193	0.917588	0.370184	0.073*
C2	0.3901 (8)	0.9998 (5)	0.4940 (4)	0.0744 (13)
H2	0.527978	0.989454	0.526168	0.089*
C3	0.2048 (10)	1.0585 (5)	0.5398 (5)	0.0809 (14)
H3	0.217209	1.087708	0.603605	0.097*
C4	0.0040 (10)	1.0735 (6)	0.4910 (6)	0.0881 (16)
H4	−0.120484	1.111766	0.522454	0.106*
C5	−0.0157 (8)	1.0328 (5)	0.3967 (5)	0.0719 (12)
H5	−0.153823	1.046666	0.362644	0.086*
C6	0.1645 (6)	0.9718 (4)	0.3513 (4)	0.0546 (9)
C7	0.1450 (7)	0.9267 (4)	0.2468 (4)	0.0633 (10)
H7A	−0.007793	0.950423	0.219937	0.076*
H7B	0.217153	0.975013	0.182875	0.076*
C8	0.1009 (5)	0.7017 (4)	0.3286 (3)	0.0482 (8)
H8A	0.026654	0.735502	0.389173	0.058*
C9	−0.0620 (6)	0.7084 (5)	0.2400 (4)	0.0611 (10)
H9	−0.008 (6)	0.699 (4)	0.172 (3)	0.037 (8)
C10	0.2440 (5)	0.5566 (4)	0.3859 (3)	0.0476 (8)
H10A	0.150657	0.498208	0.421186	0.057*
C11	0.3150 (6)	0.3931 (4)	0.2879 (4)	0.0561 (9)
H11A	0.165365	0.411913	0.257767	0.067*
H11B	0.337935	0.325059	0.36611	0.067*
C12	0.4659 (6)	0.3379 (4)	0.2092 (3)	0.0503 (8)
C13	0.4293 (8)	0.2342 (4)	0.1763 (4)	0.0670 (11)
H13	0.310555	0.201766	0.201653	0.08*
C14	0.5683 (12)	0.1783 (5)	0.1059 (4)	0.0892 (17)
H14	0.542361	0.10872	0.083301	0.107*
C15	0.7406 (11)	0.2238 (5)	0.0698 (4)	0.0932 (19)
H15	0.835425	0.184028	0.023807	0.112*
C16	0.7784 (10)	0.3278 (6)	0.0999 (5)	0.0901 (16)
H16	0.896847	0.360001	0.073233	0.108*
C17	0.6396 (7)	0.3852 (5)	0.1702 (4)	0.0654 (11)
H17	0.664789	0.456099	0.190964	0.079*
C18	0.4063 (6)	0.5461 (4)	0.4815 (3)	0.0500 (8)
H18	0.466 (6)	0.617 (4)	0.468 (3)	0.047 (10)
C19	0.5894 (7)	0.4113 (4)	0.5148 (4)	0.0610 (10)
H19	0.639934	0.401921	0.440031	0.073*
C20	0.7869 (8)	0.4047 (5)	0.5847 (4)	0.0720 (12)
H20A	0.905371	0.326088	0.584967	0.086*
H20B	0.83281	0.486366	0.548454	0.086*
C21	0.9370 (10)	0.3558 (6)	0.7752 (5)	0.0816 (14)
H21A	0.899021	0.380842	0.844858	0.098*
H21B	1.033691	0.407696	0.734726	0.098*
C22	1.0583 (8)	0.2059 (5)	0.8137 (4)	0.0650 (11)
C23	0.9571 (8)	0.1086 (5)	0.8176 (4)	0.0736 (12)
H23	0.811032	0.135291	0.789624	0.088*
C24	1.0668 (11)	−0.0274 (6)	0.8619 (5)	0.0908 (16)
H24	0.993585	−0.091671	0.864127	0.109*
C25	1.2793 (12)	−0.0704 (6)	0.9024 (5)	0.0972 (17)
H25	1.352814	−0.163216	0.933199	0.117*
C26	1.3828 (11)	0.0258 (8)	0.8969 (6)	0.1033 (19)
H26	1.529392	−0.00179	0.924048	0.124*
C27	1.2763 (10)	0.1614 (7)	0.8525 (5)	0.0865 (15)
H27	1.351725	0.225148	0.848137	0.104*
C28	0.3143 (8)	0.6491 (5)	0.6315 (4)	0.0668 (11)
H28A	0.282475	0.739621	0.572718	0.08*
H28B	0.464796	0.622288	0.663545	0.08*
C29	0.1661 (6)	0.6508 (4)	0.7273 (3)	0.0516 (9)
C30	0.2390 (8)	0.5861 (5)	0.8436 (4)	0.0676 (11)
H30	0.385812	0.538776	0.864268	0.081*
C31	0.0997 (10)	0.5896 (5)	0.9303 (4)	0.0791 (14)
H31	0.153835	0.547459	1.009112	0.095*
C32	−0.1145 (9)	0.6533 (5)	0.9026 (5)	0.0779 (14)
H32	−0.208567	0.653325	0.961922	0.093*
C33	−0.1938 (8)	0.7179 (5)	0.7873 (5)	0.0707 (12)
H33	−0.341591	0.763003	0.767561	0.085*
C34	−0.0539 (8)	0.7156 (4)	0.7012 (4)	0.0627 (10)
H34	−0.108763	0.759096	0.622597	0.075*

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: None declared.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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