# Crystal structure of (3a*S*,4*R*,5*S*,6*R*,6a*S*)-4,5,6-trihydroxy-1-phenyl-3,3a,4,5,6,6a-hexahydro-1*H*-cycopent[*c*]-isoxazole, $C_{12}H_{15}N_1O_4$

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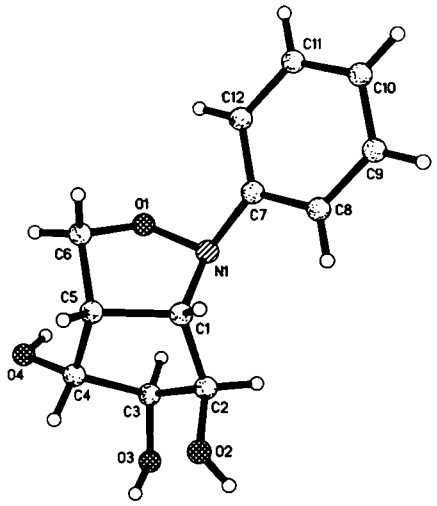


**Fig. 1.** Plot of the molecule.

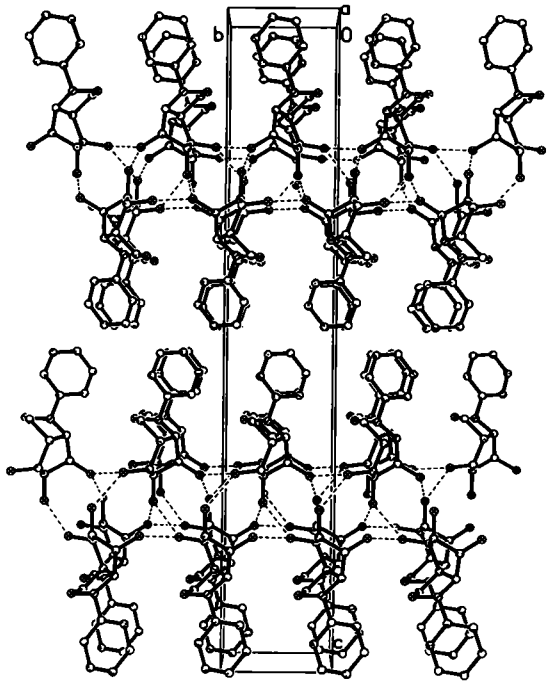


**Fig. 2.** View of the molecule assemble.

**Abstract**

$C_{12}H_{15}NO_4$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 5.7551(7)$ Å, $b = 5.7518(8)$ Å, $c = 34.459(5)$ Å, $V = 1140.7$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.039$, $wR_{all}(F^2) = 0.150$, $T = 293$ K.

**Source of material**

The title compound has been obtained by intramolecular nitrone cycloaddition [1]. Starting from 6-deoxy-6-iodo-2,3,4-tri-O-acetyl-α-D-mannopyranoside [1-4], reaction with zinc and vitamin $B_{12}$ as a catalyst afforded the corresponding 5-hexenose [5], which was transformed into the title compound as a single diastereomer by addition of *N*-phenyl-hydroxylamine and O-deprotection with sodium carbonate in methanol. Purification by flash chromatography on silica and crystallization from methanol gave the isoxazolidine in the form of colorless crystals [mp 438 K, $[\alpha]_{20}^{D} = -266$ (c = 1.0555, MeOH)].

**Discussion**

In the crystal structure strong intermolecular hydrogen bonds are present, with all three hydroxy groups acting as a donor to one and an acceptor to another neighbor. The H—O distances vary between 1.92 Å and 1.98 Å and the O—H···O angles are in the range of 155° to 160°. Along the *c*-axis , we observe alternating layers of polar regions which are represented by the network of the hydrogen bonds, and non-polar regions where the phenyl moieties are assembled (Fig. 2). Also, there is a face-to-face stacking of the phenyl groups along the *a*-axis.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colourless block, size 0.35 × 0.35 × 1.0 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| μ: | 1.04 cm$^{-1}$ |
| Diffractometer, scan mode: | Nicolet P3, Wyckoff |
| $2\theta_{max}$: | 59.98° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 1983, 1983 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 1756 |
| $N(param)_{refined}$: | 158 |
| Programs: | SHELXS-86 [6], SHELXL-93 [7] |

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**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 4*a* | 0.2527(3) | -0.2132(3) | 0.86412(5) | 0.033 |
| H(2) | 4*a* | 0.377(2) | -0.327(3) | 0.7754(6) | 0.052 |
| H(2A) | 4*a* | 0.5468(3) | -0.0890(3) | 0.81251(5) | 0.033 |
| H(3) | 4*a* | 0.245(4) | 0.042(4) | 0.7401(1) | 0.053 |
| H(3A) | 4*a* | 0.3771(3) | 0.2698(3) | 0.80235(5) | 0.032 |
| H(4) | 4*a* | 0.046(2) | 0.4813(3) | 0.8042(9) | 0.059 |
| H(4A) | 4*a* | -0.0534(3) | 0.0523(3) | 0.78663(5) | 0.035 |
| H(5) | 4*a* | -0.0988(3) | -0.0480(4) | 0.84900(5) | 0.038 |
| H(6A) | 4*a* | -0.0663(4) | 0.3715(5) | 0.87037(6) | 0.052 |
| H(6B) | 4*a* | -0.0114(4) | 0.1775(5) | 0.90150(6) | 0.052 |
| H(8) | 4*a* | 0.6046(4) | -0.2857(4) | 0.89267(6) | 0.046 |
| H(9) | 4*a* | 0.8005(5) | -0.3864(5) | 0.94905(7) | 0.057 |
| H(10) | 4*a* | 0.7948(5) | -0.1417(5) | 1.00234(7) | 0.063 |
| H(11) | 4*a* | 0.5996(5) | 0.2066(5) | 0.99868(7) | 0.060 |
| H(12) | 4*a* | 0.4139(4) | 0.3182(4) | 0.94208(6) | 0.047 |

**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| O(1) | 4*a* | 0.2741(3) | 0.3152(3) | 0.87758(4) | 0.0539(9) | 0.0281(7) | 0.0387(7) | 0.0029(7) | -0.0068(7) | -0.0038(6) |
| C(1) | 4*a* | 0.2688(3) | -0.0620(3) | 0.85128(5) | 0.0320(8) | 0.0270(7) | 0.0246(7) | -0.0028(8) | 0.0000(7) | 0.0007(6) |
| N(1) | 4*a* | 0.4069(3) | 0.1040(3) | 0.87460(4) | 0.0371(8) | 0.0274(7) | 0.0270(7) | -0.0018(7) | -0.0017(6) | -0.0013(6) |
| O(2) | 4*a* | 0.2848(3) | -0.2827(2) | 0.79195(4) | 0.0420(8) | 0.0264(6) | 0.0348(7) | -0.0035(6) | 0.0039(6) | -0.0070(5) |
| C(2) | 4*a* | 0.3768(3) | -0.0832(3) | 0.81112(5) | 0.0253(7) | 0.0253(8) | 0.0306(8) | -0.0009(7) | 0.0006(6) | -0.0017(7) |
| O(3) | 4*a* | 0.3281(3) | 0.1406(3) | 0.75028(4) | 0.0410(8) | 0.0399(8) | 0.0257(6) | -0.0107(7) | 0.0039(5) | 0.0023(6) |
| C(3) | 4*a* | 0.2930(3) | 0.1378(3) | 0.79113(5) | 0.0279(8) | 0.0263(8) | 0.0249(7) | -0.0052(7) | 0.0025(6) | 0.0000(6) |
| O(4) | 4*a* | -0.0553(3) | 0.3854(3) | 0.79911(5) | 0.0359(7) | 0.0351(7) | 0.0470(8) | 0.0061(7) | -0.0061(7) | 0.0029(6) |
| C(4) | 4*a* | 0.0374(3) | 0.1571(3) | 0.80316(5) | 0.0282(8) | 0.0300(9) | 0.0287(8) | -0.0007(8) | -0.0014(7) | 0.0018(7) |
| C(5) | 4*a* | 0.0307(3) | 0.0605(4) | 0.84538(5) | 0.0271(8) | 0.0378(9) | 0.0290(8) | -0.0017(8) | 0.0056(7) | 0.0023(8) |
| C(6) | 4*a* | 0.0354(4) | 0.2425(5) | 0.87672(6) | 0.045(1) | 0.053(1) | 0.0323(9) | 0.013(1) | 0.0046(9) | -0.0034(9) |
| C(7) | 4*a* | 0.4936(3) | 0.0294(4) | 0.91119(5) | 0.0276(8) | 0.0366(9) | 0.0264(8) | -0.0054(8) | 0.0002(7) | 0.0009(7) |
| C(8) | 4*a* | 0.6062(4) | -0.1842(4) | 0.91368(6) | 0.043(1) | 0.037(1) | 0.0351(9) | 0.001(1) | -0.0035(9) | 0.0003(8) |
| C(9) | 4*a* | 0.7213(5) | -0.2457(5) | 0.94773(7) | 0.047(1) | 0.046(1) | 0.051(1) | 0.003(1) | -0.012(1) | 0.009(1) |
| C(10) | 4*a* | 0.7189(5) | -0.0995(5) | 0.97958(7) | 0.053(1) | 0.066(2) | 0.038(1) | -0.002(1) | -0.014(1) | 0.005(1) |
| C(11) | 4*a* | 0.6030(5) | 0.1088(5) | 0.97721(7) | 0.052(1) | 0.064(2) | 0.033(1) | -0.002(1) | -0.010(1) | -0.008(1) |
| C(12) | 4*a* | 0.4906(4) | 0.1761(4) | 0.94329(6) | 0.041(1) | 0.044(1) | 0.0339(9) | -0.001(1) | -0.0022(8) | -0.0062(9) |

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