

**[3*R*-(3 $\beta$ ,4 $\beta$ ,5 $\beta$ )]-3-(Acetylamino)-3,4-dihydro-4-hydroxy-5-(hydroxymethyl)-furan-2(5*H*)-one monohydrate****William Clegg,\* Sarah L. Heath,†  
Richard F. W. Jackson‡ and  
Nicholas J. Palmer**School of Natural Sciences (Chemistry),  
University of Newcastle upon Tyne, Newcastle  
upon Tyne NE1 7RU, England† Current address: Department of Chemistry,  
University of Manchester, Manchester M13 9PL,  
England‡ Current address: Department of Chemistry,  
Dainton Building, University of Sheffield, Brook  
Hill, Sheffield S3 7HF, England

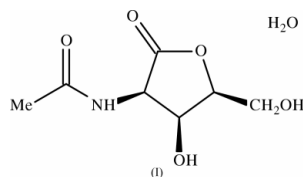
Correspondence e-mail: w. clegg@ncl.ac.uk

**Key indicators**Single-crystal X-ray study  
 $T = 160\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.038  
 $wR$  factor = 0.082  
Data-to-parameter ratio = 9.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

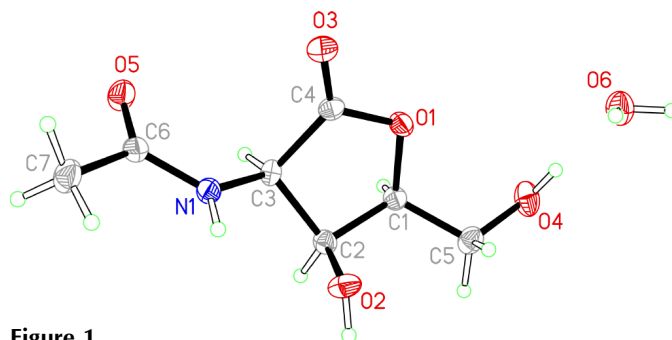
The title compound,  $\text{C}_7\text{H}_{11}\text{NO}_5 \cdot \text{H}_2\text{O}$ , a hydrated stereoisomer of the *N*-acetylated lactone of polyoxamic acid, contains a lactone ring with an envelope conformation, all substituents being *cis*. Intermolecular hydrogen bonds involving all possible donors link the lactone molecules into sheets, which are interlinked by the water molecules to give a three-dimensional network.

**Comment**

The title lactone, (I), was prepared as part of a project concerned with the synthesis of  $\beta$ -hydroxy- $\alpha$ -amino acids *via* (aryltio)nitrooxiranes (Jackson *et al.*, 1995). Its crystal structure has been determined in order to confirm the relative stereochemistry of the substituents on the lactone ring, postulated as all-*cis*. The compound (Fig. 1) is a hydrated stereoisomer of the *N*-acetylated lactone of polyoxamic acid, the crystal structure of which has already been reported (Saksena *et al.*, 1986).



A search of the Cambridge Structural Database (Version 5.24 and two updates, June 2003; Allen, 2002) finds 30 crystallographically characterized  $\gamma$ -lactones with a nitrogen substituent on the C atom adjacent to the carbonyl group, two of which are closely related to (I). One of these is the isomeric *N*-acetylated lactone of polyoxamic acid mentioned above, and the other has an azido substituent instead of the acetylamino group (Baird *et al.*, 1987). Both of these have a mixture of *cis* and *trans* substituents on the ring, with the nitrogen substituent *trans* to the hydroxymethyl group, and the hydroxy

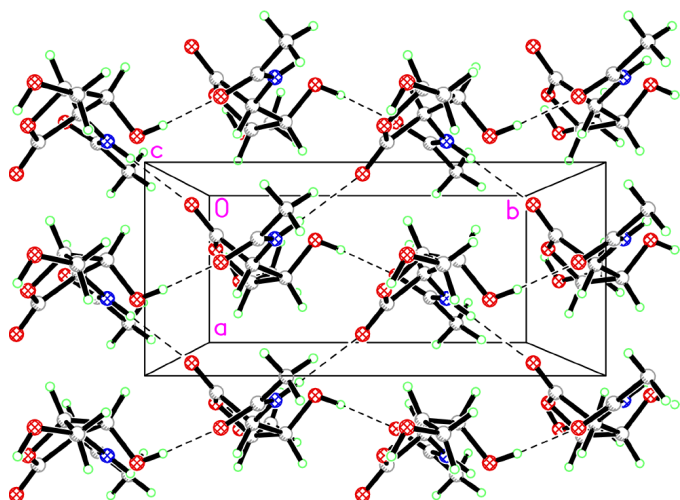
**Figure 1**

The structure of the asymmetric unit of (I), with atom labels and 50% probability ellipsoids for non-H atoms.

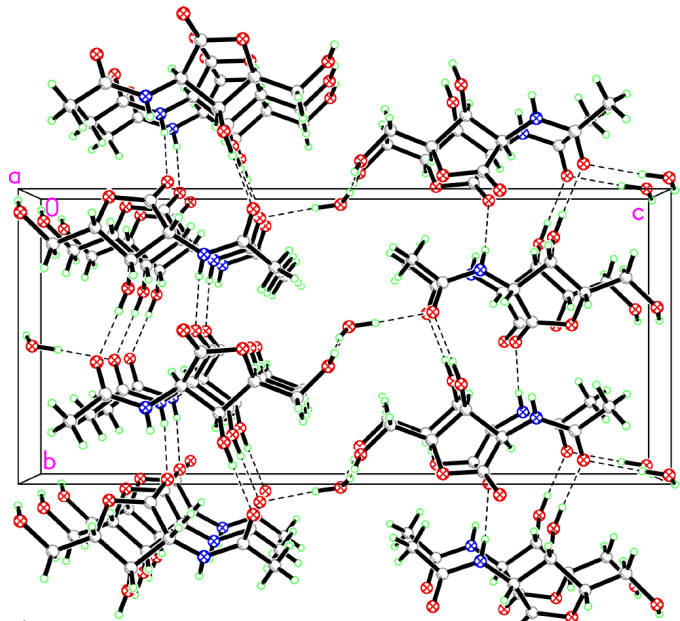
Received 23 June 2003

Accepted 24 June 2003

Online 30 June 2003



**Figure 2**  
Sheets of lactone molecules formed by intermolecular hydrogen bonds, viewed along the *c* axis.



**Figure 3**  
The crystal packing, viewed along the *a* axis, showing the linking of molecular sheets through hydrogen bonds with water molecules.

group *cis* to one of these and *trans* to the other.

The lactone ring in (I) has an envelope conformation, with atom C2 as the flap angle; C2 lies 0.558 (4) Å out of the mean plane of the other four atoms (r.m.s. deviation 0.021 Å), and this mean plane makes a dihedral angle of 35.0 (2)° with the plane formed by atoms C1, C2 and C3. The acetylamino and hydroxymethyl substituents are equatorial, while the hydroxy substituent on C2 is axial.

Details of the hydrogen bonds are given in Table 1. The N—H and both O—H groups serve as donors, together with both O—H groups of the water molecule, and the acceptors are the two carbonyl O atoms (O5 accepts two hydrogen bonds), the OH atom O4 and the water O atom. All hydrogen bonds are intermolecular. Four hydrogen bonds per lactone molecule (O2 and N1 as donors, and O3 and O5 as acceptors) link these

into sheets, as shown in Fig. 2. Interactions with the water molecules crosslink the sheets into the full three-dimensional network, as shown in Fig. 3.

## Experimental

The compound was prepared as described by Jackson *et al.* (1995), and was recrystallized from ethanol, which is presumably the source of the water of crystallization.

### Crystal data

C<sub>7</sub>H<sub>11</sub>NO<sub>5</sub>·H<sub>2</sub>O  
*M<sub>r</sub>* = 207.18  
 Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 4.5292 (5) Å  
*b* = 9.7807 (10) Å  
*c* = 21.622 (2) Å  
*V* = 957.83 (18) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.437 Mg m<sup>-3</sup>

Mo *K*α radiation  
 Cell parameters from 3459 reflections  
 $\theta$  = 1.9–28.2°  
 $\mu$  = 0.13 mm<sup>-1</sup>  
*T* = 160 (2) K  
 Needle, colourless  
 0.30 × 0.08 × 0.04 mm

### Data collection

Bruker SMART 1K CCD diffractometer  
 Thin-slice  $\omega$  scans  
 5877 measured reflections  
 1352 independent reflections  
 1101 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.047  
 $\theta_{\max}$  = 28.4°  
 $h$  = -5 → 5  
 $k$  = -9 → 12  
 $l$  = -26 → 27

### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.038  
*wR*(*F*<sup>2</sup>) = 0.082  
*S* = 1.04  
 1352 reflections  
 149 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.0129P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.25 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXTL*  
 Extinction coefficient: 0.016 (3)

**Table 1**  
Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2...O5 <sup>i</sup>	0.83 (3)	1.90 (3)	2.733 (2)	176 (3)
O4—H4...O6	0.87 (4)	1.78 (4)	2.652 (3)	178 (3)
N1—H1...O3 <sup>ii</sup>	0.87 (3)	2.24 (3)	3.104 (3)	173 (3)
O6—H6A...O4 <sup>iii</sup>	0.84 (5)	1.90 (5)	2.729 (3)	168 (4)
O6—H6B...O5 <sup>iv</sup>	0.88 (3)	1.95 (3)	2.834 (3)	175 (3)

Symmetry codes: (i) 1 - *x*, ½ + *y*, ½ - *z*; (ii) 2 - *x*, ½ + *y*, ½ - *z*; (iii) 1 + *x*, *y*, *z*; (iv) ½ - *x*, 1 - *y*, ½ + *z*.

Carbon-bound H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds), and with *U*<sub>iso</sub> constrained to be 1.2 (1.5 for methyl groups) times *U*<sub>eq</sub> of the carrier atom. H atoms bonded to N and O were located in a difference map and were refined freely with individual isotropic displacement parameters. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration was assumed from the known configuration of the starting material, *viz.* 2,3-isopropylidene-L-threitol.

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

We thank the EPSRC and Pfizer Central Research for financial support.

## References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Baird, P. D., Dho, J. C., Fleet, G. W. J., Peach, J. M., Prout, K. & Smith, P. W. (1987). *J. Chem. Soc. Perkin Trans. 1*, pp. 1785–1791.
- Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Jackson, R. F. W., Palmer, N. J., Wythes, M. J., Clegg, W. & Elsegood, M. R. J. (1995). *J. Org. Chem.* **60**, 6431–6440.
- Saksena, A. K., Lovey, R. G., Girijavallabhan, V. M., Ganguly, A. K. & McPhail, A. T. (1986). *J. Org. Chem.* **51**, 5024–5028.
- Sheldrick, G. M. (2001). *SHELXTL*. Version 6. Bruker AXS Inc., Madison, Wisconsin, USA.