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Structure of 2,3,4,5-Tetra-*O*-acetyl-6-amino-6-deoxy-D-mannonolactam

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Abstract. $C_{14}H_{19}NO_9$, $M_r = 345.31$, orthorhombic, $P2_12_12_1$, $a = 8.742\ (1)$, $b = 10.982\ (1)$, $c = 17.302\ (2)$ Å, $V = 1661.1\ (2)$ Å^3, $Z = 4$, $D_m = 1.40$, $D_x = 1.38$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.11$ mm^{-1}, $F(000) = 728$, $T = 295$ K, $R = 0.051$ for 1848 unique observed reflections. The seven-membered lactam ring adopts a chair conformation with C3, C6 and N displaced by 0.677 (6), −1.007 (7), −0.323 (7) Å, respectively, from the plane through C1, C2, C4 and C5. The molecules are linked together by N—H···O hydrogen bonds involving the amido N and the acetyl O9 atoms.

Experimental. 2,3,4,5-Tetra-*O*-acetyl-6-amino-6-deoxy-D-mannonolactam was prepared as described by Kefurt, Kefurtová & Jový (1989) and crystals obtained by recrystallization from a 1/1 mixture of chloroform/ethanol. The density was determined by flotation in iodomethane/toluene mixture at 298 K.

Data collection and structure refinement parameters are listed in Table 1.* Structure solved by direct methods and anisotropically refined by block-diagonal least squares in three blocks. H atoms were localized from a difference Fourier synthesis and

Table 1. *Data collection and structure refinement parameters*

Crystal dimensions (mm)	0.42 × 0.27 × 0.18
Diffractometer	Enraf–Nonius CAD-4, graphite monochromator
Scan technique	$\omega/2\theta$
Number and θ range (°) for lattice parameters	23, 13.07–19.23
Range of h, k and l	0→10, −13→13, −20→20
Max. value of $(\sin\theta)/\lambda$ (Å^{-1})	0.595
Standard reflections	022, 130
Monitored interval (min^{-1})	120
Intensity fluctuation (%)	−0.9
Total reflections measured, 2θ range (°)	6261, $2\theta < 50$
R_{int}	0.056
Unique observed reflections	1848
Criterion for observed reflections	$I > 1.96\sigma(I)$
Function minimized	$\sum(\|F_o\| - \|F_c\|)^2$
Weighting scheme	Unit
Parameters refined	195
R	0.051
S	1.12
$(\Delta/\sigma)_{max}$	0.004
Max. and min. $\Delta\rho$ (e Å^{-3})	0.21, −0.26
Source of atomic scattering factors	*SHELX76* (Sheldrick, 1976)
Programs used	*SHELXS86* (Sheldrick, 1986); *SHELX76* (Sheldrick, 1976); *PARST* (Nardelli, 1982); *SDP-Plus* (B. A. Frenz & Associates, Inc., 1985)
Computers used	PDP11/73, PC AT 286

* List of structure factors, anisotropical thermal parameters, H-atom positions and isotropic thermal parameters, and bond distances and angles involving H atoms, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54718 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0112]

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Table 2. *Atomic coordinates* ($\times 10^4$) *for non-*H *atoms and equivalent isotropic thermal parameters* ($Å^2 \times 10^4$)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	*y*	*z*	U_{eq}
O1	435 (4)	1430 (4)	1189 (2)	568 (12)
O2	−1093 (4)	−404 (3)	1795 (2)	412 (10)
O3	−2536 (4)	1060 (3)	2336 (2)	546 (11)
O4	2147 (4)	−1181 (3)	1596 (2)	380 (9)
O5	682 (5)	−2728 (4)	1165 (2)	668 (14)
O6	1517 (3)	−914 (3)	3657 (2)	358 (9)
O7	3035 (4)	−2269 (3)	4254 (2)	529 (11)
O8	4438 (3)	129 (3)	3621 (2)	391 (9)
O9	5927 (4)	−1316 (3)	3108 (2)	499 (11)
N	2056 (5)	1659 (4)	2197 (2)	418 (12)
C1	899 (5)	1112 (4)	1820 (3)	404 (13)
C2	192 (5)	9 (4)	2236 (3)	371 (14)
C3	1281 (5)	−1069 (4)	2303 (3)	347 (13)
C4	2421 (5)	−989 (4)	2968 (2)	285 (12)
C5	3469 (5)	111 (4)	2939 (3)	347 (13)
C6	2666 (6)	1334 (4)	2950 (3)	420 (15)
C7	−2384 (6)	267 (5)	1863 (3)	420 (14)
C8	−3559 (6)	−143 (6)	1296 (3)	549 (17)
C9	1698 (6)	−2027 (5)	1072 (3)	478 (16)
C10	2627 (13)	−1907 (9)	346 (5)	887 (33)
C11	1958 (6)	−1594 (4)	4274 (3)	384 (13)
C12	947 (8)	−1369 (6)	4943 (4)	524 (20)
C13	5650 (5)	−633 (4)	3624 (3)	412 (15)
C14	6546 (9)	−468 (8)	4357 (5)	702 (25)

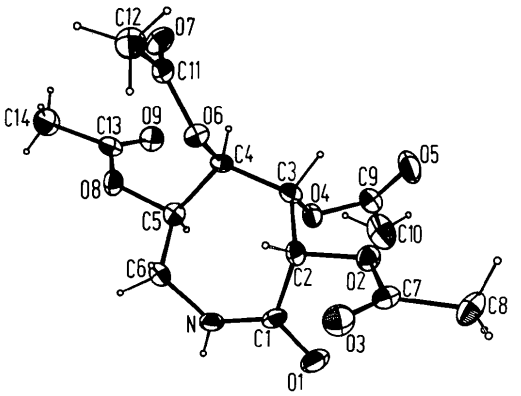

Fig. 1. View of a molecule of the lactam with atom numbering.

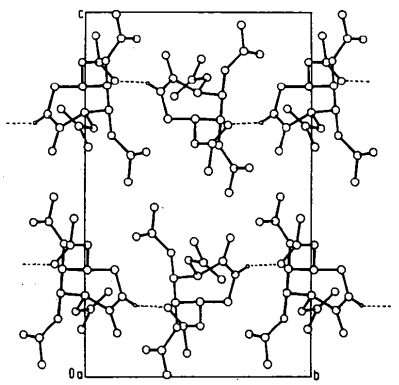

Fig. 2. Packing scheme. Hydrogen bonds are designated by dashed lines.

Table 3. *Bond lengths* (Å) *and angles* (°)

O1—C1	1.216 (6)	N—C6	1.452 (6)
O2—C2	1.432 (6)	C1—C2	1.539 (7)
O2—C7	1.353 (6)	C2—C3	1.524 (6)
O3—C7	1.202 (6)	C3—C4	1.525 (6)
O4—C3	1.444 (6)	C4—C5	1.517 (6)
O4—C9	1.356 (6)	C5—C6	1.516 (6)
O5—C9	1.186 (7)	C7—C8	1.490 (8)
O6—C4	1.433 (5)	C9—C10	1.502 (11)
O6—C11	1.359 (6)	C11—C12	1.477 (9)
O7—C11	1.199 (6)	C13—C14	1.502 (10)
O8—C5	1.453 (6)	N—HN	0.85 (5)
O8—C13	1.350 (5)	O9^{i}···HN	2.13 (5)
O9—C13	1.191 (6)	O9^{i}···N	2.887 (6)
N—C1	1.345 (6)		

C2—O2—C7	115.8 (4)	C4—C5—C6	115.2 (4)
C3—O4—C9	118.2 (4)	O8—C5—C6	104.4 (4)
C4—O6—C11	117.8 (3)	N—C6—C5	112.1 (4)
C5—O8—C13	116.9 (4)	O2—C7—O3	123.1 (5)
C1—N—C6	127.0 (4)	O3—C7—C8	126.2 (5)
O1—C1—N	123.9 (5)	O2—C7—C8	110.7 (4)
N—C1—C2	115.3 (4)	O4—C9—O5	124.8 (5)
O1—C1—C2	120.8 (4)	O5—C9—C10	125.1 (6)
O2—C2—C1	108.4 (4)	O4—C9—C10	110.0 (6)
C1—C2—C3	113.4 (4)	O6—C11—O7	122.7 (5)
O2—C2—C3	106.5 (4)	O7—C11—C12	126.6 (5)
O4—C3—C2	109.2 (4)	O6—C11—C12	110.7 (4)
C2—C3—C4	114.9 (4)	O8—C13—O9	123.2 (4)
O4—C3—C4	107.6 (4)	O9—C13—C14	127.5 (5)
O6—C4—C3	105.7 (3)	O8—C13—C14	109.7 (5)
C3—C4—C5	114.6 (3)	C6—N—HN	120 (3)
O6—C4—C5	108.4 (3)	C1—N—HN	112 (3)
O8—C5—C4	109.6 (4)	N—HN···O9^{i}	148 (4)

Symmetry code: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

expected geometry. All H atoms were isotropically refined. Absorption corrections were not made.

Atomic parameters are given in Table 2, bond lengths and angles in Table 3. A molecule of the lactam is drawn in Fig. 1, and the crystal packing is shown in Fig. 2.

Related literature. Other seven-membered lactams have been reported: ε-caprolactam (Winkler & Dunitz, 1975), and 2-aza-*A*-homo-5α-cholestan-1-one (Suginome & Furusaki, 1979). In these structures the lactam ring also adopts a chair conformation.

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