

difference Fourier synthesis] and refinement [using full-matrix least squares on  $F$  (Sheldrick, 1976)]. At isotropic convergence, final absorption corrections (min. 0.895, max. 1.171) applied empirically using *DIFABS* (Walker & Stuart, 1983). C—H and N—H bonds were constrained to be 1.08 and 1.00 Å, respectively; HCH and HCS angles in the dmso molecules were constrained to be tetrahedral. At final convergence,  $R = 0.0228$ ,  $wR = 0.0338$ ,  $S = 1.164$  for 257 refined parameters,  $(\Delta/\sigma)_{\max}$  in final cycle 0.66, max. and min. residues in final  $\Delta F$  synthesis 0.83,  $-0.73 \text{ e \AA}^{-3}$  respectively. The weighting scheme  $w^{-1} = \sigma^2(F) + 0.002487F^2$  gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Re (Cromer & Mann, 1968). Atomic coordinates are listed in Table 1, while selected bond lengths, angles and torsion angles appear in Table 2.\* The atom-numbering scheme for the quadruply protonated cyclam is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985): a packing diagram of the structure, produced using *PLUTO* (Motherwell, 1976), appears

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52228 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Diaquabis(oxo-5 prolinato)zinc(II)

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**Abstract.** [Zn(C<sub>5</sub>H<sub>6</sub>NO<sub>3</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>],  $M_r = 357.6$ , monoclinic,  $C_2$ ,  $a = 14.834$  (8),  $b = 5.888$  (3),  $c = 7.920$  (6) Å,  $\beta = 98.34$  (5)°,  $V = 684$  (1) Å<sup>3</sup>,  $Z = 2$ ,  $D_x$

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as Fig. 2. Molecular-geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

**Related literature.** Diprotonated cyclam has been reported as the diperchlorate salt (Nave & Truter, 1974). The complex *trans*-[Re(O)<sub>2</sub>(cyclam)]Cl<sub>2</sub>·2(BPh<sub>3</sub>·H<sub>2</sub>O) (Blake, Greig & Schröder, 1988), containing neutral cyclam coordinated to dioxo-Re<sup>V</sup>, also has an extensive hydrogen-bonding network.

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$= 1.735 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.7107 \text{ \AA}$ ,  $\mu = 1.88 \text{ mm}^{-1}$ ,  $F(000) = 368$ ,  $T = 293$  (1) K,  $R = 0.016$  for 1070 independent observed reflections. The mol-

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Tableau 1. Coordonnées atomiques relatives, facteurs de température isotropes équivalents et écarts-type

$$B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}abc\cos\gamma + \beta_{13}accos\beta + \beta_{23}bccos\alpha).$$

	x	y	z	$B_{eq}$ (Å <sup>2</sup> )
Zn	$\frac{1}{2}$	$\frac{1}{2}$	0	1,867 (3)
N(1)	0,38137 (9)	1,0456 (3)	0,4327 (2)	2,28 (3)
C(2)	0,3515 (1)	0,8943 (3)	0,2919 (2)	1,96 (2)
C(3)	0,2924 (1)	1,0500 (3)	0,1638 (2)	2,54 (3)
C(4)	0,2681 (1)	1,2512 (3)	0,2688 (2)	2,22 (3)
C(5)	0,3359 (1)	1,2399 (3)	0,4292 (2)	2,16 (3)
O(6)	0,3457 (1)	1,3882 (3)	0,5406 (2)	3,42 (3)
C(7)	0,4285 (1)	0,7909 (3)	0,2111 (2)	1,86 (2)
O(8)	0,50829 (8)	0,8602 (3)	0,2452 (2)	2,57 (2)
O(9)	0,40371 (8)	0,6334 (3)	0,1057 (2)	2,77 (2)
O(10)	0,5670 (1)	0,2799 (3)	0,1615 (2)	3,03 (3)

Tableau 2. Longueurs (Å), angles des liaisons (°) et écarts-type

Les astérisques signalent les liaisons hydrogène.

Zn—O(9)	1,925 (1)	C(3)—C(4)	1,520 (3)
Zn—O(10)	1,984 (1)	C(4)—C(5)	1,503 (2)
N(1)—C(2)	1,446 (2)	C(5)—O(6)	1,235 (2)
N(1)—C(5)	1,327 (2)	C(7)—O(8)	1,244 (2)
N(1)···O(8 <sup>iii</sup> )	3,028 (2)	C(7)—O(9)	1,266 (2)
C(2)—C(3)	1,543 (2)	O(10)···O(6 <sup>v</sup> )	2,600 (2)*
C(2)—C(7)	1,516 (2)	O(10)···O(8 <sup>iii</sup> )	2,733 (2)*
O(9)—Zn—O(9 <sup>i</sup> )	131,83 (7)	N(1)—C(5)—C(4)	109,3 (1)
O(9)—Zn—O(10)	108,67 (6)	N(1)—C(5)—O(6)	126,1 (2)
O(9)—Zn—O(10 <sup>i</sup> )	102,30 (6)	C(4)—C(5)—O(6)	124,6 (2)
O(10)—Zn—O(10 <sup>i</sup> )	98,44 (6)	C(2)—C(7)—O(8)	121,9 (2)
C(2)—N(1)—C(5)	114,7 (1)	C(2)—C(7)—O(9)	114,1 (1)
N(1)—C(2)—C(3)	102,9 (1)	O(8)—C(7)—O(9)	124,0 (2)
N(1)—C(2)—C(7)	114,0 (1)	Zn—O(9)—C(7)	115,0 (1)
C(3)—C(2)—C(7)	110,6 (1)	N(1)—H(1)···O(8 <sup>iii</sup> )	172 (4)*
C(2)—C(3)—C(4)	105,1 (1)	O(10)—H(10)···O(8 <sup>iii</sup> )	162 (3)*
C(3)—C(4)—C(5)	104,3 (1)	O(10)—H'(10)···O(6 <sup>v</sup> )	163 (3)*

Code de symétrie: (i)  $1-x, y, -z$ ; (ii)  $1-x, y, 1-z$ ; (iii)  $x, -1+y, z$ ; (iv)  $1-x, -1+y, 1-z$ .

ecule has a binary axis. The Zn atom is tetrahedrally coordinated to four O atoms [Zn—O = 1,925 (1) and 1,984 (1) Å]. The absolute configuration of the title compound and that of 5-oxoproline have been determined. The structure can be regarded as being constituted of layers which spread out along the  $x = 0$  and  $x = \frac{1}{2}$  planes. Molecules belonging to the same layer are linked together by O—H···O and N—H···O hydrogen bonds. This compound has been studied because it was previously described as a salt of L-pyroglyutamic acid (5-oxoproline).

**Partie expérimentale.** Cristal en forme de tronc de pyramide:  $0,20 \times 0,37 \times 0,45$  mm. Diffractomètre Enraf-Nonius CAD-4. Dimensions de la maille déterminées avec 25 réflexions telles que  $8,08 \leq \theta \leq 19,75^\circ$ .  $0,039 \leq (\sin\theta)/\lambda \leq 0,704 \text{ \AA}^{-1}$ . Balayage  $\theta/2\theta$  d'amplitude  $s$  (°) =  $0,80 + 0,35 \text{ tg}\theta$ .  $-20 \leq h \leq 20$ ,  $0 \leq k \leq 8$ ,  $0 \leq l \leq 11$ . Réflexions de contrôle de l'intensité: 422, 621 et 403. Variations non significatives de

$I$  au cours des mesures.  $\sigma(I)/I$  moyen (contrôle) = 0,0031. 1087 réflexions indépendantes mesurées, 17 inobservées [ $I < 3\sigma(I)$ ]. Méthode de l'atome lourd. Affinement sur  $F$ , programme à matrice complète. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974). Paramètres affinés:  $x, y, z$  de tous les atomes,  $\beta_{ij}$  de Zn, O, N et C. Corrections d'absorption: programme *DIFABS* (Walker & Stuart, 1983). Valeurs maximale et minimale du coefficient de correction: 1,15 et 0,89. Coefficient d'extinction secondaire isotrope:  $g = 1,35 (3) \times 10^{-5}$ .  $R = 0,016$ ,  $wR = 0,022$ ,  $w = 1/\sigma^2(F)$ ,  $S = 0,99$ .  $(\Delta/\sigma)_{\max} = 0,01$ ,  $|\Delta\rho|_{\max} = 0,20 (4) \text{ e \AA}^{-3}$ . Programmes de calcul du système *SDP* (B. A. Frenz & Associates Inc., 1982). Fig. 1 et 2: programme *ORTEPII* (Johnson, 1976). Ordinateur DEC PDP11/44.

Lorsqu'on remplace les coordonnées  $x, y, z$  de chacun des atomes respectivement par  $1-x, 1-y$  et  $1-z$ , on obtient pour le facteur  $R$  la valeur 0,028.

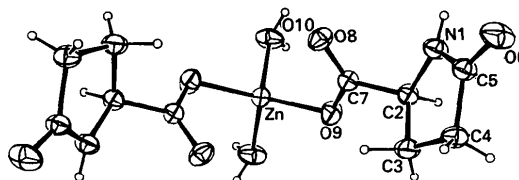
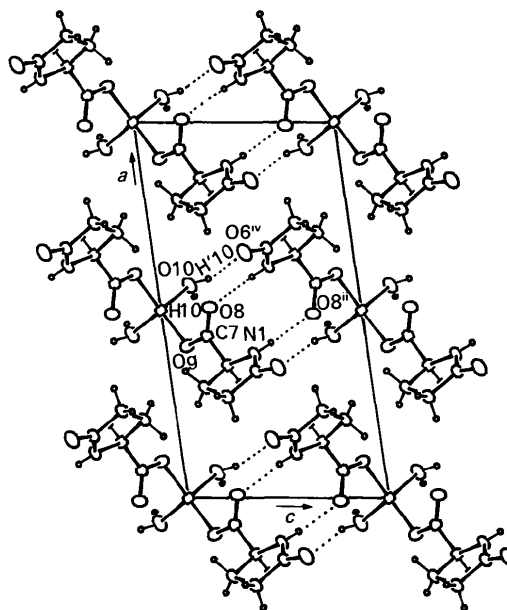


Fig. 1. Vue en perspective de la molécule et numéros attribués à ses atomes.

Fig. 2. Dessin de la structure vue selon [010]. Les traits en pointillé correspondent aux liaisons hydrogène. La liaison O(10)—H(10)···O(8<sup>iii</sup>) n'est pas représentée.

On peut donc admettre que la Fig. 1 représente les configurations absolues de la molécule étudiée et de la molécule d'acide L-pyroglutamique. Les deux atomes de carbone asymétriques possèdent la configuration *S*.

Les coordonnées atomiques relatives et les facteurs de température isotropes équivalents sont rapportés dans le Tableau 1,\* les longueurs et les angles des liaisons dans le Tableau 2. La Fig. 1 représente une vue en perspective de la molécule et la Fig. 2 une vue de la structure selon [010].

**Littérature associée.** Structure cristalline et moléculaire de l'acide pyroglutamique (oxo-5 proline) (Pattabhi & Venkatesan, 1974). Ce mémoire décrit le racémique. Structure de l'acide L-pyroglutamique

\* Les listes des facteurs de structure observés et calculés, des coefficients d'agitation thermique anisotrope, des coordonnées des atomes d'hydrogène, des distances C—H, N—H et O—H, des distances interatomiques intermoléculaires et des angles de torsion ont été déposées aux archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 52267: 9 pp.). On peut en obtenir des copies en s'adressant à: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

(van Zoeren, Oonk & Kroon, 1978). Structure cristalline d'un composé mixte d'acide L-glutamique et d'acide L-pyroglutamique  $C_5H_9NO_4 \cdot C_5H_7NO_3 \cdot H_2O$  et affinement de la structure de l'acide pyroglutamique (Taira & Watson, 1977). L'acide pyroglutamique étudié est le racémique. Structure cristalline et moléculaire de l'*allo*-hydroxy-4 L-proline dihydrate (Shamala, Guru Row & Venkatesan, 1976).

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## Structure of a Triterpene Extracted from *Austroplenckia populnea* (Celastraceae), Methyl 3-Oxofriedelan-20 $\alpha$ -oate

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**Abstract.**  $C_{31}H_{50}O_3$ ,  $M_r = 470.74$ , orthorhombic,  $P2_12_12_1$ ,  $a = 6.815$  (2),  $b = 16.127$  (3),  $c = 24.695$  (4) Å,  $V = 2714$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.152$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.668$  cm<sup>-1</sup>,  $F(000) = 1040$ ,  $T = 298$  K,  $R = 0.055$  for 1454 observed reflections. The rings adopt chair conformations. The bond angles are as expected within experimental error and the mean  $sp^2$  and  $sp^3$  bond angles are 120.0 (4) and 111.1 (3)°, respectively.

**Experimental.** The data-collection and refinement parameters are summarized in Table 1.

The crystal structure was solved using direct methods and difference Fourier techniques. In the final cycles of block-matrix least-squares refinement all non-H atoms were refined anisotropically. H atoms were located geometrically, all with the common isotropic temperature factor  $U = 0.12$  Å<sup>2</sup>. Scattering factors for non-H atoms were taken from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965) with corrections for anomalous dispersion from Cromer & Liberman (1970). Programs used: *SHELXS86* (Sheldrick, 1986), *SHELX76* (Sheldrick, 1976),