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 (van Zoeren, Oonk & Kroon, 1978). Structure cristalline d'un composé mixte d'acide L-glutamique et d'acide L-pyroglutamique $C_5H_9NO_4.C_5H_7NO_3.H_2O$ 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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* 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.

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Structure of a Triterpene Extracted from *Austroplenckia populnea* (Celastraceae), Methyl 3-Oxofriedelan-20α-oate

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Abstract. $C_{31}H_{50}O_3$, $M_r = 470{\cdot}74$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}815\,(2)$, $b = 16{\cdot}127\,(3)$, $c = 24{\cdot}695\,(4)$ Å, $V = 2714\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}152$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}668$ cm^{-1}, $F(000) = 1040$, $T = 298$ K, $R = 0{\cdot}055$ 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 $120{\cdot}0\,(4)$ and $111{\cdot}1\,(3)^\circ$, 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 $U = 0{\cdot}12$ Å^2. 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: *SHELXS*86 (Sheldrick, 1986), *SHELX*76 (Sheldrick, 1976),

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Table 1. *Experimental details*

(*a*) Data collection[i,ii]	
Mode	ω–2θ
Scan rate (° min^{-1})	2·99–10
θ range (°)	0–25
Range of *hkl*	$0 < h < 8$, $0 < k < 19$, $0 < l < 29$
Total reflections measured	3042
Unique reflections	2742
$R_{\rm int}$	0·026
Approx. crystal dimensions (mm)	0·20 × 0·25 × 0·45
(*b*) Structure refinement[iii]	
Reflections used [$I > 3\sigma(I)$]	1454
No. of variables	308
R, *wR*	0·055, 0·059
Max. shift/e.s.d.	0·04
Max., min. density in final difference map (e Å^{-3})	0·22, −0·23
S	3·24

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with $9{\cdot}55 < \theta < 19{\cdot}76°$. (ii) Enraf–Nonius CAD-4 diffractometer with graphite monochromator. Two standard reflections $(1,\overline{13},0)$ and $2\bar{7}4$ measured every hour showed no significant variations. No absorption correction. (iii) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = [\sigma^2(F_o) + 0{\cdot}001F_o^2]^{-1}$.

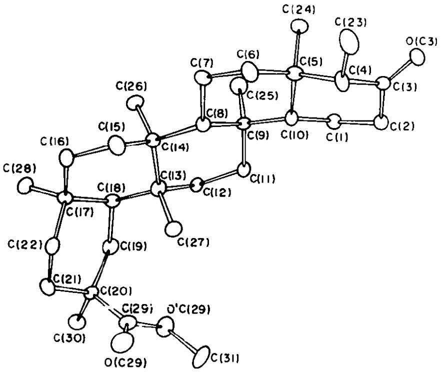

Fig. 1. Perspective view of the molecule showing the atom labelling.

ORTEP (Johnson, 1965). Most of the calculations were performed on a VAX 11/870 computer at the Instituto de Física e Química de São Carlos.

Atomic coordinates are listed in Table 2* and bond lengths in Table 3. Fig. 1 is an *ORTEP* perspective drawing of the molecule showing the atom labelling.

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Table 2. *Final positional coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j B_{ij}(\mathbf{a}_i.\mathbf{a}_j).$$

	x	*y*	*z*	$B_{\rm eq}$ (Å^2)
C(1)	0·142 (1)	0·3696 (3)	0·0833 (2)	3·9 (2)
C(2)	0·266 (1)	0·2992 (4)	0·0583 (3)	4·5 (3)
C(3)	0·273 (1)	0·3063 (4)	−0·0019 (3)	4·4 (3)
C(4)	0·330 (1)	0·3890 (3)	−0·0248 (3)	4·3 (2)
C(5)	0·1774 (9)	0·4560 (4)	−0·0030 (2)	3·9 (2)
C(6)	0·237 (1)	0·5421 (3)	−0·0233 (2)	4·1 (2)
C(7)	0·136 (1)	0·6134 (4)	0·0052 (2)	4·3 (2)
C(8)	0·1773 (8)	0·6105 (3)	0·0658 (2)	3·1 (2)
C(9)	0·0914 (8)	0·5293 (3)	0·0906 (2)	3·0 (2)
C(10)	0·1936 (9)	0·4555 (3)	0·0603 (2)	3·3 (2)
C(11)	0·1479 (9)	0·5266 (3)	0·1506 (2)	3·6 (2)
C(12)	0·1035 (9)	0·6077 (3)	0·1824 (2)	3·4 (2)
C(13)	0·2047 (8)	0·6846 (3)	0·1575 (2)	3·1 (2)
C(14)	0·134 (1)	0·6938 (3)	0·0973 (2)	3·6 (2)
C(15)	0·252 (1)	0·7650 (3)	0·0712 (3)	4·5 (2)
C(16)	0·302 (1)	0·8482 (3)	0·0992 (3)	4·7 (3)
C(17)	0·1996 (9)	0·8498 (3)	0·1623 (2)	3·2 (2)
C(18)	0·1442 (8)	0·7643 (3)	0·1890 (2)	2·9 (2)
C(19)	0·1883 (8)	0·7666 (3)	0·2513 (2)	3·3 (2)
C(20)	0·3741 (9)	0·8098 (3)	0·2734 (2)	3·5 (2)
C(21)	0·423 (1)	0·8898 (4)	0·2430 (3)	4·5 (3)
C(22)	0·406 (1)	0·8797 (4)	0·1811 (3)	4·1 (2)
C(23)	0·354 (1)	0·3876 (4)	−0·0862 (3)	6·8 (3)
C(24)	−0·029 (1)	0·4335 (4)	−0·0244 (3)	4·4 (2)
C(25)	−0·1376 (9)	0·5223 (4)	0·0876 (3)	4·6 (2)
C(26)	−0·0926 (9)	0·7141 (4)	0·0934 (3)	4·1 (2)
C(27)	0·4314 (9)	0·6710 (4)	0·1610 (2)	4·0 (2)
C(28)	0·045 (1)	0·9150 (4)	0·1791 (3)	5·1 (3)
C(29)	0·339 (1)	0·8337 (4)	0·3333 (3)	4·9 (3)
C(30)	0·553 (1)	0·7528 (4)	0·2749 (3)	4·1 (2)
C(31)	0·689 (1)	0·6278 (5)	0·3036 (3)	7·3 (4)
O(C29)	0·7144 (8)	0·7724 (3)	0·2579 (2)	7·1 (2)
O′(C29)	0·5193 (7)	0·6824 (3)	0·2995 (2)	5·1 (2)
O(C3)	0·2383 (7)	0·2469 (3)	−0·0315 (2)	5·4 (2)

Table 3. *Bond distances* (Å) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·544 (9)	C(13)—C(14)	1·570 (8)
C(1)—C(10)	1·538 (8)	C(13)—C(18)	1·558 (8)
C(2)—C(3)	1·492 (9)	C(13)—C(27)	1·563 (8)
C(3)—C(4)	1·500 (9)	C(14)—C(15)	1·543 (8)
C(3)—O(C3)	1·228 (8)	C(14)—C(26)	1·581 (9)
C(4)—C(5)	1·593 (9)	C(15)—C(16)	1·549 (8)
C(4)—C(23)	1·525 (9)	C(16)—C(17)	1·559 (8)
C(5)—C(6)	1·531 (8)	C(17)—C(18)	1·574 (8)
C(5)—C(10)	1·567 (8)	C(17)—C(22)	1·558 (9)
C(5)—C(24)	1·546 (9)	C(17)—C(28)	1·545 (9)
C(6)—C(7)	1·514 (9)	C(18)—C(19)	1·568 (8)
C(7)—C(8)	1·523 (8)	C(19)—C(20)	1·545 (8)
C(8)—C(9)	1·560 (8)	C(20)—C(21)	1·529 (8)
C(8)—C(14)	1·580 (8)	C(20)—C(29)	1·547 (9)
C(9)—C(10)	1·569 (8)	C(20)—C(30)	1·527 (9)
C(9)—C(11)	1·532 (8)	C(21)—C(22)	1·542 (9)
C(9)—C(25)	1·566 (8)	C(29)—O(C29)	1·219 (9)
C(11)—C(12)	1·555 (8)	C(29)—O′(C29)	1·308 (8)
C(12)—C(13)	1·547 (8)	C(31)—O′(C29)	1·46 (1)

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* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52164 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.