

Table 2. *Interatomic distances (Å) and bond angles (°) with e.s.d.'s in parentheses*

Te(1)–Te(2)	5.368 (2)	Te(3)–O(1) (b)	1.99 (5)
Te(1)–Te(3)	5.083 (3)	–O(2) (a)	1.89 (1)
Te(3)–Te(4)	3.543 (2)	–O(3) (d)	2.29 (3)
Te(3)–Te(1')	3.513 (2)	–O(4)	2.59 (6)
S—O(3')	1.47 (3)	–O(1') (c)	1.99 (3)
–O(4)	1.51 (7)	–O(5')	2.86 (1)
–O(5)	1.50 (5)		

Te(3)–O(1)–Te(1')	124 (1)
Te(3)–O(2)–Te(4)	140 (1)
O(1)–Te(3)–O(2) (ab)	96 (2)
O(2)–Te(3)–O(1') (ac)	81 (1)
O(2)–Te(3)–O(3) (ad)	95 (1)
O(1)–Te(3)–O(1') (bc)	87 (2)
O(1)–Te(3)–O(3) (bd)	140 (3)
O(3)–Te(3)–O(1') (cd)	133 (2)
O(3')–S—O(3'')	108 (1)
O(3')–S—O(4)	111 (3)
O(3')–S—O(5)	108 (2)
O(4)–S—O(5)	112 (3)

located on the vertices of a distorted trigonal bipyramid, with equatorial Te–O distances equal to (a), (b) and axial distances equal to (c), (d) [see Table 2, values labelled according to Zemmann (1971) and Lindqvist

(1973)]. The average Te–O distances are 1.957 and 2.04 Å for the CN's 3+1 and 3 respectively. The average angles in the bipyramid are 96, 101 and 133°, the ideal values being 120, 90 and 180°. The average S–O distance in the sulphate anion is 1.487 Å, and the average O–S–O angle 109.7°. The shortest Te–O distance to the closer sulphate anion is 2.29 and to the more distant one 2.89 Å.

References

- BURNHAM, C. W. (1962). *Carnegie Inst. Wash. Yearb.* **61**, 132.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1963). *ORFLS*. Oak Ridge National Laboratory Report ORNL-TM-305.
- CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.
- HUBKOVÁ, H., LOUB, J. & SYNEČEK, V. (1966). *Coll. Czech. Chem. Commun.* **31**, 4353–4362.
- LINDQVIST, O. (1973). *The Oxygen Coordination of Tellurium(IV) and Tellurium(VI)*. Univ. of Göteborg, Sweden.
- WEISS, Z. (1973). *FOURIER*. Report 23/3/1/002. VVUÚ, Ostrava-Radvanice.
- ZEMANN, J. (1971). *Mh. Chem.* **102**, 1209–1216.

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Cyclo-*N*-methyl-L-alanyl-L-alanyl (c-*N*-Me-L-Ala-L-Ala)

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Abstract. C₁₇H₁₂O₂N₂, *P*₂₁₂₁, *a* = 5.9775 (11), *b* = 9.5370 (21), *c* = 14.6981 (22) Å, *V*_c = 837.9 Å³, *Z* = 4, *D*_c = 1.24 g cm⁻³. The diketopiperazine ring assumes a boat conformation with both methyl substituents quasi-axial.

Introduction. Data were collected from a crystal of dimensions approximately 0.4 × 0.3 × 0.3 mm. Systematic absences of *h*00 for *h* = 2*n* + 1, 0*k*0 for *k* = 2*n* + 1 and 00*l* for *l* = 2*n* + 1 indicated space group *P*₂₁₂₁. Cell dimensions were measured with Cu *K*α radiation on a Siemens four-circle diffractometer by the five-values measuring technique as described, for example, by Allen, Roger & Troughton (1971). 995 reflexions, of which 954 had *I* ≥ 3σ, were measured in the range 0 ≤ θ ≤ 70° with a scan width of 0.6–1.0° determined from the resolution function of the instrument. Data reduction was performed by means of the program *SIEM1* written for the Siemens diffractometer. Normalized structure factors were calculated and phases determined by the program *MULTAN* (Ger-

main, Main & Woolfson, 1971). All 11 non-H atoms were found by calculation of an *E* map from the phase set with highest ABSFOM (1.13). Three cycles of full-matrix least-squares refinement with isotropic temperature factors reduced *R* (= ∑||*F*_o| – |*F*_c||/∑|*F*_o|) to 0.11 following which a difference Fourier synthesis revealed unambiguously all 12 H atoms. Further anisotropic refinement with elimination of seven very strong reflexions for which the agreement between |*F*_o| and |*F*_c| was very poor reduced *R* to its final value of 0.0374 for all observed reflexions.*

Atomic coordinates and temperature factors from the final cycle of refinement are given in Tables 1 and 2. All Fourier and refinement calculations were carried out with the X-RAY 70 system on the PDP10 computer of the Institut Laue–Langevin.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31880 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Scattering factors used were, for C, N and O, from *International Tables for X-ray Crystallography* (1968) and, for H, from Stewart, Davidson & Simpson (1965).

Discussion. The crystal structure of *c*-N-Me-L-Ala-L-Ala has been determined as part of a study of the conformations of *N*-methylated cyclic dipeptides in the solid state by X-ray diffraction (Timmins, 1975) and in solu-

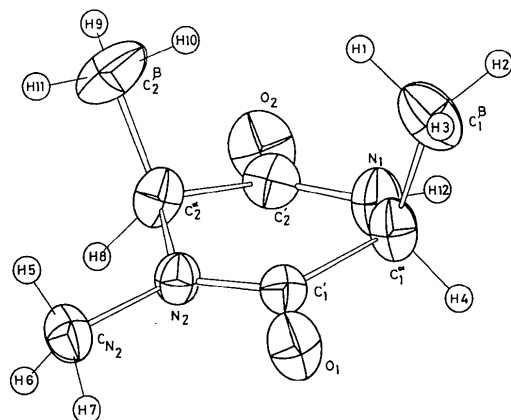


Fig. 1. ORTEP plot of the *c*-N-Me-L-Ala-L-Ala molecular conformation. Viewing direction parallel to *a*.

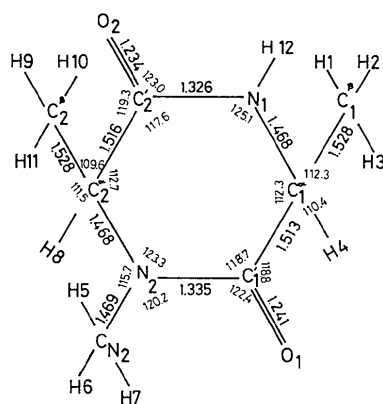


Fig. 2. Atomic labelling, bond lengths and bond angles for non-hydrogen atoms. Average standard deviations are 0.004 Å for bond lengths and 0.2° for bond angles.

Table 2. Atomic coordinates and isotropic thermal parameters ($\times 10^4$) for hydrogen atoms (e.s.d.'s in parentheses)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
H(1)	4242 (77)	6290 (44)	6976 (27)	52 (13)
H(2)	3827 (78)	6049 (45)	8013 (27)	53 (12)
H(3)	2771 (105)	7238 (52)	7544 (32)	96 (19)
H(4)	-429 (58)	5616 (32)	7621 (21)	16 (8)
H(5)	578 (71)	6621 (41)	4376 (25)	49 (13)
H(6)	-1455 (60)	5439 (36)	4313 (23)	29 (10)
H(7)	-2056 (62)	6815 (34)	4767 (22)	33 (10)
H(8)	724 (54)	3644 (33)	4908 (20)	19 (8)
H(9)	4992 (76)	3849 (45)	4978 (26)	56 (13)
H(10)	4593 (71)	5345 (42)	5664 (29)	44 (12)
H(11)	3900 (66)	5297 (40)	4525 (27)	42 (11)
H(12)	1577 (59)	3507 (32)	7496 (21)	16 (9)

tion by NMR (Davies & Khaled, 1976). The crystal structure of the unmethylated compound, *c*-L-Ala-L-Ala, shows a boat conformation with the C^α substituents quasi-equatorial (Sletten, 1972); but NMR studies of the title compound suggest a boat conformation in solution with quasi-axial substituents. The present work confirms this, as shown by the ORTEP plot (Fig. 1).

Bond lengths and angles involving non-H atoms are given in Fig. 2, which also shows the atomic labelling. The average C-H bond length is 1.02 (5) Å and the N-H bond length 0.80 (8) Å. The N-Me bond length of 1.469 (3) Å is slightly longer than that in *c*-Sar-Val (Timmins, 1975) but not significantly greater than in *c*-Sar-Sar (Groth, 1969). Comparison with *c*-L-Ala-L-Ala shows no significant differences in bond lengths and angles.

The ring conformation shown in Fig. 1 is described precisely by the torsion angles given in Table 3. It is seen that equivalent torsion angles differ by only $\sim 4^\circ$ and thus there is very little twist in the conformation. As in other cyclic dipeptides, the strain imposed by closing two *cis* peptide bonds into a ring imposes a degree of non-planarity on these bonds, as shown by the values of $\omega \neq 0^\circ$. The tendency of the molecule to take up a conformation with both C^α substituents quasi-axial must be related to the unfavourable interaction between C^β and C(N) in the quasi-equatorial conformation.

Table 1. Atomic coordinates ($\times 10^4$) and anisotropic thermal parameters ($\times 10^3$) for non-hydrogen atoms (e.s.d.'s in parentheses)

	<i>x</i>	<i>y</i>	<i>z</i>	u_{11}	u_{22}	u_{33}	u_{12}	u_{13}	u_{23}
C(1) $^\alpha$	892 (6)	5512 (3)	7153 (2)	52 (2)	32 (1)	28 (1)	5 (1)	-1 (1)	-1 (1)
C(1) $^\beta$	2935 (7)	6339 (4)	7473 (3)	62 (2)	47 (2)	51 (2)	0 (2)	-22 (2)	-7 (2)
C(1)'	-65 (4)	6156 (3)	6296 (2)	33 (1)	29 (1)	30 (1)	-1 (1)	0 (1)	0 (1)
O(1)	-1114 (4)	7275 (2)	6355 (1)	65 (1)	37 (1)	37 (1)	21 (1)	-6 (1)	-7 (1)
N(1)	1422 (5)	4020 (2)	7037 (2)	59 (2)	28 (1)	32 (1)	5 (1)	1 (1)	7 (1)
C(2) $^\alpha$	1750 (5)	4290 (3)	5396 (2)	45 (2)	25 (1)	33 (1)	4 (1)	6 (1)	-1 (1)
C(2) $^\beta$	4086 (6)	4744 (4)	5097 (3)	48 (2)	48 (2)	66 (2)	12 (2)	24 (2)	11 (2)
C(2)'	1921 (5)	3410 (3)	6253 (2)	39 (1)	27 (1)	43 (1)	-1 (1)	-2 (1)	3 (1)
O(2)	2542 (4)	2179 (2)	6195 (1)	70 (1)	25 (1)	57 (1)	9 (1)	-6 (1)	2 (1)
N(2)	250 (4)	5497 (2)	5505 (1)	35 (1)	27 (1)	26 (1)	3 (1)	1 (1)	1 (1)
C(N2)	-635 (6)	6099 (3)	4658 (2)	45 (2)	37 (1)	29 (1)	-4 (1)	-5 (1)	4 (1)

Table 3. *Torsion angles*

The convention used is that of the IUPAC-IUB Commission on Biochemical Nomenclature (1970).

[C(2)'-N(1)-C(1) ^z -C(1)']	ϕ_1	26.4°
[C(1)'-N(2)-C(2) ^z -C(2)']	ϕ_2	30.4
[N(1)-C(1) ^z -C(1)'-N(2)]	ψ_1	-18.6
[N(2)-C(2) ^z -C(2)'-N(1)]	ψ_2	-22.6
[C(1) ^z -C(1)'-N(2)-C(2) ^z]	ω_1	-9.3
[C(2) ^z -C(2)'-N(1)-C(1) ^z]	ω_2	-5.3

As with *c*-Sar-Val, the molecules are held together by a linear array of hydrogen bonds of length 2.90 (1) Å, each molecule having one donor site [N(1)-H] and one acceptor site [C(1)'=O(1)] with the second carbonyl group [C(2)'=O(2)] not being hydrogen bonded. The closest H...H contacts are 2.14 Å for H(1)...H(10) (intramolecular) and 2.41 Å for H(5)...H(7) (intermolecular, related by 2_1 screw parallel to *a*).

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Utahin, a Ditropolonofuran

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Abstract. C₂₀H₂₀O₅, isolated from *Juniperus utahensis* Lemm, triclinic, space group $P\bar{1}$, with $Z=2$, $a=13.106$ (3), $b=10.724$ (2), $c=7.640$ (2) Å, $\alpha=98.45$ (2), $\beta=125.03$ (3), $\gamma=99.33$ (4)°, $V=828.4$ Å³, $D_c=1.356$ g cm⁻³. The structure was determined by the application of direct methods and refined by full-matrix least squares to a final R of 0.041 for the 1257 observed intensities collected with a Philips PW 1100 computer-controlled diffractometer. The tropolone systems are only approximately planar and exhibit bond-length alternations indicating some double-bond fixation both for C-C and C-O bonds. Each part of the ditropolonoid is involved in intramolecular hydrogen bonds; only one of the parts is involved in intermolecular hydrogen bonds. The latter hold together dimers of centrosymmetrically related molecules.

Introduction. C₂₀H₂₀O₅ has been isolated from *Juniperus utahensis* Lemm, and from spectral data and chemical properties was established to be a symmetrical diisopropyliditropolonofuran (Baggaley & Norin, 1968). An investigation was undertaken to determine the complete structure and to study the delocalization of the π -electron system of this ditropolonoid.

Cell dimensions and intensities were measured from a crystal of approximate volume 0.0005 mm³ on the

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References

- ALLEN, F. H., ROGERS, D. & TROUGHTON, P. G. H. (1971). *Acta Cryst.* B31, 2561-2565.
- DAVIES, D. B. & KHALED, M. A. (1976). *J. Chem. Soc. Perkin II*. In the press.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A27, 368-376.
- GROTH, P. (1969). *Acta Chem. Scand.* 23, 3155-3162.
- International Tables for X-ray Crystallography* (1968). Vol. III, 2nd ed., pp. 201-209. Birmingham: Kynoch Press.
- IUPAC-IUB COMMISSION ON BIOCHEMICAL NOMENCLATURE (1970). *Biochemistry*, 9, 3471-3479.
- SLETTEN, E. (1970). *J. Amer. Chem. Soc.* 92, 172-177.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175-3187.
- TIMMINS, P. A. (1975). *Acta Cryst.* B31, 2561-2565.

Philips PW 1100 computer-controlled diffractometer with graphite-monochromatized Cu $K\alpha$ radiation. The lattice constants were determined by least-squares refinement of the setting angles of 25 accurately centred reflexions. The ω - 2θ scan technique was employed to measure reflexions out to $\theta=65^\circ$ at a scan speed of 0.016° s⁻¹. Backgrounds were estimated by stationary counting at ± 0.75 2θ from the peak maxima. Three reference reflexions were measured approximately every 90 min. No systematic variation was detected. 2836 unique reflexions were measured. Of these, the 1255 satisfying the condition $\sigma(I)/I \leq 0.25$ were selected for subsequent refinement. Lorentz and polarization factors were applied but no correction for absorption was made.

The absolute scale and overall temperature factor for the calculation of normalized structure factor magnitudes were estimated by Wilson's (1942) method. The structure was solved by a modified version of the *MULTAN* direct phase-determination procedure (Germain, Main & Woolfson, 1970) from the 344 reflexions with $|E| \geq 1.45$. An E map computed from well phased reflexions displayed 20 of the non-H atoms as prominent peaks. The structure was refined by the full-matrix least-squares method. When the R value reached 0.072 with anisotropic temperature factors, a