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Conformational Similarities and Structures of Two Isomeric Ten-Membered Ring Lactams, $C_{13}H_{25}NO$: (I) 4,4,7,7-Tetramethylazacyclodecan-2-one and (II) 6,6,9,9-Tetramethylazacyclodecan-2-one[*]

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Abstract. (I) $M_r = 211{\cdot}4$, monoclinic, $C2/c$, $a = 19{\cdot}002\,(10)$, $b = 16{\cdot}224\,(3)$, $c = 9{\cdot}8983\,(13)$ Å, $\beta = 121{\cdot}14\,(2)°$, $V = 2612\,(2)$ Å^3 at 138 K, $a = 19{\cdot}18\,(2)$, $b = 16{\cdot}458\,(9)$, $c = 9{\cdot}941\,(4)$ Å, $\beta = 121{\cdot}33\,(4)°$, $V = 2680\,(3)$ Å^3 at 298 K; $Z = 8$, $D_m = 1{\cdot}06$, $D_x = 1{\cdot}047$ g cm^{-3}, Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}5418$ Å, $\mu = 4{\cdot}4$ cm^{-1}, $F(000) = 944$, $R = 0{\cdot}035$ for 2217 observed reflections, $R = 0{\cdot}049$ for 2686 unique reflections. (II) $M_r = 211{\cdot}4$, monoclinic, $P2_1/c$, $a = 8{\cdot}6282\,(15)$, $b = 17{\cdot}790\,(10)$, $c = 9{\cdot}768\,(3)$ Å, $\beta = 120{\cdot}855\,(12)°$, $V = 1287{\cdot}1\,(9)$ Å^3 at 138 K, $a = 8{\cdot}728\,(8)$, $b = 17{\cdot}938\,(12)$, $c = 9{\cdot}825\,(8)$ Å, $\beta = 121{\cdot}15\,(7)°$, $V = 1316\,(2)$ Å^3 at 298 K; $Z = 4$, $D_m = 1{\cdot}07$, $D_x = 1{\cdot}067$ g cm^{-3}, Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}5418$ Å, $\mu = 4{\cdot}5$ cm^{-1}, $F(000) = 472$, $R = 0{\cdot}052$ for 2019 observed reflections, $R = 0{\cdot}077$ for 2641 unique reflections. After a reversal of the relative positions of the amide nitrogen and carbonyl positions, a close similarity is observed in the conformations of compounds (I), (II) and the unsubstituted parent compound pelargolactam. Both (I) and (II) have a *trans* amide group with the amide bond distorted from planarity. The molecules are linked by N—H···O hydrogen bonds to form chains along **c** in (I) and parallel to **a** in (II).

Introduction. Smolíková, Havel, Vašíčková, Vítek, Svoboda & Bláha (1974) have studied the effect of geminal methyl substitution on the solution conformations of nine- and ten-membered lactams. The predicted effect of such substitution is a decreased number of conformers, with *cis* or *trans* amide conformers being favored to varying degrees dependent on the location of the substitutions. Another characteristic of medium-ring lactams is the non-planarity of the amide bond (Dunitz & Winkler, 1975; Ealick & van der Helm, 1977; Ealick, Washecheck & van der Helm, 1976; Hossain, Baker & van der Helm, 1981). We report here the structures of the 4,4,7,7- and 6,6,9,9-tetramethyl derivatives of azacyclodecan-2-one.

Experimental. Both compounds obtained from Dr K. Bláha; crystals grown in thermal gradient with aqueous ethanol as solvent; D_m measured by flotation in KI solutions; (I) colorless plate, $0{\cdot}12 \times 0{\cdot}19 \times 0{\cdot}31$ mm; (II) colorless needle, $0{\cdot}1 \times 0{\cdot}1 \times 0{\cdot}42$ mm; Enraf–Nonius CAD-4 diffractometer with liquid N_2 low-temp. device; 48 reflections with $12 < \theta < 35°$, Cu $K\alpha_1$ ($\lambda = 1{\cdot}54051$ Å) at 138 K, 24 reflections with $5 < \theta < 15°$, Mo $K\alpha_1$ ($\lambda = 0{\cdot}70926$ Å) at 298 K used to refine cell constants; systematic absences: (I) hkl, $h + k = 2n + 1$, $h0l$, $l = 2n + 1$; (II) $h0l$, $l = 2n + 1$, $0k0$, $k = 2n + 1$; no absorption correction applied; $2\theta_{\max} = 150°$; (I) $-23 \le h \le 23$, $0 \le k \le 20$, $0 \le l \le 12$, (II) $-10 \le h \le 10$, $0 \le k \le 22$, $0 \le l \le 12$; three standard reflections measured after every 7200 s of X-ray exposure showed no deterioration; (I) 2686 unique reflections, 2217 observed, (II) 2641 unique reflections, 2019 observed [$I \ge 2\sigma(I)$]; structures solved by direct methods with *SHELX* (Sheldrick, 1976);

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[*] Alternative names: 4,4,7,7-tetramethylazocan-2-one and 6,6,9,9-tetramethylazocan-2-one.

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function minimized during refinement was $\sum w(|F_o| - |F_c|)^2$, with $w = 1/\sigma(F_o)^2$; H atoms located on difference Fourier maps and refined isotropically; non-H atoms refined anisotropically. (I) $R = 0\cdot049$, $R_{\rm observed} = 0\cdot035$, $R_w = 0\cdot045$, $S = 2\cdot21$; Δ/σ max. in final cycle 6%, av. 2%; final difference map e$^-$ max., min. $\pm0\cdot3$ Å^{-3}. (II) $R = 0\cdot077$, $R_{\rm observed} = 0\cdot052$, $R_w = 0\cdot061$, $S = 1\cdot77$; Δ/σ max. in final cycle 9%, av. 4%; final difference map e$^-$ max., min. $+0\cdot3$, $-0\cdot4$ Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); all calculations performed with *SHELX* (Sheldrick, 1976).

Discussion. Final parameter values for the two structures are given in Table 1.* Bond distances and angles are listed in Table 2. Equivalent bond lengths and angles are very similar in the two structures, and compare well with the libration-corrected values observed in azacyclodecan-2-one (pelargolactam) hemihydrochloride (Winkler & Dunitz, 1975*b*). For instance, the –N–C(H$_2$)–, the >C=O, the –N(H)–C(O)– and the –C(O)–C(H$_2$)– distances differ by only 0·004, 0·002, 0·004 and 0·003 Å, respectively, between the two molecules.

Stereoviews of (I) and (II) are given in Figs. 1 and 2, respectively. In both molecules, the amide group is *trans*. The close similarity in the conformations of the two compounds can be ascertained by reversing the relative positions of the amide nitrogen and carbonyl atoms. This places the methyl substituents at the same relative positions with respect to the amide bond. This comparison is made in Table 3. This same ring conformation was also found in the parent compound, azacyclodecan-2-one (pelargolactam), and Smolíková *et al.* (1974), from IR measurements, predicted this to be the main conformer for the 4,4,7,7-tetramethyl derivative and one of the conformers for the 6,6,9,9-tetramethyl derivative, in solution. It should be noted that these observed conformations place the dimethyl-substituted carbon atoms C(7) in (I) and C(6) in (II) in the most stable *Y* conformation (+*g*, +*g*) but the atoms C(4) in (I) and C(9) in (II) in the less stable *W* conformation (−*g*, +*g*). Topologically the same short transannular H···H distances occur in the two compounds: H(2C5)···H(1C8) 2·30 (3) and H(1C6)···H(1C9) 2·15 (3) Å in (I), H(1C8)···H(2C5) 2·23 (3) and H(1C7)···H(1C4) 2·06 (3) Å in (II). The conformation observed in (I) and (II) is dissimilar from the one found in the AgNO$_3$ adduct of cyclodecene (Ganis & Dunitz, 1967).

Table 1. *Positional and thermal parameters with e.s.d.'s in parentheses*

$$U_{\rm eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a^*_ia^*_j\mathbf{a}_i.\mathbf{a}_j.$$

(I)	$x(\times10^5)$	$y(\times10^5)$	$z(\times10^5)$	$U_{\rm eq}$ (Å$^2\times10^4$)
N(1)	83019 (6)	3951 (6)	34368 (11)	133 (2)
C(2)	86453 (7)	−426 (6)	27795 (12)	125 (3)
O(2)	84873 (5)	851 (5)	14143 (9)	164 (2)
C(3)	92202 (7)	−7164 (7)	38174 (13)	137 (3)
C(4)	88336 (7)	−15949 (7)	35330 (13)	132 (3)
C(5)	79853 (7)	−15817 (7)	33932 (13)	126 (3)
C(6)	72449 (7)	−13798 (7)	17499 (13)	127 (2)
C(7)	64724 (7)	−10508 (7)	17081 (13)	142 (3)
C(8)	66019 (7)	−1636 (8)	23649 (14)	155 (3)
C(9)	68056 (8)	4892 (7)	14844 (14)	162 (3)
C(10)	76102 (8)	9526 (7)	25354 (15)	164 (3)
C(11)	87819 (8)	−19944 (8)	20831 (16)	182 (3)
C(12)	94264 (8)	−20940 (8)	50080 (17)	200 (3)
C(13)	62584 (8)	−16084 (9)	26996 (15)	188 (3)
C(14)	57522 (8)	−10645 (9)	−91 (15)	191 (3)
H(N1)†	8422 (8)	262 (9)	439 (2)	31 (4)

(II)	$x(\times10^4)$	$y(\times10^5)$	$z(\times10^4)$	$U_{\rm eq}$ (Å$^2\times10^4$)
N(1)	3373 (2)	21502 (8)	2056 (2)	126 (4)
C(2)	4132 (2)	26766 (10)	1599 (2)	134 (4)
O(2)	3852 (2)	27085 (8)	223 (2)	190 (4)
C(3)	5419 (3)	32075 (11)	2887 (3)	171 (5)
C(4)	7031 (3)	27858 (11)	4241 (2)	158 (5)
C(5)	8018 (2)	22687 (11)	3666 (2)	140 (5)
C(6)	8428 (2)	14641 (10)	4351 (2)	128 (4)
C(7)	6688 (2)	10308 (10)	3932 (2)	120 (4)
C(8)	5409 (2)	8410 (10)	2157 (2)	122 (4)
C(9)	3383 (2)	7482 (10)	1603 (2)	121 (4)
C(10)	2382 (2)	15120 (11)	1055 (2)	146 (5)
C(11)	3123 (3)	4014 (12)	2911 (2)	154 (5)
C(12)	2503 (3)	2354 (12)	135 (3)	168 (5)
C(13)	9477 (3)	10574 (12)	3695 (3)	164 (5)
C(14)	9613 (3)	14905 (13)	6181 (2)	172 (5)
N(N1)‡	363 (3)	2138 (11)	306 (3)	22 (5)

† $x\times10^4$, $y\times10^4$, $z\times10^3$, $U\times10^3$.
‡ $x\times10^3$, $y\times10^4$, $z\times10^3$, $U\times10^3$.

Table 2. *Bond distances* (Å) *and angles* (°)

(I) E.s.d.'s are 0·002 Å for lengths and 0·1–0·2° for angles

N(1)–C(10)	1·459	C(4)–C(11)	1·531	C(7)–C(13)	1·536
N(1)–C(2)	1·339	C(4)–C(12)	1·535	C(7)–C(14)	1·535
C(2)–O(2)	1·241	C(4)–C(5)	1·545	C(7)–C(8)	1·546
C(2)–C(3)	1·511	C(5)–C(6)	1·538	C(8)–C(9)	1·543
C(3)–C(4)	1·561	C(6)–C(7)	1·542	C(9)–C(10)	1·530

C(10)–N(1)–C(2)	122·9	C(4)–C(5)–C(6)	116·0
N(1)–C(2)–O(2)	122·4	C(5)–C(6)–C(7)	116·1
N(1)–C(2)–C(3)	114·6	C(6)–C(7)–C(13)	110·0
O(2)–C(2)–C(3)	123·0	C(6)–C(7)–C(14)	108·4
C(2)–C(3)–C(4)	115·5	C(13)–C(7)–C(14)	108·7
C(3)–C(4)–C(11)	110·6	C(13)–C(7)–C(8)	108·5
C(3)–C(4)–C(12)	105·4	C(14)–C(7)–C(8)	109·6
C(11) C(4)–C(12)	109·2	C(6)–C(7)–C(8)	111·6
C(11)–C(4)–C(5)	111·3	C(7)–C(8)–C(9)	115·7
C(12)–C(4)–C(5)	108·1	C(8)–C(9)–C(10)	114·1
C(3)–C(4)–C(5)	112·0	C(9)–C(10)–N(1)	112·3

(II) E.s.d.'s are 0·002–0·003 Å for lengths and 0·2° for angles

N(1)–C(10)	1·455	C(4)–C(5)	1·541	C(7)–C(8)	1·541
N(1)–C(2)	1·343	C(5)–C(6)	1·542	C(8)–C(9)	1·551
C(2)–O(2)	1·239	C(6)–C(13)	1·532	C(9)–C(11)	1·535
C(2)–C(3)	1·508	C(6)–C(14)	1·539	C(9)–C(12)	1·532
C(3)–C(4)	1·536	C(6)–C(7)	1·545	C(9)–C(10)	1·550

C(10)–N(1)–C(2)	122·6	C(14)–C(6)–C(7)	107·4
N(1)–C(2)–O(2)	122·4	C(5)–C(6)–C(7)	111·9
N(1)–C(2)–C(3)	115·8	C(6)–C(7)–C(8)	116·5
O(2)–C(2)–C(3)	121·8	C(7)–C(8)–C(9)	116·4
C(2)–C(3)–C(4)	111·5	C(8)–C(9)–C(11)	111·8
C(3)–C(4)–C(5)	113·8	C(8)–C(9)–C(12)	108·6
C(4)–C(5)–C(6)	116·3	C(11)–C(9)–C(12)	109·0
C(5)–C(6)–C(13)	108·1	C(11)–C(9)–C(10)	110·3
C(5)–C(6)–C(14)	110·1	C(12)–C(9)–C(10)	106·1
C(13)–C(6)–C(14)	108·5	C(8)–C(9)–C(10)	110·8
C(13)–C(6)–C(7)	110·8	C(9)–C(10)–N(1)	114·9

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* Lists of structure factors, anisotropic thermal parameters and H-atom positional and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39699 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The amide torsion angles, −167·1 (2) and −168·0 (2)°, for (I) and (II), respectively, indicate significant non-planarity of the amide bond. The non-planarity parameters (Winkler & Dunitz, 1971) given in Table 3 show that the distortion is due primarily to out-of-plane bonding at the N atom, as is most common for amide and peptide bonds. Similar, but more pronounced, non-planarity has been observed in azacyclononan-2-one (caprylolactam) (Winkler & Dunitz, 1975*a*) and in its 4,4,7,7-tetramethyl derivative (Hossain, Baker & van der Helm, 1981), where decreasing the ring to nine atoms results in increased strain on the amide bond. Winkler & Dunitz (1971) (and Dunitz & Winkler, 1975) point out that the out-of-plane deformations of amide bonds require very little energy, so that increasing the ring size further might lead to less obvious changes in the planarity of the amide bond.

Packing interactions of (I) and (II) are shown in Figs. 3 and 4, respectively. In both cases, the N atom donates

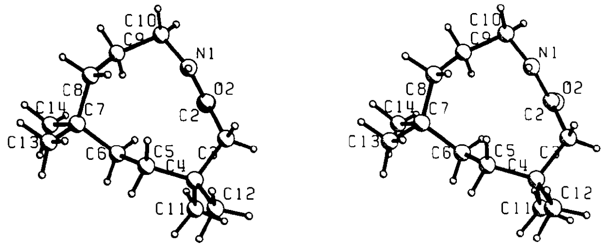

Fig. 1. Stereoview of (I) with numbering scheme.

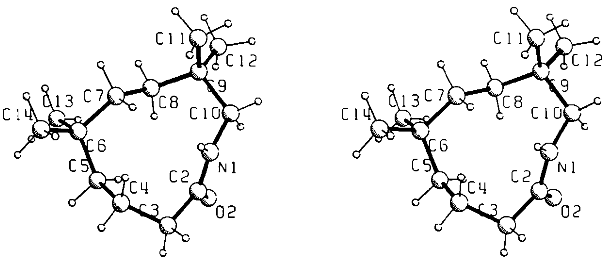

Fig. 2. Stereoview of (II) with numbering scheme.

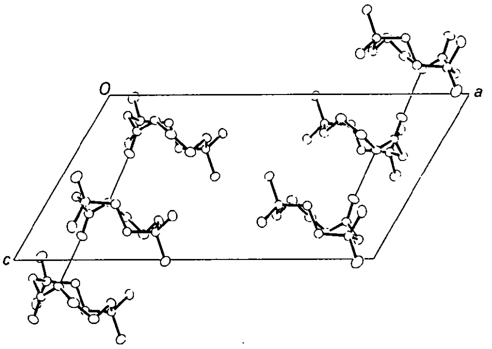

Fig. 3. Layer of hydrogen-bonded molecules (I) parallel to the *ac* plane. The fine lines represent the N(1)···O(2) hydrogen bonds.

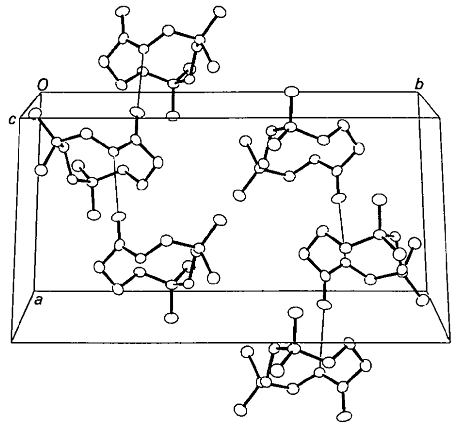

Fig. 4. Packing of (II) showing the chains of hydrogen-bonded molecules running parallel to the *a* axis. The fine lines represent the N(1)···O(2) hydrogen bonds.

Table 3. *Selected torsion angles and amide non-planarity parameters* (°)*for the* 4,4,7,7 (I) *and* 6,6,9,9 (II) *tetramethyl derivatives and for azacyclodecan-2-one hemihydrochloride* (III) (*Winkler & Dunitz*, 1975*b*); *e.s.d.'s are:* (I) 0·2° (1° *for angles to* H); (II) 0·2–0·3° (1–2° *for angles to* H); (III) 1–2° *for angles not involving* H *atoms*

		(I)		(II)	(III)
$\omega(1)$	C(10)–N(1)–C(2)–C(3)	−167·1	C(3)–C(2)–N(1)–C(10)	−168·0	−168·3
	N(1)–C(2)–C(3)–C(4)	96·3	C(2)–N(1)–C(10)–C(9)	106·7	95·4
	C(2)–C(3)–C(4)–C(5)	−45·4	N(1)–C(10)–C(9)–C(8)	−41·5	−49·5
	C(3)–C(4)–C(5)–C(6)	84·4	C(10)–C(9)–C(8)–C(7)	89·2	84·1
	C(4)–C(5)–C(6)–C(7)	−158·1	C(9)–C(8)–C(7)–C(6)	−153·2	−147·1
	C(5)–C(6)–C(7)–C(8)	71·3	C(8)–C(7)–C(6)–C(5)	67·1	78·5
	C(6)–C(7)–C(8)–C(9)	59·9	C(7)–C(6)–C(5)–C(4)	60·2	50·2
	C(7)–C(8)–C(9)–C(10)	−123·4	C(6)–C(5)–C(4)–C(3)	−132·4	−118·5
	C(8)–C(9)–C(10)–N(1)	52·4	C(5)–C(4)–C(3)–C(2)	52·6	52·1
	C(9)–C(10)–N(1)–C(2)	72·8	C(4)–C(3)–C(2)–N(1)	61·0	75·5
$\omega(2)$	O(2)–C(2)–N(1)–H(N1)	177	O(2)–C(2)–N(1)–H(N1)	178	178
$\omega(3)$	O(2)–C(2)–N(1)–C(10)	11·3	O(2)–C(2)–N(1)–C(10)	9·3	10·7
$\omega(4)$	C(3)–C(2)–N(1)–H(N1)	−1	C(3)–C(2)–N(1)–H(N1)	1	−1

	(I)	(II)	(III)
$\chi_C = (\pi+\omega_1-\omega_3)(\mathrm{mod}2\pi)$	1·6 (3)	2·7 (4)	1 (1)
$\chi_N = (\pi+\omega_2-\omega_3)(\mathrm{mod}2\pi)$	−14 (1)	−11 (2)	−13 (5)
$\tau = (\omega_1+\omega_2)/2$	5 (1)	5 (1)	−5 (3)

in a hydrogen bond to the O atom of a glide-plane related molecule to form chains of molecules. The bond lengths are: N(1)···O(2) at $x, -y, \frac{1}{2}+z$ 2·888 (2) Å (I), N(1)···O(2) at $x, \frac{1}{2}-y, \frac{1}{2}+z$ 2·914 (3) Å (II).

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Structure of 2-Isopropylcrotonic Acid, $C_7H_{12}O_2$

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Abstract. $M_r = 128·172$, monoclinic, $P2_1/c$, $a = 5·507$ (1), $b = 10·206$ (1), $c = 13·717$ (1) Å, $\beta = 97·15$ (1)°, $V = 764·96$ Å^3, $Z = 4$, $D_x = 1·113$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·861$ cm^{-1}, $F(000) = 280$, $T = 295$ K, $R = 0·045$ for 884 observed reflections. The molecules are hydrogen-bonded in pairs to form centrosymmetric dimers. The molecules are inclined with respect to the *c*-glide planes, so that stereochemical control of reactions on the (010) planes is expected to be more difficult than if the molecules were parallel to the glide planes.

Introduction. The stereochemistry of addition to alkenes can be controlled by forcing the reaction to occur on one surface of a single crystal (Holland & Richardson, 1980; Chenchaiah, Holland & Richardson, 1982). 2-Isopropylcrotonic acid is a precursor to a number of biochemically interesting compounds, but its structure has not been previously determined. We have solved its structure to determine which way the alkene portion of the molecule is aligned with respect to the crystallographic symmetry elements, as this knowledge is essential in order to choose the appropriate surface for asymmetric reaction.

Experimental. White needles from slow evaporation of a 20% aqueous acetone solution, 0·1 × 0·1 × 0·5 mm, sealed in a thin-walled capillary, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$, lattice parameters from 25 reflections with $8 < \theta < 15°$, one standard reflection measured every hour – max. variation 2·8%, 1672 reflections (h −6→6, k 0→12, l 0–16) with $\theta < 25°$, 1344 independent, 884 with $I > 3\sigma(I)$, Lp correction, no absorption correction, direct methods, refinement by full-matrix least squares on F's, all non-H atoms anisotropic, all H atoms located on a difference map. Six peaks about the C(7) methyl group indicated twofold disorder. H-atom positions refined, C(7)—H distances and H—C(7)—H angles constrained to be 1·08 Å and 109·5°, isotropic temperature factors refined for methyl H atoms as a group and for other H atoms as a group, 130 parameters less 12 constraints, max. shift/σ = 0·51 on final cycle, $w = [\sigma^2(F) + 0·0011F^2]^{-1}$, $R = 0·045$, $R_w = 0·051$, final difference map with no features greater than 0·15 e Å^{-3} or less than −0·20 e Å^{-3}, scattering factors from *International Tables for X-ray Crystallography* (1974), computer programs *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1976).

Discussion. The atomic coordinates are listed in Table 1, and bond distances and angles are given in Table 2.

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