located in an asymmetrical potential energy well. A recently published high resolution solid-state ^{13}C CP-MAS NMR spectrum of (I) lends support to this claim since two clearly resolved ^{13}C peaks ($\Delta\sigma_i = 5{\cdot}3$ p.p.m.) are seen for the carbonyl carbon and the enol carbon (Imashiro, Maeda, Takegoshi, Terao & Saika, 1982), indicating that these atoms are not being dynamically averaged. If the proton were rapidly exchanging between two equivalent positions, a single averaged isotropic chemical shift would be seen. Attempts to obtain a ^{13}C CP-MAS NMR spectrum of (II) have been unsuccessful owing to the facile phase change to (I) during sample preparation.

A small difference between the structures of (I) and (II) is found in the torsion angles between the least-squares planes of the central hydrogen-bonded ring and the phenyl rings. In (I), the two phenyl torsion angles are 4 and 17° (Hollander, Templeton & Zalkin, 1973), but in (II) they are 8 (3) and 24 (3)°.

The unit-cell packing patterns of (I) and (II) are shown viewed along their short axes, *c* and *b* respectively, in Fig. 2. Despite the strikingly similar unit-cell parameters and identical space groups [(I), *Pbca*, $Z = 8$, $a = 10{\cdot}853$ (1), $b = 24{\cdot}441$ (1), $c = 8{\cdot}7559$ (1) Å; Jones (1976)] the packing patterns show no significant similarities. There are no unusually short intermolecular contacts in (II) and no evidence for any intermolecular hydrogen bonds. It has been suggested that form (I) may be stabilized by a weak intermolecular hydrogen bond between C(2)—H and O(1) on neighboring *a*-glide-related molecules (D. Y. Curtin, private communication). The C(2)···O(1) distance in (I) is 3·70 Å (C—H···O angle, 141°), and no such contact is found in (II).

This crystal structure and the previously reported structures of (I) provide the first opportunity to study the effect of two different solid-state environments on the structure of a DKM compound. For DBM, we have found that for room-temperature X-ray data, the solid-state packing patterns are markedly different but the intramolecular geometries, including the position of the enol H atom, are virtually identical. These results are in contrast to those of Herbstein, Kapon, Reisner, Duesler, Paul & Curtin (1985) who found that in the multiple polymorphs of naphthazarin the properties of the enolized proton and of its hydrogen bonds are very sensitive to differences in crystal packing patterns.

Facile solvent-assisted (Cardew & Davey, 1985) and solid-state phase changes from (II) to (I) were observed. A detailed study of these transformation mechanisms is in progress.

Helpful discussions with Professor Doyle Britton, University of Minnesota, are gratefully acknowledged.

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Structures of (+)-(*R*)-α-Methylbenzylammonium (+)-(1*S*,6*R*,7*S*)-Bicyclo[4.1.0]hept-2-ene-7-carboxylate (I) and (+)-(*R*)-α-Methylbenzylammonium (+)-(1*R*,8*S*,9*R*)-Bicyclo[6.1.0]non-2-ene-9-carboxylate (II)

By Kazunori Yanagi and Tadatoshi Aratani

Takatsuki Research Laboratory, Sumitomo Chemical Co. Ltd, 2-10-1, *Tsukahara, Takatsuki, Osaka* 569, *Japan*

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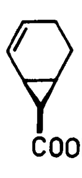
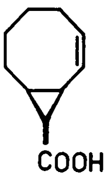
Abstract. (I): $C_8H_{12}N^+.C_8H_9O_2^-$, $M_r = 259{\cdot}35$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}582$ (1), $b = 19{\cdot}537$ (2), $c = 6{\cdot}159$ (1) Å, $V = 1513{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}14$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu(\text{Cu}\,K\alpha) = 0{\cdot}56$ mm^{-1}, $T = 295$ K, $F(000) = 560$, $R = 0{\cdot}045$ for 1218 observed reflections. (II): $C_8H_{12}N^+.C_{10}H_{13}O_2^-$,

0108-2701/87/020263-04$01.50

M_r = 287·41, monoclinic, $P2_1$, a = 15·625 (1), b = 5·639 (2), c = 9·717 (1) Å, β = 101·87 (1)°, V = 837·8 Å^3, Z = 2, D_x = 1·14 Mg m^{-3}, Cu $K\alpha$, λ = 1·5418 Å, μ(Cu $K\alpha$) = 0·55 mm^{-1}, T = 295 K, $F(000)$ = 312, R = 0·049 for 960 observed reflections. Both title complexes consist of cationic and anionic components linked by three hydrogen bonds, forming infinite helical chains parallel to the c axis [complex (I)] or b axis [complex (II)]. The absolute configurations of the anionic components of (I) and (II) were deduced from the known absolute configuration of (+)-(R)-α-methylbenzylammonium.

Introduction. In the presence of olefins the decomposition of an alkyl diazoacetate catalyzed by a chiral Schiff base–copper complex gives an optically active alkyl cyclopropanecarboxylate (Nozaki, Takaya, Moriuti & Noyori, 1968; Aratani, Yoneyoshi & Nagase, 1982; Aratani, 1985). For instance, the title anionic components, (+)-bicyclo[4.1.0]-hept-2-ene-7-carboxylic acid (I*a*) and (+)-bicyclo-[6.1.0]non-2-ene-9-carboxylic acid (II*a*), were prepared by the reaction of ethyl diazoacetate with 1,3-cyclohexadiene and 1,3-cyclooctadiene, respectively. As part of a series of studies on the steric influence of the reaction (Yanagi, Minobe & Aratani, 1986), the crystal structures of the title complexes have been determined by X-ray analysis and the absolute configurations of the anionic components have been ascertained from that of the cation.


(I*a*)


(II*a*)

Experimental. Complex (I): crystal 0·4 × 0·3 × 0·3 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu$K\alpha$ radiation. Cell dimensions refined by least-squares fitting of θ values of 25 reflections with $20 < \theta < 27°$. ω–2θ scan technique, scan angle 1·0° + 0·15° tanθ, three reference reflections monitored every 80 min throughout data collection showed no significant deterioration. Correction for Lorentz and polarization effects, but not for absorption. 1703 unique reflections measured with $2 < \theta < 70°$; 1218 with $I > 3\sigma(I)$ used for structure determination. h 0 to 15, k 0 to 23, l 0 to 7. Structure solved by direct methods with *MITHRIL* (Gilmore, 1984). H atoms located on a difference Fourier map. Structure refined by full-matrix least squares; $\sum w(|F_o| - |F_c|)^2$ minimized. For H atoms, the thermal parameters were refined isotropically; non-H atoms refined anisotropically. Final R = 0·045, wR = 0·064 for 257 variables, $w = 4I/[\sigma^2(I) + (0·05I)^2]$, S = 2·083, $(\Delta/\sigma)_{max}$ = 0·21, extinction coefficient refined to 4·3 (9) × 10^{-6}. Max. and min. peaks in final difference Fourier map 0·17 and −0·13 e Å^{-3}, respectively. Complex (II): crystal 0·4 × 0·3 × 0·3 mm. Correction for Lorentz and polarization effects, but not for absorption, or secondary extinction. 1758 unique reflections measured with $2 < \theta < 70°$; 960 with $I > 3\sigma(I)$. h −19 to 19, k 0 to 6, l 0 to 11. Structure solved by direct methods with *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H positions refined with a fixed thermal parameter (6 Å^2). Final R = 0·049, wR = 0·060 for 265 variables, $w = 4I/[\sigma^2(I) + (0·05I)^2]$, S = 1·902, $(\Delta/\sigma)_{max}$ = 0·28, max. and min. peaks in final difference Fourier map 0·17 and −0·17 e Å^{-3}. Other details same as for complex (I).

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations were carried out using *MITHRIL* and the *SDP* program system (Frenz, 1983).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Compound (I)				
O(1)	0·1714 (2)	0·43595 (9)	0·5249 (4)	5·53 (4)
O(2)	0·1735 (2)	0·3731 (1)	0·2276 (4)	6·27 (5)
N(1)	0·3452 (2)	0·5187 (1)	0·4406 (4)	4·45 (5)
C(1)	0·1181 (3)	0·2526 (2)	0·4921 (8)	6·39 (8)
C(2)	0·1167 (4)	0·2019 (2)	0·6706 (10)	9·5 (1)
C(3)	0·0323 (4)	0·1950 (3)	0·7985 (9)	11·0 (1)
C(4)	−0·0658 (4)	0·2298 (2)	0·7575 (12)	10·8 (1)
C(5)	−0·0812 (3)	0·2568 (2)	0·5436 (11)	8·6 (1)
C(6)	0·0153 (3)	0·2829 (2)	0·4323 (7)	6·19 (8)
C(7)	0·0957 (2)	0·3264 (1)	0·5435 (6)	4·90 (6)
C(8)	0·1514 (2)	0·3818 (1)	0·4231 (5)	4·50 (6)
C(9)	0·4426 (2)	0·4754 (1)	0·4568 (5)	4·77 (6)
C(10)	0·4422 (3)	0·4377 (2)	0·6746 (6)	6·50 (8)
C(11)	0·5401 (2)	0·5182 (1)	0·4174 (5)	4·42 (6)
C(12)	0·5992 (3)	0·5114 (2)	0·2316 (6)	5·68 (7)
C(13)	0·6881 (3)	0·5507 (2)	0·1955 (7)	6·70 (9)
C(14)	0·7187 (3)	0·5984 (2)	0·3451 (8)	7·1 (1)
C(15)	0·6611 (3)	0·6058 (2)	0·5356 (8)	7·51 (9)
C(16)	0·5732 (3)	0·5661 (2)	0·5722 (7)	6·30 (8)
Compound (II)				
O(1)	0·3821 (2)	0·5	0·7109 (3)	6·10 (8)
O(2)	0·4301 (2)	0·2198 (7)	0·5870 (3)	5·84 (8)
N(1)	0·5075 (2)	0·7905 (8)	0·6594 (3)	4·73 (8)
C(1)	0·2686 (3)	0·1552 (11)	0·8100 (5)	5·1 (1)
C(2)	0·2517 (3)	−0·0717 (12)	0·8767 (5)	6·8 (1)
C(3)	0·1802 (4)	−0·1540 (13)	0·9106 (6)	8·1 (2)
C(4)	0·0877 (4)	−0·0602 (19)	0·8821 (7)	12·2 (2)
C(5)	0·0684 (4)	0·1742 (20)	0·8221 (6)	10·9 (2)
C(6)	0·0559 (3)	0·1808 (22)	0·6630 (7)	11·1 (3)
C(7)	0·1325 (3)	0·1123 (17)	0·6057 (5)	8·2 (2)
C(8)	0·2152 (3)	0·2442 (11)	0·6741 (5)	5·6 (1)
C(9)	0·3036 (3)	0·1397 (11)	0·6758 (4)	5·2 (1)
C(10)	0·3761 (3)	0·2982 (10)	0·6589 (4)	4·6 (1)
C(11)	0·5763 (3)	0·8174 (10)	0·7914 (5)	4·9 (1)
C(12)	0·5359 (3)	0·9580 (12)	0·8971 (4)	5·8 (1)
C(13)	0·6568 (3)	0·9370 (11)	0·7601 (4)	5·1 (1)
C(14)	0·7395 (3)	0·8318 (12)	0·8009 (5)	6·3 (1)
C(15)	0·8108 (3)	0·9359 (16)	0·7665 (7)	8·3 (2)
C(16)	0·8046 (3)	1·1370 (17)	0·6934 (6)	8·3 (2)
C(17)	0·7264 (3)	1·2524 (12)	0·6584 (5)	7·2 (2)
C(18)	0·6511 (3)	1·1493 (12)	0·6876 (5)	5·8 (1)

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms of complexes (I) and (II) are listed in Table 1.* Bond distances, bond angles and selected torsion angles are given in Table 2. Fig. 1 is an *ORTEP* drawing (Johnson, 1976) of both complexes with atomic numbering. From the known configuration of (+)-(*R*)-α-methylbenzylammonium, the absolute structures of the anionic components of (I) and (II) were determined to be (1*S*, 6*R*, 7*S*) and (1*R*, 8*S*, 9*R*), respectively. In complex (I), the conformation of the cyclohexene ring takes nearly the 1,3-diplanar form. This conformation is a rather higher energy form than the monoplanar form which is the most stable conformation of cyclohexene (Bucourt, 1974). The values for the torsion angle C(1)—C(2)=C(3)—C(4) in (I) and (II) are 7·4 (8) and 6·7 (11)° respectively.

The crystal structures of complexes (I) and (II) are shown in Figs. 2 and 3. In both structures, the cationic components are linked by three hydrogen bonds to anionic components related by a twofold screw axis, giving rise to an infinite helical chain parallel to the *c* axis [complex (I)] or *b* axis [complex (II)]. Hydrogen-bond distances are listed in Table 3. No other intermolecular contacts shorter than the sum of the van der Waals radii have been detected.

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

Compound (I)		Compound (II)	
O(1)–C(8)	1·256 (3)	O(1)–C(10)	1·241 (5)
O(2)–C(8)	1·247 (3)	O(2)–C(10)	1·279 (5)
N(1)–C(9)	1·492 (3)	N(1)–C(11)	1·502 (5)
C(1)–C(2)	1·480 (6)	C(1)–C(2)	1·480 (7)
C(1)–C(6)	1·469 (4)	C(1)–C(8)	1·497 (6)
C(1)–C(7)	1·503 (4)	C(1)–C(9)	1·517 (5)
C(2)–C(3)	1·329 (8)	C(2)–C(3)	1·314 (7)
C(3)–C(4)	1·431 (8)	C(3)–C(4)	1·510 (12)
C(4)–C(5)	1·432 (9)	C(4)–C(5)	1·451 (14)
C(5)–C(6)	1·485 (5)	C(5)–C(6)	1·518 (11)
C(6)–C(7)	1·488 (4)	C(6)–C(7)	1·471 (10)
C(7)–C(8)	1·487 (4)	C(7)–C(8)	1·521 (8)
C(9)–C(10)	1·530 (4)	C(8)–C(9)	1·498 (6)
C(9)–C(11)	1·504 (3)	C(9)–C(10)	1·479 (6)
C(11)–C(12)	1·371 (4)	C(11)–C(12)	1·532 (6)
C(11)–C(16)	1·400 (4)	C(11)–C(13)	1·511 (6)
C(12)–C(13)	1·375 (5)	C(13)–C(14)	1·404 (6)
C(13)–C(14)	1·366 (5)	C(13)–C(18)	1·383 (6)
C(14)–C(15)	1·386 (6)	C(14)–C(15)	1·360 (8)
C(15)–C(16)	1·370 (5)	C(15)–C(16)	1·331 (9)
		C(16)–C(17)	1·364 (9)
		C(17)–C(18)	1·393 (7)
C(2)–C(1)–C(6)	116·5 (3)	C(2)–C(1)–C(8)	124·0 (5)
C(2)–C(1)–C(7)	118·9 (4)	C(2)–C(1)–C(9)	117·1 (5)
C(6)–C(1)–C(7)	60·1 (2)	C(8)–C(1)–C(9)	59·6 (3)
C(1)–C(2)–C(3)	121·1 (4)	C(1)–C(2)–C(3)	130·7 (6)
C(2)–C(3)–C(4)	122·5 (4)	C(2)–C(3)–C(4)	131·4 (7)
C(3)–C(4)–C(5)	117·0 (5)	C(3)–C(4)–C(5)	120·3 (7)
C(4)–C(5)–C(6)	116·1 (4)	C(4)–C(5)–C(6)	113·8 (9)
C(1)–C(6)–C(5)	117·7 (3)	C(5)–C(6)–C(7)	115·8 (6)
C(1)–C(6)–C(7)	61·1 (2)	C(6)–C(7)–C(8)	112·9 (6)
C(5)–C(6)–C(7)	122·7 (4)	C(1)–C(8)–C(7)	119·4 (5)
C(1)–C(7)–C(6)	58·9 (2)	C(1)–C(8)–C(9)	60·8 (3)
C(1)–C(7)–C(8)	120·3 (3)	C(7)–C(8)–C(9)	120·7 (5)
C(6)–C(7)–C(8)	120·4 (3)	C(1)–C(9)–C(8)	59·5 (3)
O(1)–C(8)–O(2)	123·5 (2)	C(1)–C(9)–C(10)	118·8 (4)
O(1)–C(8)–C(7)	117·3 (2)	C(8)–C(9)–C(10)	119·1 (5)
O(2)–C(8)–C(7)	119·2 (2)	O(1)–C(10)–O(2)	122·5 (4)
N(1)–C(9)–C(10)	109·2 (2)	O(1)–C(10)–C(9)	120·3 (4)
N(1)–C(9)–C(11)	110·1 (2)	O(2)–C(10)–C(9)	117·3 (5)
C(10)–C(9)–C(11)	114·3 (2)	N(1)–C(11)–C(12)	107·6 (4)
C(9)–C(11)–C(12)	121·5 (2)	N(1)–C(11)–C(13)	110·6 (3)
C(9)–C(11)–C(16)	120·3 (3)	C(12)–C(11)–C(13)	112·3 (4)
C(12)–C(11)–C(16)	118·1 (3)	C(11)–C(13)–C(14)	120·7 (5)
C(11)–C(12)–C(13)	121·5 (3)	C(11)–C(13)–C(18)	121·3 (4)
C(12)–C(13)–C(14)	120·1 (4)	C(14)–C(13)–C(18)	117·9 (5)
C(13)–C(14)–C(15)	119·7 (3)	C(13)–C(14)–C(15)	120·1 (6)
C(14)–C(15)–C(16)	120·1 (3)	C(14)–C(15)–C(16)	121·7 (6)
C(11)–C(16)–C(15)	120·5 (3)	C(15)–C(16)–C(17)	120·2 (6)
		C(16)–C(17)–C(18)	120·1 (6)
		C(13)–C(18)–C(17)	119·8 (5)
C(6)–C(1)–C(2)–C(3)	−13·6 (7)	C(8)–C(1)–C(2)–C(3)	−53·6 (9)
C(2)–C(1)–C(6)–C(5)	−4·3 (6)	C(2)–C(1)–C(8)–C(7)	7·0 (8)
C(1)–C(2)–C(3)–C(4)	7·4 (8)	C(1)–C(2)–C(3)–C(4)	6·7 (11)
C(2)–C(3)–C(4)–C(5)	17·4 (7)	C(2)–C(3)–C(4)–C(5)	−8·7 (11)
C(3)–C(4)–C(5)–C(6)	−34·4 (6)	C(3)–C(4)–C(5)–C(6)	83·4 (8)
C(4)–C(5)–C(6)–C(1)	27·8 (6)	C(4)–C(5)–C(6)–C(7)	−62·5 (11)
		C(5)–C(6)–C(7)–C(8)	−50·9 (12)
		C(6)–C(7)–C(8)–C(1)	83·7 (8)

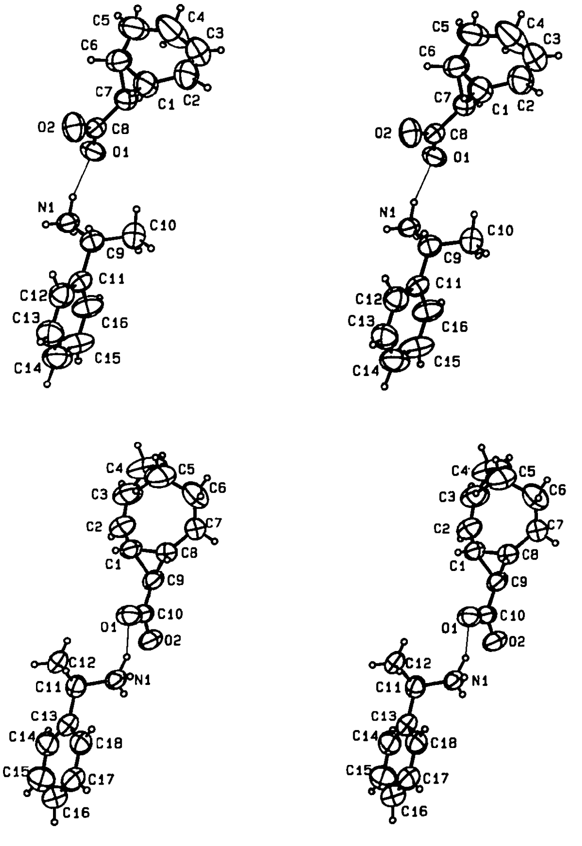


Fig. 1. *ORTEP* drawings (Johnson, 1976) of complex (I) (upper) and (II) (lower) with the atom numbering. The ellipsoids are drawn at the 50% probability level.

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms, and positional and thermal parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43360 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

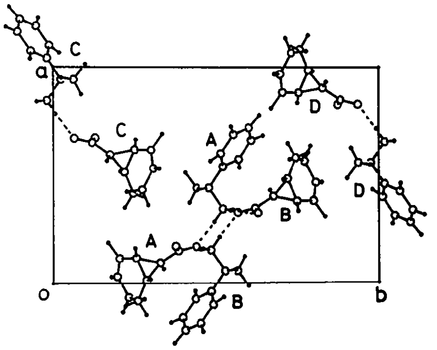

Fig. 2. The crystal structure of (I) viewed along the *c* axis. The hydrogen bonds are indicated by broken lines. Symmetry code: (*A*) x, y, z; (*B*) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (*C*) $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$; (*D*) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$.

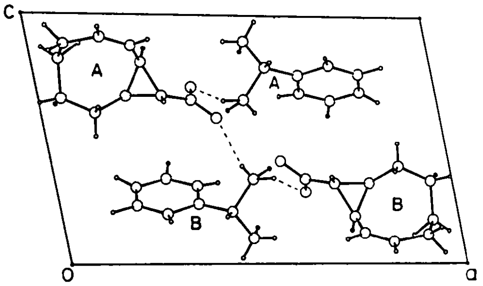

Fig. 3. The crystal structure of (II) viewed along the *b* axis. The hydrogen bonds are indicated by broken lines. Symmetry code: (*A*) x, y, z; (*B*) $1-x, -\frac{1}{2}+y, 1-z$.

Table 3. *Hydrogen-bond distances* (Å)

Compound (I)		Compound (II)	
N(1)–H···O(1)	2·767 (3)	N(1)–H···O(1)	2·679 (5)
N(1)–H···O(2^{i})	2·717 (3)	N(1)–H···O(2^{iii})	2·733 (5)
N(1)–H···O(2^{iii})	2·766 (3)	N(1)–H···O(2^{iv})	2·793 (4)

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

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Substituent Effects of π Acceptors to Prismane. Structure of Methyl 2,3,5,6-Tetramethyl-4-phenylprismanecarboxylate* and Theoretical Calculations on Formylprismane

BY HERMANN IRNGARTINGER, DIETMAR KALLFASS, EDWIN LITTERST AND ROLF GLEITER

Organisch-Chemisches Institut der Universität, Im Neuenheimer Feld 270, *D*-6900 *Heidelberg, Federal Republic of Germany*

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Abstract. $C_{18}H_{20}O_2$, $M_r = 268·36$, triclinic, $P\bar{1}$, $a = 7·275$ (1), $b = 10·244$ (2), $c = 10·350$ (2) Å, $\alpha = 81·20$ (2), $\beta = 80·63$ (2), $\gamma = 77·70$ (2)°, $V = 738·0$ (3) Å^3, $Z = 2$, $D_x = 1·21$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 0·0720$ mm^{-1}, $F(000) = 288$, $T = 89$ (2) K, $R = 0·059$ for 2632 unique observed reflections [$I > 3\sigma(I)$]. The carbonyl group in the title compound has a nearly bisected conformation relative to the adjacent three-membered ring of the prismane system. This is attributed to favourable electronic interactions. As a result the distal bond of this ring is shortened [1·499 (2) Å] and the vicinal bonds are lengthened [1·541 (2) and 1·564 (2) Å]. MO and *ab*

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* IUPAC name: methyl 2,3,5,6-tetramethyl-4-phenyltetracyclo-[2.2.0.0^{2,6}.0^{3,5}]hexanecarboxylate.