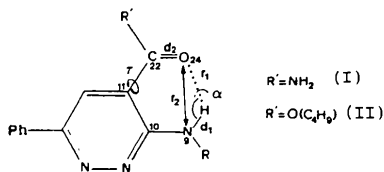


Table 2. Selected parameters of the intramolecular hydrogen bond observed in the 4-carbamoyl and 4-butoxycarbonyl analogs



	Compound (I)		Compound (II)
	'10'	'100'	
d_1 (Å)	1.098 (6)	1.112 (6)	0.980 (3)
d_2 (Å)	1.237 (9)	1.228 (9)	1.200 (4)
r_1 (Å)	1.830 (6)	1.818 (3)	2.004 (3)
r_2 (Å)	2.642 (9)	2.649 (7)	2.722 (4)
α (°)	127.1 (4)	127.7 (4)	128.3 (1)
τ (°)	6.6 (12)	7.9 (11)	0.4 (6)
Deviations (Å) from the mean plane of the pyridazine ring			
C(22)	0.008	0.006	0.025
O(24)	0.145	0.162	0.020
N(9)	0.063	0.026	-0.057
H(9)	0.091	0.172	-0.097
E.s.d.'s (Å)	0.013	0.002	0.010

Table 3. Intermolecular hydrogen-bond distances (Å) and angles (°) for compound (I)

N...H—O	N...O	H—O	N...H	\angle N...H—O
N(4')...H(992)—O(99 ⁱⁱ)	2.877 (7)	1.176 (4)	1.734 (6)	162.5 (3)
N(14')...H(991)—O(99 ⁱⁱⁱ)	2.905 (7)	0.954 (4)	2.087 (6)	142.8 (3)
N(104')...H(552)—O(55 ^v)	2.903 (8)	1.083 (4)	1.849 (6)	163.0 (2)
N—H...N	N...N	N—H	H...N	\angle N—H...N
N(23')—H(232)...N(115')	2.954 (9)	1.088 (6)	2.026 (6)	141.3 (4)
N—H...O	N...O	N—H	H...O	\angle N—H...O
N(23')—H(231)...O(55 ^{iv})	2.868 (7)	1.067 (6)	1.872 (3)	153.7 (4)
N(123')—H(123)...O(99')	3.047 (6)	1.089 (6)	1.984 (3)	167.7 (2)
Symmetry code: (i) x, y, z ; (ii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $-x - \frac{1}{2}, y, z - \frac{1}{2}$; (iv) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (v) $x - \frac{1}{2}, -y + 1, -z + \frac{1}{2}$.				

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Structure of a Tricyclo[4.3.0.0^{3,7}]nonane Derivative

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Abstract. 4-(5,8-Diacetyl-6,7-dihydroxy-3-methyltricyclo[4.3.0.0^{3,7}]non-1-yl)-2-butanone, C₁₈H₂₆O₅, $M_r = 322.40$, monoclinic, $P2_1/c$, $a = 13.762$ (2), $b = 10.507$ (2), $c = 12.110$ (1) Å, $\beta = 97.338$ (8)°, $V =$

The morpholino ring of analog (I) is in the chair form and the methylene groups 7 and 8 are in the *anti* position [N(4)—C(7)—C(8)—N(9): 170.0 (6) and 168.4 (6)° respectively]. In the 4-butoxycarbonyl derivative, these groups are in the +synclinal conformation [N(4)—C(7)—C(8)—N(9): 57.7 (5)°].

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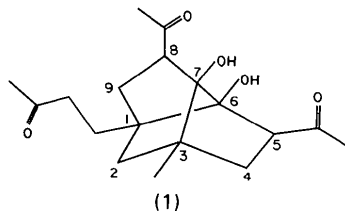
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1736.7 (4) Å³, $Z = 4$, $D_x = 1.233$ Mg m⁻³, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 0.69$ mm⁻¹, $F(000) = 696$, $T = 293$ K, $R = 0.050$ for 2761 unique reflections. It has been confirmed that the compound formed by Michael

reaction of 3-methyl-1,2-cyclopentanedione with excess methyl vinyl ketone has the novel tricyclononane skeleton. Endocyclic bond angles at positions 1, 3, 6 and 7 are remarkably decreased, 94.0 (1)–100.7 (2)°, while the exocyclic angles are increased, 118.2 (1)–116.9 (1)°, indicating a considerable strain in the skeleton. The C–C bond lengths lie in a similar range as found in some 8,9,10-trinorbornane rings: 1.529 (1)–1.557 (3) Å. The two six-membered rings take boat conformations, and the three five-membered rings pseudo C₂ conformations.

Introduction. 4-(5,8-Diacetyl-6,7-dihydroxy-3-methyl-tricyclo[4.3.0.0^{3,7}]non-1-yl)-2-butanone (1) was obtained as a Michael-reaction adduct of 1 mol of 3-methyl-1,2-cyclopentanedione with 3 mol of methyl vinyl ketone under basic conditions (Utaka, Fujii & Takeda, unpublished). Since such a Michael reaction is unprecedented and the structure of the adduct could not be unambiguously determined by spectral analyses (IR, ¹H and ¹³C NMR), X-ray structure analysis of (1) has been carried out, and its novel tricyclo[4.3.0.0^{3,7}]nonane skeleton has been confirmed.



Experimental. Colorless prismatic crystal (slow evaporation from ether solution) elongated along *c* with developed faces {100}, dimensions 0.30 × 0.28 × 0.35 mm. Rigaku AFC-5 four-circle diffractometer. Lattice parameters determined with 20 reflections in the range 10 < 2θ < 47° by least-squares method; intensities measured up to sinθ/λ 0.5753 Å⁻¹, ω–2θ scan method [scan speed 4° min⁻¹ in ω, scan range (2θ): 1.2° + 0.15° tanθ], Ni-filtered Cu Kα, 40 kV, 200 mA (rotating anode), background measured for 4 s on either side of the peak. Three standard reflections measured for every 57 reflections, fluctuation within 1.5% in *F*. Lorentz and polarization corrections; no absorption correction. 3068 reflections measured, all 2761 unique reflections (ranging over *h* = –15 to 15, *k* = 0 to 12, *l* = 0 to 13) used for refinement; 2627 reflections larger than 1.0σ(*F_o*). *R*_{int} = 0.010 for 149 *hk0* reflections. Structure solved by *MULTAN78*, and refined (anisotropically for non-H atoms) by block-diagonal least squares; ∑w(|*F_o*| – |*F_c*|)² minimized, with *w* = 1.0/|σ(*F_o*)² – 0.0364|*F_o*| + 0.0028|*F_o*|² for |*F_o*| > 0, *w* = 1.6983 for |*F_o*| = 0; H-atom positions determined from difference Fourier map, and refined isotropically by least squares. Extinction correction for ten strongest

Table 1. *Final atomic parameters* (× 10⁴) with *e.s.d.*'s in parentheses

$$B_{eq} = (4/3) \sum_i \beta_{ii} / a_i^*{}^2.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
O(1)	3934.6 (8)	5674 (1)	4785.6 (9)	4.43 (5)
O(2)	2112.6 (7)	7387.1 (9)	4635.0 (8)	3.74 (4)
O(3)	1977 (1)	5128 (1)	3255 (1)	5.23 (6)
O(4)	–640 (1)	7871 (1)	7515 (2)	8.58 (9)
O(5)	4360 (1)	8315 (1)	5011 (1)	6.08 (6)
C(1)	3208 (1)	5618 (1)	5510 (1)	3.32 (6)
C(2)	2472 (1)	4538 (1)	5141 (1)	3.64 (6)
C(3)	1517 (1)	5018 (1)	5555 (1)	3.65 (6)
C(4)	1868 (1)	6122 (1)	6331 (1)	3.15 (6)
C(5)	2611 (1)	5552 (1)	7273 (1)	3.62 (6)
C(6)	3589 (1)	5551 (2)	6769 (1)	3.79 (7)
C(7)	4022 (1)	6883 (2)	6975 (1)	4.36 (7)
C(8)	3262 (1)	7767 (1)	6307 (1)	3.74 (6)
C(9)	2575 (1)	6812 (1)	5622 (1)	3.10 (5)
C(10)	2408 (1)	4365 (1)	3895 (1)	4.19 (7)
C(11)	2910 (2)	3234 (2)	3489 (2)	6.6 (1)
C(12)	1049 (1)	6904 (1)	6719 (1)	3.66 (6)
C(13)	505 (1)	6189 (1)	7535 (2)	4.31 (7)
C(14)	–306 (1)	6901 (2)	7962 (2)	4.98 (8)
C(15)	–715 (1)	6356 (2)	8940 (2)	6.5 (1)
C(16)	4290 (1)	4477 (2)	7147 (2)	5.48 (9)
C(17)	3722 (1)	8670 (2)	5542 (1)	4.49 (8)
C(18)	3357 (2)	9999 (2)	5448 (2)	6.6 (1)

reflections [*I*_{corr} = *I*_o(1 + 1.29 × 10⁻⁵*I*_c)]. *R* = 0.050 for 2761 reflections, *wR* = 0.062, *S* = 2.18, (Δ/σ)_{max} in final refinement cycle 0.3; max. and min. Δρ in final difference Fourier map 0.30 and –0.25 e Å⁻³; atomic scattering factors from *International Tables for X-ray Crystallography* (1974), computations carried out at the Crystallographic Research Center, Institute for Protein Research, Osaka University, and at the Okayama University Computer Center; programs *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *HBL5-V* (Ashida, 1973), *MOLCON* (Fujii, 1979) and *ORTEP* (Johnson, 1965).

Discussion. Final atomic parameters are listed in Table 1.* A projection of the molecular structure is shown in Fig. 1. Bond lengths and angles are listed in Table 2, and torsion angles relevant to the conformation of the tricyclononane ring in Table 3.

The tricyclononane skeleton is constituted of two six-membered rings and three five-membered rings [rings (1) and (2), and rings (3), (4) and (5), respectively, in Table 3]. The endocyclic torsion angles show that both six-membered rings take boat conformations as found in an 8,9,10-trinorbornane group (Léger, Colleter & Carpy, 1982). Overtwisting from the ideal boat form (Hendrickson, 1961) amounts to 24.6° at χ₃ for ring (1) and 24.4° at χ₉ for ring (2). The

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42566 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

five-membered rings take pseudo C_2 conformations, differing from the C_s conformation found in the 8,9,10-trinorbornane group (Léger *et al.*, 1982). The largest overtensing from the ideal C_2 conformation (Hendrickson, 1961) is 17.4° at χ_{18} in ring (4).

The C—C bond lengths 1.529 (1)–1.557 (3) Å in the tricyclononane skeleton lie in a similar range to that found in some 8,9,10-trinorbornane rings (Léger *et al.*, 1982; Beckwith, Rodgers & Wagner, 1982). Endocyclic bond angles at C(1), C(4), C(6) and C(9) are remarkably decreased, 94.0 (1)– 100.7 (2) $^\circ$, while the exocyclic angles at these atoms are much increased,

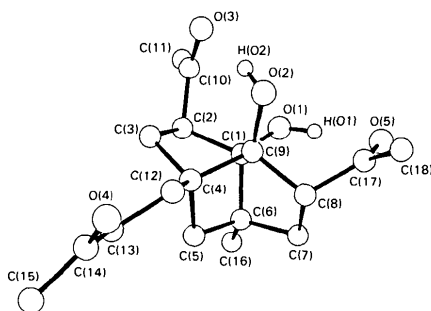


Fig. 1. View of the molecule with numbering of atoms used in the tables. H atoms attached to C atoms are omitted.

Table 2. Bond lengths (Å) and angles ($^\circ$) with *e.s.d.*'s in parentheses

O(1)—C(1)	1.413 (2)	C(4)—C(12)	1.517 (2)
O(2)—C(9)	1.416 (1)	C(5)—C(6)	1.547 (3)
O(3)—C(10)	1.216 (2)	C(6)—C(7)	1.529 (3)
O(4)—C(14)	1.216 (3)	C(6)—C(16)	1.517 (3)
O(5)—C(17)	1.212 (2)	C(7)—C(8)	1.548 (3)
C(1)—C(2)	1.548 (3)	C(8)—C(9)	1.545 (2)
C(1)—C(6)	1.549 (3)	C(8)—C(17)	1.520 (3)
C(1)—C(9)	1.543 (2)	C(10)—C(11)	1.489 (3)
C(2)—C(3)	1.550 (3)	C(12)—C(13)	1.513 (3)
C(2)—C(10)	1.511 (3)	C(13)—C(14)	1.490 (3)
C(3)—C(4)	1.532 (2)	C(14)—C(15)	1.489 (3)
C(4)—C(5)	1.552 (2)	C(17)—C(18)	1.483 (3)
C(4)—C(9)	1.557 (1)		
O(1)—C(1)—C(2)	109.9 (2)	C(7)—C(6)—C(16)	114.3 (2)
O(1)—C(1)—C(6)	115.8 (2)	C(6)—C(7)—C(8)	103.9 (2)
O(1)—C(1)—C(9)	118.2 (1)	C(7)—C(8)—C(9)	102.5 (1)
C(2)—C(1)—C(6)	112.2 (2)	C(7)—C(8)—C(17)	112.8 (2)
C(2)—C(1)—C(9)	105.4 (1)	C(9)—C(8)—C(17)	110.6 (2)
C(6)—C(1)—C(9)	94.3 (1)	O(2)—C(9)—C(1)	118.1 (1)
C(1)—C(2)—C(3)	102.6 (2)	O(2)—C(9)—C(4)	114.8 (1)
C(1)—C(2)—C(10)	109.2 (2)	O(2)—C(9)—C(8)	110.7 (1)
C(3)—C(2)—C(10)	114.7 (2)	C(1)—C(9)—C(4)	94.0 (1)
C(2)—C(3)—C(4)	103.4 (1)	C(1)—C(9)—C(8)	105.2 (1)
C(3)—C(4)—C(5)	106.6 (1)	C(4)—C(9)—C(8)	112.9 (1)
C(3)—C(4)—C(9)	100.6 (1)	O(3)—C(10)—C(2)	121.3 (2)
C(3)—C(4)—C(12)	114.3 (1)	O(3)—C(10)—C(11)	121.7 (2)
C(5)—C(4)—C(9)	100.7 (1)	C(2)—C(10)—C(11)	117.1 (2)
C(5)—C(4)—C(12)	115.3 (1)	C(4)—C(12)—C(13)	112.5 (2)
C(9)—C(4)—C(12)	117.4 (1)	C(12)—C(13)—C(14)	115.6 (2)
C(4)—C(5)—C(6)	103.6 (1)	O(4)—C(14)—C(13)	121.6 (2)
C(1)—C(6)—C(5)	100.7 (2)	O(4)—C(14)—C(15)	121.1 (2)
C(1)—C(6)—C(7)	101.2 (2)	C(13)—C(14)—C(15)	117.3 (2)
C(1)—C(6)—C(16)	116.9 (2)	O(5)—C(17)—C(8)	121.2 (2)
C(5)—C(6)—C(7)	106.0 (2)	O(5)—C(17)—C(18)	120.7 (2)
C(5)—C(6)—C(16)	115.7 (2)	C(8)—C(17)—C(18)	118.1 (2)

118.2 (1)– 116.9 (1) $^\circ$. Such a correlation between the decreased internal angles and the increased external angles is also observed in the 5,7,8,9-tetraoxatri-cyclo[4.2.1.0^{2,6}]nonane ring (Henke & Keul, 1975), indicating a considerable strain in these skeletons. The 5- and 8-acetyl groups take *exo* conformations (χ_{27} and χ_{33} in Table 3, respectively), intramolecular short contacts with the H atoms at 5 and 8 positions being H(2)···C(16) 2.90 (2), H(2)···H(16B) 2.34 (3); H(8)···C(12) 2.88 (1), H(8)···H(12A) 2.32 (2) Å.

The hydroxyl groups at 6 and 7 participate in intramolecular hydrogen bonds: O(1)···O(5) 2.842 (1), O(1)—H(O1) 0.90 (3), H(O1)···O(5) 1.99 (3) Å,

Table 3. Torsion angles ($^\circ$) concerning the conformation of the tricyclononane skeleton

Endocyclic		
(I) Ring (1) through C(1), C(2), C(3), C(4), C(5) and C(6)		
χ_1	C(1)—C(2)—C(3)—C(4)	−12.8 (2)
χ_2	C(2)—C(3)—C(4)—C(5)	−60.4 (2)
χ_3	C(3)—C(4)—C(5)—C(6)	84.6 (2)
χ_4	C(4)—C(5)—C(6)—C(1)	−20.7 (2)
χ_5	C(5)—C(6)—C(1)—C(2)	−54.7 (2)
χ_6	C(6)—C(1)—C(2)—C(3)	77.8 (2)
(II) Ring (2) through C(4), C(5), C(6), C(7), C(8) and C(9)		
χ_7	C(6)—C(7)—C(8)—C(9)	−10.0 (2)
χ_8	C(5)—C(6)—C(7)—C(8)	−62.9 (2)
χ_9	C(4)—C(5)—C(6)—C(7)	84.4 (2)
χ_{10}	C(9)—C(4)—C(5)—C(6)	−20.0 (2)
χ_{11}	C(8)—C(9)—C(4)—C(5)	−55.2 (1)
χ_{12}	C(7)—C(8)—C(9)—C(4)	75.6 (2)
(III) Ring (3) through C(1), C(2), C(3), C(4) and C(9)		
χ_1		−12.8 (2)
χ_{13}	C(2)—C(3)—C(4)—C(9)	44.2 (1)
χ_{14}	C(3)—C(4)—C(9)—C(1)	−56.2 (1)
χ_{15}	C(4)—C(9)—C(1)—C(2)	48.9 (1)
χ_{16}	C(9)—C(1)—C(2)—C(3)	−23.5 (2)
(IV) Ring (4) through C(9), C(4), C(5), C(6) and C(1)		
χ_4		−20.7 (2)
χ_{17}	C(5)—C(6)—C(1)—C(9)	53.8 (2)
χ_{18}	C(6)—C(1)—C(9)—C(4)	−65.5 (1)
χ_{19}	C(1)—C(9)—C(4)—C(5)	53.1 (1)
χ_{20}		−20.0 (2)
(V) Ring (5) through C(1), C(6), C(7), C(8) and C(9)		
χ_{20}	C(7)—C(8)—C(9)—C(1)	−25.5 (2)
χ_{21}	C(8)—C(9)—C(1)—C(6)	49.5 (2)
χ_{22}	C(9)—C(1)—C(6)—C(7)	−55.1 (2)
χ_{23}	C(1)—C(6)—C(7)—C(8)	41.8 (2)
χ_7		−10.0 (2)
Exocyclic		
χ_{24}	O(1)—C(1)—C(2)—C(3)	−151.9 (2)
χ_{25}	O(1)—C(1)—C(6)—C(5)	178.1 (2)
χ_{26}	C(10)—C(2)—C(3)—C(4)	−131.1 (2)
χ_{27}	C(10)—C(2)—C(1)—C(6)	−160.1 (2)
χ_{28}	C(2)—C(3)—C(4)—C(12)	171.0 (1)
χ_{29}	C(6)—C(5)—C(4)—C(12)	−147.4 (1)
χ_{30}	C(4)—C(5)—C(6)—C(16)	−147.8 (2)
χ_{31}	C(8)—C(7)—C(6)—C(16)	168.4 (2)
χ_{32}	C(6)—C(7)—C(8)—C(17)	−129.0 (2)
χ_{33}	C(4)—C(9)—C(8)—C(17)	−163.9 (1)
χ_{34}	C(7)—C(8)—C(9)—O(2)	−154.1 (1)
χ_{35}	C(5)—C(4)—C(9)—O(2)	176.6 (1)
χ_{36}	O(1)—C(1)—C(2)—C(10)	−29.8 (2)
χ_{37}	C(17)—C(8)—C(9)—O(2)	−33.7 (2)
χ_{38}	O(1)—C(1)—C(9)—O(2)	51.2 (2)

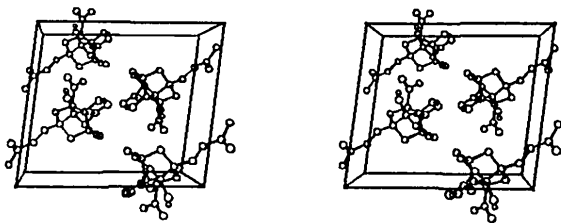


Fig. 2. Stereoscopic view of the molecular packing in the cell. The *a* axis points from left to right, the *b* axis onto the plane of the paper, and the *c* axis downwards. H atoms attached to C atoms are omitted.

O(1)—H(O1)···O(5) 157 (3), H(O1)···O(5)—C(17) 108.3 (9)°; O(2)···O(3) 2.888 (2), O(2)—H(O2) 0.82 (2), H(O2)···O(3) 2.10 (2) Å, O(2)—H(O2)···O(3) 164 (2), H(O2)···O(3)—C(10) 104.7 (6)°. This finding explains shifts observed in the IR and ¹³C NMR spectra for the 5- and 8-acetyl groups: 1695 cm⁻¹ (KBr) in IR and 215.5 and 215.7 p.p.m. (CDCl₃, Me₄Si) in ¹³C NMR in comparison with 1710 cm⁻¹ and 208.7 p.p.m. for the 13-acetyl group.

Molecular packing in the crystal is shown in Fig. 2. The molecules are held together by van der Waals interactions with normal intermolecular contacts.

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Structures of an Antithrombotic Agent and its Dimethyl Derivative: 4-Methoxy-*N,N'*-bis(3-pyridylmethyl)isophthalamide Monohydrate (I) and 4-Methoxy-*N,N'*-dimethyl-*N,N'*-bis(3-pyridylmethyl)isophthalamide Hemihydrate (II)

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Abstract. (I): C₂₁H₂₀N₄O₃·H₂O, *M_r* = 394.37, monoclinic, *P*2₁/*c*, *a* = 6.354 (2), *b* = 14.043 (1), *c* = 21.418 (3) Å, β = 92.33 (2)°, *V* = 1909.9 (6) Å³, *Z* = 4, *D_x* = 1.37 Mg m⁻³, Mo *K*α, λ = 0.7107 Å, μ = 0.058 mm⁻¹, *F*(000) = 832, *T* = 293 K, *R* = 0.066 for 1830 observed reflections. (II): C₂₃H₂₄N₄O₃·½H₂O, *M_r* = 413.41, monoclinic, *P*2₁/*n*, *a* = 31.649 (4), *b* = 11.753 (2), *c* = 11.501 (2) Å, β = 93.11 (2)°, *V* =

4272 (1) Å³, *Z* = 8, *D_x* = 1.28 Mg m⁻³, Mo *K*α, λ = 0.7107 Å, μ = 0.05 mm⁻¹, *F*(000) = 1752, *T* = 293 K, *R* = 0.065 for 5369 observed reflections. A considerable change in the shape of the basic molecular skeleton is caused by the *N*-methylation in (II), an inactive compound, to be compared with the planar shape observed in (I), an antithrombotic agent (picotamide). The two independent molecules in (II) are substantially

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