C2—N1—C6	118.93 (10)	N1—C6—C5	117.48 (10)
N3—C2—N1	129.22 (10)	C8—N7—C5	103.72 (9)
N3—C2—H2	114.7 (9)	N7—C8—N9	113.77 (10)
N1—C2—H2	116.1 (9)	C8—N9—C4	106.15 (9)
C2—N3—C4	110.18 (9)	C8—N9—C22	129.36 (10)
N3—C4—N9	126.23 (10)	C4—N9—C22	124.37 (10)
N3—C4—C5	127.73 (10)	O21—C21—O31	125.27 (11)
N9—C4—C5	106.02 (9)	O21—C21—C22	124.18 (11)
C4—C5—N7	110.33 (10)	O31—C21—C22	110.55 (9)
C4—C5—C6	116.44 (10)	N9—C22—C21	110.33 (9)
N7—C5—C6	133.18 (10)	C21—O31—C31	115.12 (9)
N16—C6—N1	118.29 (10)	O31—C31—C32	107.19 (10)
N16—C6—C5	124.21 (10)		

C6—N1—C2—N3	−0.6 (2)	C6—C5—N7—C8	−177.89 (12)
N1—C2—N3—C4	−0.9 (2)	C5—N7—C8—N9	0.30 (13)
C2—N3—C4—N9	−177.06 (11)	N7—C8—N9—C4	0.12 (14)
C2—N3—C4—C5	1.1 (2)	N7—C8—N9—C22	−176.11 (11)
N3—C4—C5—N7	−177.76 (11)	N3—C4—N9—C8	178.00 (11)
N9—C4—C5—N7	0.70 (13)	C5—C4—N9—C8	−0.49 (12)
N3—C4—C5—C6	0.0 (2)	N3—C4—N9—C22	−5.5 (2)
N9—C4—C5—C6	178.48 (9)	C5—C4—N9—C22	175.98 (10)
C2—N1—C6—N16	−179.72 (10)	C8—N9—C22—C21	104.65 (13)
C2—N1—C6—C5	1.8 (2)	C4—N9—C22—C21	−70.96 (14)
C4—C5—C6—N16	−179.93 (11)	O21—C21—C22—N9	−28.5 (2)
N7—C5—C6—N16	−2.8 (2)	O31—C21—C22—N9	152.62 (9)
C4—C5—C6—N1	−1.5 (2)	O21—C21—O31—C31	−1.9 (2)
N7—C5—C6—N1	175.64 (12)	C22—C21—O31—C31	177.04 (9)
C4—C5—N7—C8	−0.61 (13)	C21—O31—C31—C32	−177.14 (10)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N16—H161···N7^{i}	0.93 (2)	2.15 (2)	3.072 (1)	172.4 (14)
N16—H162···N1^{ii}	0.91 (2)	2.08 (2)	2.976 (1)	166.3 (15)

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

Data collection: Enraf–Nonius CAD-4 software. Cell refinement: Enraf–Nonius CAD-4 software. Data reduction: *DREADD* (Blessing, 1987). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*.

We thank Mr Flemming Hansen for technical assistance with the data collection and Dr Sine Larsen for valuable discussions and suggestions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1182). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Elucidation of the Mechanism of Transposition of Tricyclo[*m*.*n*.0.0$^{2,m+1}$]-alka-2,3,*m*+2-triol Derivatives: the Stereochemistry of *cis-anti-cis*-1-Acetoxytricyclo[7.3.0.0^{2,7}]dodecan-8-one

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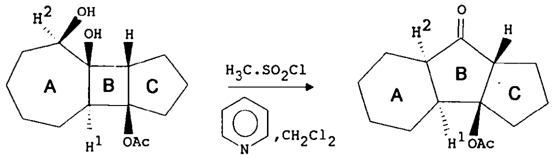
Abstract

The crystal structure analysis of the title compound (8-oxotricyclo[7.3.0.0^{2,7}]dodec-1-yl acetate, $C_{14}H_{20}O_3$) shows that in the transposition reaction causing its formation, the relative positions of the protons at the ring junctions are maintained, demonstrating that the process is a concerted one. The conformation and relevant structural parameters of the molecule, as found in the crystal, are compared with those calculated by molecular mechanics for the isolated molecule, providing evidence that packing effects exert no significant influence on the molecular conformation.

Comment

During studies of new approaches for obtaining polycyclic cyclopentane derivatives (Jamart-Grégoire, Brosse, Ianelli, Nardelli & Caubère, 1991, 1993; Ianelli, Nardelli, Belletti, Jamart-Grégoire, Brosse & Caubère, 1993) we needed to elucidate the mechanism of the following transposition reaction.



(1) (2)

To do this, it was important to know if the relative position of the H^1 and H^2 protons was preserved during the process. The NMR spectrum of the title compound, (2), showed that the *A*/*B* ring junction was *cis*, but it was not possible to determine the relative position, *syn* or *anti*, of the *A* and *C* rings. The crystal structure analysis of (2) was therefore undertaken mainly to clarify this point.

As shown by the *ORTEP* (Johnson, 1965) drawing in Fig. 1, the three rings have an *anti* configuration and substitution at both junctions is *cis*. The configuration at the C atoms involved in the *A*/*B* ring junction has been preserved during the transposition process, showing that the process itself was a concerted one. The configurations at the chiral centres are *S*(C2), *R*(C7), *R*(C8) and *S*(C9), but the enantiomorph is also present in the crystal since the space group is centrosymmetric. The conformations at the two junctions are not identical; the C2—C7 interaction is more open, *i.e.* the bonds tend to be *gauche*, while at C8—C9 the bonds tend to be eclipsed.

Table 2 compares relevant structural descriptors of the molecule in the crystal with those calculated from the optimized geometries obtained for the isolated molecule by the *PCMODEL-MMX* (Serena Software, 1989) and *HYPERCHEM-MM+* (Autodesk, Inc., 1992) force fields, starting from the experimental coordinates and using the program default parameters. From these data it appears that the largest discrepancies are observed when the atoms C11, C14 and O2 are involved, these atoms being among those affected by the largest thermal motion (or disorder).

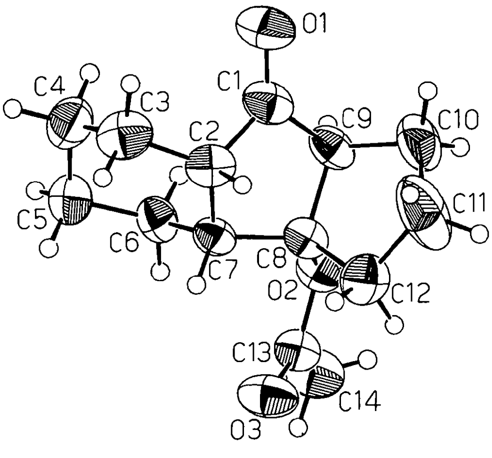


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule with ellipsoids drawn at the 50% probability level.

The relevant parameters describing the conformation of the three ring systems are Q_T, the total puckering amplitude according to Cremer & Pople (1975), and the dihedral angles between the weighted least-squares planes through the rings. These parameters have the following values (values calculated from the force-field optimized structures are given in square brackets, first for *MMX* and then *MM+*):

Ring	Q_T	Conformation
A	0.534(3) [0.552, 0.552]	Chair
B	0.396(3) [0.385, 0.382]	Twisted envelope
C	0.264(4) [0.387, 0.388]	Twisted envelope

	Dihedral angle (°)
A^B	64.7(1) [66.0, 65.7]
B^C	72.8(1) [79.2, 79.5]
C^A	68.3(1) [68.5, 68.4]

The acetoxy group is antiperiplanar with respect to the *B*/*C* ring junction and this orientation is imposed by intramolecular steric hindrance, as indicated by the non-bonded energy profile obtained by rotating the group about the C8—O2 bond; the profile shows a well defined minimum for the observed conformation.

The analysis of 'thermal' motion, carried out in terms of the Schomaker & Trueblood (1968) **TLS** rigid-body approximation using the *THMV* program (Trueblood, 1984), gives an overall residual disagreement index R_{wU} of 0.174, which is rather high, as is usually the case when high thermal motion or disorder is present, but a significant improvement [R_{wU} = 0.109] is observed if the internal motions of atoms O1, O3, C10, C11, C12 and C14 are considered according to Dunitz & Withe (1973).

Experimental

Crystal data

$C_{14}H_{20}O_3$
M_r = 236.31
Monoclinic
$P2_1/n$
a = 16.299 (3) Å
b = 8.041 (1) Å
c = 10.148 (2) Å
β = 106.56 (1)°
V = 1274.8 (4) Å^3
Z = 4
D_x = 1.231 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 30 reflections
θ = 18–36°
μ = 0.650 mm^{-1}
T = 293 (2) K
Small prisms
0.30 × 0.26 × 0.23 mm
Colourless

Data collection

Siemens AED diffractometer
θ–2θ scans
Absorption correction: none

θ_{max} = 70.24°
$h = -13 \rightarrow 19$
$k = -9 \rightarrow 9$
$l = -10 \rightarrow 12$

2559 measured reflections
2426 independent reflections
976 observed reflections
[$I > 2\sigma(I)$]
$R_{\rm int} = 0.0149$

1 standard reflection
monitored every 50 reflections
intensity variation: within statistical fluctuation

Refinement

Refinement on F^2
$R(F) = 0.0453$
$wR(F^2) = 0.1033$
$S = 0.735$
2425 reflections
212 parameters
All H-atom parameters refined excepting those of the C10, C11 and C12 methylene groups
$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.032$

$\Delta\rho_{\rm max} = 0.219$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.180$ e Å^{-3}
Extinction correction: *SHELXL* (Sheldrick, 1993)
Extinction coefficient: 0.0049 (3)
Atomic scattering factors from *International Tables for X-ray Crystallography* [1974, Vol. IV, Tables 2.2A, 2.3.1 (O, C) and 2.2C (H)]

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

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
O1	−0.1542 (1)	0.3407 (3)	0.5321 (2)	0.0933 (11)
O2	0.1024 (1)	0.0797 (2)	0.7496 (2)	0.0531 (8)
O3	0.1245 (1)	−0.0741 (3)	0.9413 (2)	0.0768 (9)
C1	−0.1031 (2)	0.2707 (3)	0.6256 (3)	0.0602 (12)
C2	−0.1230 (2)	0.1619 (4)	0.7334 (3)	0.0563 (12)
C3	−0.2098 (2)	0.0752 (5)	0.6919 (4)	0.0731 (16)
C4	−0.2131 (2)	−0.0705 (5)	0.5962 (4)	0.0745 (15)
C5	−0.1375 (2)	−0.1875 (4)	0.6525 (4)	0.0713 (16)
C6	−0.0531 (2)	−0.0957 (4)	0.6744 (3)	0.0563 (14)
C7	−0.0447 (2)	0.0476 (4)	0.7757 (3)	0.0476 (11)
C8	0.0298 (2)	0.1658 (3)	0.7761 (3)	0.0483 (10)
C9	−0.0058 (2)	0.2821 (3)	0.6534 (3)	0.0548 (13)
C10	0.0298 (2)	0.4564 (4)	0.6985 (3)	0.0806 (16)
C11	0.0448 (3)	0.4565 (4)	0.8470 (4)	0.1331 (25)
C12	0.0604 (2)	0.2807 (4)	0.9017 (3)	0.0699 (14)
C13	0.1437 (2)	−0.0368 (4)	0.8400 (3)	0.0563 (12)
C14	0.2152 (3)	−0.1105 (7)	0.7970 (5)	0.0769 (18)

Table 2. *Comparison of selected bond distances* (Å), *bond angles* (°) *and torsion angles* (°) *determined by experiment and calculation*

Calculated values are for the isolated molecule.

	X-ray	*PCMODEL-MMX*	*HYPERCHEM-MM+*
O1—C1	1.208 (3)	1.209	1.208
O2—C8	1.461 (3)	1.418	1.412
O2—C13	1.348 (3)	1.349	1.344
O3—C13	1.195 (4)	1.210	1.208
C1—C2	1.507 (5)	1.517	1.516
C1—C9	1.533 (5)	1.521	1.521
C2—C7	1.532 (4)	1.538	1.539
C7—C8	1.541 (4)	1.546	1.549
C8—C9	1.533 (4)	1.546	1.546
C8—O2—C13	118.5 (2)	124.2	118.8
O1—C1—C9	124.2 (3)	124.8	124.8
O1—C1—C2	126.7 (3)	125.6	125.6
C2—C1—C9	109.1 (3)	109.6	109.6
C1—C2—C7	102.5 (2)	102.6	102.6
C1—C2—C3	116.1 (3)	116.6	116.5
C3—C2—C7	115.9 (3)	115.3	115.4
C2—C7—C6	110.9 (2)	111.3	111.1
C6—C7—C8	112.8 (2)	111.9	111.8
C2—C7—C8	102.9 (2)	103.4	103.5
O2—C8—C7	112.7 (2)	111.6	112.1
C7—C8—C12	116.3 (2)	116.6	115.9
C7—C8—C9	105.0 (2)	104.5	104.6
O2—C8—C12	110.4 (2)	109.9	110.5
O2—C8—C9	106.2 (2)	109.1	108.5
C9—C8—C12	105.4 (2)	104.5	104.7
C1—C9—C8	104.2 (2)	104.6	104.8
C8—C9—C10	106.9 (2)	106.3	106.2
C1—C9—C10	113.1 (3)	112.7	112.7
C8—O2—C13—C14	−179.9 (3)	179.4	179.9
C13—O2—C8—C9	−177.2 (2)	179.9	177.3
C2—C1—C9—C10	−112.1 (3)	−113.3	−112.4
C9—C1—C2—C3	−154.2 (3)	−151.6	−152.2
C1—C2—C7—C8	39.2 (3)	37.5	37.4
C1—C2—C7—C6	−81.7 (3)	−82.7	−82.8
C3—C2—C7—C8	166.7 (3)	165.3	165.2
C3—C2—C7—C6	45.8 (3)	45.0	45.0
C2—C7—C8—O2	−153.0 (2)	−155.1	−153.9
C6—C7—C8—C12	−162.2 (2)	−162.6	−162.3
O2—C8—C9—C1	140.6 (2)	141.3	140.7
C7—C8—C9—C1	21.1 (3)	21.8	20.9
C7—C8—C9—C10	141.1 (2)	141.3	140.4
O2—C8—C9—C10	−99.3 (2)	−99.2	−99.8
C12—C8—C9—C1	−102.2 (3)	−101.2	−101.3
C12—C8—C9—C10	17.8 (3)	18.2	18.2

Data were collected using local programs. Cell refienement was performed using *LQPARM* (Nardelli & Mangia, 1984). The integrated intensities were obtained by a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) peak-profile analysis procedure. All reflections were corrected for Lorentz and polarization effects, but not for absorption. The structure was solved (*SHELXS*86; Sheldrick, 1985) by direct methods and refined by anisotropic full-matrix least squares. All the H atoms were localized from a $\Delta\rho$ map, except those of the (probably disordered) C11 methylene group and were refined isotropically except those of the C10, C11, C12 methylene groups which were placed in geometrically calculated positions, riding on the attached C atoms. Refinement (*SHELXL*93; Sheldrick, 1993) was on F^2 for all reflections except one possibly affected by systematic errors. The calculations were carried out on the ENCORE91 and Gould-Powernode 6040 computers of the Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *PARST* (Nardelli, 1983*b*); *PARSTCIF* (Nardelli, 1991).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1109). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methyl (3a*R*,4*R*,7*S*,7a*S*)-1,3-Dioxo-7-phenyl-1,3,3a,4,7,7a-hexahydro-4-isobenzofurancarboxylate

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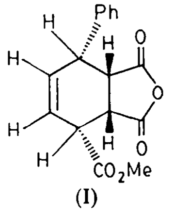
Abstract

The structure of the title compound, $C_{16}H_{14}O_5$, has been determined by X-ray diffraction. The molecule contains four chiral centres; both enantiomers are present in the crystal as a result of the centrosymmetric space group. As expected for a Diels–Alder adduct, the ring-junction H atoms display a *cis* relationship. The conformation of the six-membered ring is an ideal boat, whereas the five-membered ring is significantly flattened.

Comment

The Diels–Alder reaction has played an important role in the development of synthetic, mechanistic and theoretical organic chemistry. The remarkable stereo- and regioselectivities associated with cycloaddition provide various polyfunctionalized molecules containing multiple chiral centres which are of potential use in protein catalysis (Suckling, Tedford, Bence, Irvine & Stimson, 1993; Braisted & Schultz, 1990; Hilvert, Hill, Nared & Auditor, 1989). In order to construct a hierarchy and unambiguously assign the stereochemistry of the four contiguous chiral centres, the crystal structure determination of methyl (3a*R*,4*R*,7*S*,7a*S*)-1,3-dioxo-7-phenyl-1,3,3a,4,7,7a-hexahydro-4-isobenzofurancarboxylate (I) was undertaken. The compound is the Diels–Alder adduct of (*E*,*E*)-1-methoxycarbonyl-4-phenyl-1,3-butadiene with maleic anhydride and was prepared by heating the reactants in a xylene solution.



(I)

The results of the present structure analysis are in agreement with those of the structure determinations of (1*R*,6*R*,10*S*)-10-[(2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl)oxy]bicyclo[4.4.0]decane-2,5,8-trione (Gupta, Raynor, Stoodley, Slawin & Williams, 1988) and (2*Z*,4*E*)-1,3,6-triacetoxyhexa-2,4-diene (Areces, Roman, Pozo & Serrano, 1993), based on X-ray, spectral and chemical evidence. The *ORTEP*II (Johnson, 1976) view (Fig. 1) shows that the molecule consists of a bicyclic core (*AB*) with a *cis* relationship between the H atoms [H1—C1—C2—H2 torsion angle −3.8°]. There are four chiral centres [configurations *S*(C1), *R*(C2), *R*(C3), *S*(C6)]; the enantiomorph is also present since the space group is centrosymmetric. The bond distances [C—C bond lengths range from 1.492 (3) to 1.558 (3) Å] and angles quoted in Table 2 are as expected. Ring *A* is in an almost ideal boat conformation, with C3 and C6 displaced by 0.588 (2) and 0.579 (2) Å, respectively, in the same direction from the least-squares plane through the remaining endocyclic atoms. For this ring, the *Q* and θ puckering parameters (Cremer & Pople, 1975) are 0.675 (3) Å and 89.8 (2)°, respectively. The five-membered ring *B* is significantly flattened (the sum of absolute values of torsion angles is 16.7°), with a maximum deviation of 0.030 (3) Å at C1, and is nearly

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