C1	0.1168 (3)	0.1461 (1)	−0.3538 (3)	0.032 (2)
C2	0.1847 (3)	0.1787 (1)	−0.3630 (3)	0.034 (2)
C3	0.1978 (3)	0.2065 (1)	−0.2958 (3)	0.037 (2)
C4	0.1432 (3)	0.2035 (1)	−0.2155 (3)	0.036 (2)
C5	0.0736 (3)	0.1722 (1)	−0.2024 (3)	0.036 (2)
C6	0.0596 (3)	0.1448 (1)	−0.2714 (3)	0.034 (2)
C7	0.3159 (4)	0.2066 (2)	−0.4573 (4)	0.058 (3)
C8	0.1032 (4)	0.2328 (2)	−0.0711 (3)	0.051 (3)
C9	−0.0751 (4)	0.1132 (2)	−0.1883 (4)	0.060 (3)
C10	0.0332 (3)	0.0680 (1)	−0.4224 (3)	0.032 (2)
C11	0.0638 (3)	0.0463 (1)	−0.3465 (3)	0.036 (2)
C12	0.0097 (4)	0.0130 (1)	−0.3151 (3)	0.042 (2)
C13	−0.0772 (3)	0.0018 (1)	−0.3633 (3)	0.043 (2)
C14	−0.1080 (3)	0.0220 (1)	−0.4418 (3)	0.041 (2)
C15	−0.0533 (3)	0.0542 (1)	−0.4722 (3)	0.036 (2)
C16	0.1727 (5)	0.0456 (2)	−0.2152 (4)	0.077 (3)
C17	−0.1076 (5)	−0.0532 (2)	−0.2627 (4)	0.074 (3)
C18	−0.1709 (4)	0.0672 (2)	−0.5934 (4)	0.063 (3)
C19	0.0468 (3)	0.1424 (1)	−0.5379 (3)	0.033 (2)
C20	0.0921 (3)	0.1535 (1)	−0.6207 (3)	0.034 (2)
C21	0.0418 (3)	0.1790 (1)	−0.6818 (3)	0.040 (2)
C22	−0.0523 (3)	0.1933 (1)	−0.6587 (3)	0.039 (2)
C23	−0.1011 (3)	0.1828 (1)	−0.5790 (3)	0.039 (2)
C24	−0.0510 (3)	0.1576 (1)	−0.5193 (3)	0.035 (2)
C25	0.2434 (4)	0.1544 (2)	−0.7121 (4)	0.058 (3)
C26	−0.0630 (4)	0.2309 (2)	−0.7987 (4)	0.057 (3)
C27	−0.1892 (4)	0.1562 (2)	−0.4123 (4)	0.063 (3)
C28	0.2273 (3)	0.0933 (1)	−0.4838 (3)	0.040 (2)
C29*	0.4495 (11)	0.4957 (5)	0.0073 (11)	0.106 (10)

* Occupancy 0.5, scattering factor equal to the average of O and C.

Table 2. *Selected intermolecular distances* (Å) *and angles* (°)

P—C1	1.813 (4)	P—C10	1.806 (4)
P—C19	1.797 (4)	P—C28	1.804 (4)
C1—P—C10	112.3 (2)	C1—P—C19	104.3 (2)
C1—P—C28	110.7 (2)	C10—P—C19	113.6 (2)
C10—P—C28	103.6 (2)	C19—P—C28	112.5 (2)

H atoms were fixed at idealized positions with $U_{iso} = 1.3 \times U_{eq}$ of the parent atom. $\sigma(F^2) = [\sigma^2(I) + (0.4I)^2]^{0.5}/\mathrm{Lp}$. Function minimized was $\Sigma w(|F_o|-|F_c|)^2$. Two peaks, C(29) and its equivalent lying across an inversion centre, were included as a molecule of ethanol, disordered and at half occupancy.

We thank the SERC for a grant (to RMH).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, and a diagram of the unit-cell contents have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71292 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1047]

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Three Isomers of Tricyclo[10.3.0.0^{4,9}]pentadecane-2,10-dione, (I), (II) and (III), and *cis-cisoid-cis*-9-Hydroxy-9-methyltricyclo-[9.3.0.0^{3,7}]tetradecan-2-one, (IV)

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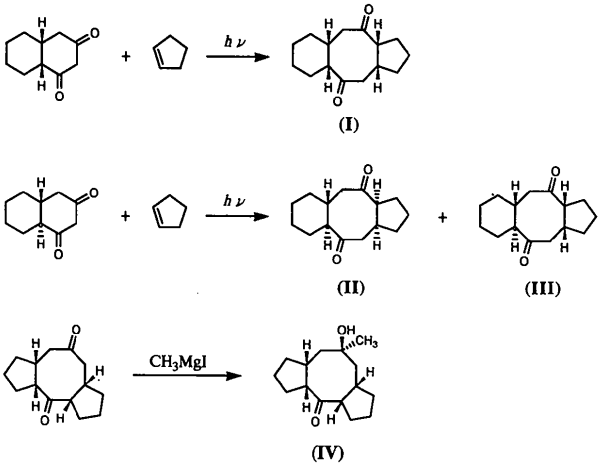
Abstract

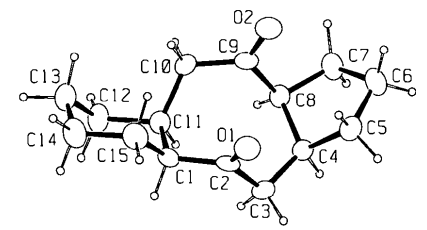
The crystal structures of the four title compounds, which were obtained during studies of the preparation and reactions of 6–8–5 and 5–8–5 fused-ring compounds, have been determined by X-ray diffraction. (I), (II) and (III) are *cis-cisoid-cis*, *trans-cisoid-cis* and *trans-transoid-cis* isomers of the 6–8–5 fused-ring compound, respectively. The eight-membered ring takes a boat–chair form in compounds (I) to (IV).

Comment

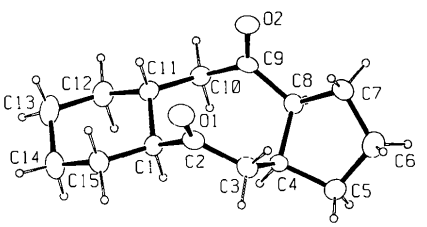
Syntheses of 5–8–5 fused-ring compounds and the conformations of their eight-membered rings have been investigated previously (Okumoto, Ohba, Saito, Umehara & Hishida, 1988; Umehara *et al.*, 1990). The syntheses of the 6–8–5 fused-ring compounds (I)–(III) (Umehara *et al.*, 1993) and methylation of



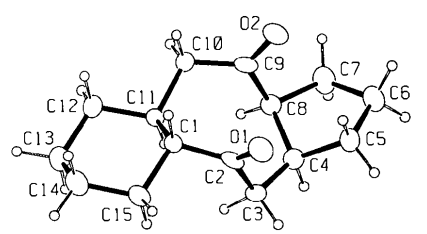
the 5–8–5 fused-ring compounds will be reported later. Symmetry of the boat–chair form in (I)–(III) indicates that the intramolecular strain of the 5–8–5 fused-ring compounds is much reduced by replacement of one of the five-membered rings with a cyclohexane ring. There is an intermolecular hydrogen bond in (IV), O1···O2($x-\frac{1}{2}$, $\frac{1}{2}-y$, $1-z$) = 3.018 (4) Å.



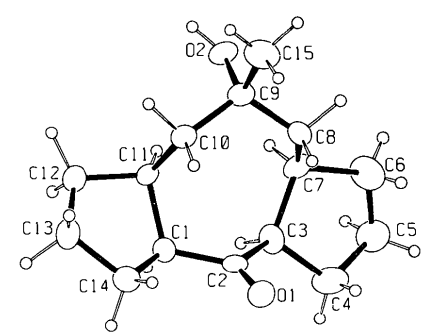
(I)



(II)



(III)



(IV)

Fig. 1. *ORTEP*II drawings (Johnson, 1976) of the molecules with 30% probability ellipsoids. H atoms are represented by circles of radius 0.08 Å.

Experimental

Compound (I)

Crystal data

$C_{15}H_{22}O_2$
M_r = 234.3
Orthorhombic
Pbca
a = 13.250 (1) Å
b = 10.054 (1) Å
c = 19.496 (1) Å
V = 2597.2 (3) Å^3
Z = 8
D_x = 1.199 Mg m^{-3}
M.p. = 393–397 K

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–15°
μ = 0.072 mm^{-1}
T = 298 (2) K
Prism
0.60 × 0.50 × 0.40 mm
Colourless

Data collection

Rigaku AFC-5 four-circle diffractometer
θ–2θ scans
Absorption correction: by integration from crystal shape
T_{min} = 0.965, T_{max} = 0.979
2986 measured reflections
2986 independent reflections

1348 observed reflections [$|F_o| > 3\sigma(|F_o|)$]
θ_{max} = 27.5°
h = 0 → 17
k = −13 → 0
l = 0 → 25
5 standard reflections monitored every 100 reflections
intensity variation: 0.984–1.006%

Refinement

Refinement on F
Final R = 0.046
wR = 0.043
S = 3.85
1348 reflections
243 parameters
All H-atom parameters refined

$w = 1/\sigma^2$
$(\Delta/\sigma)_{max}$ = 0.207
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (II)

Crystal data

$C_{15}H_{22}O_2$
M_r = 234.3
Monoclinic
Pc
a = 10.326 (3) Å
b = 6.706 (2) Å
c = 9.761 (5) Å
β = 101.05 (4)°
V = 663.4 (4) Å^3
Z = 2
D_x = 1.173 Mg m^{-3}
M.p. = 441–442 K

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–15°
μ = 0.071 mm^{-1}
T = 299 (2) K
Prism
0.50 × 0.50 × 0.30 mm
Colourless

Data collection

Rigaku AFC-5 four-circle diffractometer

R_{int} = 0.025
θ_{max} = 27.5°

ω scans
Absorption correction:
by integration from crystal shape
T_{min} = 0.963, T_{max} = 0.980
1618 measured reflections
1526 independent reflections
965 observed reflections
$[|F_o| > 3\sigma(|F_o|)]$

Refinement

Refinement on F
Final R = 0.045
wR = 0.049
S = 2.15
965 reflections
243 parameters
All H-atom parameters refined

$h = -13 \rightarrow 13$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 12$
5 standard reflections
monitored every 100 reflections
intensity variation: 1.000–1.008%

$w = [\sigma^2(|F_o|) + (0.015|F_o|)^2]^{-1}$
$(\Delta/\sigma)_{max}$ = 0.098
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (III)

Crystal data

$C_{15}H_{22}O_2$
M_r = 234.3
Monoclinic
$P2_1/n$
a = 18.730 (3) Å
b = 9.630 (2) Å
c = 7.218 (1) Å
β = 94.92 (2)°
V = 1297.1 (4) Å^3
Z = 4
D_x = 1.200 Mg m^{-3}
M.p. = 377–378 K

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–15°
μ = 0.072 mm^{-1}
T = 297 (2) K
Plate
0.70 × 0.40 × 0.10 mm
Colourless

Data collection

Rigaku AFC-5 four-circle diffractometer
ω scans
Absorption correction:
by integration from crystal shape
T_{min} = 0.958, T_{max} = 0.992
3224 measured reflections
2992 independent reflections
1225 observed reflections
$[|F_o| > 3\sigma(|F_o|)]$

R_{int} = 0.013
θ_{max} = 27.5°
$h = -24 \rightarrow 24$
$k = -12 \rightarrow 0$
$l = 0 \rightarrow 9$
5 standard reflections
monitored every 100 reflections
intensity variation: 0.978–1.002%

Refinement

Refinement on F
Final R = 0.048
wR = 0.035
S = 3.15
1225 reflections
243 parameters
All H-atom parameters refined

$w = 1/\sigma^2$
$(\Delta/\sigma)_{max}$ = 0.152
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (IV)

Crystal data

$C_{15}H_{24}O_2$
M_r = 236.4
Orthorhombic
$Pbcn$
a = 14.276 (1) Å
b = 10.984 (2) Å
c = 17.117 (3) Å
V = 2684.1 (6) Å^3
Z = 8
D_x = 1.170 Mg m^{-3}
M.p. = 393–395 K

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 23 reflections
θ = 10–15°
μ = 0.070 mm^{-1}
T = 299 (2) K
Plate
0.60 × 0.30 × 0.10 mm
Colourless

Data collection

Rigaku AFC-5 four-circle diffractometer
ω scans
Absorption correction:
by integration from crystal shape
T_{min} = 0.984, T_{max} = 0.995
3088 measured reflections
3088 independent reflections

836 observed reflections
$[|F_o| > 3\sigma(|F_o|)]$
θ_{max} = 27.5°
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 14$
$l = -22 \rightarrow 0$
5 standard reflections
monitored every 100 reflections
intensity variation: 0.990–1.004%

Refinement

Refinement on F
Final R = 0.054
wR = 0.033
S = 2.33
836 reflections
251 parameters
All H-atom parameters refined

$w = 1/\sigma^2$
$(\Delta/\sigma)_{max}$ = 0.227
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
(I)				
O1	0.4427 (2)	0.4233 (2)	0.7485 (1)	0.0611 (9)
O2	0.6568 (2)	0.6016 (2)	0.7877 (1)	0.065 (1)
C1	0.5525 (2)	0.2963 (3)	0.6765 (2)	0.043 (1)
C2	0.5033 (3)	0.3320 (3)	0.7439 (2)	0.046 (1)
C3	0.5314 (3)	0.2511 (3)	0.8060 (2)	0.052 (1)
C4	0.5971 (2)	0.3227 (3)	0.8594 (2)	0.044 (1)
C5	0.5446 (2)	0.4328 (4)	0.9005 (2)	0.053 (1)
C6	0.6299 (3)	0.5054 (4)	0.9378 (2)	0.064 (2)
C7	0.7282 (3)	0.4633 (4)	0.9015 (2)	0.058 (1)
C8	0.6967 (2)	0.3904 (3)	0.8357 (2)	0.043 (1)
C9	0.6824 (2)	0.4872 (3)	0.7767 (2)	0.045 (1)
C10	0.7044 (2)	0.4433 (3)	0.7045 (2)	0.048 (1)
C11	0.6682 (2)	0.3052 (3)	0.6818 (1)	0.043 (1)
C12	0.7151 (3)	0.2652 (4)	0.6126 (2)	0.060 (2)
C13	0.6730 (3)	0.3441 (4)	0.5521 (2)	0.066 (2)
C14	0.5598 (3)	0.3314 (4)	0.5486 (2)	0.068 (2)
C15	0.5110 (3)	0.3738 (4)	0.6156 (2)	0.062 (1)

(II)				
O1	0.0	0.3236 (4)	0.0	0.057 (1)
O2	−0.0291 (3)	−0.1345 (3)	−0.1591 (3)	0.0495 (9)
C1	0.1310 (3)	0.3687 (5)	−0.1725 (4)	0.039 (1)
C2	0.0001 (4)	0.3511 (5)	−0.1218 (4)	0.041 (1)
C3	−0.1278 (4)	0.3772 (5)	−0.2236 (5)	0.047 (1)
C4	−0.1458 (3)	0.2753 (5)	−0.3660 (4)	0.038 (1)
C5	−0.2869 (4)	0.3058 (6)	−0.4500 (5)	0.056 (2)
C6	−0.3679 (4)	0.1313 (7)	−0.4138 (6)	0.071 (2)
C7	−0.2722 (4)	−0.0212 (6)	−0.3339 (5)	0.057 (2)
C8	−0.1368 (3)	0.0458 (6)	−0.3587 (4)	0.039 (1)
C9	−0.0194 (4)	−0.0322 (5)	−0.2585 (4)	0.037 (1)
C10	0.1148 (4)	0.0229 (6)	−0.2870 (4)	0.041 (1)
C11	0.1948 (4)	0.1613 (5)	−0.1784 (4)	0.041 (1)
C12	0.3347 (4)	0.1825 (7)	−0.2100 (5)	0.059 (2)
C13	0.4218 (4)	0.3234 (8)	−0.1084 (6)	0.074 (2)
C14	0.3574 (4)	0.5262 (8)	−0.1114 (6)	0.074 (2)
C15	0.2219 (4)	0.5085 (7)	−0.0728 (5)	0.055 (1)
(III)				
O1	0.2207 (1)	0.2154 (2)	0.5766 (3)	0.0573 (9)
O2	0.2060 (1)	0.3693 (2)	0.1413 (3)	0.0591 (9)
C1	0.3138 (1)	0.3819 (3)	0.6057 (4)	0.036 (1)
C2	0.2368 (2)	0.3373 (3)	0.6067 (4)	0.041 (1)
C3	0.1811 (2)	0.4426 (3)	0.6554 (4)	0.042 (1)
C4	0.1315 (2)	0.4918 (3)	0.4684 (4)	0.044 (1)
C5	0.0791 (2)	0.3830 (3)	0.3972 (4)	0.051 (1)
C6	0.0472 (2)	0.4524 (4)	0.2165 (5)	0.058 (1)
C7	0.0986 (2)	0.5721 (4)	0.1764 (5)	0.059 (1)
C8	0.1654 (2)	0.5574 (3)	0.3190 (4)	0.041 (1)
C9	0.2227 (2)	0.4674 (3)	0.2451 (4)	0.042 (1)
C10	0.2999 (2)	0.5023 (3)	0.2933 (4)	0.044 (1)
C11	0.3236 (1)	0.5196 (3)	0.5015 (4)	0.035 (1)
C12	0.4026 (2)	0.5636 (4)	0.5222 (4)	0.052 (1)
C13	0.4321 (2)	0.5739 (4)	0.7260 (5)	0.059 (1)
C14	0.4244 (2)	0.4349 (4)	0.8249 (5)	0.066 (2)
C15	0.3449 (2)	0.3909 (3)	0.8106 (4)	0.051 (1)
(IV)				
O1	0.7735 (2)	0.2650 (3)	0.4737 (2)	0.057 (1)
O2	0.4300 (2)	0.1575 (3)	0.4187 (2)	0.055 (1)
C1	0.6685 (3)	0.1701 (4)	0.5637 (3)	0.046 (2)
C2	0.7204 (3)	0.1797 (5)	0.4874 (2)	0.044 (2)
C3	0.7046 (3)	0.0783 (4)	0.4288 (3)	0.049 (2)
C4	0.7876 (3)	0.0573 (5)	0.3761 (3)	0.088 (3)
C5	0.7470 (4)	−0.0074 (6)	0.3061 (3)	0.087 (3)
C6	0.6601 (4)	0.0572 (6)	0.2926 (3)	0.120 (4)
C7	0.6189 (3)	0.0962 (5)	0.3727 (3)	0.055 (2)
C8	0.5750 (3)	0.2208 (5)	0.3653 (2)	0.054 (2)
C9	0.4951 (3)	0.2573 (4)	0.4202 (3)	0.047 (2)
C10	0.5250 (3)	0.2834 (4)	0.5050 (3)	0.045 (2)
C11	0.5594 (3)	0.1749 (4)	0.5548 (2)	0.042 (2)
C12	0.5311 (3)	0.1903 (5)	0.6411 (3)	0.063 (2)
C13	0.6022 (4)	0.2794 (5)	0.6753 (3)	0.065 (2)
C14	0.6902 (3)	0.2696 (5)	0.6247 (3)	0.066 (2)
C15	0.4495 (4)	0.3719 (5)	0.3877 (3)	0.071 (3)

Table 2. *Selected bond lengths* (Å) *and torsion angles* (°)

	(I)	(II)	(III)	(IV)
C1—C11	1.539 (4)	1.545 (5)	1.543 (4)	1.566 (6)
C4—C8	1.555 (4)	1.543 (5)	1.560 (4)	
C3—C7				1.567 (7)
C2—C1—C11—C10	−57.1 (3)	65.6 (4)	−65.1 (3)	−50.4 (5)
C3—C4—C8—C9	54.4 (4)	−48.7 (4)	52.6 (4)	
C2—C3—C7—C8				15.8 (6)

The calculations were carried out either on a FACOM M-780/10 computer using the *UNICS*III (Sakurai & Kobayashi, 1979) program system or on a MIPS 3230 workstation with the *Xtal*3.0 (Hall & Stewart, 1990) program system.

In (II), the systematic absences *h*0*l*, *l* odd suggested that the space group is *Pc* or *P*2/*c*. Since $Z = 2$ and the molecule does not have a centre of inversion or a twofold axis, the space group was determined to be *Pc*.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71252 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1027]

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Structure of a Bis-spiroacetal, *cis*-14-Phenylsulfonyl-1,7,9-trioxadispiro[5.1.5.3]-hexadecane

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Abstract

The crystal structure of the title compound has been determined by X-ray crystallography. The two terminal rings have chair conformations, while the central ring is disordered between distorted boat and chair structures.