GF thanks NSERC (Canada) for Grants in Aid of Research.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms and torsion angles, a plot of the structure of molecule *B* and plots showing the patterns of hydrogen bonding have been deposited with the IUCr (Reference: NA1185). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chehna, M., Pradere, J.-P., Vicens, J., Toupet, L. & Quiniou, H. (1988). *Bull. Soc. Chim. Fr.* pp. 897–900.

Enraf–Nonius (1992). *CAD-4-PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Harrison, W. A. & Kulka, M. (1973). US Patent 3 725 427.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1994). *Structure Correlations*, Vol. 2, edited by H.-B. Bürgi & J. D. Dunitz, Appendix A: *Typical Interatomic Distances in Organic Compounds and Organometallic and Coordination Complexes of the d- and f-Block Metals*. Weinheim: VCH.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Spek, A. L. (1994*a*). *PLATON. Molecular Geometry Program*. Version of July 1994. Univ. of Utrecht, The Netherlands.

Spek, A. L. (1994*b*). *PLUTON. Molecular Graphics Program*. Version of July 1994. Univ. of Utrecht, The Netherlands.

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Four Isomeric Dioxolane Derivatives of D-Camphorquinone

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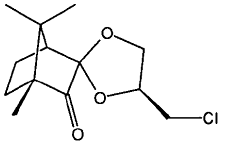
Abstract

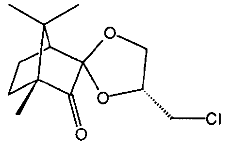
The bicyclo[2.2.1]heptane unit is essentially invariant in all four structures, reflecting its rigid nature. The four isomers (1*S*,2*S*,4*R*,4′*S*)-, (1*S*,2*S*,4*R*,4′*R*)-, (1*S*,2*R*,4*R*,4′*R*)- and (1*S*,2*R*,4*R*,4′*S*)-4,7,7-trimethylbicyclo[2.2.1]heptan-3-one-2-spiro-2′-(4′-chloromethyl-1′,3′-dioxolane), $C_{13}H_{19}ClO_3$, have different positions of substitution by the CH_2Cl group in the ethylene segment of the dioxolane ring. In each case, this ring has an envelope conformation but the identity of the flap atom varies. All four molecules have a similar overall shape and hence give similar packing patterns, resulting in close correspondence or simple relationships for the four sets of unit-cell parameters and the space groups of the structures.

Comment

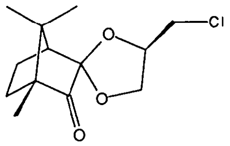
The four isomers, (1)–(4), whose structures are reported here are the major products of the reaction of 3-chloropropane-1,2-diol [$HOCH_2CH(OH)CH_2Cl$] with D-camphorquinone. They are produced by dioxolane formation involving the less sterically hindered of the two carbonyl groups of the quinone, with respective yields for (1)–(4) of 23, 14, 24 and 14%. The corresponding four products obtained in the same reaction from attack on the more hindered carbonyl had yields of 3–10%. All the products were separated by HPLC (Ellis, Golding, Maude & Watson, 1991). The crystal structures of the four major products have been determined to confirm the assignments made on the basis of high-field proton NMR spectroscopy.



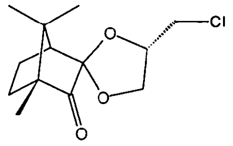
(1)



(2)



(3)



(4)

The structure of the bicyclo[2.2.1]heptane unit is essentially invariant in all four structures, as it is in several hundred other structures in the Cambridge Structural Database (CSD; Allen, Davies, Galloy, Johnson, Kennard, Macrae, Mitchell, Mitchell, Smith & Watson, 1991), reflecting its rigid nature. The four structures differ in the conformation and substitution of their dioxolane rings. All are envelopes, but the identity of the flap atom varies, as is shown by the torsion angles and mean-plane parameters in Table 9; the two disorder components of compound (2) also have different conformations. The dioxolane ring displays a variety of conformations in over 800 structures in the CSD (Allen *et al.*, 1991). In each of the structures (1)–(4), the conformation adopted extends the CH_2Cl substituent away from the bicyclic core. This, despite the different substitution and stereochemistry of the four molecules, makes

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them all of rather similar overall external form; for example, least-squares superposition of the bicyclic units brings all four Cl atoms within 2 Å of each other. The molecules thus have similar packing arrangements, resulting in unit-cell parameters that fall within a small range for both *a* and *b* and represent an approximate doubling of the *c* axes of the orthorhombic structures (3) and (4) compared with those of the monoclinic structures (1) and (2); the two monoclinic cells have *β* angles close to 90°. The molecules have a similar orientation relative to the unit-cell axes in each structure.

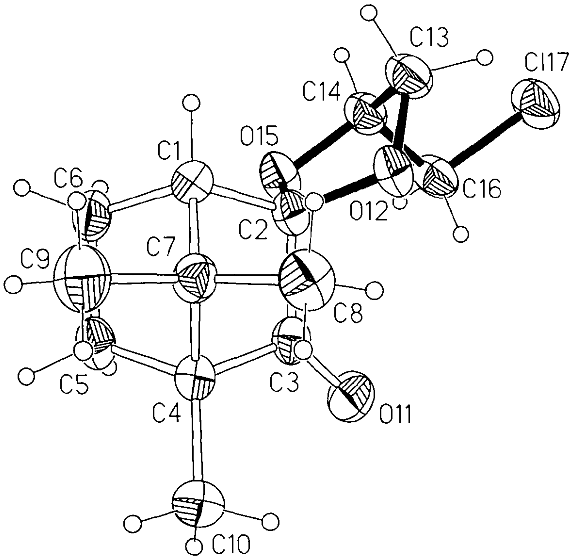

Fig. 1. Structure of compound (1). Atom labels and 50% ellipsoids are shown in all figures, in which the bicyclo[2.2.1]heptane unit is always in the same orientation. H atoms are represented as spheres of arbitrary radii.

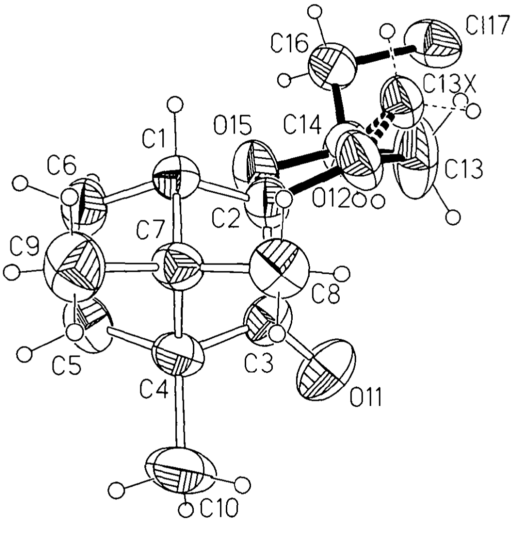

Fig. 2. Structure of compound (2). Both components of the disordered dioxolane ring are shown.

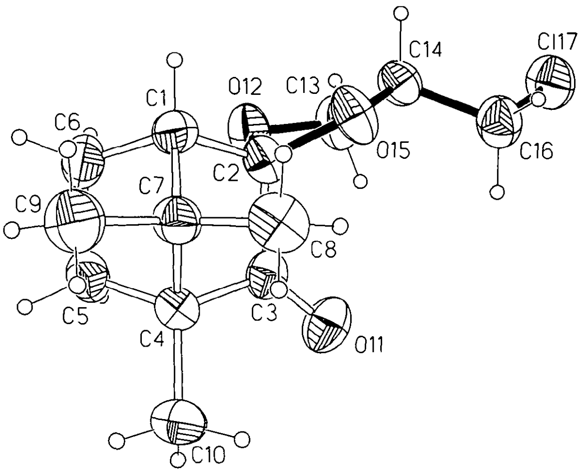

Fig. 3. Structure of compound (3).

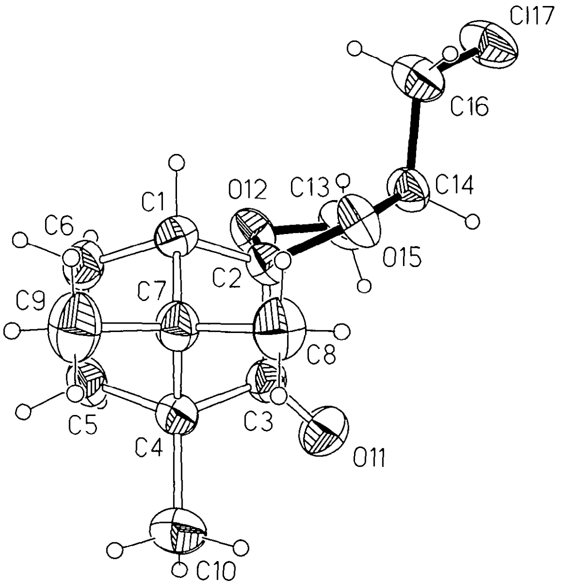

Fig. 4. Structure of compound (4).

Experimental

The preparation of the compounds has already been described (Ellis, Golding, Maude & Watson, 1991). They were recrystallized from aqueous methanol solution.

Compound (1)

Crystal data

$C_{13}H_{19}ClO_3$
$M_r = 258.73$
Monoclinic
$P2_1$
$a = 7.4728\ (14)$ Å
$b = 7.0341\ (12)$ Å
$c = 12.356\ (2)$ Å
$\beta = 92.90\ (2)°$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10.56–12.45°$
$\mu = 0.289\ \mathrm{mm}^{-1}$
$T = 240\ (2)$ K
Block

$V = 648.7\ (2)$ Å^3
$Z = 2$
$D_x = 1.325$ Mg m^{-3}

0.48 × 0.28 × 0.24 mm
Colourless

Data collection

Stoe Siemens diffractometer
ω/θ scans with on-line profile fitting (Clegg, 1981)
Absorption correction: none
3566 measured reflections
2287 independent reflections
2149 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0276$
$\theta_{\rm max} = 24.99°$
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0282$
$wR(F^2) = 0.0762$
$S = 1.054$
2287 reflections
158 parameters
Isotropic H atoms refined with a riding model
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.1598P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.001$

$\Delta\rho_{\rm max} = 0.173$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.175$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin 2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.0100\ (31)$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
C1	0.2836 (3)	0.5487 (3)	0.7615 (2)	0.0304 (4)
C2	0.2635 (2)	0.6670 (3)	0.65743 (13)	0.0283 (4)
C3	0.2740 (2)	0.8734 (3)	0.70297 (15)	0.0281 (4)
C4	0.2982 (2)	0.8543 (3)	0.82544 (15)	0.0295 (4)
C5	0.4924 (3)	0.7764 (3)	0.8430 (2)	0.0354 (5)
C6	0.4807 (3)	0.5679 (3)	0.8034 (2)	0.0373 (5)
C7	0.1836 (2)	0.6724 (3)	0.84377 (14)	0.0303 (4)
C8	−0.0171 (3)	0.7026 (4)	0.8166 (2)	0.0393 (5)
C9	0.2032 (3)	0.5959 (4)	0.9606 (2)	0.0465 (6)
C10	0.2591 (3)	1.0343 (3)	0.8870 (2)	0.0426 (5)
O11	0.2691 (2)	1.0160 (2)	0.64893 (12)	0.0411 (4)
O12	0.0983 (2)	0.6396 (2)	0.59787 (10)	0.0329 (3)
C13	0.1362 (3)	0.5078 (3)	0.5137 (2)	0.0369 (5)
C14	0.3182 (3)	0.5746 (3)	0.4808 (2)	0.0338 (4)
O15	0.4012 (2)	0.6339 (2)	0.58329 (10)	0.0358 (4)
C16	0.3138 (3)	0.7433 (3)	0.4055 (2)	0.0386 (5)
Cl17	0.21572 (8)	0.67850 (8)	0.27543 (4)	0.0517 (2)

Table 2. *Selected geometric parameters* (Å, °) *for* (1)

C2—O12	1.418 (2)	C14—O15	1.443 (2)
C2—O15	1.431 (2)	C14—C16	1.507 (3)
O12—C13	1.432 (2)	C16—Cl17	1.791 (2)
C13—C14	1.515 (3)		
O12—C2—O15	106.31 (13)	O15—C14—C13	101.85 (15)
C2—O12—C13	105.43 (14)	C16—C14—C13	114.7 (2)
O12—C13—C14	102.0 (2)	C2—O15—C14	108.55 (13)
O15—C14—C16	108.0 (2)	C14—C16—Cl17	110.39 (14)

Compound (2)

Crystal data

$C_{13}H_{19}ClO_3$
$M_r = 258.73$
Monoclinic
$P2_1$
$a = 7.2200\ (13)$ Å
$b = 7.6439\ (15)$ Å
$c = 12.148\ (3)$ Å
$\beta = 93.90\ (2)°$
$V = 668.9\ (2)$ Å^3
$Z = 2$
$D_x = 1.285$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10.68–12.45°$
$\mu = 0.280$ mm^{-1}
$T = 240\ (2)$ K
Block
0.44 × 0.40 × 0.28 mm
Colourless

Data collection

Stoe Siemens diffractometer
ω/θ scans with on-line profile fitting (Clegg, 1981)
Absorption correction: none
4174 measured reflections
2375 independent reflections
2273 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0158$
$\theta_{\rm max} = 25.03°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0284$
$wR(F^2) = 0.0756$
$S = 1.088$
2375 reflections
168 parameters
Isotropic H atoms refined with a riding model
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.1103P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.001$

$\Delta\rho_{\rm max} = 0.159$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.140$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin 2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.0227\ (33)$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

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

	x	y	z	$U_{\rm eq}$
C1	0.8920 (2)	0.5103 (2)	0.73858 (14)	0.0421 (4)
C2	0.7468 (3)	0.4855 (3)	0.64304 (15)	0.0440 (4)
C3	0.5637 (2)	0.4977 (2)	0.7042 (2)	0.0432 (4)
C4	0.6215 (2)	0.5328 (3)	0.82408 (14)	0.0445 (4)
C5	0.6991 (3)	0.7217 (3)	0.8239 (2)	0.0558 (5)
C6	0.8864 (3)	0.7049 (3)	0.7706 (2)	0.0541 (5)
C7	0.8017 (3)	0.4199 (3)	0.83678 (15)	0.0419 (4)
C8	0.7647 (3)	0.2239 (3)	0.8227 (2)	0.0501 (5)
C9	0.9141 (3)	0.4466 (3)	0.9472 (2)	0.0630 (6)
C10	0.4698 (3)	0.4996 (4)	0.9027 (2)	0.0702 (7)
O11	0.4100 (2)	0.4837 (2)	0.66032 (14)	0.0642 (4)
O12	0.7615 (2)	0.3231 (2)	0.58833 (11)	0.0560 (4)
C13†	0.6690 (19)	0.3576 (9)	0.4765 (4)	0.085 (3)
C13*X*‡	0.7831 (14)	0.3366 (8)	0.4788 (5)	0.044 (2)
C14	0.6947 (3)	0.5379 (4)	0.45706 (14)	0.0579 (5)
O15	0.7519 (2)	0.6156 (2)	0.56110 (11)	0.0584 (4)
C16	0.8133 (3)	0.6133 (3)	0.3746 (2)	0.0529 (5)
Cl17	0.73625 (7)	0.53893 (9)	0.23916 (4)	0.0626 (2)

† Occupancy 0.61 (2).
‡ Occupancy 0.39 (2).

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

C2—O15	1.409 (2)	C13*X*—C14	1.680 (7)
C2—O12	1.416 (2)	C14—O15	1.432 (2)
O12—C13*X*	1.354 (6)	C14—C16	1.478 (3)
O12—C13	1.496 (7)	C16—Cl17	1.793 (2)
C13—C14	1.413 (6)		
O15—C2—O12	106.31 (14)	O15—C14—C16	106.9 (2)
C13*X*—O12—C2	114.3 (3)	O12—C13*X*—C14	99.0 (4)
C2—O12—C13	103.2 (4)	O15—C14—C13*X*	99.1 (3)
C14—C13—O12	105.6 (3)	C16—C14—C13*X*	103.4 (3)
C13—C14—O15	106.9 (2)	C2—O15—C14	108.1 (2)
C13—C14—C16	125.6 (5)	C14—C16—Cl17	109.9 (2)

Compound (3)

Crystal data

$C_{13}H_{19}ClO_3$
$M_r = 258.73$
Orthorhombic
$P2_12_12_1$
$a = 7.0866\ (14)$ Å
$b = 7.813\ (2)$ Å
$c = 23.956\ (4)$ Å
$V = 1326.4\ (5)$ Å^3
$Z = 4$
$D_x = 1.296$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10.37–12.38°$
$\mu = 0.283$ mm^{-1}
$T = 240\ (2)$ K
Block
0.48 × 0.30 × 0.24 mm
Colourless

Data collection

Stoe Siemens diffractometer
ω/θ scans with on-line profile fitting (Clegg, 1981)
Absorption correction: none
3907 measured reflections
2355 independent reflections
2070 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0177$
$\theta_{\rm max} = 25.04°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -28 \rightarrow 28$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0285$
$wR(F^2) = 0.0714$
$S = 1.046$
2353 reflections
158 parameters
Isotropic H atoms refined with a riding model
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 0.2666P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.002$

$\Delta\rho_{\rm max} = 0.155$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.150$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.0068\ (11)$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 5. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (3)

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

	x	y	z	$U_{\rm eq}$
C1	0.0717 (3)	0.4174 (3)	0.37542 (8)	0.0410 (4)
C2	0.1980 (3)	0.3982 (2)	0.32402 (8)	0.0391 (4)
C3	0.3975 (3)	0.4220 (2)	0.34989 (8)	0.0373 (4)
C4	0.3686 (3)	0.4378 (3)	0.41231 (7)	0.0382 (4)
C5	0.2949 (3)	0.2594 (3)	0.43102 (8)	0.0475 (5)
C6	0.0906 (3)	0.2499 (3)	0.40885 (8)	0.0507 (5)
C7	0.1838 (3)	0.5470 (3)	0.41207 (7)	0.0395 (4)
C8	0.2115 (3)	0.7244 (3)	0.38643 (9)	0.0498 (5)
C9	0.0983 (4)	0.5718 (3)	0.47053 (8)	0.0589 (6)
C10	0.5396 (3)	0.5011 (3)	0.44360 (9)	0.0546 (6)
O11	0.5429 (2)	0.4284 (2)	0.32386 (6)	0.0533 (4)
O12	0.1816 (2)	0.2361 (2)	0.29814 (5)	0.0496 (4)
C13	0.2366 (3)	0.2601 (3)	0.24106 (8)	0.0507 (5)
C14	0.1665 (3)	0.4390 (3)	0.22819 (7)	0.0416 (4)
O15	0.1600 (2)	0.5217 (2)	0.28202 (5)	0.0480 (4)
C16	0.2949 (3)	0.5428 (3)	0.19158 (8)	0.0498 (5)
Cl17	0.30134 (9)	0.45044 (8)	0.12308 (2)	0.0600 (2)

Table 6. *Selected geometric parameters* (Å, °) *for* (3)

C2—O12	1.415 (2)	C14—O15	1.443 (2)
C2—O15	1.420 (2)	C14—C16	1.502 (3)
O12—C13	1.434 (2)	C16—Cl17	1.793 (2)
C13—C14	1.515 (3)		
O12—C2—O15	106.39 (14)	O15—C14—C13	103.97 (14)
C2—O12—C13	106.16 (14)	C16—C14—C13	114.7 (2)
O12—C13—C14	103.0 (2)	C2—O15—C14	108.86 (14)
O15—C14—C16	107.4 (2)	C14—C16—Cl17	109.42 (14)

Compound (4)

Crystal data

$C_{13}H_{19}ClO_3$
$M_r = 258.73$
Orthorhombic
$P2_12_12_1$
$a = 7.364\ (2)$ Å
$b = 7.484\ (2)$ Å
$c = 24.168\ (5)$ Å
$V = 1332.0\ (6)$ Å^3
$Z = 4$
$D_x = 1.290$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 11.55–12.53°$
$\mu = 0.281$ mm^{-1}
$T = 240\ (2)$ K
Block
0.65 × 0.48 × 0.30 mm
Colourless

Data collection

Stoe Siemens diffractometer
ω/θ scans with on-line profile fitting (Clegg, 1981)
Absorption correction: none
4477 measured reflections
2347 independent reflections
2257 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0176$
$\theta_{\rm max} = 25.04°$
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -28 \rightarrow 28$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0269$
$wR(F^2) = 0.0720$
$S = 1.030$
2345 reflections
158 parameters
Isotropic H atoms refined with a riding model
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.2549P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.002$

$\Delta\rho_{\rm max} = 0.187$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.198$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.0131\ (15)$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 7. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (4)

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

	x	y	z	U_{eq}
C1	0.1043 (2)	0.4572 (2)	0.61386 (6)	0.0342 (3)
C2	0.2579 (2)	0.4624 (2)	0.57131 (6)	0.0321 (3)
C3	0.4280 (2)	0.4675 (2)	0.60939 (6)	0.0336 (3)
C4	0.3585 (2)	0.4571 (2)	0.66829 (6)	0.0338 (3)
C5	0.2811 (3)	0.2640 (2)	0.67299 (7)	0.0413 (4)
C6	0.1046 (2)	0.2674 (2)	0.63845 (7)	0.0418 (4)
C7	0.1811 (2)	0.5704 (2)	0.66240 (6)	0.0356 (3)
C8	0.2174 (3)	0.7675 (2)	0.64843 (7)	0.0449 (4)
C9	0.0603 (3)	0.5665 (3)	0.71418 (8)	0.0548 (5)
C10	0.4969 (3)	0.5076 (3)	0.71197 (7)	0.0538 (5)
O11	0.58299 (14)	0.4758 (2)	0.59351 (5)	0.0515 (3)
O12	0.2617 (2)	0.31076 (14)	0.53659 (4)	0.0401 (3)
C13	0.3566 (3)	0.3644 (2)	0.48748 (7)	0.0472 (4)
C14	0.3011 (2)	0.5583 (2)	0.48052 (6)	0.0381 (3)
O15	0.2500 (2)	0.61384 (14)	0.53561 (4)	0.0404 (3)
C16	0.1360 (2)	0.5836 (3)	0.44410 (7)	0.0480 (4)
Cl17	0.19854 (7)	0.54743 (8)	0.37314 (2)	0.0592 (2)

Table 8. *Selected geometric parameters* (Å, °) *for* (4)

C2—O12	1.411 (2)	C14—O15	1.445 (2)
C2—O15	1.426 (2)	C14—C16	1.513 (2)
O12—C13	1.435 (2)	C16—Cl17	1.796 (2)
C13—C14	1.517 (2)		
O12—C2—O15	106.27 (11)	O15—C14—C13	104.07 (12)
C2—O12—C13	106.06 (12)	C16—C14—C13	113.62 (15)
O12—C13—C14	103.19 (12)	C2—O15—C14	108.56 (11)
O15—C14—C16	106.90 (13)	C14—C16—Cl17	109.30 (12)

Table 9. *Dioxolane ring torsion angles* (°), *deviation* (Å) *of flap atom from mean plane of remaining atoms, and r.m.s. deviation* (Å) *for this plane*

Results are given for both components of the disordered structure of (2).

	(1)	(2*a*)	(2*b*)	(3)	(4)
C2—O12—C13—C14	−39.4 (2)	28.5 (9)	−22.1 (6)	−34.4 (2)	−34.5 (2)
O12—C13—C14—O15	35.9 (2)	−15.0 (9)	33.3 (6)	25.0 (2)	23.8 (2)
C13—C14—O15—C2	−20.0 (2)	−4.4 (6)	−34.0 (4)	−7.1 (2)	−4.9 (2)
C14—O15—C2—O12	−3.6 (2)	23.1 (2)	23.1 (2)	−14.0 (2)	−16.3 (2)
O15—C2—O12—C13	27.6 (2)	−31.3 (5)	2.4 (5)	30.8 (2)	32.2 (2)
Flap atom	C13	O12	C14	O12	O12
Δ	0.576	0.454	0.574	0.469	0.476
r.m.s. Δ	0.016	0.019	0.010	0.032	0.022

For all compounds, data collection: *DIF*4 (Stoe & Cie, 1988); cell refinement: *DIF*4; data reduction: local programs; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXL*93 and local programs.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Macrae, C. F., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.

Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.

Ellis, M. K., Golding, B. T., Maude, A. B. & Watson, W. P. (1991). *J. Chem. Soc. Perkin Trans.* 1, pp. 747–755.

Sheldrick, G. M. (1990). *SHELXTL User's Manual.* Version 4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

Stoe & Cie (1988). *DIF4. Diffractometer Control Program.* Version 7.04. Stoe & Cie, Darmstadt, Germany.

Acta Cryst. (1995). C**51**, 1829–1832

Bicyclic [*b*]-Heteroannulated Pyridazine Derivatives. 3. Partially Hydrogenated Triazolo[4,3-*b*]pyridazines

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Abstract

The structures of 6-benzyloxy-7,8-dihydro-8-phenyl-3-trifluoromethyl-*s*-triazolo[4,3-*b*]pyridazine, (1), and its isomer, 5-benzyl-5,6,7,8-tetrahydro-8-phenyl-3-trifluoromethyl-*s*-triazolo[4,3-*b*]pyridazin-6-one, (2), $C_{19}H_{15}F_3N_4O$, have been solved. The two molecules differ in the bonding at N5 and in the conformation of the pyridazine ring which contains this atom. This partially hydrogenated six-membered ring is puckered in both molecules, adopting a half-chair shape in (1) and a twist-boat shape in (2). In both molecules, the triazole ring and the mean plane of the pyridazine ring are not coplanar.

Comment

Many derivatives of bicyclic [*b*]-heteroannulated pyridazine are known as compounds of interesting biological activity. In particular, triazolo[4,3-*b*]pyridazines are classified as ligands of the benzodiazepine receptor with partial agonist activity (Lippa, Crichtett, Sano, Klepner, Greenblatt, Coupet & Beer, 1979; Albright, Moran, Wright Jr, Collins, Beer, Lippa & Greenblatt, 1981;

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