Data collection

Rigaku AFC-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1765 measured reflections
1604 independent reflections
1286 observed reflections [$F > 3\sigma(F)$]

$\theta_{\max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 7$
3 standard reflections monitored every 50 reflections intensity variation: 5%

Refinement

Refinement on F
Final $R = 0.058$
$wR = 0.035$
$S = 3.01$
1286 reflections
155 parameters
All H-atom parameters refined

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.0009$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Program(s) used to solve structure: *TEXSAN* (Molecular Structure Corporation, 1985). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

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

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

	x	y	z	$U_{\rm eq}$
O1	0.5468 (2)	0.8058 (2)	0.4457 (3)	0.0499
O2	0.2754 (2)	0.7567 (2)	0.2255 (4)	0.0698
C1	0.5549 (3)	0.5625 (3)	0.5254 (5)	0.0431
C2	0.5940 (3)	0.6671 (3)	0.3704 (5)	0.0436
C3	0.3703 (4)	0.7974 (4)	0.4363 (7)	0.0623
C4	0.3061 (7)	0.8636 (7)	0.0881 (10)	0.0943
C5	0.7819 (3)	0.7013 (3)	0.3407 (5)	0.0465
C6	0.8973 (5)	0.7681 (5)	0.5611 (6)	0.0679
C7	0.8324 (5)	0.5595 (4)	0.2416 (8)	0.0707
C8	0.7993 (5)	0.8105 (5)	0.1797 (7)	0.0690

Table 2. *Geometric parameters* (Å, °)

O1—C2	1.445 (3)	C2—C5	1.542 (4)
O1—C3	1.409 (4)	C5—C6	1.522 (4)
O2—C3	1.382 (4)	C5—C7	1.524 (5)
O2—C4	1.420 (7)	C5—C8	1.534 (5)
C1—C2	1.497 (4)	C1—C1$^{\rm i}$	1.310 (4)
C2—O1—C3	114.1 (2)	C2—C5—C6	110.9 (3)
C3—O2—C4	113.6 (3)	C2—C5—C7	109.0 (2)
C2—C1—C1$^{\rm i}$	125.6 (3)	C2—C5—C8	108.5 (3)
O1—C2—C1	110.0 (2)	C6—C5—C7	110.1 (3)
O1—C2—C5	107.26 (19)	C6—C5—C8	109.5 (3)
C1—C2—C5	115.4 (2)	C7—C5—C8	108.9 (3)
O1—C3—O2	114.5 (3)		

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

The work was supported partly by a Grant-in-Aid for Scientific Research from the Ministry of Education, Science and Culture.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry including bond distances and angles involving H atoms and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71228 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1056]

References

Arai, M., Nemoto, T., Ohashi, Y. & Nakamura, E. (1992). *Synlett*, pp. 309-310

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.

Acta Cryst. (1993). C**49**, 1802–1804

Structure of *endo*-(5*S**,6*S**,11*R**,12*R**)-5,6,11,12-Tetrahydro-5,6-dihydroxy-4,11,12-trimethoxy-9,13,13-trimethyl-6,10-methano-8(7*H*)-benzocyclodecenone

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(*Received* 23 *February* 1993; *accepted* 19 *April* 1993)

Abstract

It has been confirmed that the title molecule has three fused rings consisting of a six-membered, an eight-membered and a benzene ring. The six-membered ring has a twisted C=C double bond; the dihedral angle between the planes of C10, C11, C15 and C13, C12, C18 is 22.1 (3)° [IUPAC numbering: C11—C10—C(methano) and C8—C9—C(methyl), respectively]. The eight-membered ring adopts a chair-boat conformation.

Comment

The title compound was obtained by Lewis acid promoted eight-membered ring cyclization between C9 and C10. The details of the synthesis will be published elsewhere (Tsuruta, Waizumi, Nakamura, Horiguchi & Kuwajima, 1993). In order to confirm the tricarbocyclic skeleton and the configuration of the substituents, the structure was determined by X-ray diffraction. The molecular structure is shown in Fig. 1.

The eight-membered ring adopts a chair-boat conformation possessing the same stereochemistry as natural taxanes. The six-membered ring has a C11=C12 double bond at a bridgehead site. This causes a substantial twist in the double bond and so the dihedral angle between planes 1 and 2, composed of C10, C11, C15 and C13, C12, C18, respectively, becomes 22.1 (3)°. The crystal structure is shown in Fig. 2. The two hydrogen bonds, O1—HO1···O3 and O1···HO2—O2, which connect the molecules, are related by a twofold screw axis and form a ribbon along the *b* axis. The O1—HO1···O3 and O1···HO2—O2 distances are 3.085 (3) and 3.089 (3) Å, respectively.

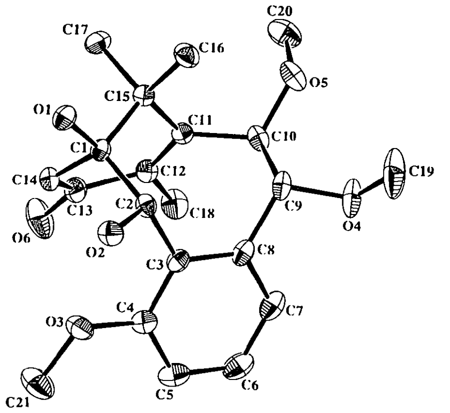

Fig. 1. A perspective view of the molecule (30% probability ellipsoids) with the numbering scheme, excluding H atoms.

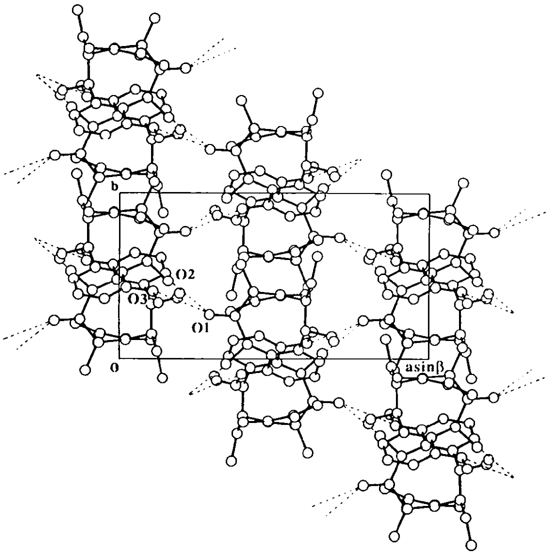

Fig. 2. Crystal structure viewed along the *c* axis. Hydrogen bonds are represented as dashed lines.

Experimental

Crystal data

$C_{21}H_{28}O_6$
M_r = 376.45
Monoclinic
D_x = 1.292 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.541840 Å
$P2_1/n$
a = 15.2330 (8) Å
b = 8.1208 (5) Å
c = 15.6685 (8) Å
β = 93.132 (4)°
V = 1935.4 (2) Å^3
Z = 4
Cell parameters from 25 reflections
θ = 26.84–27.48°
μ = 0.732 mm^{-1}
T = 296.15 K
Prism
0.45 × 0.20 × 0.09 mm
Colorless

Data collection

Rigaku AFC-5*R* four-circle diffractometer
Profile data from $\theta/2\theta$ scans
Absorption correction: none
3476 measured reflections
3341 independent reflections
2349 observed reflections [$F > 3.0\sigma(F)$]
$R_{\rm int}$ = 0.03216
$\theta_{\rm max}$ = 62.5°
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 9$
$l = -18 \rightarrow 17$
3 standard reflections monitored every 100 reflections intensity variation: 0.2%

Refinement

Refinement on F
Final R = 0.0523
wR = 0.0648
S = 2.044
2349 reflections
322 parameters
H-atom parameters not refined
$w^{-1} = \sigma(F_o)^2$
$(\Delta/\sigma)_{\rm max}$ = 0.38
$\Delta\rho_{\rm max}$ = 0.36 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.62 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: AFC-5*R* software (Molecular Structure Corporation, 1988). Cell refinement: AFC-5*R* software. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

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

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

	x	y	z	$U_{\rm eq}$
O1	0.28960 (11)	0.2680 (3)	0.31173 (13)	0.0420
O2	0.34322 (12)	−0.0308 (3)	0.30199 (13)	0.0409
O3	0.39284 (13)	−0.0933 (3)	0.14753 (13)	0.0477
O4	0.68689 (13)	0.1330 (3)	0.39264 (14)	0.0610
O5	0.62055 (14)	0.4548 (3)	0.39422 (13)	0.0524
O6	0.43370 (17)	0.3555 (4)	0.05489 (14)	0.0723
C1	0.37312 (16)	0.2597 (3)	0.27395 (17)	0.0312
C2	0.41266 (17)	0.0871 (3)	0.29983 (18)	0.0314
C3	0.49124 (17)	0.0232 (3)	0.25165 (17)	0.0323
C4	0.47848 (19)	−0.0703 (4)	0.17718 (19)	0.0393
C5	0.5484 (2)	−0.1381 (4)	0.1359 (2)	0.0514
C6	0.6326 (2)	−0.1109 (5)	0.1686 (3)	0.0582
C7	0.6477 (2)	−0.0157 (4)	0.2403 (2)	0.0502
C8	0.57862 (18)	0.0521 (4)	0.28249 (19)	0.0377
C9	0.60146 (18)	0.1699 (4)	0.35629 (19)	0.0405
C10	0.60303 (19)	0.3494 (4)	0.32288 (18)	0.0385
C11	0.51935 (17)	0.3857 (3)	0.26991 (16)	0.0313
C12	0.52014 (18)	0.3796 (4)	0.18403 (17)	0.0346
C13	0.43728 (19)	0.3463 (4)	0.13268 (18)	0.0400
C14	0.35884 (19)	0.2847 (4)	0.17754 (19)	0.0367
C15	0.43057 (18)	0.4025 (3)	0.31155 (17)	0.0333
C16	0.4328 (2)	0.4034 (5)	0.4096 (2)	0.0445

C17	0.3903 (2)	0.5705 (4)	0.2854 (2)	0.0466
C18	0.6010 (2)	0.3862 (5)	0.1326 (2)	0.0496
C19	0.6937 (3)	0.1047 (10)	0.4787 (3)	0.1038
C20	0.6389 (3)	0.6185 (5)	0.3698 (3)	0.0632
C21	0.3749 (3)	−0.1621 (7)	0.0645 (3)	0.0698

Table 2. *Geometric parameters* (Å, °)

O6—C13	1.219 (4)	C7—C8	1.387 (4)
C1—C2	1.570 (4)	C8—C9	1.526 (4)
C1—C14	1.528 (4)	C9—C10	1.549 (4)
C1—C15	1.550 (4)	C10—C11	1.512 (4)
C2—C3	1.540 (4)	C11—C12	1.347 (4)
C3—C4	1.397 (4)	C11—C15	1.539 (4)
C3—C8	1.411 (4)	C12—C13	1.484 (4)
C4—C5	1.389 (5)	C12—C18	1.509 (5)
C5—C6	1.374 (5)	C13—C14	1.504 (4)
C6—C7	1.373 (5)		
O1—C1—C2	105.8 (2)	O4—C9—C10	107.6 (2)
O1—C1—C14	108.7 (2)	C8—C9—C10	109.9 (2)
O1—C1—C15	107.7 (2)	O5—C10—C9	107.9 (2)
C2—C1—C14	113.9 (2)	O5—C10—C11	115.5 (2)
C2—C1—C15	111.7 (2)	C9—C10—C11	109.9 (2)
C14—C1—C15	108.9 (2)	C10—C11—C12	119.2 (2)
O2—C2—C1	109.3 (2)	C10—C11—C15	121.2 (2)
O2—C2—C3	112.5 (2)	C12—C11—C15	118.9 (2)
C1—C2—C3	118.1 (2)	C11—C12—C13	119.6 (3)
C2—C3—C4	121.1 (2)	C11—C12—C18	125.8 (3)
C2—C3—C8	121.3 (2)	C13—C12—C18	114.3 (2)
C4—C3—C8	117.5 (3)	O6—C13—C12	121.4 (3)
O3—C4—C3	116.3 (3)	O6—C13—C14	119.6 (3)
O3—C4—C5	121.8 (3)	C12—C13—C14	118.7 (2)
C3—C4—C5	121.9 (3)	C1—C14—C13	115.6 (2)
C4—C5—C6	119.2 (3)	C1—C15—C11	105.3 (2)
C5—C6—C7	120.5 (3)	C1—C15—C16	111.4 (2)
C6—C7—C8	121.1 (3)	C1—C15—C17	110.7 (2)
C3—C8—C7	119.8 (3)	C11—C15—C16	116.9 (2)
C3—C8—C9	122.4 (2)	C11—C15—C17	108.1 (2)
C7—C8—C9	117.5 (3)	C16—C15—C17	104.4 (3)
O4—C9—C8	109.7 (2)		
C14—C1—C2—C3	46.1 (3)	C8—C9—C10—C11	50.7 (3)
C15—C1—C2—C3	−77.8 (3)	C9—C10—C11—C12	−99.7 (3)
C2—C1—C14—C13	−85.7 (3)	C9—C10—C11—C15	70.6 (3)
C15—C1—C14—C13	39.6 (3)	C10—C11—C15—C1	−116.5 (3)
C2—C1—C15—C11	63.5 (3)	C12—C11—C15—C1	53.8 (3)
C14—C1—C15—C11	−63.2 (3)	C11—C12—C13—O6	172.9 (3)
C1—C2—C3—C8	92.7 (3)	C11—C12—C13—C14	−12.9 (4)
C2—C3—C8—C9	−10.0 (4)	O6—C13—C14—C1	173.5 (3)
C3—C8—C9—C10	−82.0 (3)	C12—C13—C14—C1	−0.9 (4)

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 71260 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1058]

References

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

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

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

Tsuruta, K., Waizumi, N., Nakamura, T., Horiguchi, Y. & Kuwajima, I. (1993). In preparation.

Acta Cryst. (1993). C**49**, 1804–1806

Quinuclidine–Trifluoromonoborane: a (1/1) Lewis Base–Lewis Acid Complex

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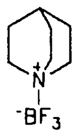
(*Received* 10 *February* 1993; *accepted* 13 *April* 1993)

Abstract

In quinuclidine-trifluoroborane a crystallographic mirror plane bisects the staggered quinuclidine and boron trifluoride groups. The B—N distance [1.601 (7) Å] is identical to that in pyridine-trifluoroborane [1.602 (4) Å; Topel, Hensen & Bats (1984). *Acta Cryst.* C**40**, 828-830] despite the markedly lower basicity of the aromatic amine [pK_a 5.25 (H_2O, 298 K) *versus* 10.95 for quinuclidine].

Comment

Several novel *N*-fluoroquinuclidinium salts have been synthesized recently (Banks, Du Boisson, Morton & Tsiliopoulos, 1988; Banks & Sharif, 1991) as part of the continuing search for safe, easily handled, non-gaseous electrophilic fluorinating agents for use in organic synthesis. The title molecule (I) was prepared in order to determine if changes occur in the quinuclidine moiety on lone-pair donation to a typical Lewis acid. Although the C—N bonds of the title molecule [1.486 (6) and 2×1.514 (5) Å] are longer than the 1.472 Å assumed for quinuclidine (Fourme, 1979) it is impossible to relate these differences to lone-pair donation as both quinuclidine geometries fall in a range defined by known complexes. These complexes include bis(quinuclidine)Br$^+$ (Blair, Parris, Hii & Brock, 1983), bis(quinuclidine)I$^+$ (Brock, Fu, Blair, Chen & Lovell, 1988) and a quinuclidine-carbon tetrabromide charge-transfer complex (Blackstock, Lorand & Kochi, 1987).



(I)

Interestingly, the title compound is isomorphous with the 1:1 complex formed by quinuclidine with sulfur trioxide (Kubas, Larson & Ryan, 1979) [$P2_1/m$, a = 7.955 (4), b = 8.829 (3), c = 6.100 (4) Å, β = 96.03 (4)°].

Acta Crystallographica Section C
ISSN 0108-2701 ©1993