tion of the coordination geometry, it will not necessarily prevent the formation of a square-pyramidal complex. The synthesis of molecules containing the $Me_4C_4O_4^{4-}$ unit (including $Me_4C_4O_4W{=}O$, a potentially square-pyramidal molecule) will be undertaken.

Experimental

Crystal data

$C_8H_{16}O_4$
M_r = 176.2
Monoclinic
$P2_1/m$
a = 10.850 (5) Å
b = 8.206 (4) Å
c = 10.780 (4) Å
β = 114.07 (3)°
V = 876.0 (7) Å^3
Z = 4
D_x = 1.34 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 35 reflections
θ = 8–20°
μ = 0.099 mm^{-1}
T = 130 K
Prism
0.23 × 0.10 × 0.06 mm
Colourless

Data collection

Syntex $P2_1$ diffractometer
ω scans
Absorption correction: none
1322 measured reflections
1245 independent reflections
843 observed reflections [$F>4.0\sigma(F)$]
R_{int} = 0.011

θ_{max} = 22.5°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 18$
$l = -11 \rightarrow 10$
2 standard reflections monitored every 198 reflections intensity variation: ±1.5%

Refinement

Refinement on F
Final R = 0.041
wR = 0.049
S = 1.13
843 reflections
158 parameters
$w = [\sigma^2(F)+0.0007F^2]^{-1}$

$(\Delta/\sigma)_{max}$ = 0.06
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

The diffractometer was equipped with a modified LT-1 low-temperature apparatus and a graphite monochromator was used. Data were collected using ω scans over a 0.90° range at a scan rate of 2.00° min^{-1} and a ±0.9° offset for backgrounds. Anisotropic thermal parameters were used for all non-H atoms.

The observation of the condition $0k0$, $k = 2n$ suggested the space groups $P2_1$ or $P2_1/m$. The structure was solved and successfully refined in the latter space group.

H atoms bonded to C atoms were included at calculated positions, using a riding model with C—H 0.96 Å and fixed isotropic thermal parameters of 0.025 Å^2. Hydroxyl H atoms were located in a final difference map and refined with the additional constraint that O—H be 0.95(2) Å. Some of these were disordered; isotropic thermal parameters for such atoms were allowed to refine.

Computer programs from *SHELXTL* (Sheldrick, 1985) were used.

We thank the donors of the Petroleum Research Fund, administered by the American Chemical Society, for financial support.

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 55246 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1001]

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Structure of the Allene $C_{39}H_{32}P_2{}^{2+}.2CF_3O_3S^-$

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Abstract

In 1,3-propadienebis(triphenylphosphonium) bis(trifluoromethanesulfonate), the cation consists of two phosphane ligands which are connected by a —CH=C=CH— chain. It has point-group symmetry 2, typical of allenes. The central atom of the C_3 chain lies on the symmetry axis.

Comment

The molecule (1) was synthesized as part of a study aimed at stabilizing small molecules of formally 'naked' C atoms by complexation. It can be used to produce a molecular compound that can be considered as a C_3 chain stabilized by two phosphane ligands which act as electron donors by dehydrogenation of the two non-aromatic

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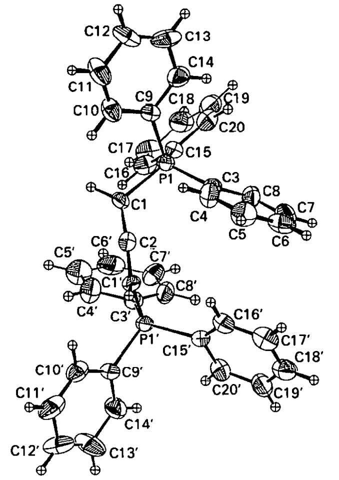
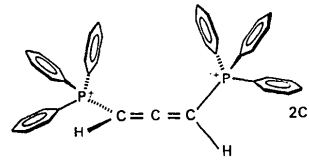


Fig. 1. View of the molecule showing the atomic numbering system. The displacement ellipsoids are plotted at the 30% probability level.

protons. Similar compounds containing C_2 and C_4 chains have already been synthesized using a phosphane ligand as the electron donor and a borane ligand as the electron acceptor (Behl, 1990; Bestmann & Behl, 1989).



(1)

Experimental

Crystal data

$C_{39}H_{32}P_2^{2+}.2CF_3O_3S^-$
$M_r = 860.77$
Orthorhombic
*Ac*2*a*
$a = 19.9686\ (7)$ Å
$b = 16.7330\ (6)$ Å
$c = 11.8636\ (4)$ Å
$V = 3964.0\ (4)$ Å^3
$Z = 4$
$D_x = 1.442$ Mg m^{-3}

Mo $K\alpha_1$ radiation
$\lambda = 0.70930$ Å
Cell parameters from 32 reflections
$\theta = 4.5$-11°
$\mu = 0.2800$ mm^{-1}
$T = 293$ K
Flat plate
0.49 × 0.23 × 0.10 mm
Colourless

Data collection

Rebuilt Philips PW1100 diffractometer (Gomm, 1991)
$\theta/2\theta$ scans
Absorption correction: Gaussian by integration from crystal shape (Busing & Levy, 1957)
$T_{min} = 0.926$, $T_{max} = 0.971$
8847 measured reflections
2215 independent reflections

2052 observed reflections [$F > 1.0\sigma(F)$]
$R_{int} = 0.0128$
$\theta_{max} = 24.5°$
$h = -23 \rightarrow 23$
$k = -4 \rightarrow 19$
$l = -13 \rightarrow 13$
6 standard reflections monitored every 100 reflections
intensity variation: 1.4%

Refinement

Refinement on F
Final $R = 0.041$
$wR = 0.027$
$S = 2.66$
2052 reflections
259 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_{obs})]$

$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974 Vol. IV, Table 2.2B)

Data collection: local software (Gomm, 1991). Cell refinement: local software. Data reduction: local software. Program(s) used to solve structure: *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989). Program(s) used to refine structure: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Software used to prepare material for publication: *CRYSTAN* (Burzlaff & Rothammel, 1988).

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

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

	x	y	z	U_{eq}
P(1)	0.53181 (5)	0.07430 (9)	0.20069 (8)	0.0425
C(1)	0.4891 (2)	0.0080 (2)	0.1073 (3)	0.0453
C(2)	0.5000	0.0116 (4)	0.0000	0.0427
C(3)	0.6033 (2)	0.1152 (2)	0.1304 (3)	0.0477
C(4)	0.6523 (2)	0.0630 (3)	0.1010 (4)	0.0697
C(5)	0.7087 (2)	0.0892 (3)	0.0458 (4)	0.0840
C(6)	0.7162 (2)	0.1682 (4)	0.0234 (4)	0.0760
C(7)	0.6686 (3)	0.2199 (3)	0.0518 (4)	0.0783
C(8)	0.6108 (2)	0.1945 (3)	0.1066 (4)	0.0623
C(9)	0.5589 (2)	0.0173 (3)	0.3196 (3)	0.0447
C(10)	0.5507 (2)	−0.0629 (3)	0.3245 (4)	0.0650
C(11)	0.5703 (3)	−0.1051 (3)	0.4167 (5)	0.0897
C(12)	0.5983 (2)	−0.0669 (4)	0.5064 (5)	0.0817
C(13)	0.6079 (3)	0.0128 (4)	0.5017 (4)	0.0877
C(14)	0.5899 (2)	0.0555 (3)	0.4083 (4)	0.0750
C(15)	0.4725 (2)	0.1476 (2)	0.2444 (3)	0.0457
C(16)	0.4062 (2)	0.1408 (3)	0.2144 (3)	0.0613
C(17)	0.3594 (2)	0.1933 (3)	0.2578 (4)	0.0747
C(18)	0.3781 (3)	0.2515 (3)	0.3318 (4)	0.0740
C(19)	0.4438 (3)	0.2587 (3)	0.3624 (4)	0.0733
C(20)	0.4913 (2)	0.2077 (3)	0.3180 (4)	0.0620
S(1)	0.14671 (5)	0.35048 (9)	0.13894 (9)	0.0598
O(1)	0.1355 (2)	0.3900 (2)	0.0345 (3)	0.0957
O(2)	0.0989 (1)	0.3689 (2)	0.2230 (2)	0.0720
O(3)	0.1636 (2)	0.2677 (2)	0.1316 (3)	0.0993
C(21)	0.2215 (3)	0.3980 (4)	0.1942 (6)	0.1007
F(1)	0.2368 (1)	0.3706 (3)	0.2958 (4)	0.1547
F(2)	0.2123 (2)	0.4752 (2)	0.2039 (4)	0.1497
F(3)	0.2712 (2)	0.3869 (3)	0.1290 (4)	0.2080

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

P(1)—C(1)	1.785 (4)	C(12)—C(13)	1.349 (7)
P(1)—C(3)	1.789 (4)	C(13)—C(14)	1.366 (6)
P(1)—C(9)	1.786 (4)	C(15)—C(16)	1.374 (5)
P(1)—C(15)	1.783 (4)	C(15)—C(20)	1.384 (5)
C(1)—C(2)	1.292 (3)	C(16)—C(17)	1.382 (6)
C(3)—C(4)	1.358 (6)	C(17)—C(18)	1.363 (6)
C(3)—C(8)	1.365 (6)	C(18)—C(19)	1.366 (6)
C(4)—C(5)	1.373 (6)	C(19)—C(20)	1.380 (6)
C(5)—C(6)	1.358 (6)	S(1)—O(1)	1.422 (3)
C(6)—C(7)	1.328 (7)	S(1)—O(2)	1.414 (3)
C(7)—C(8)	1.392 (6)	S(1)—O(3)	1.428 (3)
C(9)—C(10)	1.354 (6)	S(1)—C(21)	1.815 (6)
C(9)—C(14)	1.379 (5)	C(21)—F(1)	1.326 (7)
C(10)—C(11)	1.359 (6)	C(21)—F(2)	1.309 (7)
C(11)—C(12)	1.361 (7)	C(21)—F(3)	1.272 (6)

C(3)—P(1)—C(1)	109.2 (2)	C(14)—C(13)—C(12)	120.9 (5)
C(9)—P(1)—C(1)	107.7 (2)	C(13)—C(14)—C(9)	119.7 (5)
C(9)—P(1)—C(3)	109.3 (2)	C(16)—C(15)—P(1)	120.5 (3)
C(15)—P(1)—C(1)	106.9 (2)	C(20)—C(15)—P(1)	120.2 (3)
C(15)—P(1)—C(3)	113.7 (2)	C(20)—C(15)—C(16)	119.0 (4)
C(15)—P(1)—C(9)	109.8 (2)	C(17)—C(16)—C(15)	120.1 (4)
C(2)—C(1)—P(1)	120.2 (3)	C(18)—C(17)—C(16)	120.6 (4)
C(1)—C(2)—C(1)^i	174.7 (7)	C(19)—C(18)—C(17)	119.8 (4)
C(4)—C(3)—P(1)	116.7 (3)	C(20)—C(19)—C(18)	120.3 (5)
C(8)—C(3)—P(1)	123.8 (4)	C(19)—C(20)—C(15)	120.2 (4)
C(8)—C(3)—C(4)	119.5 (4)	O(2)—S(1)—O(1)	114.0 (2)
C(5)—C(4)—C(3)	120.6 (5)	O(3)—S(1)—O(1)	115.8 (2)
C(6)—C(5)—C(4)	119.7 (5)	O(3)—S(1)—O(2)	114.4 (2)
C(7)—C(6)—C(5)	120.3 (5)	C(21)—S(1)—O(1)	103.9 (3)
C(8)—C(7)—C(6)	121.0 (5)	C(21)—S(1)—O(2)	101.9 (3)
C(7)—C(8)—C(3)	118.9 (5)	C(21)—S(1)—O(3)	104.7 (3)
C(10)—C(9)—P(1)	121.8 (3)	F(1)—C(21)—S(1)	111.5 (4)
C(14)—C(9)—P(1)	119.5 (4)	F(2)—C(21)—S(1)	110.4 (5)
C(14)—C(9)—C(10)	118.7 (4)	F(2)—C(21)—F(1)	107.1 (6)
C(11)—C(10)—C(9)	121.0 (5)	F(3)—C(21)—S(1)	111.0 (5)
C(12)—C(11)—C(10)	120.3 (5)	F(3)—C(21)—F(1)	108.8 (6)
C(13)—C(12)—C(11)	119.3 (5)	F(3)—C(21)—F(2)	108.0 (6)

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

Trifluoromethanesulfonic acid anhydride was dissolved in benzol. An equimolar quantity of 1,3-bis(triphenylphoshoranylidene)acetone (Bestmann & Schlosser, 1979) in benzolic solution was added dropwise. The precipitate was then filtered and recrystallized in a (1:1) mixture of dichloromethane and diethyl ether.

One common isotropic displacement parameter was refined for all the H atoms. Although all the H-atom positions could be recognized in the difference Fourier maps, their free parameters were fixed at calculated positions (C—H 0.95 Å). The polar y direction was fixed by keeping $\Sigma f_i y_i$ constant. The absolute configuration was determined by measuring the Friedel pairs with k indices between -4 and 4 and by refinement of the Flack parameter.

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 55215 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1002]

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Acta Cryst. (1992). C**48**, 1906–1908

{2*S*-[2α,3α,3aβ,6β(*R**),7α,7aα]}-6-(3-Benzyloxy-2-propyl)-2-hydroxy-2,3,7-trimethylhexahydro-4*H*-furo[3,2-*c*]pyran-4-one, a Rearrangement Product of Pyranone Derivatives in the Tirandamycin A Series

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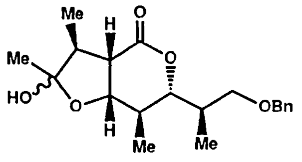
Abstract

The six- and five-membered heterocyclic rings are *cis*-fused and adopt twist-boat and half-chair conformations, respectively. The average bond distances are: Csp^3—Csp^3 1.520 (6), Csp^3—Csp^2 1.494 (6), C—C_{benz} 1.360 (10) and Csp^3—O 1.425 (5) Å; the C=O and Csp^2—O bond lengths are 1.214 (5) and 1.324 (5) Å, respectively.

Comment

During studies for the total synthesis of tirandamycin A (DeShong, Ramesh, Elango & Perez, 1985) and the pheromone of the male swift moth *Hepialus hecta* L. (DeShong, Lin & Perez, 1986), it was demonstrated that pyranones derived from oxidation of furfuryl alcohols rearranged during treatment with HF yielding lactone (1). Confirmation of the structure of lactone (1) was made using the single-crystal X-ray analysis described in this report.



(1)

The structure consists of discrete molecules separated by normal van der Waals distances. The molecules

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