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Structure of 1,3-Bis(triphenylphosphonium)acetone Bis(trifluoromethanesulfonate)

ANDREAS BRAM AND HANS BURZLAFF

Institut für Angewandte Physik, Lehrstuhl für Kristallographie, Universität Erlangen-Nürnberg, Bismarckstrasse 10, D-W-8520 Erlangen, Germany

DARIUSCH HADAWI AND HANS-JUERGEN BESTMANN

Institut für Organische Chemie, Universität Erlangen-Nürnberg, Henkestrasse 42, D-W-8520 Erlangen, Germany

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Abstract

In 2-oxo-1,3-propandiylbis(triphenylphosphonium) bis-(trifluoromethanesulfonate) the cation consists of two phosphane moieties which are connected by an acetone chain —CH_2—CO—CH_2— with the point-group symmetry 2. The central CO group lies on the twofold axis. The structure is stabilized by two SO_3CF_3 anions.

Comment

The title compound can be used to prepare arylmethylene- and alkylidene-acetylmethylidenetriphenylphosphoranes (Bestmann & Schlosser, 1979), which are utilized in many ylid reactions (Cooke & Goswami, 1977; Bestmann, 1965).
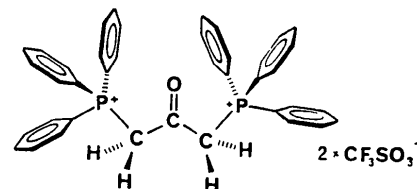
Experimental

Crystal data

$C_{39}H_{34}OP_2^{2+}.2CF_3O_3S^-$	$D_x = 1.425$ Mg m^{-3}
$M_r = 878.79$	Mo $K\alpha_1$ radiation
Monoclinic	$\lambda = 0.70930$ Å
$A2/a$	Cell parameters from 40 reflections
$a = 20.529\ (2)$ Å	$\theta = 7.2\text{–}16.4°$
$b = 17.075\ (2)$ Å	$\mu = 0.274$ mm^{-1}
$c = 11.820\ (1)$ Å	$T = 298$ K
$\beta = 98.65\ (1)°$	Brick shape
$V = 4096\ (1)$ Å^3	$0.42 \times 0.17 \times 0.16$ mm
$Z = 4$	Colourless

Data collection

Rebuilt Philips PW1100 diffractometer (Gomm, 1991)	$R_{int} = 0.038$
	$\theta_{max} = 24.5°$
$\theta/2\theta$ scans	$h = -24 \rightarrow 24$
Absorption correction: none	$k = 0 \rightarrow 19$
	$l = -13 \rightarrow 13$
7082 measured reflections	4 standard reflections
3416 independent reflections	monitored every 100
2717 observed reflections	reflections
[$F > 2.0\sigma(F)$]	intensity variation: 1.8%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.01$
Final $R = 0.061$	$\Delta\rho_{max} = 0.42$ e Å^{-3}
$wR = 0.059$	$\Delta\rho_{min} = -0.42$ e Å^{-3}
$S = 1.49$	Atomic scattering factors
2717 reflections	from *International Tables*
315 parameters	*for X-ray Crystallography*
All H-atom parameters refined with common U_{iso}	(1974, Vol. IV, Table 2.2B)
$w = 1/[\sigma(F_o)]$	

Data collection: local software (Gomm, 1991). Cell refinement: local software (Gomm, 1991). Data reduction: local software. Program(s) used to solve structure: *SIR*88 (Burla, Camalli, Cascerano, 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).
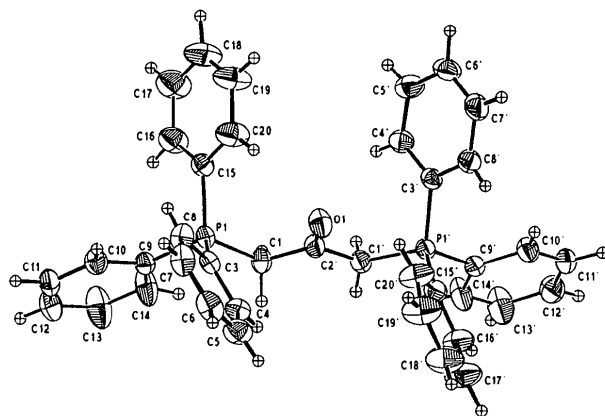

Fig. 1. View of the cation showing the atomic numbering system. The displacement ellipsoids are plotted at the 35% propability level.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)*

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

	x	y	z	U_{eq}
P(1)	0.69791 (5)	0.18583 (5)	0.20206 (7)	0.0407
O(1)	0.7500	0.2483 (2)	0.0000	0.0649
C(1)	0.7293 (2)	0.1297 (2)	0.0945 (3)	0.0482
C(2)	0.7500	0.1760 (3)	0.0000	0.0490
C(3)	0.7591 (2)	0.2551 (2)	0.2632 (3)	0.0434
C(4)	0.8247 (2)	0.2398 (2)	0.2609 (3)	0.0531
C(5)	0.8715 (2)	0.2904 (3)	0.3158 (4)	0.0691
C(6)	0.8535 (3)	0.3546 (3)	0.3728 (4)	0.0690
C(7)	0.7890 (3)	0.3693 (2)	0.3744 (3)	0.0657
C(8)	0.7408 (2)	0.3206 (2)	0.3204 (3)	0.0560
C(9)	0.6818 (2)	0.1187 (2)	0.3112 (3)	0.0426
C(10)	0.6705 (2)	0.1465 (2)	0.4155 (3)	0.0536
C(11)	0.6595 (2)	0.0964 (2)	0.5000 (3)	0.0571
C(12)	0.6594 (3)	0.0183 (3)	0.4828 (4)	0.0675
C(13)	0.6701 (4)	−0.0100 (3)	0.3810 (4)	0.0971
C(14)	0.6816 (3)	0.0389 (2)	0.2942 (4)	0.0792
C(15)	0.6222 (2)	0.2324 (2)	0.1429 (3)	0.0492
C(16)	0.5640 (2)	0.1943 (3)	0.1458 (4)	0.0710
C(17)	0.5057 (3)	0.2268 (4)	0.0946 (5)	0.0961
C(18)	0.5064 (3)	0.2961 (3)	0.0400 (5)	0.0953
C(19)	0.5629 (3)	0.3348 (3)	0.0361 (5)	0.0920
C(20)	0.6219 (3)	0.3031 (3)	0.0869 (5)	0.0769
S(1)	0.60965 (6)	0.49694 (6)	0.33941 (9)	0.0609
O(2)	0.6513 (2)	0.5262 (3)	0.2651 (3)	0.1137
O(3)	0.6266 (2)	0.5202 (2)	0.4532 (3)	0.1269
O(4)	0.5948 (2)	0.4158 (2)	0.3302 (4)	0.1162
C(21)	0.5332 (4)	0.5408 (5)	0.2930 (8)	0.1251
F(1)	0.4864 (2)	0.5200 (3)	0.3461 (6)	0.1890
F(2)	0.5368 (3)	0.6161 (3)	0.2966 (7)	0.2551
F(3)	0.5134 (3)	0.5211 (4)	0.1830 (5)	0.2397

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

P(1)—C(1)	1.787 (4)	C(12)—C(13)	1.345 (7)
P(1)—C(3)	1.796 (3)	C(13)—C(14)	1.371 (6)
P(1)—C(9)	1.794 (3)	C(15)—C(16)	1.365 (6)
P(1)—C(15)	1.792 (4)	C(15)—C(20)	1.377 (5)
O(1)—C(2)	1.233 (6)	C(16)—C(17)	1.374 (7)
C(1)—C(2)	1.483 (5)	C(17)—C(18)	1.348 (7)
C(3)—C(4)	1.376 (6)	C(18)—C(19)	1.343 (8)
C(3)—C(8)	1.389 (5)	C(19)—C(20)	1.377 (6)
C(4)—C(5)	1.380 (6)	S(1)—O(2)	1.407 (4)
C(5)—C(6)	1.367 (7)	S(1)—O(3)	1.395 (3)
C(6)—C(7)	1.351 (7)	S(1)—O(4)	1.419 (3)
C(7)—C(8)	1.374 (6)	S(1)—C(21)	1.752 (8)
C(9)—C(10)	1.373 (5)	C(21)—F(1)	1.274 (9)
C(9)—C(14)	1.376 (5)	C(21)—F(2)	1.288 (9)
C(10)—C(11)	1.360 (5)	C(21)—F(3)	1.345 (9)
C(11)—C(12)	1.348 (6)		
C(3)—P(1)—C(1)	109.5 (2)	C(14)—C(13)—C(12)	121.4 (4)
C(9)—P(1)—C(1)	107.1 (2)	C(13)—C(14)—C(9)	119.4 (4)
C(9)—P(1)—C(3)	109.0 (1)	C(16)—C(15)—P(1)	119.4 (3)
C(15)—P(1)—C(1)	110.1 (2)	C(20)—C(15)—P(1)	121.2 (3)
C(15)—P(1)—C(3)	112.2 (2)	C(20)—C(15)—C(16)	119.3 (4)
C(15)—P(1)—C(9)	108.7 (2)	C(17)—C(16)—C(15)	120.3 (4)
C(2)—C(1)—P(1)	115.1 (3)	C(18)—C(17)—C(16)	119.6 (5)
C(1)—C(2)—O(1)	122.3 (2)	C(19)—C(18)—C(17)	121.3 (5)
C(1)—C(2)—C(1^i)	115.5 (4)	C(20)—C(19)—C(18)	119.9 (5)
C(4)—C(3)—P(1)	119.6 (3)	C(19)—C(20)—C(15)	119.6 (5)
C(8)—C(3)—P(1)	120.2 (3)	O(3)—S(1)—O(2)	114.5 (3)
C(8)—C(3)—C(4)	120.0 (3)	O(4)—S(1)—O(2)	116.3 (3)
C(5)—C(4)—C(3)	119.1 (4)	O(4)—S(1)—O(3)	111.8 (3)
C(6)—C(5)—C(4)	120.9 (5)	C(21)—S(1)—O(2)	104.7 (4)
C(7)—C(6)—C(5)	119.7 (4)	C(21)—S(1)—O(3)	105.2 (4)
C(8)—C(7)—C(6)	121.3 (4)	C(21)—S(1)—O(4)	102.7 (3)
C(7)—C(8)—C(3)	119.0 (4)	F(1)—C(21)—S(1)	115.7 (6)
C(10)—C(9)—P(1)	119.9 (3)	F(2)—C(21)—S(1)	111.8 (6)
C(14)—C(9)—P(1)	121.6 (3)	F(2)—C(21)—F(1)	107.8 (9)
C(14)—C(9)—C(10)	118.5 (4)	F(3)—C(21)—S(1)	108.8 (7)

C(11)—C(10)—C(9)	120.6 (4)	F(3)—C(21)—F(1)	105.4 (7)
C(12)—C(11)—C(10)	120.6 (4)	F(3)—C(21)—F(2)	106.8 (7)
C(13)—C(12)—C(11)	119.5 (4)		

Symmetry code: (i) $\tfrac{3}{2} - x, y, -z$.

Reaction of trifluoromethane sulfonic acid anhydride with 1,3-bis(triphenylphosphoranylidene)acetone in benzene, followed by fractional crystallization in dichloromethane and diethyl ether, yielded the title compound as well as 1,3-propadienediylbis(triphenylphosphonium) bis(trifluoromethanesulfonate) (Bram, Burzlaff, Hadawi & Bestmann, 1992).

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 71102 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1045]

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Structure of 2-(α-Hydroxybenzyl)-8-methyl-8-azabicyclo[3.2.1]octan-3-one

Jun Li, J. Wilson Quail, Guo Zhü Zheng and Marek Majewski

Department of Chemistry, University of Saskatchewan, Saskatoon, Saskatchewan, Canada S7N 0W0

Abstract

The unambiguous structure of the title compound (which was formed in the reaction of the lithium enolate of tropinone with benzaldehyde) has been determined to be the *exo–anti* diastereoisomer. The piperidine ring adopts a flattened-chair conformation with the carbonyl group pushed away from the ethylene bridge. There is an intermolecular hydrogen bond between the hydroxyl group