

## Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.0181$
$\omega$ -2 $\theta$ scans	$\theta_{\text{max}} = 22.45^\circ$
Absorption correction: none	$h = 0 \rightarrow 17$
1589 measured reflections	$k = 0 \rightarrow 14$
1526 independent reflections	$l = -11 \rightarrow 11$
1075 observed reflections [ $I > 2\sigma(I)$ ]	3 standard reflections
	frequency: 60 min
	intensity decay: none

## Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$R(F) = 0.0675$	$\Delta\rho_{\text{max}} = 0.457 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.1194$	$\Delta\rho_{\text{min}} = -0.342 \text{ e } \text{\AA}^{-3}$
$S = 1.062$	Atomic scattering factors
1526 reflections	from <i>International Tables for Crystallography</i> (1992),
157 parameters	Vol. C, Tables 4.2.6.8 and 6.1.1.4)
$w = 1/[\sigma^2(F_o^2) + (0.1011P)^2 + 6.6075P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j$$

	x	y	z	$U_{\text{eq}}$
Cl	0.31683 (10)	0.84307 (13)	0.31915 (15)	0.0561 (6)
Mg	1/2	0.7231 (2)	3/4	0.0358 (7)
OW1	0.3988 (3)	0.7266 (3)	0.6112 (4)	0.0546 (12)
O1	0.5487 (3)	0.8514 (3)	0.6615 (4)	0.0638 (13)
O2	0.3896 (4)	0.8505 (5)	0.4039 (6)	0.132 (3)
O3	0.2782 (4)	0.7527 (5)	0.3360 (9)	0.145 (3)
O4	0.3389 (5)	0.8529 (7)	0.2031 (6)	0.157 (3)
O5	0.2579 (5)	0.9145 (6)	0.3318 (8)	0.155 (3)
N1	1/2	0.5628 (5)	3/4	0.039 (2)
N2	0.5862 (3)	0.6692 (4)	0.6174 (4)	0.0480 (13)
C1	0.5493 (4)	0.5133 (4)	0.6802 (5)	0.047 (2)
C2	0.5509 (5)	0.4111 (5)	0.6790 (7)	0.070 (2)
C3	1/2	0.3615 (8)	3/4	0.080 (3)
C4	0.5990 (4)	0.5781 (5)	0.6059 (6)	0.052 (2)
C5	0.6583 (5)	0.5315 (7)	0.5249 (7)	0.084 (3)
C6	0.6275 (5)	0.7440 (7)	0.5495 (7)	0.077 (2)
C7	0.6165 (9)	0.8395 (8)	0.5988 (14)	0.160 (6)
C8	0.5296 (8)	0.9411 (6)	0.7103 (13)	0.145 (5)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Mg—OW1	2.069 (4)	Mg—N1	2.182 (7)
		Mg—N2	2.231 (5)
OW1 <sup>i</sup> —Mg—OW1	177.4 (3)	O1—Mg—N2	72.5 (2)
OW1—Mg—O1 <sup>i</sup>	90.7 (2)	N1—Mg—N2	70.8 (2)
OW1—Mg—O1	87.2 (2)	OW1—Mg—N2 <sup>i</sup>	89.3 (2)
OW1—Mg—N1	91.3 (2)	O1—Mg—N2 <sup>i</sup>	145.9 (2)
O1—Mg—N1	143.2 (1)	N2—Mg—N2 <sup>i</sup>	141.6 (3)
OW1—Mg—N2	91.6 (2)		

Symmetry code: (i)  $1 - x, y, \frac{3}{2} - z$ .

H atoms were allowed to ride on their parent atoms with displacement factors 1.5 times those of their parent C atoms. The H atom of the water molecule was located and refined with  $U$  fixed at  $0.10 \text{ \AA}^2$ .

Data collection: CAD-4 VAX/PC Fortran System (Enraf–Nonius, 1988). Cell refinement: CAD-4 VAX/PC Fortran System. Data reduction: Xtal3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Pro-

gram(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: SHELXL93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1014). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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### Dimorphism in Actinide Phosphides: Tetrakis[bis(2-dimethylphosphinoethyl)-phosphido]thorium(IV)

SIMON J. COLES, PETER G. EDWARDS,  
MICHAEL B. HURSTHOUSE AND PAUL W. READ

Department of Chemistry, University of Wales, Cardiff,  
PO Box 912, Cardiff CF1 3TB, Wales

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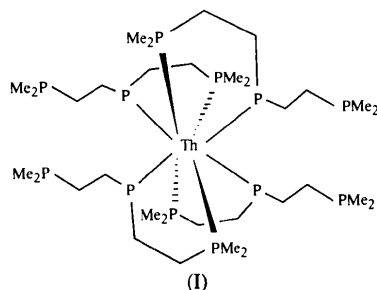
## Abstract

The first example of dimorphism in actinide dialkylphosphides has been observed in the title complex, [Th(C<sub>8</sub>H<sub>20</sub>P<sub>3</sub>)<sub>4</sub>]. The Th atom is eight-coordinate with coordination geometry which may be described as a bi-capped trigonal prism, and is bonded to four phosphido

P atoms, which have approximately planar coordination geometry, and four tertiary phosphine P atoms. The average Th—P<sub>phosphido</sub> and Th—P<sub>phosphino</sub> distances are 2.883 (2) and 3.091 (2) Å, respectively.

### Comment

The determination of the crystal structure of the actinide dialkylphosphido complex [Th{P(CH<sub>2</sub>CH<sub>2</sub>PMe<sub>2</sub>)<sub>2</sub>}<sub>4</sub>] (Edwards, Harman, Hursthouse & Parry, 1992) showed that the Th atom is eight-coordinate with one anomalously long metal–phosphine bond. This long interaction is thought to be responsible for the unique reactivity of the complex (Edwards, Parry & Read, 1994). On recrystallization of the complex from petroleum ether, a new material with identical analytical properties was obtained as crystals of a different morphology. Dialkylphosphido ligands are known to exhibit different bonding modes, such as  $\sigma$  or  $\pi$  bonding (Baker, Whitney & Wreford, 1983), which may have an important influence on the properties of such compounds. Dimorphism has not been reported previously for dialkylphosphido complexes and the causes of dimorphism are of intrinsic interest. For these reasons we have determined the X-ray single-crystal structure of the title complex, (I).



The complex contains eight-coordinate Th in a geometry best described as a bicapped trigonal prism distorted towards a bicapped octahedron. The prism has one trigonal face defined by the atoms P(1), P(10) and P(11) and the second defined by P(4), P(7) and P(8). The prism is capped by the phosphido P atoms P(2) and P(5). A source of distortion is the difference in the thorium–phosphino and the thorium–phosphido bond lengths. Of the four phosphido P atoms which coordinate to the Th atom, one, P(11), has a coordination geometry which is planar within experimental error, while the other three [P(2), P(5) and P(8)] have coordination geometries which deviate only slightly from planarity (the sum of the bond angles around each being 353.5, 351.7 and 357.4°, respectively).

The metal–ligand bond lengths correspond well with those found for the previously reported dimorph (Edwards, Harman, Hursthouse & Parry, 1992). The average Th—P<sub>phosphido</sub> bond length [2.883 (2) Å] is similar to that observed in [Cp<sub>2</sub>Th{P(Cy)<sub>2</sub>}<sub>2</sub>] [2.87 (2) Å] (Cp =

$\eta^5$ -C<sub>5</sub>H<sub>5</sub>, Cy = *cyclo*-C<sub>6</sub>H<sub>11</sub>) (Wroblewski, Ryan, Wasserman, Salazer, Paine & Moody, 1986). The average Th—P<sub>phosphino</sub> bond length [3.091 (2) Å] is a little shorter than that observed in [(PhCH<sub>2</sub>)<sub>4</sub>Th(dmpe)] [3.155 (10) Å] (Ph = C<sub>6</sub>H<sub>5</sub>, dmpe = Me<sub>2</sub>PCH<sub>2</sub>CH<sub>2</sub>PMe<sub>2</sub>) (Edwards, Andersen & Zalkin, 1984). The difference between the average lengths of the formally anionic phosphido–thorium bonds and the formally neutral phosphino–thorium bonds is as expected (Domaille, Foxman, McNeese & Wreford, 1980).

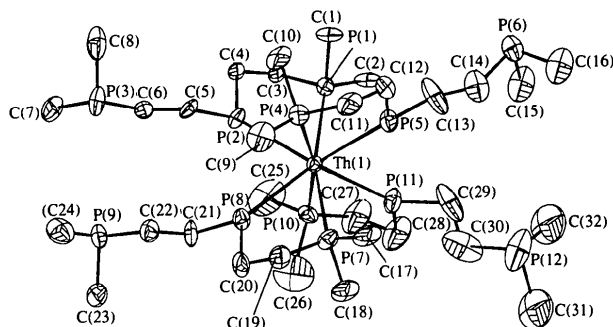


Fig. 1. The molecular structure of the title complex with 50% probability ellipsoids. H atoms have been omitted for clarity.

### Experimental

#### Crystal data

[Th(C<sub>8</sub>H<sub>20</sub>P<sub>3</sub>)<sub>4</sub>]

$M_r = 1068.64$

Monoclinic

$P2_1$

$a = 15.919 (5) \text{ \AA}$

$b = 10.307 (9) \text{ \AA}$

$c = 16.23 (2) \text{ \AA}$

$\beta = 104.93 (4)^\circ$

$V = 2574 (3) \text{ \AA}^3$

$Z = 2$

$D_x = 1.379 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 250 reflections

$\theta = 2.08\text{--}24.93^\circ$

$\mu = 3.290 \text{ mm}^{-1}$

$T = 120 \text{ K}$

Needle

$0.1 \times 0.1 \times 0.08 \text{ mm}$

Red

#### Data collection

Enraf–Nonius diffractometer

with FAST area detector

MADNES scans (Pflugrath & Messerschmidt, 1989)

Absorption correction:

refined from  $\Delta F$

(DIFABS; Walker &

Stuart, 1983)

10 577 measured reflections

6696 independent reflections

3278 observed reflections

$[F_o > 4\sigma(F_o)]$

$R_{\text{int}} = 0.0754$

$\theta_{\text{max}} = 24.93^\circ$

$h = -17 \rightarrow 17$

$k = -9 \rightarrow 11$

$l = -17 \rightarrow 18$

#### Refinement

Refinement on  $F^2$

$R(F) = 0.0447$

$wR(F^2) = 0.1026$

$S = 0.515$

$(\Delta/\sigma)_{\text{max}} = 0.004$

$\Delta\rho_{\text{max}} = 1.21 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.93 \text{ e \AA}^{-3}$

Extinction correction: none

6676 reflections  
380 parameters  
Only H-atom  $U$ 's refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0448P)^2]$   
where  $P = [\max(F_o^2, 0) + 2(F_c^2)]/3$

Atomic scattering factors  
from *International Tables*  
for *X-ray Crystallography*  
(1974, Vol. IV)

P(8)—C(21)	1.903 (9)	P(9)—C(24)	1.837 (4)
P(9)—C(23)	1.837 (4)	P(9)—C(22)	1.842 (4)
P(10)—C(27)	1.74 (2)	P(10)—C(25)	1.840 (11)
P(10)—C(26)	1.874 (13)	P(11)—C(29)	1.843 (13)
P(11)—C(28)	1.919 (12)	P(12)—C(32)	1.829 (5)
P(12)—C(31)	1.851 (4)	P(12)—C(30)	1.854 (4)
C(3)—C(4)	1.591 (14)	C(5)—C(6)	1.559 (11)
C(11)—C(12)	1.52 (2)	C(13)—C(14)	1.485 (13)
C(19)—C(20)	1.543 (13)	C(21)—C(22)	1.488 (14)
C(27)—C(28)	1.60 (2)	C(29)—C(30)	1.47 (2)
P(11)—Th(1)—P(8)	120.81 (9)	P(11)—Th(1)—P(2)	141.23 (8)
P(8)—Th(1)—P(2)	72.46 (8)	P(11)—Th(1)—P(5)	70.82 (8)
P(8)—Th(1)—P(5)	142.63 (8)	P(2)—Th(1)—P(5)	122.85 (9)
P(11)—Th(1)—P(4)	130.43 (8)	P(8)—Th(1)—P(4)	88.35 (9)
P(2)—Th(1)—P(4)	82.49 (9)	P(5)—Th(1)—P(4)	63.31 (9)
P(11)—Th(1)—P(10)	62.09 (8)	P(8)—Th(1)—P(10)	81.84 (9)
P(2)—Th(1)—P(10)	86.87 (9)	P(5)—Th(1)—P(10)	128.95 (10)
P(4)—Th(1)—P(10)	167.30 (8)	P(11)—Th(1)—P(1)	88.74 (10)
P(8)—Th(1)—P(1)	132.45 (8)	P(2)—Th(1)—P(1)	62.51 (8)
P(5)—Th(1)—P(1)	79.03 (9)	P(4)—Th(1)—P(1)	99.73 (8)
P(10)—Th(1)—P(1)	81.30 (8)	P(11)—Th(1)—P(7)	81.36 (10)
P(8)—Th(1)—P(7)	61.64 (9)	P(2)—Th(1)—P(7)	130.77 (9)
P(5)—Th(1)—P(7)	88.20 (10)	P(4)—Th(1)—P(7)	79.50 (9)
P(10)—Th(1)—P(7)	102.58 (9)	P(1)—Th(1)—P(7)	165.90 (7)
C(2)—P(1)—C(1)	100.2 (4)	C(2)—P(1)—C(3)	101.3 (4)
C(1)—P(1)—C(3)	99.5 (4)	C(2)—P(1)—Th(1)	125.6 (3)
C(1)—P(1)—Th(1)	111.4 (3)	C(3)—P(1)—Th(1)	114.9 (3)
C(5)—P(2)—C(4)	101.8 (4)	C(5)—P(2)—Th(1)	127.6 (3)
C(4)—P(2)—Th(1)	124.1 (3)	C(8)—P(3)—C(7)	99.9 (4)
C(8)—P(3)—C(6)	101.0 (3)	C(7)—P(3)—C(6)	98.3 (4)
C(10)—P(4)—C(9)	103.1 (5)	C(10)—P(4)—C(11)	99.1 (5)
C(9)—P(4)—C(11)	105.6 (5)	C(10)—P(4)—Th(1)	113.8 (4)
C(9)—P(4)—Th(1)	119.5 (4)	C(11)—P(4)—Th(1)	113.3 (4)
C(12)—P(5)—C(13)	101.4 (5)	C(12)—P(5)—Th(1)	123.3 (4)
C(13)—P(5)—Th(1)	127.0 (4)	C(16)—P(6)—C(14)	92.0 (5)
C(16)—P(6)—C(15)	95.2 (3)	C(14)—P(6)—C(15)	97.8 (5)
C(17)—P(7)—C(19)	103.9 (5)	C(17)—P(7)—C(18)	101.2 (5)
C(19)—P(7)—C(18)	101.2 (6)	C(17)—P(7)—Th(1)	121.9 (4)
C(19)—P(7)—Th(1)	115.1 (3)	C(18)—P(7)—Th(1)	110.8 (4)
C(20)—P(8)—C(21)	100.5 (4)	C(20)—P(8)—Th(1)	125.9 (3)
C(21)—P(8)—Th(1)	131.0 (3)	C(24)—P(9)—C(23)	99.7 (3)
C(24)—P(9)—C(22)	99.0 (4)	C(23)—P(9)—C(22)	102.0 (4)
C(27)—P(10)—C(25)	103.7 (7)	C(27)—P(10)—C(26)	101.1 (6)
C(25)—P(10)—C(26)	99.9 (6)	C(27)—P(10)—Th(1)	115.3 (4)
C(25)—P(10)—Th(1)	120.1 (4)	C(26)—P(10)—Th(1)	114.0 (4)
C(29)—P(11)—C(28)	101.4 (6)	C(29)—P(11)—Th(1)	133.5 (5)
C(28)—P(11)—Th(1)	125.3 (4)	C(32)—P(12)—C(31)	90.1 (6)
C(32)—P(12)—C(30)	92.8 (6)	C(31)—P(12)—C(30)	97.9 (6)
C(4)—C(3)—P(1)	106.8 (6)	C(3)—C(4)—P(2)	110.3 (6)
C(6)—C(5)—P(2)	117.9 (6)	C(5)—C(6)—P(3)	113.7 (5)
C(12)—C(11)—P(4)	111.4 (7)	C(11)—C(12)—P(5)	110.2 (6)
C(14)—C(13)—P(5)	126.4 (7)	C(13)—C(14)—P(6)	116.4 (6)
C(20)—C(19)—P(7)	114.1 (7)	C(19)—C(20)—P(8)	107.6 (6)
C(22)—C(21)—P(8)	114.2 (6)	C(21)—C(22)—P(9)	115.7 (5)
C(28)—C(27)—P(10)	112.6 (9)	C(27)—C(28)—P(11)	104.8 (8)
C(30)—C(29)—P(11)	117.7 (8)	C(29)—C(30)—P(12)	118.0 (7)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}$
Th(1)	0.2488 (1)	1.0002 (1)	0.2537 (1)	0.026 (1)
P(1)	0.3450 (2)	0.8997 (2)	0.4324 (2)	0.030 (1)
P(2)	0.3821 (2)	0.8074 (2)	0.2632 (2)	0.035 (1)
P(3)	0.5533 (2)	0.6532 (2)	0.0998 (2)	0.045 (1)
P(4)	0.3893 (2)	1.1817 (2)	0.2187 (2)	0.038 (1)
P(5)	0.2791 (2)	1.2406 (2)	0.3494 (2)	0.041 (1)
P(6)	0.2787 (2)	1.3968 (2)	0.6142 (2)	0.057 (1)
P(7)	0.1399 (2)	1.1566 (3)	0.0999 (2)	0.043 (1)
P(8)	0.2266 (2)	0.8947 (2)	0.0851 (2)	0.039 (1)
P(9)	0.2993 (2)	0.5306 (2)	-0.0416 (1)	0.049 (1)
P(10)	0.1257 (2)	0.7732 (2)	0.2674 (2)	0.036 (1)
P(11)	0.1011 (2)	1.0560 (2)	0.3184 (2)	0.045 (1)
P(12)	-0.0615 (2)	1.4184 (3)	0.3154 (2)	0.106 (2)
C(1)	0.4456 (5)	0.9921 (11)	0.4768 (5)	0.047 (2)
C(2)	0.3018 (6)	0.8941 (9)	0.5223 (6)	0.047 (2)
C(3)	0.3917 (6)	0.7340 (8)	0.4341 (6)	0.036 (2)
C(4)	0.4462 (6)	0.7323 (8)	0.3645 (5)	0.036 (2)
C(5)	0.4565 (6)	0.7932 (7)	0.1959 (6)	0.038 (3)
C(6)	0.4802 (3)	0.6543 (8)	0.1712 (2)	0.034 (3)
C(7)	0.5591 (6)	0.4763 (3)	0.0857 (6)	0.060 (4)
C(8)	0.6588 (3)	0.6808 (9)	0.1762 (5)	0.072 (4)
C(9)	0.4023 (7)	1.1954 (11)	0.1109 (6)	0.067 (4)
C(10)	0.5017 (6)	1.1432 (9)	0.2832 (8)	0.060 (4)
C(11)	0.3809 (7)	1.3519 (9)	0.2553 (7)	0.060 (4)
C(12)	0.3646 (7)	1.3552 (8)	0.3435 (7)	0.049 (4)
C(13)	0.2705 (9)	1.2672 (10)	0.4582 (7)	0.084 (4)
C(14)	0.2581 (7)	1.3938 (9)	0.4970 (2)	0.065 (4)
C(15)	0.1865 (3)	1.2937 (6)	0.6230 (7)	0.080 (4)
C(16)	0.2202 (4)	1.5505 (4)	0.6115 (8)	0.080 (4)
C(17)	0.1330 (7)	1.3299 (9)	0.1019 (8)	0.073 (3)
C(18)	0.0233 (7)	1.1109 (9)	0.0764 (8)	0.073 (3)
C(19)	0.1631 (7)	1.1228 (9)	0.0006 (7)	0.053 (4)
C(20)	0.1694 (7)	0.9768 (9)	-0.0180 (6)	0.056 (4)
C(21)	0.2289 (7)	0.7203 (8)	0.0472 (7)	0.047 (4)
C(22)	0.2815 (6)	0.7006 (4)	-0.0154 (6)	0.048 (4)
C(23)	0.1944 (3)	0.4881 (10)	-0.1151 (4)	0.052 (3)
C(24)	0.3639 (3)	0.5581 (9)	-0.1188 (3)	0.072 (5)
C(25)	0.1690 (8)	0.6076 (9)	0.2890 (11)	0.109 (7)
C(26)	0.0378 (9)	0.7433 (13)	0.1675 (8)	0.119 (6)
C(27)	0.0647 (9)	0.7951 (11)	0.3414 (9)	0.087 (4)
C(28)	0.0153 (8)	0.9321 (10)	0.3306 (9)	0.087 (4)
C(29)	0.0578 (9)	1.2067 (10)	0.3528 (8)	0.084 (4)
C(30)	-0.0302 (7)	1.2467 (4)	0.3067 (9)	0.132 (7)
C(31)	-0.1809 (2)	1.3971 (14)	0.2804 (10)	0.144 (6)
C(32)	-0.0648 (7)	1.3993 (4)	0.4266 (3)	0.144 (6)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Th(1)—P(11)	2.870 (4)	Th(1)—P(8)	2.881 (4)
Th(1)—P(2)	2.882 (3)	Th(1)—P(5)	2.898 (3)
Th(1)—P(4)	3.079 (3)	Th(1)—P(10)	3.097 (3)
Th(1)—P(1)	3.089 (3)	Th(1)—P(7)	3.100 (3)
P(1)—C(2)	1.768 (11)	P(1)—C(1)	1.843 (9)
P(1)—C(3)	1.860 (9)	P(2)—C(5)	1.812 (11)
P(2)—C(4)	1.866 (8)	P(3)—C(8)	1.835 (4)
P(3)—C(7)	1.843 (4)	P(3)—C(6)	1.841 (4)
P(4)—C(10)	1.867 (10)	P(4)—C(9)	1.819 (11)
P(4)—C(11)	1.868 (10)	P(5)—C(12)	1.822 (10)
P(5)—C(13)	1.827 (13)	P(6)—C(16)	1.832 (4)
P(6)—C(14)	1.846 (4)	P(6)—C(15)	1.847 (4)
P(7)—C(17)	1.790 (10)	P(7)—C(19)	1.777 (12)
P(7)—C(18)	1.856 (11)	P(8)—C(20)	1.887 (9)

The sample was mounted in Dow Corning high vacuum grease under nitrogen because of its sensitivity to air. The structure was solved *via* heavy-atom methods using *SHELXS86* (Sheldrick, 1990) and refined using *SHELXL93* (Sheldrick, 1993). In order to achieve a stable refinement and sensible geometry parameters, it was necessary to constrain some of the PR<sub>2</sub> C—P bonds to standard values [1.84 (2)  $\text{\AA}$ ]. Refinement of all 10 577 data gave  $R_1$  and  $wR$  values of 0.0950 and 0.1061, respectively, where  $wR_2 = [\sum\{w(F_o^2 - F_c^2)^2\}/\sum\{w(F_o^2)^2\}]^{1/2}$  and  $R_1 = \sum(F_o - F_c)/\sum F_o$ . Bad agreements, edge reflections and reflections occurring behind the beamstop were omitted from the final refinement. A Flack (1983) parameter of 0.003 (10) confirms the correct absolute structure.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: JZ1000). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *syn*-[2,2-Bis(3-isopropyl- $\eta^5$ -cyclopentadienyl)propane]dichlorotitanium

IGOR URAZOWSKI, SHAEN MKOYAN AND LEV ATOVMIAN

*Institute of Chemical Physics, Russian Academy of Sciences, 142432 Chernogolovka, Russia*

BERNARD GAUTHERON,\* ROLAND BROUSSIER AND PATRICE PERRON

*Laboratoire de Synthèse et d'Electrosynthèse Organométalliques, URA 1685, Université de Bourgogne, 6 boulevard Gabriel, 21000 Dijon, France*

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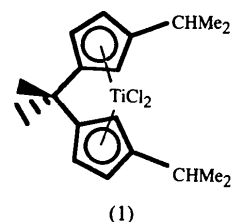
## Abstract

The title complex,  $[\text{TiCl}_2(\text{C}_{19}\text{H}_{26})]$ , belongs to the short-bridged *ansa*-metallocene family, members of which are useful in polymerization processes. It was obtained in a three-step reaction sequence from 2,2-bis(cyclopentadienyl)propane in moderate overall yield. The most important structural feature is the distortion in

the angles caused by the short bridge; the two isopropyl groups do not contribute significantly to the intramolecular tension in the molecule.

## Comment

The *ansa*-metallocene complexes of Group IV transition metals are of interest because of their activity in terminal olefin polymerization processes. Most of the literature in this area, including reports of structure determinations, concerns *ansa*-metallocene molecules with  $C_2$  symmetry (*anti* isomers) which are isolated as racemic mixtures, and which are useful in isospecific polymerization processes (Halterman, 1992). In contrast, there are few reports of the structures of the corresponding molecules with  $C_s$  symmetry (*meso* or *syn* isomers) (Erikson, Fronczek & McLaughlin, 1990; Erker, Wilker, Krüger & Goddard, 1992), such as the title complex, (1).



The crystal structure of (1) shows important distortion caused by the short C(CH<sub>3</sub>)<sub>2</sub> bridge. The ring C—C bond distances are in the range 1.397 (9)–1.412 (7) Å, which is, however, typical for titanium-dicyclopentadienyl complexes (Tkachev & Atovmyan, 1972). The ring angles lie in the range 105.5 (4)–110.9 (5)°, with the smallest angles, 105.5 (4) and 105.9 (5)°, at the ring atoms C(4) and C(6), which are bonded to the bridging group and to the isopropyl substituent, respectively. The mean Ti—C(ring) distance of 2.379 Å is practically the same as in 1,1'-(2,2-propylidene)dicyclopentadienyldimethyltitanium (2.378 Å; Nifant'ev, Churakov, Urazowski,

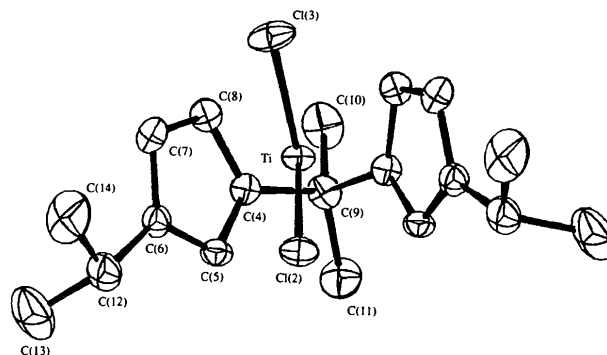


Fig. 1. Molecular structure of the title complex with the atomic labelling scheme. Displacement ellipsoids are shown at 50% probability.