2260 reflections
104 parameters
H atoms riding, except for those of the water molecule
$w = 1/[\sigma^2(F_o^2) + (0.0195P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
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)*

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

	x	y	z	U_{eq}
Pt	0	0	0	0.02928 (10)
Cl	0.2426 (2)	0.14633 (14)	0.0609 (2)	0.0449 (3)
O1	−0.4189 (6)	0.2777 (4)	−0.0870 (6)	0.0575 (10)
O2	0.3679 (6)	−0.1445 (5)	0.4703 (7)	0.0602 (11)
N1	−0.1187 (5)	0.1796 (4)	0.1844 (6)	0.0333 (9)
N2	−0.1296 (6)	0.3599 (4)	0.4820 (6)	0.0413 (10)
N3	0.1382 (6)	0.1485 (5)	0.5359 (6)	0.0481 (11)
C1	−0.0291 (7)	0.2262 (5)	0.4091 (7)	0.0363 (11)
C2	−0.2970 (7)	0.2810 (5)	0.1065 (8)	0.0403 (12)
C3	−0.0892 (9)	0.4291 (6)	0.7130 (8)	0.0544 (14)
C4	−0.3222 (8)	0.4011 (5)	0.2997 (8)	0.0462 (13)

Table 2. *Selected geometric parameters $(\text{Å}, °)$*

Pt—N1	2.022 (3)	N2—C1	1.332 (5)
Pt—Cl	2.2968 (13)	N2—C4	1.448 (6)
O1—C2	1.224 (5)	N2—C3	1.460 (6)
N1—C2	1.363 (6)	N3—C1	1.304 (6)
N1—C1	1.366 (5)	C2—C4	1.510 (6)
N1^i—Pt—N1	180.0	C4—N2—C3	123.2 (4)
N1—Pt—Cl	89.95 (11)	N3—C1—N2	124.3 (4)
Cl^i—Pt—Cl	180.0	N3—C1—N1	123.2 (4)
C2—N1—C1	107.6 (4)	N2—C1—N1	112.5 (4)
C2—N1—Pt	126.2 (3)	O1—C2—N1	126.7 (4)
C1—N1—Pt	126.1 (3)	O1—C2—C4	124.6 (4)
C1—N2—C4	109.0 (4)	N1—C2—C4	108.7 (4)
C1—N2—C3	125.5 (4)	N2—C4—C2	101.5 (4)

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

Table 3. *Hydrogen-bonding geometry $(\text{Å}, °)$*

D—H$\cdots$A	D—H	H$\cdots$A	D$\cdots$A	D—H$\cdots$A
O2—H1$\cdots$O1^i	0.85 (8)	1.91 (8)	2.741 (5)	167 (7)
O2—H2$\cdots$Cl^{ii}	0.78 (8)	2.48 (7)	3.197 (4)	152 (7)
N3—H3A$\cdots$Cl^{iii}	0.86	2.65	3.391 (4)	146
N3—H3B$\cdots$O2	0.86	1.97	2.778 (6)	157

Symmetry codes: (i) $-x, -y, -z$; (ii) $1 - x, -y, 1 - z$; (iii) $x, y, 1 + z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP-Plus* (Frenz, 1985). Program(s) used to solve structure: *MULTAN11/82* (Main *et al.*, 1982). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1117). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Beurskens, P. T., Perales, A., Martín-Gil, F. J. & Martín-Gil, J. (1988). *Monatsh. Chem.* **119**, 1189–1199.

Du Pré, S. & Mendel, H. (1955). *Acta Cryst.* **8**, 311–313.

Enraf–Nonius (1989). *CAD-4 Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Frenz, B. A. (1985). *SDP-Plus Structure Determination Package.* Version 3.0. Enraf–Nonius, Delft, The Netherlands.

Hodgson, D. J. (1977). *Prog. Inorg. Chem.* **23**, 211–254.

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

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Universities of York, England, and Louvain, Belgium.

Martín-Gil, F. J. & Martín-Gil, J. (1987). *Inorg. Chim. Acta,* **137**, 131–134.

Matos Beja, A., Paixão, J. A., Martín-Gil, J. & Salgado, M. A. (1991). *Acta Cryst.* C47, 2333–2336.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A24, 351–359.

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

Udupa, M. R. & Krebs, B. (1979). *Inorg. Chim. Acta,* **33**, 241–244.

(Tetrahydrofuran-O)bis(η^5-1,2,4-triiso-propylcyclopentadienyl)strontium

DAVID J. BURKEY AND TIMOTHY P. HANUSA

Department of Chemistry, Vanderbilt University, Nashville, TN 37235, USA. E-mail: hanusat@ctrvax.vanderbilt.edu
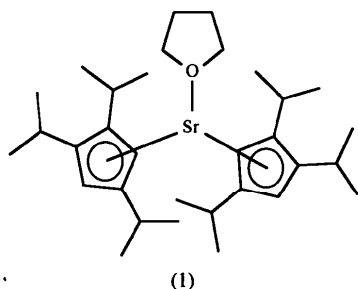
Abstract

The title compound, $[Sr(C_{14}H_{23})_2(C_4H_8O)]$, has twofold crystallographic symmetry and possesses a bent geometry with a Cp—Sr—Cp angle of 139.4 (1)°, where Cp indicates a cyclopentadienyl ring (Cp) centroid. The Sr—O distance is 2.571 (3) Å and the average Sr—C$_{Cp}$ distance is 2.838 (7) Å.

Comment

We have reported previously the synthesis and characterization of a series of alkaline earth metallocenes containing the 1,2,4-triisopropylcyclopentadienyl ligand, $[X\{(C_3H_7)_3C_5H_2\}_2(thf)_n]$ (X = Mg–Ba; n = 0–2; thf is tetrahydrofuran) (Burkey, Williams & Hanusa, 1993; Burkey, Hanusa & Huffman, 1994). During the course of related work, we obtained X-ray quality crystals of one of these complexes, namely $[Sr\{(C_3H_7)_3C_5H_2\}_2(thf)]$,

(1). Considering the paucity of structural information available for organometallic strontium complexes (Hanusa, 1993; Hays, Hanusa & Nile, 1996), we determined the X-ray crystal structure of (1) and present the results here.

(1)

The structure of (1) contains a crystallographically-imposed twofold rotation axis through the Sr—O bond (Fig. 1). The two $[(C_3H_7)_3C_5H_2]^-$ ligands and the thf ligand are arranged in a pseudo-trigonal-planar fashion; the rotation axis requires that the angles subtended by the two ring centroids (Cp) and the thf O atom at the Sr atom sum to 360°. The Cp—Sr—Cp angle in (1) is 139.4 (1)°, which is noticeably larger than the angle of 131.9 (6)° found for the analogous disolvated barium metallocene $[Ba\{(C_3H_7)_3C_5H_2\}_2(thf)_2]$ (Burkey, Williams & Hanusa, 1993). Both these values, however, are much smaller than the Cp—Ca—Cp angle for the unsolvated calcium metallocene $[Ca\{(C_3H_7)_3C_5H_2\}_2]$, which is almost linear [169.7 (4)°; Burkey, Hanusa

& Huffman, 1994]. The cyclopentadienyl ligands in (1) are somewhat displaced from η^5 coordination; the Sr—C bond lengths range from 2.785 (3) for Sr(1)—C(4) to 2.889 (3) Å for Sr(1)—C(2). A similar range of Sr—C distances is present in $[Sr\{('Bu)C_5H_4\}_2(thf)_2]$ [2.797 (11)–2.905 (10) Å; Gardiner, Raston & Kennard, 1991]. The slight ring displacement in (1) brings the Sr atom into close contact with the isopropyl substituent on C(4), as evidenced by the unusually short Sr(1)··· C(13) separation of 3.380 (4) Å.

The Sr—Cp distance in (1) is 2.572 (3) Å, which is similar to the related distance in $[Sr\{(Me_3Si)_2$-$C_5H_3\}_2(thf)]$ of 2.55 (5) Å (Engelhardt, Junk, Raston & White, 1988). The Sr—O bond length in (1) of 2.569 (3) Å is longer than expected, however, considering that the analogous distance in $[Sr\{(Me_3Si)_2C_5H_3\}_2(thf)]$ is 2.49 (3) Å. In $[Sr\{('Bu)$-$C_5H_4\}_2(thf)_2]$, the Sr—O distances are 2.536 (6) and 2.533 (7) Å, which are approximately 0.08 Å shorter than that in (1) when the estimated 0.05 Å difference between seven- and eight-coordinate Sr^{2+} is taken into account (Shannon, 1976).

Experimental

The title compound was prepared according to Burkey, Williams & Hanusa (1993).

Crystal data

$[Sr(C_{14}H_{23})_2(C_4H_8O)]$	Cu $K\alpha$ radiation
$M_r = 542.40$	$\lambda = 1.5418$ Å
Monoclinic	Cell parameters from 15
$C2/c$	reflections
$a = 13.555 (3)$ Å	$\theta = 50.2–73.4°$
$b = 13.059 (4)$ Å	$\mu = 2.480$ mm^{-1}
$c = 18.919 (2)$ Å	$T = 293.2$ K
$\beta = 107.58 (1)°$	Prism
$V = 3192 (1)$ Å^3	$0.33 \times 0.30 \times 0.25$ mm
$Z = 4$	Colorless
$D_x = 1.128$ Mg m^{-3}	
D_m not measured	

Data collection

AFC-6S diffractometer	2058 observed reflections
$\omega/2\theta$ scans	$[I > 3\sigma(I)]$
Absorption correction:	$R_{int} = 0.030$
empirical via ψ scans	$\theta_{max} = 60.07°$
(North, Phillips &	$h = 0 \rightarrow 15$
Mathews, 1968)	$k = 0 \rightarrow 15$
$T_{min} = 0.43$, $T_{max} = 0.54$	$l = -21 \rightarrow 21$
2625 measured reflections	3 standard reflections
2502 independent reflections	monitored every 150
	reflections
	intensity decay: none

Refinement

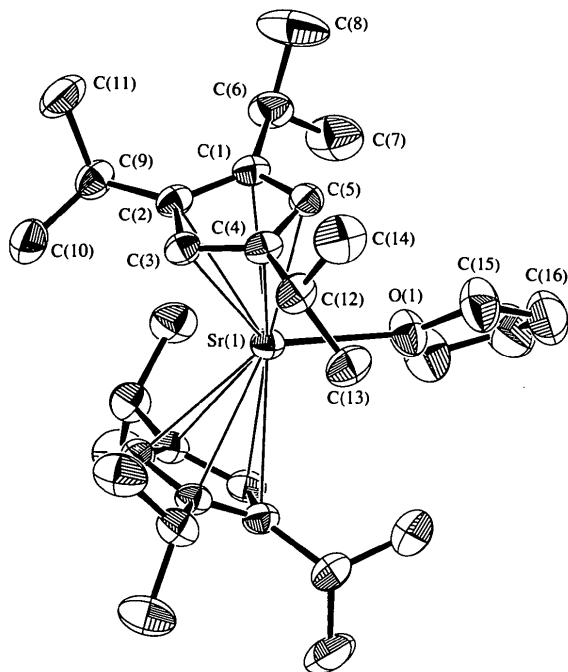|	
Refinement on F	$(\Delta/\sigma)_{max} = 0.0452$
$R = 0.0328$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
$wR = 0.0420$	$\Delta\rho_{min} = -0.40$ e Å^{-3}

Fig. 1. *ORTEP*II (Johnson, 1976) view of the non-H atoms of (1), with displacement ellipsoids drawn at the 30% probability level.

$S = 1.580$
2058 reflections
155 parameters
H-atom parameters riding
$w = 4F_o^2/\sigma^2(F_o^2)$

Extinction correction: none
Atomic scattering factors
from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$)*

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

	x	y	z	U_{eq}
Sr(1)	0	0.13280 (3)	1/4	0.0503 (1)
O(1)	0	−0.0641 (3)	1/4	0.082 (1)
C(1)	0.1510 (2)	0.1492 (2)	0.1708 (2)	0.0562 (9)
C(2)	0.1161 (2)	0.2499 (2)	0.1726 (2)	0.0540 (9)
C(3)	0.1484 (2)	0.2827 (2)	0.2475 (2)	0.0540 (9)
C(4)	0.2033 (2)	0.2035 (3)	0.2922 (2)	0.0554 (9)
C(5)	0.2048 (2)	0.1205 (2)	0.2452 (2)	0.0583 (9)
C(6)	0.1431 (3)	0.0859 (3)	0.1023 (2)	0.079 (1)
C(7)	0.0923 (4)	−0.0156 (4)	0.1007 (3)	0.119 (2)
C(8)	0.2477 (4)	0.0713 (5)	0.0906 (3)	0.149 (3)
C(9)	0.0644 (3)	0.3162 (3)	0.1063 (2)	0.068 (1)
C(10)	0.0007 (3)	0.4018 (3)	0.1223 (3)	0.100 (2)
C(11)	0.1419 (4)	0.3599 (4)	0.0705 (3)	0.106 (2)
C(12)	0.2480 (3)	0.2085 (3)	0.3755 (2)	0.071 (1)
C(13)	0.1889 (3)	0.1418 (3)	0.4150 (2)	0.086 (1)
C(14)	0.3614 (3)	0.1849 (4)	0.4021 (2)	0.109 (2)
C(15)	0.0862 (3)	−0.1277 (3)	0.2870 (3)	0.107 (2)
C(16)	0.0503 (4)	−0.2323 (4)	0.2806 (3)	0.130 (2)

Table 2. *Selected geometric parameters ($\mathring{A}$, °)*

Sr(1)—O(1)	2.571 (3)	C(1)—C(2)	1.402 (4)
Sr(1)—C(1)	2.887 (3)	C(1)—C(5)	1.427 (4)
Sr(1)—C(2)	2.889 (3)	C(2)—C(3)	1.417 (4)
Sr(1)—C(3)	2.818 (3)	C(3)—C(4)	1.399 (4)
Sr(1)—C(4)	2.785 (3)	C(4)—C(5)	1.406 (4)
Sr(1)—C(5)	2.809 (3)		
Sr(1)—O(1)—C(15)	125.5 (2)	C(2)—C(3)—C(4)	109.1 (3)
C(15)—O(1)—C(15^i)	109.0 (4)	C(3)—C(4)—C(5)	107.2 (3)
C(2)—C(1)—C(5)	107.3 (3)	C(1)—C(5)—C(4)	108.7 (3)
C(1)—C(2)—C(3)	107.7 (3)		

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

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985), *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

References

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors*. Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Burkey, D. J., Hanusa, T. P. & Huffman, J. C. (1994). *Adv. Mater. Opt. Electron.* **4**, 1–8.

Burkey, D. J., Williams, R. A. & Hanusa, T. P. (1993). *Organometallics*, **12**, 1331–1337.

Engelhardt, L. M., Junk, P. C., Raston, C. L. & White, A. H. (1988). *J. Chem. Soc. Chem. Commun.* pp. 1500–1501.

Gardiner, M. G., Raston, C. L. & Kennard, C. H. L. (1991). *Organometallics*, **10**, 3680–3686.

Hanusa, T. P. (1993). *Chem. Rev.* **93**, 1023–1036.

Hays, M. L., Hanusa, T. P. & Nile, T. A. (1996). *J. Organomet. Chem.* **514**, 73–79.

Johnson, C. K. (1976). *ORTEP*II. 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.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A24, 351–359.

Shannon, R. D. (1976). *Acta Cryst.* A32, 751–767.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Dicaesium Ethyltrimethylammonium Heptaphosphide–Ammonia (1/2)

NIKOLAUS KORBER AND JÖRG DANIELS

Institut für Anorganische Chemie der Universität Bonn, Gerhard-Domagk-Straße 1, 53121 Bonn, Germany. E-mail: korber@plumbum.chemie.uni-bonn.de

Abstract

The exchange of one Cs^+ cation of Cs_3P_7 for $NEtMe_3^+$ in liquid ammonia gives the title compound, $(NEtMe_3)Cs_2P_7.2NH_3$. The crystal structure determination shows the existence of corrugated two-dimensionally infinite $Cs_2P_7^-$ layers.

Comment

The title compound, (I), was prepared and its structure determined in the course of investigations of the reactions of alkali metal phosphides, $M_3^IP_{11}$ and $M_3^IP_7$ ($M^I =$ Na, K, Rb, Cs), with quaternary alkyl ammonium salts.

One Cs^+ cation of Cs_3P_7 was exchanged for $NEtMe_3^+$ in liquid ammonia yielding $(NEtMe_3)Cs_2P_7.2NH_3$. The

(I)