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[η^2,η^5-Dicyclopentadienyldimethylsilane(2–)]-bis(tetrahydrofuran-κO)magnesium: an Si-bridged magnesocene

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In the title Si-bridged magnesocene, [Mg($C_{12}H_{14}Si$)(C_4H_8O)$_2$], at 87 (2) K, the Mg atom is η^5- and η^2-bonded to the cyclopentadienyl rings. The C_{ipso}—Si—C_{ipso} angle is 102.63 (6)°.

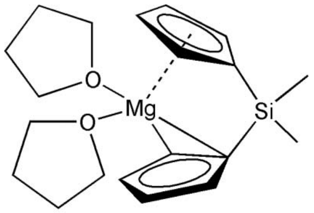
Key indicators

Single-crystal X-ray study
T = 87 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.088
Data-to-parameter ratio = 16.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The molecular structure of the title compound, $C_{20}H_{30}MgO_2Si$, (I), is shown in Fig. 1. It was synthesized as a ligand transfer reagent in our continued synthesis of *ansa*-metallocenes *e.g.* [$Me_4C_2(C_5H_4)_2Al(\mu\text{-}Cl)$]$_2$ (Shapiro *et al.*, 2005). The THF coordination to the magnesium is within the range seen in the few known *ansa*-magnesocene examples (Westerhausen *et al.*, 1998; Damrau *et al.*, 1998; Damrau, 2000; Damrau, Schaper & Brintzinger, 2002). The η^5 cyclopentadienyl (Cp) ring bonding in compound (I) can be grouped into three short [2.4045 (14)–2.4504 (14) Å] and two longer Mg—C bonds [2.5413 (15) and 2.5534 (15) Å]. This η^5 pattern can be seen in [$Me_2Si(\eta^5,\eta^1\text{-3-tBu-}C_5H_3)_2Mg(THF)_2$], (Damrau *et al.*, 1998) with an even more pronounced range between short (2.34–2.41 Å) and long bonds (2.64–2.68 Å). The η^2 interaction with the second ring displays asymmetry in the Mg—C distances (*ca* 0.22 Å), the longer interaction being between the Mg and the ring bridgehead carbon, C6. Magnesocene η^2 interactions are not common and known Mg—C distances range between 2.72 and 2.68 Å, with C—C distances of 1.32–1.46 Å (Atwood & Smith, 1974; Westerhausen *et al.*, 1998; Olmstead *et al.*, 1996; Westerhausen *et al.*, 1998; Damrau, 2000; Damrau, Schaper & Brintzinger, 2002; Damrau, Weeber & Brintzinger, 2002; Xia *et al.*, 2002, 2003; Damrau *et al.*, 2004). In compound (I) the relatively long η^2 C—C distance indicates a decrease in the π-character of the bond. The C_{ispo}—Si—C_{ipso} angle in (I) is close to tetrahedral geometry and is similar to [$Me_2Si(\eta^2,\eta^2\text{-2-Me-indenyl})_2Mg(THF)_2$] (*ca* 105.7 Å; Damrau, 2000; Damrau, Schaper & Brintzinger, 2002) and [$Me_2Si(\eta^5,\eta^2\text{-3-}^t\text{Bu-}C_5H_3)_2Mg(DME)_2$] (102.3, 103.5 Å; Damrau, 2000; Damrau, Weeber & Brintzinger, 2002).



(I)

Experimental

A solution of dibutylmagnesium in heptane (Aldrich, 13.2 mmol, 1 *M*, 8 ml) was added to an ice-bath cooled solution of $Me_2Si(C_5H_5)_2$

(Frisch, 1953; Gomez-Garcia & Royo, 1999) (1.50 g, 7.96 mmol) in heptane (45 ml). The ice bath was removed and the reaction mixture warmed to 343 K. Bubbling was observed when the reaction mixture reached a temperature of *ca* 333 K and a white precipitate gradually formed. The reaction proceeded relatively slowly, so the butane generated by the reaction was simply allowed to exit by the argon line. After stirring under argon at 343 K overnight (15 h), the product was isolated by filtration and washed with pentane (2 × 20 ml) to yield 1.06 g (63%) of $Me_2Si(C_5H_4)_2Mg$ as a white powder. A slow cooling of a supersaturated hot (*ca* 343 K) solution of the magnesocene in a THF/benzene (*ca* 1:5) mixture afforded single crystals of (I).

Crystal data

$[Mg(C_{12}H_{14}Si)(C_4H_8O)_2]$
$M_r = 354.84$
Monoclinic, $P2_1/n$
$a = 8.7260$ (4) Å
$b = 14.7521$ (7) Å
$c = 15.7421$ (7) Å
$\beta = 102.866$ (1)°
$V = 1975.55$ (16) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.16$ mm^{-1}
$T = 87$ (2) K
0.45 × 0.35 × 0.09 mm

Data collection

Bruker–Siemens SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.931$, $T_{max} = 0.982$
30193 measured reflections
3580 independent reflections
3343 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.088$
$S = 1.08$
3580 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—Mg1	2.4504 (14)	C6—Si1	1.8551 (15)
C2—Mg1	2.5534 (15)	C6—Mg1	2.5723 (15)
C3—Mg1	2.5413 (15)	C7—Mg1	2.3529 (15)
C4—Mg1	2.4385 (15)	O1—Mg1	2.0372 (11)
C5—Si1	1.8548 (15)	O2—Mg1	2.0305 (11)
C5—Mg1	2.4045 (14)		
C5—Si1—C6	102.63 (6)	O2—Mg1—O1	89.03 (4)

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.95 (aromatic CH), 0.98 (CH_3) and 0.99 Å (CH_2). For methyl H atoms, $U_{iso}(H) = 1.5U_{eq}(C)$; for all other H atoms, $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *publCIF* (Westrip, 2007).

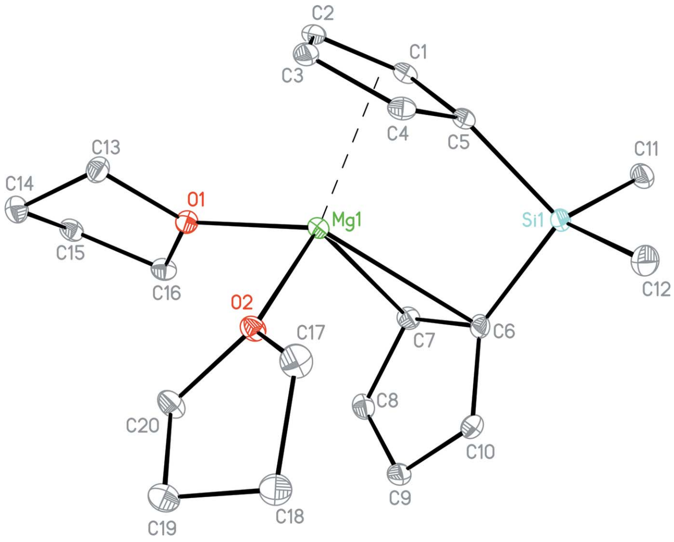


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. The dashed line indicates η^5 bonding.

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References

Atwood, J. L. & Smith, K. D. (1974). *J. Am. Chem. Soc.* **96**, 994–998.
Bruker (2001). *SMART* (Version 5.626), *SAINT-Plus* (Version 6.45a) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2002). *SADABS*. Version 2.03. Bruker AXS Inc., Madison, Wisconsin, USA.
Damrau, H. R. H., Geyer, A., Prosenc, M. H., Weeber, A., Schaper, F. & Brintzinger, H. H. (1998). *J. Organomet. Chem.* **553**, 331–343.
Damrau, H. R. H. (2000). Thesis, Universität Konstanz, Germany.
Damrau, H. R. H., Schaper, F. & Brintzinger, H. H. (2002). Private communication (refcode XILZIB). CCDC, Union Road, Cambridge, England.
Damrau, H. R. H., Schaper, F. & Brintzinger, H. H. (2004). Private communication (refcode ISAREZ). CCDC, Union Road, Cambridge, England.
Damrau, H. R. H., Weeber, A. & Brintzinger, H. H. (2002). Private communication (refcode XIMBAW). CCDC, Union Road, Cambridge, England.
Frisch, K. C. (1953). *J. Am. Chem. Soc.* **75**, 6050–6051.
Gomez-Garcia, R. & Royo, P. J. (1999). *J. Organomet. Chem.* **583**, 86–93.
Olmstead, M. M., Grigsby, W. J., Chacon, D. R., Hascall, T. & Power, P. P. (1996). *Inorg. Chim. Acta*, **251**, 273–284.
Shapiro, P. J., Lee, S.-J., Perrotin, P., Cantrell, T., Blumenfeld, A. & Twamley, B. (2005). *Polyhedron*, **24**, 1366–1381.
Westerhausen, M., Makropoulos, N., Wieneke, B., Karaghiosoff, K., Noth, H., Schwenk-Kircher, H., Knizek, J. & Seifert, T. (1998). *Eur. J. Inorg. Chem.* pp. 965–971.
Westrip, S. P. (2007). *publCIF*. In preparation.
Xia, A., Heeg, M. J. & Winter, C. H. (2002). *J. Am. Chem. Soc.* **124**, 11264–11265.
Xia, A., Knox, J. E., Heeg, M. J., Schlegel, H. B. & Winter, C. H. (2003). *Organometallics*, **22**, 4060–4069.

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