Cyclopentyl{2-[(2,6-diisopropylphenyl)amino]-4-[(2,6-diisopropylphenyl)imino]pent-2-enato-κ^2N,N'}(tetrahydrofuran-κO)magnesium(II)

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Key indicators

Single-crystal X-ray study
$T = 173$ K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.122
Data-to-parameter ratio = 14.5

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

The title compound, [Mg($C_{29}H_{41}N_2$)(C_5H_9)(C_4H_8O)], has tetrahedral coordination geometry around Mg, from three ligands. The material crystallizes in the monoclinic space group $P2_1$ and the data were obtained from a composite of two crystals. A previously reported structure of a chemically different, but structurally similar, compound is also discussed.

Comment

Monoanionic bidentate β-diketiminates [ArNC(Me)CHC\-forcelb](Me)NAr] are excellent ligands for a range of main-group and transition-metal organometallic complexes (Bourget-Merle *et al.*, 2002), especially with Ar = 2,6-iPr$_2$C$_6$H$_3$. Several recent studies have employed this β-diketiminate ligand to stabilize three- and four-coordinate monoalkyl magnesium complexes (Gibson *et al.*, 2000; Bailey *et al.*, 2000, 2001; Prust *et al.*, 2001; Dove *et al.*, 2003). These and related β-diketiminate complexes have proven to be useful synthetic precursors to lactide polymerization catalysts, as reviewed by O'Keefe *et al.* (2001). In the course of our studies of organo-chromium β-diketiminate complexes (Doherty *et al.*, 2003), we isolated crystals of the title compound, [ArNC(Me)CHC(-Me)NAr]Mg(C_5H_9)(THF) (THF is tetrahydrofuran), a complex analogous to the recently reported monoalkyl magnesium complexes cited above.

Ar = 2,6-(CHMe$_2$)C$_6$H$_3$

(I)
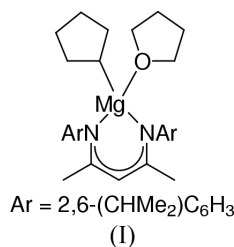
Experimental

All operations were performed in a nitrogen-filled glove-box, and all solvents were dried and deoxygenated prior to use. To a solution of CpCr[ArNC(Me)CHC(Me)NAr] (Doherty *et al.*, 2003) (0.563 mmol) in Et$_2$O was added silver trifluoromethanesulfonate (0.568 mmol). After 2 h, the solution was filtered to remove the Ag-containing by-product, and the solvent was removed *in vacuo*. The residue was extracted with pentane, the solution was filtered and the solvent was again removed *in vacuo*. The residue was dissolved in THF, and 2.0 *M* cyclopentylmagnesium chloride (0.30 ml) was added. After 20 min, the solvent was removed *in vacuo*. The residue was extracted with pentane and the solution was filtered. Crystals suitable for X-ray analysis were obtained by cooling the pentane solution to 243 K overnight.

Crystal data

$[Mg(C_{29}H_{41}N_2)(C_5H_9)(C_4H_8O)]$
$M_r = 583.17$
Monoclinic, $P2_1$
$a = 9.8292\ (7)$ Å
$b = 16.631\ (1)$ Å
$c = 11.5822\ (8)$ Å
$\beta = 109.52\ (1)°$
$V = 1784.5\ (2)$ Å^3
$Z = 2$

$D_x = 1.085$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5264
  reflections
$\theta = 1.9–25.3°$
$\mu = 0.08$ mm^{-1}
$T = 173\ (2)$ K
Irregular, red
$0.50 \times 0.20 \times 0.20$ mm

Data collection

Rigaku/ADSC CCD area-detector
  diffractometer
ω and φ scans
Absorption correction: multi-scan
  (Blessing, 1995)
  $T_{min} = 0.632$, $T_{max} = 0.849$
25 493 measured reflections

6009 independent reflections
4812 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 25.1°$
$h = 0 \rightarrow 11$
$k = -19 \rightarrow 18$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.122$
$S = 0.97$
6009 reflections
414 parameters

H atoms treated by a mixture of
  independent and constrained
  refinement
$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Initial attempts to index the data using *d*TREK* (Molecular Structure Corporation, 2001) were unsuccessful. The TwinSolve function in *CrystalClear* (Rigaku/MSC, 2003) determined that the crystal was in fact a two-component 'split crystal', with the components related by a 29.4° rotation about an axis normal to (1, 0, 0). The data for both components were integrated, including all overlapped reflections. Both the cyclopentyl and the THF ligands are disordered. In the case of the cyclopentyl ligand, the four uncoordinated atoms of the ring are disordered. Restraints on bond lengths and angles were employed to maintain reasonable geometries and each atom was refined isotropically. In the case of THF, only one atom in the ring (C36) is disordered and all non-H atoms other than the minor disordered atom were refined anisotropically. All H atoms were included in calculated positions (C—H = 0.95–1.00 Å) and treated as riding [$U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$], except for H30, the methine H atom on C30, which was located in a difference map and refined isotropically [C30—H30 = 1.06 (5) Å]. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration could not be determined and has been assigned arbitrarily. In the course of preparing this manuscript, a remarkably similar structure (Chisholm *et al.*, 2001) was discovered. This latter material differs only by the presence of an O-*tert*-butyl ligand rather than the cyclopentyl ligand reported here. The cell parameters and space group are the same. In order to ascertain that the two structures were indeed different, the coordinates of the O-^{t}Bu structure were refined with the data from the cyclopentyl structure. $R1$ and $wR2$ from the cyclopentyl structure rose from 0.051 and 0.122, respectively, to 0.124 and 0.339, respectively. Only the atoms of the O-^{t}Bu ligand shifted, and a post-refinement difference synthesis generated large residual peaks in positions inconsistent with the original O-^{t}Bu structure.
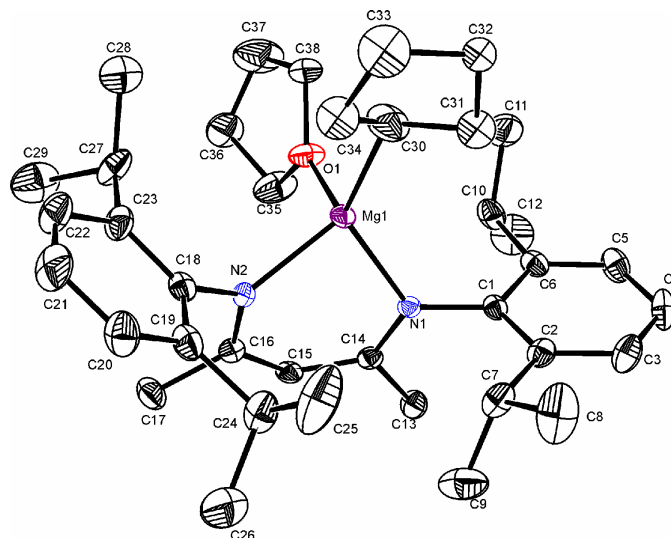

Figure 1
The molecular structure with atom labels. H atoms have been omitted, and only one component of each disordered group is shown. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *d*TREK* (Molecular Structure Corporation, 2001); cell refinement: *CrystalClear* (Rigaku/MSC, 2003); data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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