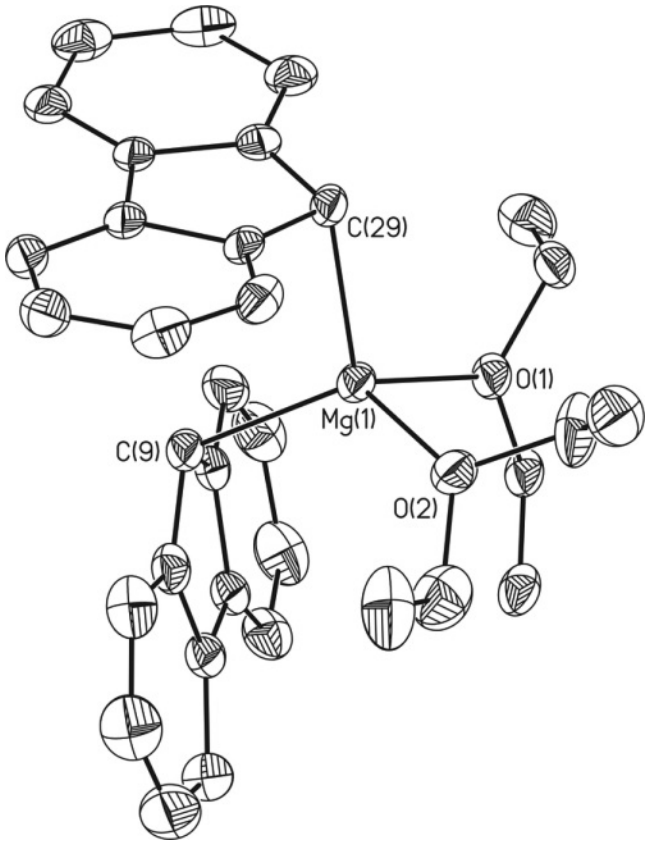
Crystal structure of *bis*(diethylether)*bis*(fluorenyl)magnesium, $[Mg(C_{13}H_9)_2(Et_2O)_2]$, $C_{34}H_{38}MgO_2$

Ulrich Behrens* and Elena Aglaia Dimos

Institut für Anorganische und Angewandte Chemie der Universität Hamburg, Martin-Luther-King-Platz 6, 20146 Hamburg, Germany

Received April 02, 2012, accepted September 04, 2012, available online November 09, 2012, CCDC no. 908387



Abstract

$C_{34}H_{38}MgO_2$, monoclinic, $P2_1/c$ (no. 14), a = 10.321(1) Å, b = 16.365(2) Å, c = 16.981(2) Å, β = 104.615(2)°, V = 2775.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.0595, $wR_{ref}(F^2)$ = 0.1665, T = 153 K.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.12×0.19×0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.93 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	29969, 5453
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3531
$N(param)_{refined}$:	344
Programs:	SHELX [2]

Source of material

Bis(fluorenyl)magnesium [1] (260 mg, 0.733 mmol) was dissolved in diethylether (5 mL) and the solution covered with a layer of *n*-hexane to give yellow crystals suitable for X-ray analysis [2].

Formula: *Bis*(diethylether)*bis*(fluorenyl)magnesium, $[Mg(C_{13}H_9)_2(Et_2O)_2]$. Solubility at room temperature: good in tetrahydrofuran, diethylether and toluene, poor in *n*-hexane.

Discussion

In *bis*(diethylether)*bis*(fluorenyl)magnesium the Mg^{2+} ion is surrounded in a distorted tetrahedral manner by two η^1-bound fluorenyl anions and two ether ligands. There is only a sigma-bond between the bridging C atom of the fluorenyl unit and the Mg atom (Mg–C 2.235 Å). In the known complex [Mg(CH_3)(sigma-fluorenyl)(tmeda)] [3], the only related Mg compound, the Mg–C(fluorenyl) bond is slightly longer (Mg–C 2.273)Å.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.9009	0.4913	0.2253	0.058
H(2)	4*e*	1.0507	0.5579	0.3306	0.071
H(3)	4*e*	0.9888	0.5996	0.4468	0.073
H(4)	4*e*	0.7692	0.5821	0.4572	0.054
H(5)	4*e*	0.4998	0.5555	0.4309	0.050
H(6)	4*e*	0.2747	0.5241	0.3807	0.062
H(7)	4*e*	0.1967	0.4662	0.2519	0.062
H(8)	4*e*	0.3441	0.4316	0.1734	0.052
H(9)	4*e*	0.621(2)	0.450(2)	0.1628(4)	0.041
H(21)	4*e*	0.3769	0.2233	0.0910	0.047
H(22)	4*e*	0.2156	0.2975	–0.0017	0.054
H(23)	4*e*	0.2756	0.4074	–0.0733	0.054
H(24)	4*e*	0.5002	0.4436	–0.0533	0.044
H(25)	4*e*	0.7734	0.4580	–0.0090	0.043
H(26)	4*e*	1.0032	0.4391	0.0385	0.052
H(27)	4*e*	1.0882	0.3308	0.1260	0.055
H(28)	4*e*	0.9450	0.2434	0.1720	0.047
H(29)	4*e*	0.668(2)	0.1978(5)	0.149(2)	0.037
H(41A)	4*e*	0.4727	0.2630	0.4096	0.043
H(41B)	4*e*	0.4456	0.3507	0.3664	0.043
H(42A)	4*e*	0.7008	0.2927	0.4547	0.062
H(42B)	4*e*	0.6170	0.3632	0.4849	0.062
H(42C)	4*e*	0.6780	0.3787	0.4083	0.062
H(43A)	4*e*	0.4661	0.1590	0.3163	0.047
H(43B)	4*e*	0.4788	0.1810	0.2268	0.047
H(44A)	4*e*	0.2788	0.2401	0.3000	0.076
H(44B)	4*e*	0.2553	0.1805	0.2230	0.076
H(44C)	4*e*	0.2965	0.2739	0.2149	0.076
H(45A)	4*e*	0.7475	0.1674	0.3117	0.072
H(45B)	4*e*	0.8192	0.1910	0.4035	0.072
H(46A)	4*e*	0.9657	0.1487	0.2911	0.087
H(46B)	4*e*	0.9213	0.0831	0.3491	0.087
H(46C)	4*e*	1.0212	0.1556	0.3877	0.087
H(47A)	4*e*	0.8985	0.3773	0.3914	0.080
H(47B)	4*e*	0.9554	0.2935	0.4360	0.080
H(48A)	4*e*	1.1004	0.2799	0.3598	0.120
H(48B)	4*e*	1.1135	0.3719	0.3927	0.120
H(48C)	4*e*	1.0324	0.3530	0.3011	0.120

* Correspondence author (e-mail: ubehrens@chemie.uni-hamburg.de)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mg(1)	4*e*	0.66595(9)	0.32312(5)	0.24635(5)	0.0376(5)	0.0274(5)	0.0221(5)	0.0017(4)	0.0087(4)	0.0008(4)
C(1)	4*e*	0.8730(3)	0.5095(2)	0.2715(2)	0.048(2)	0.046(2)	0.058(2)	0.008(2)	0.026(2)	0.007(2)
C(2)	4*e*	0.9617(3)	0.5485(2)	0.3345(2)	0.037(2)	0.066(2)	0.075(3)	0.000(2)	0.012(2)	0.006(2)
C(3)	4*e*	0.9244(3)	0.5746(2)	0.4036(2)	0.048(2)	0.062(2)	0.061(2)	–0.006(2)	–0.007(2)	0.002(2)
C(4)	4*e*	0.7945(3)	0.5644(2)	0.4099(2)	0.051(2)	0.042(2)	0.039(2)	–0.002(1)	0.007(2)	–0.002(1)
C(5)	4*e*	0.4690(3)	0.5310(2)	0.3788(2)	0.049(2)	0.031(2)	0.049(2)	0.004(1)	0.020(2)	–0.005(1)
C(6)	4*e*	0.3360(3)	0.5125(2)	0.3489(2)	0.047(2)	0.038(2)	0.075(2)	0.008(1)	0.026(2)	0.002(2)
C(7)	4*e*	0.2896(3)	0.4768(2)	0.2722(2)	0.040(2)	0.040(2)	0.072(2)	0.001(1)	0.006(2)	0.005(2)
C(8)	4*e*	0.3768(3)	0.4566(2)	0.2251(2)	0.047(2)	0.028(2)	0.046(2)	0.001(1)	–0.005(2)	0.002(1)
C(9)	4*e*	0.6274(3)	0.4565(2)	0.2222(2)	0.056(2)	0.024(1)	0.025(1)	0.000(1)	0.013(1)	0.000(1)
C(10)	4*e*	0.7413(3)	0.4968(2)	0.2765(2)	0.046(2)	0.025(1)	0.038(2)	0.006(1)	0.015(1)	0.006(1)
C(11)	4*e*	0.7008(3)	0.5274(2)	0.3455(2)	0.042(2)	0.024(1)	0.033(2)	0.003(1)	0.010(1)	0.001(1)
C(12)	4*e*	0.5590(3)	0.5133(1)	0.3314(2)	0.040(2)	0.021(1)	0.034(2)	0.003(1)	0.009(1)	0.001(1)
C(13)	4*e*	0.5136(3)	0.4734(1)	0.2544(2)	0.044(2)	0.020(1)	0.032(1)	0.003(1)	0.006(1)	0.005(1)
C(21)	4*e*	0.4029(3)	0.2678(2)	0.0625(2)	0.046(2)	0.044(2)	0.029(2)	–0.007(1)	0.014(1)	–0.011(1)
C(22)	4*e*	0.3073(3)	0.3120(2)	0.0072(2)	0.036(2)	0.059(2)	0.038(2)	–0.001(1)	0.007(1)	–0.019(2)
C(23)	4*e*	0.3430(3)	0.3776(2)	–0.0359(2)	0.048(2)	0.045(2)	0.036(2)	0.014(1)	–0.000(1)	–0.014(1)
C(24)	4*e*	0.4760(3)	0.3991(2)	–0.0240(2)	0.052(2)	0.027(1)	0.028(1)	0.004(1)	0.004(1)	–0.007(1)
C(25)	4*e*	0.8073(3)	0.4145(2)	0.0275(2)	0.051(2)	0.030(1)	0.029(2)	–0.002(1)	0.014(1)	–0.001(1)
C(26)	4*e*	0.9433(3)	0.4031(2)	0.0555(2)	0.052(2)	0.045(2)	0.039(2)	–0.013(2)	0.022(2)	–0.005(1)
C(27)	4*e*	0.9941(3)	0.3388(2)	0.1085(2)	0.036(2)	0.062(2)	0.041(2)	0.002(1)	0.012(1)	–0.005(2)
C(28)	4*e*	0.9091(3)	0.2868(2)	0.1359(2)	0.045(2)	0.041(2)	0.030(2)	0.010(1)	0.010(1)	0.003(1)
C(29)	4*e*	0.6590(3)	0.2560(2)	0.1309(2)	0.044(2)	0.026(1)	0.023(1)	0.001(1)	0.012(1)	0.002(1)
C(30)	4*e*	0.5391(3)	0.2890(2)	0.0765(2)	0.043(2)	0.029(1)	0.023(1)	–0.002(1)	0.012(1)	–0.009(1)
C(31)	4*e*	0.5743(3)	0.3555(2)	0.0309(1)	0.040(2)	0.024(1)	0.020(1)	0.001(1)	0.007(1)	–0.006(1)
C(32)	4*e*	0.7192(3)	0.3617(2)	0.0529(1)	0.042(2)	0.026(1)	0.022(1)	0.001(1)	0.010(1)	–0.005(1)
C(33)	4*e*	0.7704(3)	0.2980(2)	0.1103(1)	0.041(2)	0.029(1)	0.019(1)	0.002(1)	0.010(1)	–0.003(1)
O(1)	4*e*	0.5408(2)	0.2719(1)	0.3081(1)	0.054(1)	0.0277(9)	0.027(1)	–0.0001(8)	0.0195(9)	0.0000(8)
C(41)	4*e*	0.5117(3)	0.3059(2)	0.3815(2)	0.060(2)	0.028(1)	0.027(1)	0.003(1)	0.022(1)	0.001(1)
C(42)	4*e*	0.6378(3)	0.3379(2)	0.4372(2)	0.064(2)	0.036(2)	0.029(2)	0.009(1)	0.021(1)	0.004(1)
C(43)	4*e*	0.4530(3)	0.2029(2)	0.2750(2)	0.061(2)	0.025(1)	0.036(2)	–0.005(1)	0.021(1)	–0.003(1)
C(44)	4*e*	0.3085(3)	0.2264(2)	0.2512(2)	0.054(2)	0.046(2)	0.056(2)	–0.008(2)	0.021(2)	–0.011(2)
O(2)	4*e*	0.8370(2)	0.2771(1)	0.3228(1)	0.046(1)	0.035(1)	0.030(1)	0.0044(9)	0.0027(9)	0.0006(8)
C(45)	4*e*	0.8293(4)	0.1920(2)	0.3471(2)	0.083(3)	0.043(2)	0.062(2)	0.027(2)	0.036(2)	0.023(2)
C(46)	4*e*	0.9438(3)	0.1406(2)	0.3434(2)	0.065(2)	0.047(2)	0.061(2)	0.013(2)	0.015(2)	0.001(2)
C(47)	4*e*	0.9353(4)	0.3229(2)	0.3833(2)	0.069(2)	0.069(2)	0.058(2)	0.013(2)	0.013(2)	0.007(2)
C(48)	4*e*	1.0547(4)	0.3327(2)	0.3572(3)	0.095(3)	0.065(2)	0.091(3)	0.013(2)	0.046(3)	0.027(2)

References

1. Burns, D. M.; Iball, J.: The Crystal and Molecular Structure of Fluorene. Proc. R. Soc. London. Ser. **A227** (1995) 200-214.
2. a) Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
   b) Sheldrick, G. M.: Phase annealing in SHELX-90: direct methods for larger structures. Acta Crystallogr. **A46** (1990) 467-473.
3. Viebrock, H.; Abeln, D.; Weiss, E.: Ueber Metallalkyl- und -arylverbindungen, 51. Mittlg. Unsymmetrische Diorganomagnesiumverbindungen. MgRR´(L) und solvensgetrennte Ionenpaare mit R = Me, Et, R´= Cyclopentadienyl, Indenyl, Fluorenyl, Phenylethinyl, L = Tetramethylethylendiamin, Pentamethyldiethylentriamin. Z. Naturforsch. **49b** (1994) 89-99.