Preparation and Properties of RMgN< Compounds Having Coordinating Oxygen Atoms in the Amino Component

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Compounds of composition RMgN(CH$_2$CH$_2$OMe)$_2$ and RMg(N15C5) were prepared (HN15C5 = 1,4,7,-10-tetraoxa-13-azacyclopentadecane). Crystal structures of the compounds in which R is ethyl showed the solids to have dimeric structures with four-membered rings formed by two nitrogen and two magnesium atoms, and molecular weight measurements found RMgN(CH$_2$CH$_2$OMe)$_2$ (R = ethyl or neopentyl) to be dimeric in benzene solutions. A crystal structure was determined of a compound of composition (Et$_2$Mg)$_2$EtOMg(N15C5) that was obtained as a minor and accidental component.

Grignard reagents are not the simple RMgX that we write for them.[1] In solids[2] or in solutions,[3] the magnesium atom usually is bonded to at least four groups. In solvents (e.g., diethyl ether, THF) ordinarily used with these reagents, the heteroatoms of two or more solvent molecules are usually bonded to the magnesium. In the absence of solvents having coordinating atoms (and sometimes even in their presence), halogens can be bonded in a bridging fashion to two magnesium atoms. R$_2$Mg compounds exhibit similar behavior: heteroatoms of solvent molecules are bonded to the magnesium and, in the absence of a suitable solvent, R may be bonded in a bridging fashion to two magnesium atoms. Only with extraordinary[4] R's is magnesium bonded to only two groups. Another complication is disproportionation of organomagnesium species, exemplified by the Schlenk equilibrium of Grignard reagents (eq 1, Z = a halogen). Consequently, we often do not know the composition

$$2 \text{ R}-\text{Mg}-\text{Z} \rightleftharpoons \text{R}-\text{Mg}-\text{R} + \text{Z}-\text{Mg}-\text{Z} \qquad (1)$$
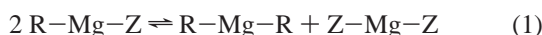
and sometimes even the identity of the species in an organomagnesium solution; even less do we know the extent to which each species participates in a reaction with some substrate.

Our objective was to prepare organomagnesium compounds that would be more structurally defined. The approach was to synthesize RMgN< compounds having in the amino component an array of coordinating groups to provide coordination to the magnesium.[5] Coordination by internal groups can be particularly effective since these groups cannot wander away as readily as

can coordinating solvent molecules. Reactivities of such RMgN< species could differ significantly from those of routine organomagnesium compounds. Besides the effect of the nitrogen atom on reactivity, efficient intramolecular coordination might reduce rates of reactions dependent on coordination to the magnesium and, by enhancing polarity of the R−Mg bond, might increase rates of reactions in which action of R as a nucleophile is important.

Results and Discussion

The work involved synthesis and characterization of organomagnesium compounds prepared by reactions (eq 2) of dialkylmagnesium compounds and secondary amines **1** and **2** (Z = H).
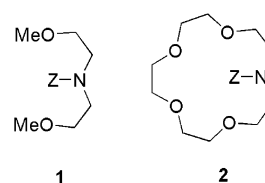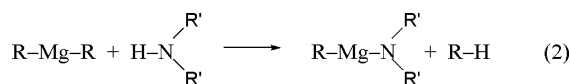
Compounds of Composition 1-MgR. Reactions of **1-H** (Z = H) with Me$_2$Mg, Et$_2$Mg, or Np$_2$Mg resulted in solids of composition **1-MgR** (Z = MgR) that readily sublime and are soluble in benzene and toluene, moderately soluble in diethyl ether and THF, and insoluble in saturated hydrocarbons. The ^{1}H NMR and ^{13}C NMR absorptions of the amine component in benzene-d_6 solutions of these solids are similar (variation in ^{1}H NMR absorptions < ca. 0.1 ppm and in ^{13}C NMR absorptions < ca. 0.6 ppm).[6,7]

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(1) For general information about organomagnesium compounds, see: Lindsell, W. E. In *Comprehensive Organometallic Chemistry*; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon: Oxford, l982; Chapter 4. Lindsell, W. E. In *Comprehensive Organometallic Chemistry II*; Abel, E. W., Stone, F. G. A., Wilkinson, G., Eds.; Pergamon: Oxford, l995; Vol. 1, Chapter 3.

(2) Bickelhaupt, F. In *Grignard Reagents: New Developments*; Richey, H. G., Jr., Ed.; Wiley: Chichester, 2000; Chapter 9.

(3) Evidence in solution[1] now includes information from EXAFS and LAXS techniques: Ertel, T. S.; Bertagnolli, H. In *Grignard Reagents: New Developments*; Richey, H. G., Jr., Ed.; Wiley: Chichester, 2000; Chapter 10.

(4) Examples are listed in ref 2.

(5) This differs from extensive studies in which coordinating groups are part of the R group of the organomagnesium compound. Gruter, G.-J. M.; van Klink, G. P. M.; Akkerman, O. S.; Bickelhaupt, F. *Chem. Rev.* l995, *95*, 2405.

(6) Some data are given in the Supporting Information.

(7) There was no indication of potential disproportionation products **1**-Mg-**1** and R$_2$Mg.
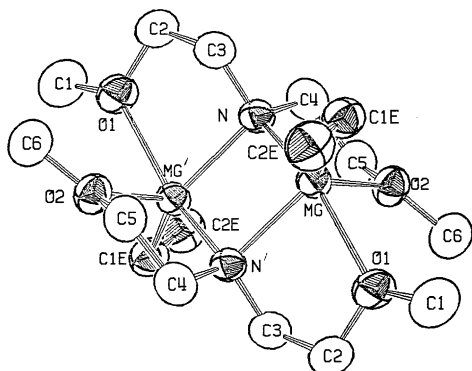
Figure 1. ORTEP drawing of **(1-MgEt)₂**. Atoms are shown with 50% probability ellipsoids.

Addition to benzene-d_6 solutions of **1-MgEt** or **1-MgNp** of 2−3 equiv of diethyl ether, DME, or TMEDA had no significant effect on positions of ^{1}H or ^{13}C NMR absorptions.[6] Since similar addition of these compounds significantly alters NMR spectra of routine organomagnesium compounds (^{1}H NMR spectra[6] of Np₂Mg, for example), they must not coordinate significantly to the magnesium atom of **1-MgEt** or **1-MgNp**. Even the particularly potent[8] donor HMPA had no significant effect on the spectra of **1-MgEt** or **1-MgNp** though it did cause small shifts in the absorptions of **1-MgMe**.[9,10] It is likely that **1-MgMe**, **1-MgEt**, and **1-MgNp** are structurally similar, but the small size of the methyl group permits some attachment of HMPA to the magnesium of **1-MgMe** that is not significant with **1-MgEt** and **1-MgNp**.

A technique developed by Fraenkel, Beckenbaugh, and Young[11] and modified by Jackman and DeBrosse[12] to measure very small pressure differences between a solvent and a solution prepared with that solvent was used to determine the vapor pressure lowering of benzene caused by **1-MgEt** and **1-MgNp**. The results indicated the degree of aggregation in 0.05−0.70 M solutions of "**1-MgR**" to be 2.00 ± 0.05. Therefore, the dominant species in solution are dimers.

The structure (ORTEP drawing in Figure 1) of **1-MgEt** determined by X-ray structural analysis of a crystal is a centrosymmetric dimer; the essential structural feature is a four-membered ring formed by two nitrogen and two magnesium atoms. Magnesium is pentavalent, its coordination sphere completed by the carbon atom of an ethyl group and chelating oxygen atoms. Bond distances and angles involving magnesium are listed in Table 1. A number of other structures having similar Mg−N four-membered rings have been reported,[13,14] though

(8) Ducom, J. *J. Organomet. Chem.* l**973**, *59*, 83.

(9) The C*H*₃Mg absorption was ca. 0.1 ppm upfield, and absorptions of the hydrogens of the amino component were ca. 0.25 ppm downfield; the *C*Mg absorption was 2.5 ppm downfield, and other C absorptions of the amino component were downfield an average of 0.5 ppm.[6]

(10) Addition of **1-H** to solutions of Np₂Mg plus 0.8 equiv of TMEDA or HMPA also gave spectra with absorptions due only to **1-MgNp**, neopentane, and free TMEDA or HMPA.

(11) Fraenkel, G.; Beckenbaugh, W. E.; Young, P. P. *J. Am. Chem. Soc.* l**976**, *98*, 6878.

(12) Jackman, L. M.; DeBrosse, C. W. *J. Am. Chem. Soc.* l**983**, *105*, 4177.

(13) Magnuson, V. R.; Stucky, G. D. *Inorg. Chem.* **1969**, *8*, 1427. Engelhardt, L. M.; Jolly, B. S.; Junk, P. C.; Raston, C. L.; Skelton, B. W.; White, A. H. *Aust. J. Chem.* **1986**, *39*, 1337. Henderson, K. W.; Mulvey, R. E.; Clegg, W.; O'Neil, P. A. *J. Organomet. Chem.* l**992**, *439*, 237. Henderson, K. W.; Mulvey, R. E.; Clegg, W.; O'Neil, P. A. *Polyhedron*, **1993**, *12*, 2535. Olmstead, M. M.; Grigsby, W. J.; Chacon, D. R.; Hascall, T.; Power, P. P. *Inorg. Chim. Acta* **1996**, *251*, 273. Yang, K.-C.; Chang, C.-C.; Huang, J.-Y.; Lin, C.-C.; Lee, G.-H.; Wang, Y.; Chiang, M. Y. *J. Organomet. Chem.* **2002**, *648*, 176. Westerhausen, M.; Bollwein, T.;

Table 1. Selected Bond Distances (Å) and Angles (deg) for the Crystal Structures

For **(1-MgEt)₂** (Figure 1)			
Bond Distances			
Mg−O(1)	2.272 (1)	Mg−N′	2.140 (1)
Mg−O(2)	2.154 (1)	Mg−C(1E)	2.142 (2)
Mg−N	2.131 (1)		
Bond Angles			
O(1)−Mg−O(2)	83.50 (5)	O(2)−Mg−C(1E)	120.93 (7)
O(1)−Mg−N	76.71 (5)	N−Mg−N′	87.77 (5)
O(1)−Mg−N′	151.02 (6)	N−Mg−C(1E)	128.51 (8)
O(1)−Mg−C(1E)	94.88 (7)	N′−Mg-C(1E)	113.82 (8)
O(2)−Mg−N	108.61 (6)	Mg−N−Mg′	92.23 (5)
O(2)−Mg−N′	78.42 (5)		
For **(2-MgEt)₂** (Figure 2)			
Bond Distances			
Mg−O(1)	2.282 (2)	Mg−N′	2.139 (2)
Mg−O(4)	2.169 (2)	Mg−C(1E)	2.178 (3)
Mg−N	2.158 (2)		
Bond Angles			
O(1)−Mg−O(4)	123.46 (8)	O(4)−Mg−C(1E)	100.8 (1)
O(1)−Mg−N	76.65 (7)	N−Mg−N′	86.19 (9)
O(1)−Mg−N′	123.69 (8)	N−Mg−C(1E)	167.5 (1)
O(1)−Mg−C(1E)	94.11 (9)	N′−Mg-C(1E)	106.1 (1)
O(4)−Mg−N	78.06 (8)	Mg−N−Mg′	93.81 (9)
O(4)−Mg−N′	103.86 (8)		
For **2-MgOEt(Et₂Mg)₂** (Figure 3)			
Bond Angles			
Mg(1)−O(1)	2.130 (3)	Mg(2)−C(30)	2.288 (5)
Mg(1)−O(2)	2.159 (3)	Mg(2)−C(40)	2.316 (5)
Mg(1)−O(3)	2.304 (3)	Mg(2)−C(50)	2.107 (7)
Mg(1)−O(4)	2.149 (3)	Mg(3)−O(5)	1.963 (3)
Mg(1)−O(5)	1.938 (3)	Mg(3)−C(30)	2.277 (5)
Mg(1)−N	2.107 (3)	Mg(3)−C(40)	2.281 (5)
Mg(2)−N	2.106 (4)	Mg(3)−C(60)	2.121 (6)
Bond Angles			
O(1)−Mg(1)−O(2)	70.7 (1)	O(5)−Mg(1)−N	100.6 (1)
O(1)−Mg(1)−O(3)	139.6 (1)	N−Mg(2)−C(30)	109.6 (2)
O(1)−Mg−O(4)	108.7 (1)	N−Mg(2)−C(40)	104.3 (2)
O(1)−Mg(1)−O(5)	103.7 (1)	N−Mg(2)−C(50)	114.6 (3)
O(1)−Mg(1)−N	79.3 (1)	C(30)−Mg(2)−C(40)	99.4 (2)
O(2)−Mg(1)−O(3)	69.5 (1)	C(30)−Mg(2)−C(50)	115.8 (3)
O(2)−Mg(1)−O(4)	97.9 (1)	C(40)−Mg(2)−C(50)	111.6 (3)
O(2)−Mg(1)−O(5)	99.3 (1)	O(5)−Mg(3)−C(30)	107.8 (2)
O(2)−Mg(1)−N	147.2 (1)	O(5)−Mg(3)−C(40)	104.9 (2)
O(3)−Mg(1)−O(4)	70.4 (1)	O(5)−Mg(3)−C(60)	109.5 (2)
O(3)−Mg(1)−O(5)	89.4 (1)	C(30)−Mg(3)−C(40)	100.8 (2)
O(3)−Mg(1)−N	136.2 (2)	C(30)−Mg(3)−C(60)	117.0 (3)
O(4)−Mg(1)−O(5)	146.8 (2)	C(40)−Mg(3)−C(60)	115.9 (2)
O(4)−Mg(1)−N	78.9 (2)		

in these examples the magnesium atom is four-coordinate. The N−Mg−N angle of **1-MgEt** is somewhat larger than in prior examples, probably due to the accommodation of a fifth group at magnesium.

The ready sublimation of **1-MgR** compounds indicated that they have vapor pressures large enough to permit obtaining routine mass spectra. Mass spectra of **1-MgEt** were recorded over the course of 32 min as the source temperature was increased linearly from 50 °C to 175 °C. A plot of peak intensities of several of the highest mass peaks versus time indicated the presence of a component that began to distill from

Makropoulos, N.; Schneiderbauer, S.; Suter, M.; Nöth, H.; Mayer, P.; Piotrowski, H.; Polborn, K.; Pfitzner, A. *Eur. J. Inorg. Chem.* **2002**, 389. Hevia, E.; Kennedy, A. R.; Mulvey, R. E.; Weatherstone, S. *Angew. Chem., Int. Ed.* **2004**, 1709.

(14) Conway, B.; Hevia, E.; Kennedy, A. R.; Mulvey, R. E.; Weatherstone, S. *Dalton Trans.* **2005**, 1532.

Table 2. Experimental Data for the Structure Determinations of the Crystalline Solids

	(1-MgEt)$_2$	**(2-MgEt)$_2$**	**(Et$_2$Mg)$_2$(2-MgOEt)**
formula	C$_{16}$H$_{38}$Mg$_2$N$_2$O$_4$	C$_{24}$H$_{50}$Mg$_2$N$_2$O$_8$	C$_{20}$H$_{45}$ Mg$_3$NO$_5$
fw (amu)	371.1	543.3	452.5
cryst syst	monoclinic	monoclinic	monoclinic
space group	$P2_1/n$	$P2_1/n$	$P2_1/c$
a (Å)	7.976(1)	8.240(9)	16.649(5)
b (Å)	14.938(3)	11.885(2)	10.092(2)
c (Å)	9.404(1)	15.073(6)	17.279(3)
α (deg)	90	90	90
β (deg)	100.61(1)	100.27(1)	113.19(2)
γ (deg)	90	90	90
vol (Å^3)	1101.2(5)	1453(3)	2669(2)
Z	2	2	4
calcd density (g/cm^3)	1.119	1.242	1.126
$F(000)$	408	592	912
cryst size (mm)	0.38 × 0.38 × 0.40	0.22 × 0.22 × 0.90	0.24 × 0.42 × 0.35
μ (cm^{-1})	1.289	1.289	1.402
θ range	3.0−42.24	3.0−44.16	3.0−41.66
no. unique reflns	1081	1661	2787
no. reflns with $I > 2\sigma(I)$	973	1327	1625
no. params refined	166	238	370
R1	0.031	0.045	0.049
wR2	0.040	0.052	0.050
goodness of fit	1.792	1.705	1.754
largest residual peak (e Å^{-3})	0.1	0.4	0.2

the probe at ca. 8 min and reached a maximum at ca. 27 min.[15] The highest significant mass peak of this component, *m/e* 342, corresponds to a dimer of **1-MgEt** minus ethyl, suggesting that the **1-MgEt** dimer also is present in the gas phase.[16]

Compounds of Composition 2-MgR. Reactions of **2-H** with Et$_2$Mg, Pr$_2$Mg, or Np$_2$Mg resulted in formation of **2-MgEt**, **2-MgPr**, and **2-MgNp** solids that are soluble in THF, sparingly soluble in benzene, and insoluble in diethyl ether.

The structure (ORTEP drawing in Figure 2) determined by X-ray structural analysis of a crystal of **2-MgEt**, like that of **1**-**MgEt**, is a centrosymmetric dimer having a four-membered ring formed by two nitrogen and two magnesium atoms. Bond distances and angles involving the magnesium are listed in Table 2. The Mg−N distances and ring bond angles are similar to those of **1-MgEt**. The coordination sphere of magnesium is completed by two oxygen atoms and an ethyl group. Bridge bonding prevails in solid **2-MgEt** as in **1-MgEt** even though the azacrown ring contains two additional oxygen atoms that might coordinate to the magnesium in a monomeric structure (**3**). Each

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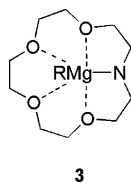
magnesium of the **2-MgEt** dimer is bonded to two oxygens of the same amino group rather than to one oxygen of each amino group as in the **1-MgEt** dimer, a difference that must be due to restrictions imposed by the geometry of the azacrown ring.

The ^{1}H NMR spectra of benzene-d_6 solutions of **2-MgEt**, **2-MgPr**, and **2-MgNp** had single sets of absorptions that are consistent with structures similar to that found for the **2-MgEt**
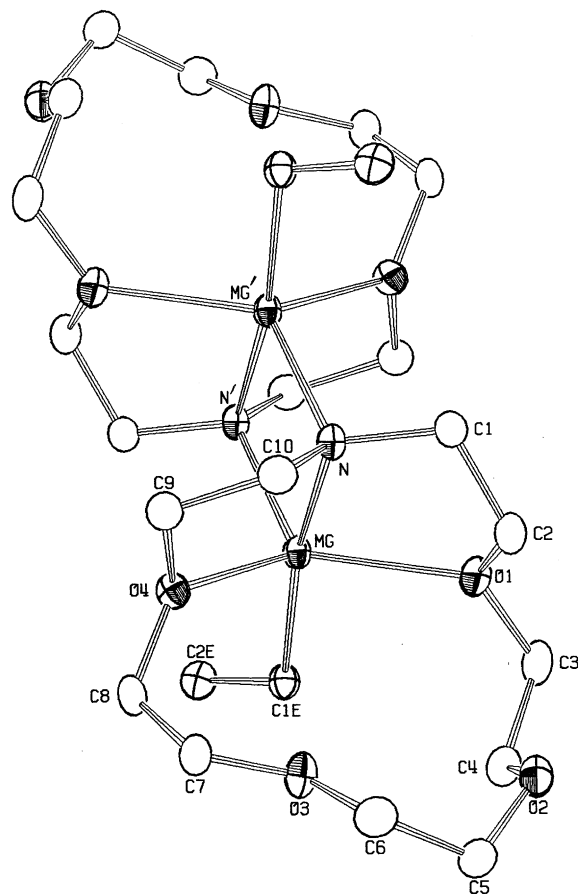
(15) Mass spectra of Np$_2$Mg had a significant peak at *m/e* 166, corresponding to monomeric Np$_2$Mg. This suggestion that Np$_2$Mg is monomeric in the gas phase is in accord with an electron diffraction study [Ashby, E. C.; Fernholt, L.; Haaland, A.; Seip, R.; Smith, R. S. *Acta Chem. Scand. A* **1980**, *34*, 213] that found monomer to be the principal component of Np$_2$Mg vapor.

Figure 2. ORTEP drawing of **(2-MgEt)$_2$**. Atoms are shown with 50% probability ellipsoids.

solid. ^{1}H NMR spectra of solutions of **2-MgR** that contained excess R$_2$Mg suggested the formation of a species having for each azacrown ring two structurally different R groups in 2:1 ratio.[17] These observations and observations with similar solu-

(16) The plot of peak intensities versus time indicated the presence of another component that began to distill from the probe within 2 min and reached a maximum at ca. 13 min. The highest significant mass peak was *m/e* 288, consistent with this component being **1-Mg-1**.
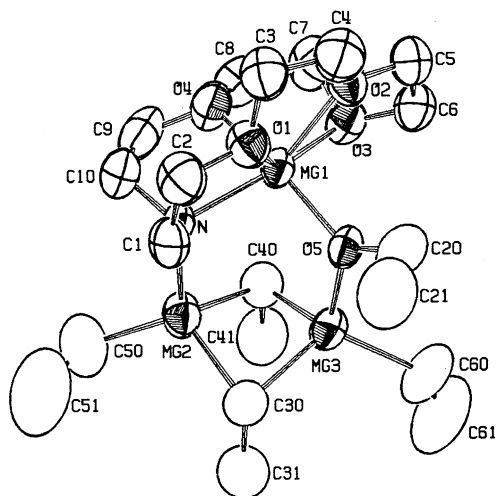
Figure 3. ORTEP drawing of **2-MgOEt(Et$_2$Mg)$_2$**. Atoms are shown with 50% probability ellipsoids.

tions formed with several related monoazacrown rings are included in the Supporting Information.

X-ray Structure of 2-MgOEt(Et$_2$Mg)$_2$. A preparation in benzene−diethyl ether of **2-MgEt** and excess Et$_2$Mg that was heated and then cooled afforded a small amount of crystalline material, which contained some crystals suitable for X-ray diffraction analysis. The crystals that diffracted satisfactorily were only a minor component of the small amount of crystalline material; indeed, the ^{1}H NMR spectrum of a solution (benzene-d_6) of all crystals had only very feeble OEt absorptions, even though such a grouping proved to be part of the structure. Although the composition of the crystals differed from that of the preparation from which they were obtained, the structure (ORTEP drawing in Figure 3) is significant in revealing possibilities for bonding of the macrocycle with organomagnesium species. The basic structural units are **2-MgOEt** and (Et$_2$Mg)$_2$. The ethoxy group probably resulted from the reaction of an ethylmagnesium species with adventitious oxygen. Such involvement of oxygen has been well-known in organomagnesium chemistry.[18] A diagrammatic representation of the structure (**4**) indicates that one magnesium of the (Et$_2$Mg)$_2$ unit is bonded

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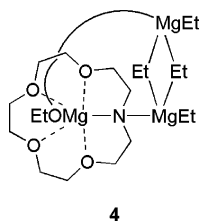
to the nitrogen atom. The other magnesium is closest to the ethoxide oxygen (1.94 Å) but within bonding distance (2.11−2.30 Å) of all four oxygens of the macrocyclic ring. Bond distances and angles involving the magnesium atoms are listed in Table 1. The Mg−Mg distance (2.74 Å) and ring bond angles (73°, 74°, 99°, and 101°) of the (Et$_2$Mg)$_2$ unit are similar to those (2.67 Å, 72°, and 108°) of polymeric Et$_2$Mg.[19]

Conclusions. The finding that **1-MgEt** and **1-MgNp** are dimeric in solution suggests that the dominant structures in

solution are similar to that found for solid **1-MgEt**.[20] The mass spectral observations indicate that this structure also may be significant in the gas phase. That **1-MgEt** forms a dimer rather than existing as a monomer (e.g., **5**) must be due to the strength

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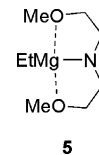
of Mg−N bridge bonding and the formation of five rather than four bonds to magnesium. The finding that strong coordinating agents do not coordinate to **1-MgR** species suggests that the magnesium−oxygen bonding seen in the crystal structure (Figure 1) also is important in solution and is not readily displaced.

Experimental Section

Procedures involving organometallic compounds were performed under an atmosphere of purified nitrogen using Schlenk techniques, a glovebox, and a vacuum line. The general procedure for preparing NMR solutions was as follows. In a glovebox, the salt and dialkylmagnesium compound were weighed into a vial equipped with a magnetic stirring bar, the solvent was added, and the mixture was stirred for a few minutes. If a homogeneous solution was present, it was transferred into an NMR tube. A preparation that remained heterogeneous was stirred for a longer time before transfer. The NMR tube had an extension of routine glass tubing that had been added to facilitate sealing with a flame. The filled tube was capped temporarily with a septum, removed from the glovebox, immersed in liquid nitrogen, and sealed at the extension.

Molecular Weight Determinations. The determinations used the same equipment and procedure that have been described in detail.[12] The only significant deviation from the reported procedure was substitution of benzil[21] for triphenylmethane as the calibration standard (benzil is considerably more soluble in benzene).

Sample for X-ray Diffraction Analysis of (1-MgEt)$_2$. A diethyl ether solution (ca. 0.15 M) of **1-MgEt** was sealed in a glass tube, which then was cooled to −10 °C. After 2 days, clear colorless crystals had formed. The tube was opened and a crystal was selected for X-ray diffraction analysis. A solution of the remaining crystals dissolved in benzene-d_6 exhibited a ^{1}H NMR spectrum identical to that given above for **1-MgEt** preparations.[6]

Sample for X-ray Diffraction Analysis of (2-MgEt)$_2$. A sample of **2-H** (130 mg, 0.59 mmol) dissolved in THF (5 mL) was added dropwise (5 min) to a stirred diethyl ether−THF solution of Et$_2$-Mg (2.8 mL, 0.42 M, 1.18 mmol), resulting in a clear solution. After 1 h, a portion of this solution was sealed in a tube and cooled to −10 °C. After 2 days, crystals were filtered from the solution.

Sample for X-ray Diffraction Analysis of 2-MgOEt(Et$_2$Mg)$_2$. A solution of **2-H** (150 mg, 0.69 mmol) in benzene (7 mL) was added to a diethyl ether solution of Et$_2$Mg (6.9 mL, 0.20 M, 1.38 mmol) in a tube that then was sealed. The tube was heated to 100 °C for 1 h and then cooled to −10 °C. The small amount of crystalline material that was present was filtered from the solution. X-ray diffraction analysis of about 40 crystals was attempted, but only 3 proved to be suitable.

X-ray Crystallographic Structure Determinations. A suitable crystal was sealed in a glass X-ray diffraction capillary. Diffraction data were collected at 23(1) °C on an Enraf-Nonius CAD4

(17) The solutions of course also can exhibit absorptions of the RH that is a product of the reaction of **2-H** and R$_2$Mg.

(18) Particularly pertinent recent references are ref 14 and the following: Kennedy, A. R.; Mulvey, R. E.; Rowlings, R. B. *J. Am. Chem. Soc.* **1998**, *120*, 7816.

(19) Weiss, E. *J. Organomet. Chem.* **1965**, *4*, 101.

(20) Several RMgNR′R″ species in which R′ and R″ are alkyl groups also have been reported to be dimeric in benzene [Coates, G. E.; Heslop, J. A. *J. Chem. Soc., A* **1966**, 26. Coates, G. E.; Heslop, J. A. *J. Chem. Soc., A* **1968**, 514. Coates, G. E.; Ridley, D. *J. Chem. Soc., A* **1967**, 56].

(21) Roddy, J. W.; Coleman, C. F. *J. Inorg. Nucl. Chem.* **1970**, *32*, 3891.

diffractometer using monochromated Mo Kα (λ = 0.70173 Å) radiation. A $\theta-2\theta$ scan mode was used at a scan speed of 1−5 deg min^{-1}. Three standard reflections were measured every hour to check on crystal orientation and stability. No orientation or stability problems were encountered. The atomic coordinates of magnesium atoms were determined by Patterson heavy atom techniques. The remaining non-hydrogen atom positions were determined from subsequent least squares refinements and difference Fourier electron density maps and refined upon. Hydrogen atoms were fixed at their initial calculated positions (C−H = 0.97 Å) and given fixed isotropic temperature factors (B = 5.0 Å^2) to give the best agreement with those located from a difference Fourier map. Details of structure collection and refinement are given in Table 2.

Supporting Information Available: Preparations of reactants and NMR observations of solutions of **1-MgR** preparations, solutions of Np$_2$Mg in the presence of donors, and **2-MgR** preparations and similar preparations with other monoazacrown ethers. This material is available free of charge via the Internet at http://pubs.acs.org.

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