

Supporting Information

**Preparation and Properties of RMgN< Compounds Having Coordinating Oxygen
Atoms in the Amino Component¹**

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This supporting information begins with observations concerning solutions prepared from reactions of **2-H** and three other monoazacrown ethers with >1 equiv of an R₂Mg compound. These results are followed by experimental information for these additional preparations and for the preparations with **1-H** and **2-H** reported in the note. Tables of information relating to the X-ray structure determinations are placed at the end.

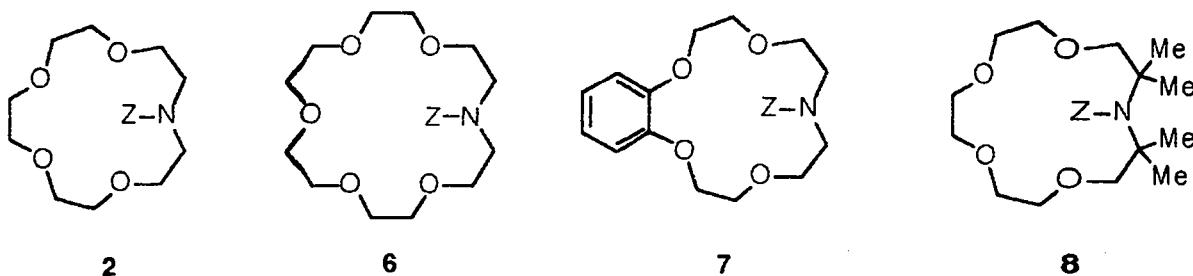
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Preparations of 2-MgR, 6-MgR, 7-MgR, or 8-MgR with Excess R₂Mg

¹H NMR spectra of solutions (benzene-*d*₆) of the **2-MgR** compounds prepared from



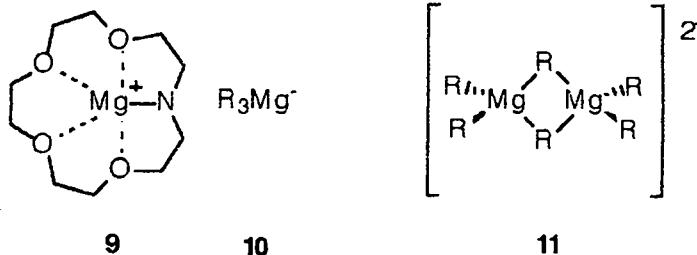
equimolar amounts of **2-H** ($Z = H$) and R_2Mg ($R = Np$, Et, Hex, *i*-Bu, and *i*-Pr) were in accord with the composition **2-MgR**, as expected for reactions of R_2Mg and a secondary amine. When more than 1 mole of R_2Mg per mole of **2-H** was used in a preparation (more R_2Mg than needed to form **2-MgR**), ¹H NMR spectra often suggested formation of a new species. Two² sets of $NpMg$ absorptions were seen when the ratio of all $NpMg$ groups to the azacrown ring was ca. 2:3 (Figure 1); the positions of neither set similar to those of Np_2Mg . Absorption areas indicated that the neopentyl groups responsible for these absorptions are in ca. 2:1 ratio. NMR observations also indicated that all 10 hydrogens of the azacrown ring are structurally different; a ¹H NMR spectrum (Figure 2) shows 7 absorption groupings in the δ 2.7-3.5 region for the hydrogens of the macrocycle, but 3 of these are double the size of the others and a ¹³C-¹H correlated NMR spectrum (Figure 2) reveals 5 discrete geminal absorption pairs. NOE experiments³ suggest that the CH_2Mg protons of both types of neopentyl groups are in close proximity to some hydrogens of the azacrown ring.

¹H NMR spectra of solutions formed with excess Np_2Mg and **6-H** or **7-H** also indicated formation of species having 2:1:1 $Np:Np':\text{azacrown}$ stoichiometries and exhibiting positions of the neopentyl absorptions similar to those observed with **2-H**.⁴ The aryl ring of **7** should make the adjoining oxygen atoms much poorer donor atoms and its field effect should affect absorption positions of nearby hydrogens. That introduction of the aryl ring has little effect on formation of the 2:1 species and on positions of the neopentyl absorptions suggests that neither a neopentyl

group nor a magnesiurn is close to the aryl ring. Similar reactions of Np_2Mg and **8-H** also led to a 2:1 species; positions of the CH_2Mg absorptions differed somewhat from those observed with the preparations with **2-H**, **6-H**, and **7-H**, not surprising since the methyl groups of **8-H** must impose a significant constraint to approach to the nitrogen.

Similar preparations from **2-H** and excess $i\text{-Pr}_2\text{Mg}$ or $i\text{-Bu}_2\text{Mg}$ and from **8-H** and excess $i\text{-Bu}_2\text{Mg}$ also exhibited two sets of alkyl absorptions. Similar preparations from **2-H** and Et_2Mg or Hex_2Mg exhibited only single sets of R absorptions. A considerably more dilute solution of a preparation from **2-H** with Et_2Mg did give two sets (2:1) of ethyl absorptions but these coalesced to a single set when the solution was heated to 80 °C. Ethyl and hexyl groups should be more prone than the bulkier isopropyl, isobutyl, or neopentyl groups to undergo exchanges that are rapid relative to the NMR time scale. In all of the reactions, only single sets of alkyl absorptions were generally seen when the alkyl:azacrown ratio was much greater than 3 (e.g., 3.5–4.0).

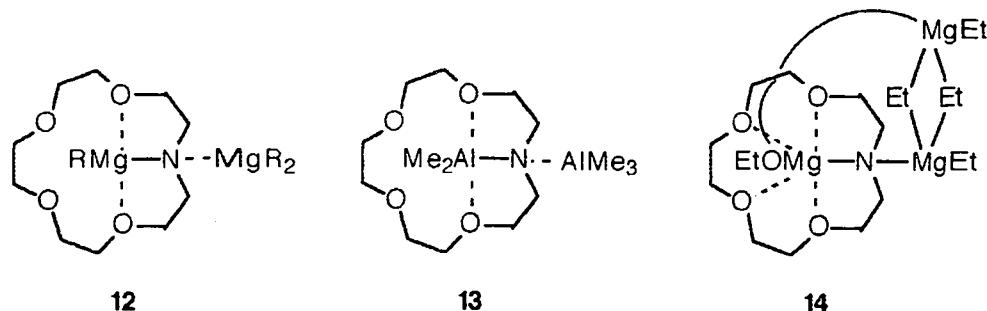
Addition of R_2Mg to species such as **2-MgR** might produce ions **9** and **10** in analogy to



reactions of R_2Mg and some neutral macrocycles that produce $\text{RMg}(\text{macrocycle})^+$ and **10**. There could be two sets of R absorptions in 2:1 ratio if the magnesate ion was a dimer (**11**), but it is unlikely that the 2:1 species is **11**. There has been no NMR evidence for dimers when a neopentylmagnesate anion has been generated from Np_2Mg and neutral macrocycles: only a single sets of neopentyl absorptions has been seen.⁵ Moreover, the positions of the absorptions do not correspond to the weighted averages of the pairs of absorptions of the 2:1 species obtained with **2**; this suggests that the difference in number of neopentyl absorptions is not due to rapid equilibration between structurally different neopentyl groups in solutions with neutral macrocycles but not in solutions involving **2**. Moreover, if ions **9** and **11** were present, the two faces of the

macrocyclic ring would be unlikely to remain distinct for times that are indicated by NMR observations to be long relative to the NMR time scale.⁶

Another possible structure is **12**, a structure consistent with the minimal effects of the aryl ring



of **7** and the larger ring of **6**. This structure is the magnesium counterpart of **13**, a solid obtained from similar experiments with **2-AlMe₂** and **Me₃Al**.⁷ It also has some similarity to the structure of the **1-MgOEt(Et₂Mg)₂** obtained in this work and shown in diagrammatic representation **14**; in both **12** and **14**, a "neutral" magnesium is bonded to the nitrogen atom, and another magnesium (bonded to R in **12** and to OEt in **14**) is bonded to the nitrogen atom and oxygen atoms of the ring.

Experimental Section¹

Procedures involving organometallic compounds were performed under an atmosphere of purified nitrogen using Schlenk techniques, a glovebox, and a vacuum line. NMR spectra were recorded at ca. 23 °C in benzene-*d*₆. ¹H NMR absorption positions are relative to internal C₆D₅H (δ 7.15); notations used: s, singlet; d, doublet; t, triplet; q, quartet; m, a more complex multiplet (or a multiplet whose weaker absorptions could not be distinguished); c, complex overlapping absorptions; b, broad. ¹³C NMR absorptions positions are relative to internal benzene-*d*₆ (δ 128.00). Solutions for NMR analysis were prepared in a glovebox and transferred into NMR tubes to which an extension of routine glass tubing had been added to facilitate sealing with a flame. An NMR tube was capped temporarily with a septum, removed from the glovebox, immersed in liquid nitrogen, and sealed at the extension.

Compounds for which preparations are not given were commercial samples. Diethyl ether and THF were distilled from sodium benzophenone ketyl under an atmosphere of nitrogen immediately prior to use. Organic halides and benzene-*d*₆ were distilled from CaH₂. Dioxane, TMEDA, DME, HMPA, and benzene were distilled from CaH₂ under an atmosphere of nitrogen. Bis(2-methoxyethyl)amine (**1 - H**) was distilled from KOH prior to use. Magnesium was 1/8 inch pieces (Cerac., Inc. 99.99%) or turnings (1 cm and less, Johnson Matthey, Puratronic Division, 99.98%). MeMgBr was sometimes obtained as a 3.0 M solution in diethyl ether (Alfa Products).

Dialkylmagnesium Compounds. Their preparation and the determination of concentrations of their solutions generally followed a procedure already described.⁸ Yields of NpMgCl were highest (ca. 75%) when relatively concentrated solutions (1-2 M) were prepared and when the preparation was heated at reflux temperature for 15 h. In the preparation of Np₂Mg, it was advantageous to use exactly one mole of dioxane per mole of potential Np₂Mg since excess dioxane forms a complex with Np₂Mg that sublimes at a slightly higher temperature than dioxane-free Np₂Mg. Np₂Mg was further purified by sublimation (80 °C, 10⁻³ Torr).

Azacrown Ethers. 1,4,7,10-Tetraoxa-13-azacyclopentadecane (**2 - H**),⁹ 5,6-benzo-1,4,7,10-tetraoxa-13-azacyclopentadecane (**7 - H**),¹⁰ and 12,12,14,14-tetramethyl-1,4,7,10-tetraoxa-13-azacyclopentadecane (**8 - H**)¹¹ were prepared using previously reported procedures. The only significant modification was in the purification of **7 - H**. The crude product (20 g) was added to hexane (80 mL), and the resulting mixture was heated to dissolve all solids, resulting in two liquid phases. When this mixture was cooled, colorless crystals of **7 - H** were deposited from the clear upper phase. A dark brown solid that formed from the dark bottom liquid phase contained some **4 - H** along with other materials. The procedure of heating with hexane was repeated at least once with the dark solid to extract additional **7 - H**. The 1,2-bis(2-hydroxyethoxy)benzene used to prepare **7 - H** was prepared as previously described.¹² The bis(1,1-dimethyl-2-hydroxyethyl)amine used to prepare **8 - H** was prepared as previously described.¹³ A sample of 1,4,7,10,13-pentaoxa-16-azacyclooctadecane (**6 - H**) was obtained from Professor George W. Gokel.

Preparation of **1-MgR.** A preparation of **1-MgEt** is typical. A solution of **1-H** (0.80 g, 6.0 mmol) in diethyl ether (10 mL) was added dropwise over 15 min to a stirred diethyl ether solution of Et₂Mg (24 mL, 0.25 M, 6.0 mmol). After addition was complete, stirring was continued for 2-3 h. Removal of the solvent at reduced pressure gave an amorphous white solid that was sublimed (100 °C, <10⁻³ Torr) to give a white solid (890 mg, 4.8 mmol, 80%). ¹H NMR spectra of the **1-MgR** solutions and also of solutions of **1-H** and of **1-Mg-1** are summarized in Table 1. ¹³C NMR spectra of the solutions are summarized in Table 2.

Preparation of **1-Mg-1.** A solution of **1-H** (0.82 g, 6.2 mmol) in diethyl ether (3 mL) was added dropwise to a stirred diethyl ether solution of Et₂Mg (10 mL, 0.21 M, 2.1 mmol). The resulting preparation was allowed to stir for 8 h after which the solvent was removed at reduced pressure. The resulting white solid was sublimed (100 °C, 10⁻³ Torr).

Addition of Donors to Benzene-d₆ Solutions of **1-MgR or **Np₂Mg**.** Addition to benzene-d₆ solutions (ca. 0.3 M) of **1-MgEt** or **1-MgNp** of up to 2-3 eq of diethyl ether, DME, TMEDA, or HMPA resulted in no significant changes in the ¹H or ¹³C (or ³¹P in the case of HMPA) NMR absorptions of **1-MgEt**, **1-MgNp**, or the donor solvent. The same was true for **1-MgMe** except for additions of HMPA which resulted in small changes as evident in the following examples. A solution was prepared from HMPA (1000 mg, 5.6 mmol) and **1-MgMe** (170 mg, 1.0 mmol) in benzene-d₆ (4 mL). ¹H NMR (200 MHz) (principal absorptions) δ -1.13 (s, CH₃Mg), 2.42 (d, *J* = 9.2 Hz, HMPA), 3.08 (t, *J* = 5.2 Hz, CH₂N), 3.29 (s, CH₃O), 3.58 (t, *J* = 5.2 Hz, CH₂O). ¹³C NMR (25 MHz) δ -16.43 (CMg), 36.67 (d, HMPA), 51.23 (CN), 58.51 (CH₃O), 73.15 (CH₂O). Another solution was prepared from HMPA (200 mg, 1.12 mmol) and **1-MgMe** (170 mg, 1.0 mmol) in benzene-d₆ (4 mL). ¹H NMR (200 MHz) (principal absorptions) δ -1.07 (s, CH₃Mg), 2.39 (d, *J* = 9.2 Hz, HMPA), 2.91 (t, *J* = 5.3 Hz, CH₂N), 3.11 (s, CH₃O), 3.34 (t, *J* = 5.3 Hz, CH₂O). ¹³C NMR (25 MHz) δ -17.72 (CMg), 36.68 (d, HMPA), 50.96 (CN), 58.61 (CH₃O), 72.95 (CH₂O). For comparison, ¹H NMR spectra of solutions prepared from Np₂Mg and the same molecules are given in Table 3.

Mass Spectrometry Studies. Mass spectra were obtained with a KRATOS MS-9/50 mass

spectrometer at 70 ev. A sublimed sample of **1-MgEt**, **1-MgNp**, or Np_2Mg was dissolved in benzene to form a ca. 0.2 M solution. A sample was introduced into the spectrometer chamber using a direct insertion probe, the tip of which had been submerged in a solution of sample under a rapid flow of nitrogen. The source temperature was increased linearly from 50 to 175 °C over a period of 30 min.

Preparations of 2-MgR, 6-MgR, 7-MgR, and 8-MgR Solutions With and Without Excess R₂Mg. The solutions used for the NMR spectra in Figure 1 were prepared as follows. A solution of **2-H** (50 mg, 0.23 mmol) in benzene-*d*₆ (2 mL) was added dropwise to a solution of Np_2Mg in benzene (2 mL). The amount of Np_2Mg was 50 mg (0.30 mmol) for A, 65 mg (0.39 mmol) for B, 75 mg (0.45 mmol) for C, 100 mg (0.60 mmol) for D, and 125 mg (0.75 mmol) for E. The ratios of neopentyl groups to azacrown rings indicated in Figure 1 were determined from areas of ¹H NMR absorptions of neopentyl groups and of **1** (rather than relying on the amounts used in the preparations). The solution used for the NMR spectra in Figure 2 was similar to C. In another preparation, a solution of **2-H** (220 mg, 1.0 mmol) in diethyl ether (5 mL) was added dropwise (5 min) to a stirred solution of Np_2Mg (165 mg, 1.0 mmol) in diethyl ether (5 mL). A white precipitate formed immediately. The ¹H NMR spectrum of a solution of the precipitate in benzene-*d*₆ was similar to A in Figure 1.

A general procedure used for most other preparations and also for some **2-H-Np₂Mg** preparations was the following. A solution of the monoazacrown ether (0.20 mmol) in benzene-*d*₆ (0.25 mL) was added to a solution of the R₂Mg compound (0.40 mmol) in benzene-*d*₆ (0.25 mL). ¹H NMR spectra of such preparations exhibited the expected ratio of R groups to azacrown rings of ca. 3. Other solutions were prepared by increasing the amount of R₂Mg or of monoazacrown ether. The following are representative spectra of preparations whose ¹H NMR spectra exhibit R/azacrown ratios of ca. 1 or 3. In those preparations with an R/azacrown ratio of 3 that exhibit two sets of R absorptions, they are always in a ratio of ca. 2 (the larger absorption of a pair is indicated by *). The preparations with R/azacrown ratios of ca. 3 are given headings in the form "**2-MgNp + Np₂Mg**."

2-MgNp. ^1H NMR (200 MHz) δ -0.13 (s, CH_2Mg), 1.51 (s, $(\text{CH}_3)_3\text{C}$), 2.5-4.0 (bc, 2-).

2-MgNp + Np₂Mg. ^1H NMR (200 MHz) δ -0.31 and -0.11* (s, CH_2Mg), 1.30 and 1.53* (s, $(\text{CH}_3)_3\text{C}$), 2.6-3.7 (c, 2-).

2-MgEt. ^1H NMR (200 MHz) δ -0.19 (bq, CH_2Mg), 1.85 (bt, CH_3), 2.7-3.8 (bc, 2-).

2-MgEt + Et₂Mg. ^1H NMR (200 MHz) δ -0.18 (b, CH_2Mg), 1.93 (b, CH_3), 2.6-3.7 (bc, 2-). In another experiment, a white precipitate formed immediately when a solution of **2-H** (120 mg, 0.55 mmol) in diethyl ether (3 mL) was added dropwise to a stirred solution of Et_2Mg (2.18 mL, 0.50 M, 1.1 mmol). A suspension of the precipitate in diethyl ether was sealed in a glass tube. The tube was heated at 85 °C for 30 min, resulting in a homogeneous solution. Crystals resulted upon cooling this tube to 0 °C. A dilute benzene-*d*₆ solution of the crystals exhibited a ^1H NMR spectrum indicating an ethyl/azacrown ratio of 3 and having two sets of ethyl absorptions in ca. 2/1 ratio. ^1H NMR (200 MHz) δ -0.35 and -0.11* (q, $J = 7.9$ and 7.7* Hz, CH_2Mg), 1.71 and 2.02* (t, 7.9 and 7.7* Hz, CH_3), 2.75-3.55 (c, 2-). When this solution was heated to 80 °C, the ^1H NMR spectrum exhibited only a single set of broad ethyl absorptions, similar to that of a more concentrated solution described above. ^1H NMR (200 MHz) δ -0.2 (b, CH_2Mg), 1.9 (b, CH_3), 2.9-3.5 (b, 2-).

2-MgHex + Hex₂Mg. ^1H NMR (200 MHz) δ -0.22 (b, CH_2Mg), 1.6-2.3 (bc, $\text{CH}_3(\text{CH}_2)_5$), 2.5-4.2 (bc, 2-).

2-Mg-i-Pr + i-Pr₂Mg. ^1H NMR (360 MHz) δ -0.01 (b overlapping m's, CHMg), 1.59 and 1.86* (d, $J = 7.6$ and 7.6* Hz, CH_3), 2.7-3.8 (bc, 2-).

2-Mg-i-Bu. ^1H NMR (200 MHz) δ -0.18 (d, CH_2Mg), 1.29 (d, $(\text{CH}_3)_2\text{C}$), 2.28 (m, CHCH_2), 2.8-4.0 (bc, 2-).

2-Mg-i-Bu + i-Bu₂Mg. ^1H NMR (200 MHz) δ -0.43 and -0.26* (d, $J = 6.2$ and 6.7* Hz, CH_2Mg), 1.22 and 1.41* (d, $J = 6.4$ Hz and 6.4 Hz, $(\text{CH}_3)_2\text{C}$), 2.21 and 2.49* (m, CHCH_2 , $J = 6.3$ and 6.6* Hz), 2.7-3.4 (bc, 2-).

6-MgNp + Np₂Mg. ^1H NMR (200 MHz) δ -0.26 and -0.12* (s, CH_2Mg), 1.33 and 1.55* (s, $(\text{CH}_3)_3\text{C}$), 2.5-4.0 (c, 3-). The spectrum also had absorptions at 0.22 (b) and 1.44 (s) that

were ca. 10% of the total neopentyl absorption.

7-MgNp + Np₂Mg. ¹H NMR (200 MHz) δ -0.30 and -0.14* (s, CH₂Mg), 1.24 and 1.41* (s, (CH₃)₃C), 2.7-3.7 (c, CH₂CH₂), 6.22 and 6.69 (m, aryl H's).

8-MgNp + Np₂Mg. ¹H NMR (200 MHz) δ -0.11 and -0.01* (b, CH₂Mg), 1.48 and 1.50 (b, (CH₃)₃C and (CH₃)₂C), 2.5-4.2 (bc, CH₂O).

8-Mg-i-Bu + i-Bu₂Mg. ¹H NMR (200 MHz) δ -0.37 and -0.18* (b, CH₂Mg), 1.38 (b, (CH₃)₂CC and (CH₃)₂CN), 2.5-2.9 (b, CHCH₂), 3.1-3.8 (bc, CH₂O).

References

(1) The following abbreviations are used: Np = neopentyl, Et = ethyl, Hex = hexyl, *i*-Bu = isobutyl, *i*-Pr = isopropyl, DME = 1,2-dimethoxyethane, TMEDA = N,N,N',N'-tetramethylethylenediamine, HMPA = hexamethylphosphoramide.

(2) The solutions of course also exhibit the absorption of neopentane, another product of the reaction of **2-H** and Np₂Mg.

(3) For example, irradiation of the multiplet at δ ca. 3.02 (using a solution similar to C in Figure 1) enhanced (1.3 %) the smaller CH₂Mg absorption and irradiation of the multiplet at δ ca. 3.11 enhanced (2.6 %) the larger CH₂Mg absorption.

(4) The NMR spectra of solutions of composition **6-MgNp**, **7-MgNp**, and **8-MgNp** (prepared with equimolar azacrown and Np₂Mg), however, showed more than one significant absorption for each type of neopentyl hydrogen.

(5) Squiller, E. P.; Whittle, R. R.; Richey, H. G., Jr. *J. Am. Chem. Soc.* **1985**, *107*, 432. Richey, H. G., Jr.; Kushlan, D. M. *J. Am. Chem. Soc.* **1987**, *109*, 2510. Pajerski, A. D.; Parvez, M.; Richey, H. G., Jr. *J. Am. Chem. Soc.* **1988**, *110*, 2660. Richey, H. G., Jr. In *Comprehensive Supramolecular Chemistry*; Atwood, J. L., Davies, J. E. D., MacNicol, D. D. Vögtle, F., Eds.; Pergamon: Oxford, 1996; Vol. 1 (G. W. Gokel, Ed.), Chapter 21. Also see Viebrock, H.; Behrens, U; Weiss, E. *Angew. Chem., Int. Ed. Engl.* **1994**, *33*, 1257.

(6) Moreover, the preparations with **2** and **6-8** are homogeneous solutions. By contrast, generation of organomagnesate anions in benzene frequently results in two liquid phases, the denser and generally smaller of which contains the majority of the solute.

(7) Pajerski, A. D.; Cleary, T. P.; Parvez, M; Gokel, G. W.; Richey, H. G., Jr.
Organometallics **1992**, *11*, 1400.

(8) Pajerski, A. D.; Squiller, E. P.; Parvez, M.; Whittle, R. R.; Richey, H. G., Jr.
Organometallics **2005**, *24*, 809. Alkyl bromides were used to prepare the hexyl and isopropyl Grignard reagents.

(9) Maeda, H.; Furuyoshi, S.; Nakatsuji, Y.; Okahara, M. *Bull. Chem. Soc. Jpn.* **1983**, *56*, 212.

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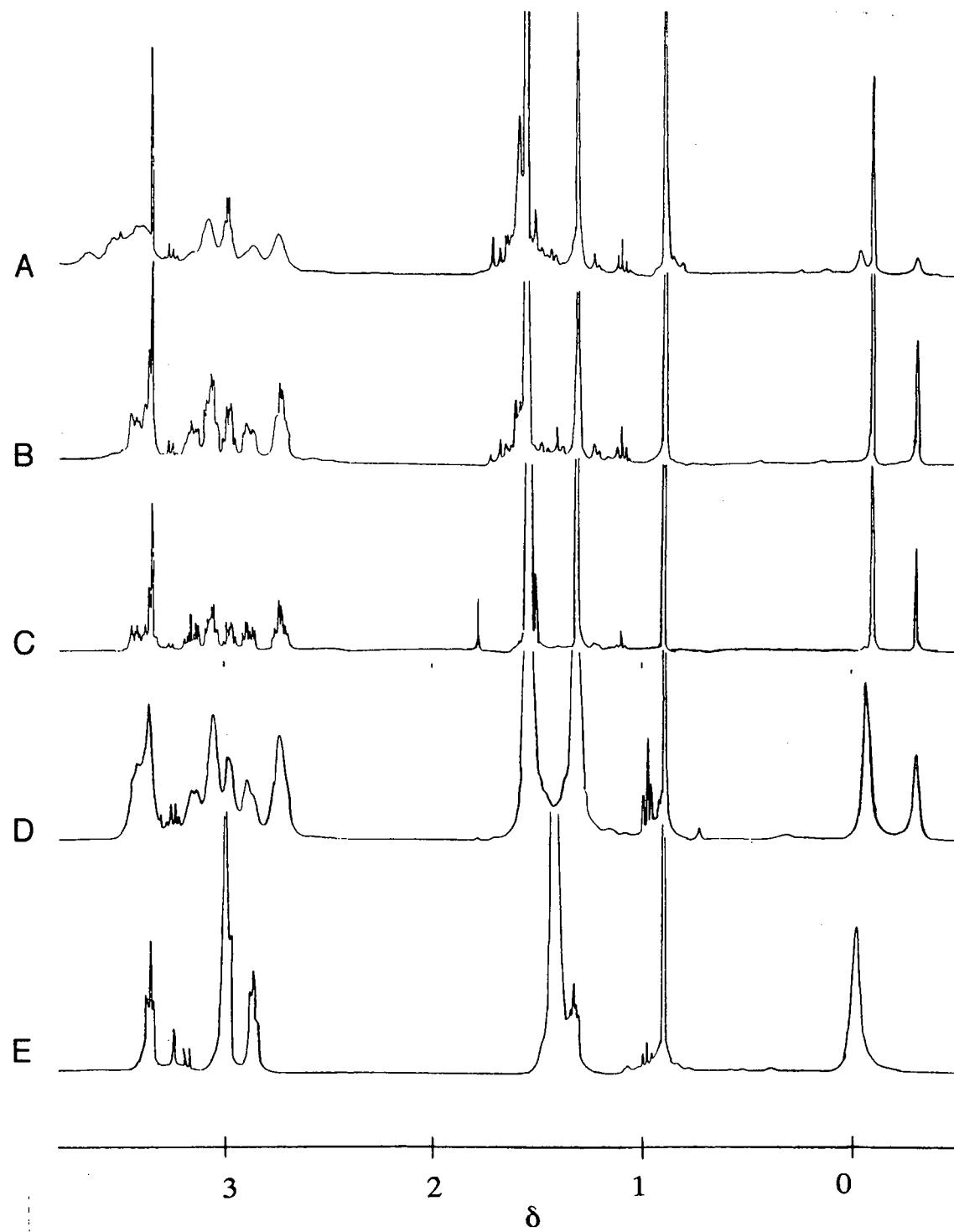


Figure 1. ^1H NMR spectra (360 MHz) of benzene- d_6 solutions prepared from **2-H** and excess Np_2Mg . Ratio of all neopentyl groups to the azacrown ring in each solution: A, 1.1; B, 2.1; C, 2.5; D, 3.0; E, 4.0.

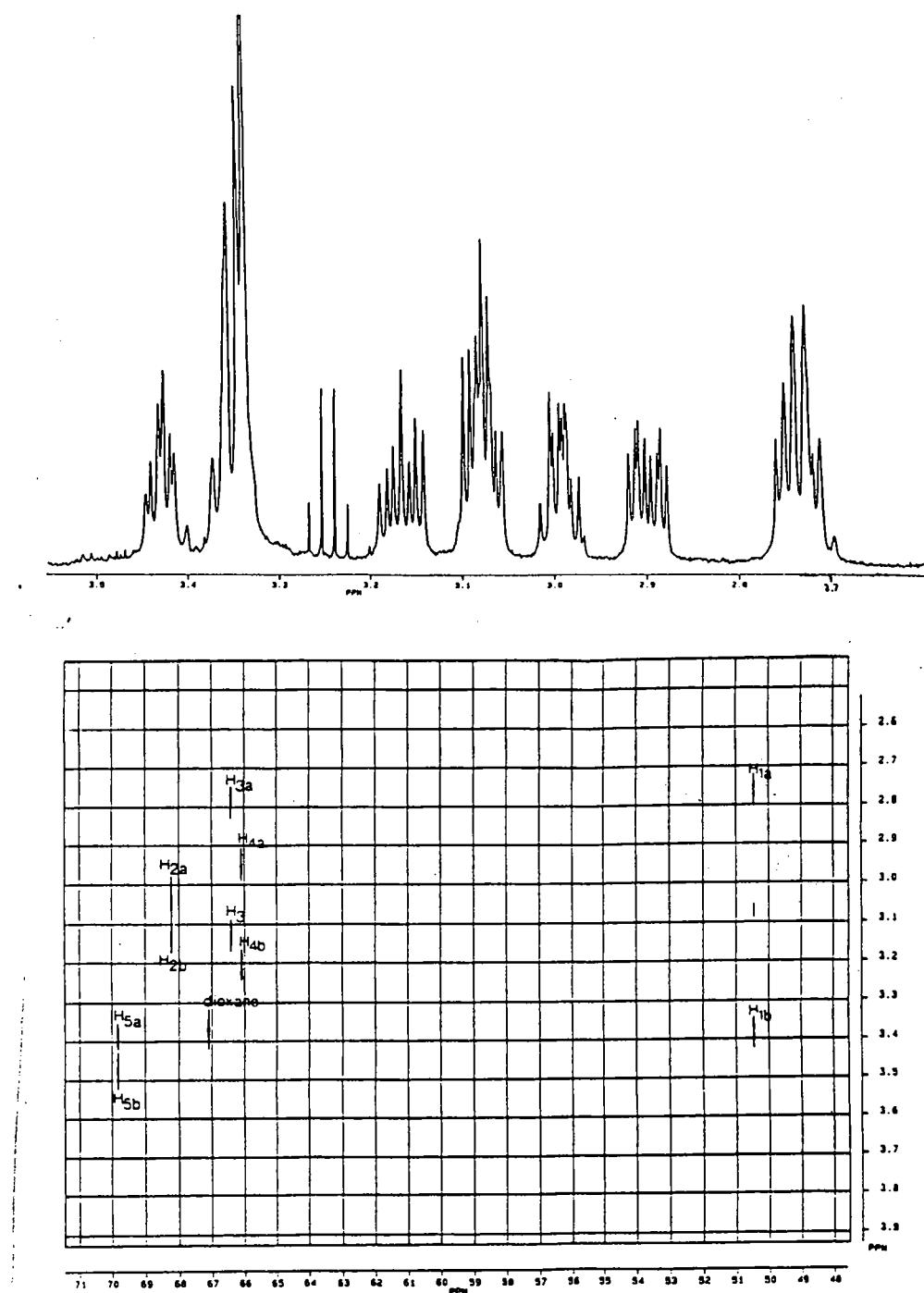


Figure 2. The upper spectrum is a portion of the ^1H NMR spectrum (500 MHz) of a benzene- d_6 solution prepared from **2-H** and excess Np_2Mg having a ratio of all neopentyl groups to azacrown rings of ca. 3. The lower spectrum is a ^1H - ^{13}C correlated (COESY) spectrum (500 MHz) of the same solution (tentative structural assignments are indicated on each absorption).

Table 1. ^1H NMR Absorptions^a of 1-MgR Compounds and 1-H

Compound	δ					
	CH ₂ O ^b	CH ₃ O ^c	CH ₂ N ^b	CH _n Mg	CH ₃ CMg	CH ₃ CCMg
1-H^d	3.29	3.07	2.69	---	---	---
1-MgMe	3.24	3.03	2.82	-1.08 ^e	---	---
1-MgEt	3.22	3.05	2.80	-0.20 ^f	1.80 ^g	---
1-MgNp	3.21	3.04	2.78	-0.01 ^e	---	1.47 ^e
1-Mg-1	3.59	3.25	3.48	---	---	---

^a Absorptions are relative to internal C₆D₅H (δ 7.15). ^b All are triplets; $J = 5.2$ Hz for all except **1-Mg-1** for which $J = 5.0$ Hz. ^c All are singlets. ^d The NH absorption is at δ 1.62. ^e A singlet. ^f A quartet, $J = 8.2$ Hz. ^g A triplet, $J = 8.2$ Hz.

Table 2. ^{13}C NMR Absorptions^a of 1-MgR Compounds and 1-H

Compound	δ					
	CN	CCN	CO	CMg	CCMg	CCCMg
1-H	49.24	72.14	57.83	---	---	---
1-MgMe	50.56	72.61	58.53	-18.93	---	---
1-MgEt	50.76	72.77	58.75	-2.96	15.47	---
1-MgNp	50.67	72.27	58.51	28.49 ^b	32.59 ^b	27.25
1-Mg-1	51.05	76.29	55.49	---	---	---

^a Absorptions are relative to C₆D₆ (δ 128.0). ^b The assignments for these absorptions may be reversed.

Table 3. ^1H NMR Data^a for Benzene- d_6 Solutions of Np_2Mg in the Presence of Donor Molecules

donor	molar ratio ^{b,c} (donor:Np ₂ Mg)	δ		donor ^d
		Np ₂ Mg CH ₃	CH ₂	
none	---	1.25	0.21	---
Et ₂ O	4	1.49	0.02	0.99 [1.10] (CH ₃), 3.30 [3.25] (CH ₂)
DME	6	1.39	0.03	3.00 [3.10] (CH ₃), 3.20 [3.23] (CH ₂)
THF	5	1.51	0.06	1.36 [1.42] (CH ₂ O), 3.20 [3.55] (CH ₂ O)
TMEDA	1	1.44	-0.05	1.60 [2.11] (CH ₃), 1.83 [2.35] (CH ₂)
HMPA	0.8	1.55	0.30	2.15 and 2.27 [2.40] (CH ₃) ^c
HMPA	1.2	1.57	0.21	2.24 and 2.29 [2.40] (CH ₃) ^c
HMPA	3.5	1.66	0.17	2.40 [2.40] (CH ₃)

^a Absorptions are relative to internal C₆D₅H (δ 7.15). ^b Np₂Mg concentration is 0.3M. Molar ratio determined by integration of NMR absorptions. ^c For an explanation of the multiple HMPA absorptions, see Chubb, J. E.; Richey, H. G., Jr. *Organometallics* **2003**, 22, 541. ^d Absorptions in brackets are of benzene- d_6 solutions (ca. 0.5 M) of the solvents alone.

X-ray Diffraction Analysis of (1-MgEt)₂

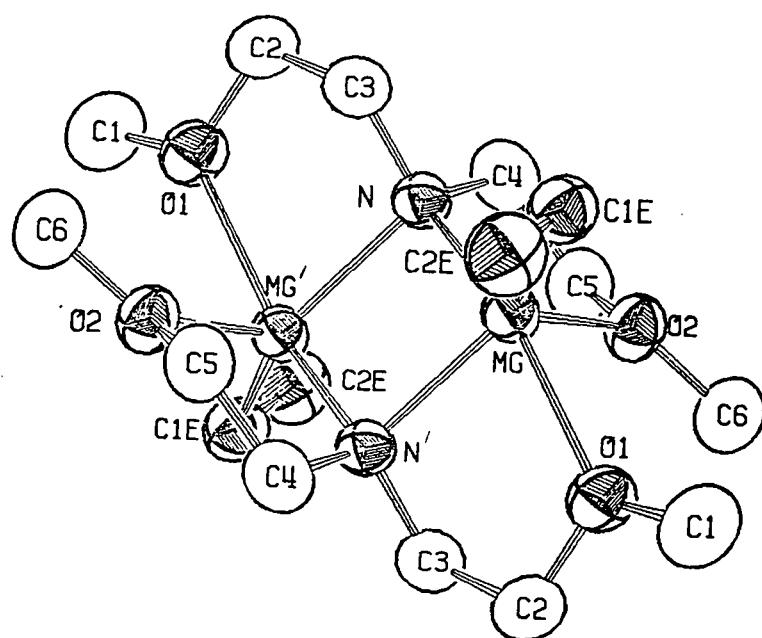


Table of Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	B(A2)
MG	0.03245(9)	0.08412(5)	0.09566(7)	2.98(2)
O1	0.2450(2)	0.0753(1)	0.2925(2)	4.21(4)
O2	-0.2321(2)	-0.1349(1)	0.0082(2)	3.80(4)
N	0.0680(2)	-0.0564(1)	0.1270(2)	3.12(4)
C1E	-0.1227(3)	0.1729(2)	0.1949(3)	4.15(6)
C2E	-0.1928(4)	0.1386(2)	0.3240(3)	5.24(7)
C1	0.2896(4)	0.1338(2)	0.4126(3)	5.99(7)
C2	0.3133(3)	-0.0129(2)	0.3181(3)	4.60(6)
C3	0.2502(3)	-0.0661(2)	0.1813(3)	4.23(6)
C4	-0.0296(4)	-0.1039(2)	0.2195(3)	4.53(6)
C5	-0.2142(3)	-0.1038(2)	0.1545(3)	4.42(6)
C6	-0.4059(3)	-0.1561(2)	-0.0518(3)	5.12(7)
H1EA	-0.213(3)	0.196(2)	0.132(3)	*****
H1EB	-0.055(3)	0.221(1)	0.223(2)	*****
H2EA	-0.262(3)	0.181(2)	0.369(3)	*****
H2EB	-0.262(3)	0.087(1)	0.304(2)	*****
H2EC	-0.105(3)	0.117(2)	0.395(2)	*****
H1A	0.407(3)	0.140(1)	0.440(2)	*****
H1B	0.245(3)	0.103(2)	0.491(2)	*****
H1C	0.228(3)	0.185(2)	0.395(2)	*****
H2A	0.265(3)	-0.034(1)	0.402(2)	*****
H2B	0.441(3)	-0.007(2)	0.338(2)	*****
H3A	0.318(3)	-0.043(1)	0.111(2)	*****
H3B	0.283(3)	-0.128(1)	0.201(2)	*****
H4A	-0.021(3)	-0.078(1)	0.315(2)	*****
H4B	0.008(3)	-0.165(1)	0.234(2)	*****
H5A	-0.285(3)	-0.142(1)	0.203(2)	*****
H5B	-0.263(3)	-0.041(1)	0.151(3)	*****
H6A	-0.447(3)	-0.199(1)	0.004(2)	*****
H6B	-0.404(3)	-0.182(2)	-0.141(2)	*****
H6C	-0.472(3)	-0.098(2)	-0.065(2)	*****

Starred atoms were refined isotropically.
 Anisotropically refined atoms are given in the form of the
 isotropic equivalent thermal parameter defined as:

$$(4/3) * [a^2*B(1,1) + b^2*B(2,2) + c^2*B(3,3) + ab(\cos \gamma)*B(1,2)
 + ac(\cos \beta)*B(1,3) + bc(\cos \alpha)*B(2,3)]$$

Table of General Temperature Factor Expressions - U's

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
MG	0.0442(4)	0.0358(4)	0.0339(4)	0.0005(4)	0.0093(3)	-0.0002(3)
O1	0.060(1)	0.058(1)	0.0383(9)	0.0007(9)	-0.0026(8)	-0.0078(8)
O2	0.0498(9)	0.0529(9)	0.0428(8)	-0.0122(8)	0.0115(7)	-0.0043(7)
N	0.044(1)	0.039(1)	0.0346(9)	0.0019(9)	0.0052(8)	0.0057(9)
C1E	0.052(1)	0.055(1)	0.051(1)	0.004(1)	0.010(1)	-0.006(1)
C2E	0.072(2)	0.072(2)	0.061(1)	-0.000(2)	0.025(1)	-0.012(1)
C1	0.077(2)	0.086(2)	0.059(2)	-0.006(2)	-0.002(2)	-0.020(2)
C2	0.055(1)	0.064(2)	0.050(1)	0.005(1)	-0.004(1)	0.007(1)
C3	0.056(1)	0.049(1)	0.053(1)	0.010(1)	0.003(1)	0.006(1)
C4	0.079(2)	0.054(1)	0.039(1)	-0.011(1)	0.009(1)	0.011(1)
C5	0.068(1)	0.060(2)	0.044(1)	-0.018(1)	0.023(1)	-0.000(1)
C6	0.059(2)	0.072(2)	0.064(2)	-0.018(1)	0.011(1)	0.002(1)

The form of the anisotropic thermal parameter is:
 $\exp[-2\pi i(ha_2U(1,1) + kb_2U(2,2) + 12c_2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3) + 2k1bcU(2,3))]$ where a,b, and c are reciprocal lattice constants.

Table of Bond Distances in Angstroms.

Atom1	Atom2	Distance	Atom1	Atom2	Distance	Atom1	Atom2	Distance
=====	=====	=====	=====	=====	=====	=====	=====	=====
MG	MG'	3.079(1)	C1E	C2E	1.517(3)	C3	H3A	0.99(2)
MG	O1	2.272(1)	C1E	H1EA	0.91(2)	C3	H3B	0.97(2)
MG	O2	2.154(1)	C1E	H1EB	0.91(2)	C4	C5	1.487(3)
MG	N	2.131(1)	C2E	H2EA	0.99(2)	C4	H4A	0.97(2)
MG	N'	2.140(1)	C2E	H2EB	0.95(2)	C4	H4B	0.96(2)
MG	C1E	2.142(2)	C2E	H2EC	0.93(2)	C5	H5A	0.98(2)
O1	C1	1.420(3)	C1	H1A	0.93(2)	C5	H5B	1.02(2)
O1	C2	1.429(2)	C1	H1B	0.99(2)	C6	H6A	0.92(2)
O2	C5	1.434(2)	C1	H1C	0.91(2)	C6	H6B	0.93(2)
O2	C6	1.432(3)	C2	C3	1.517(3)	C6	H6C	1.01(2)
N	C3	1.456(2)	C2	H2A	0.99(2)			
N	C4	1.454(3)	C2	H2B	1.00(2)			

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Bond Angles in Degrees

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
=====	=====	=====	=====	=====	=====	=====	=====	=====	=====	=====	=====
MG	MG	O1	116.68(4)	MG	N	C4	110.2(1)	H2A	C2	H2B	114.(2)
MG	MG	O2	94.67(4)	C3	N	C4	110.5(2)	N	C3	C2	113.5(2)
MG	MG	N	44.00(4)	MG	C1E	C2E	117.8(2)	N	C3	H3A	111.(1)
MG	MG	N'	43.77(4)	MG	C1E	H1EA	114.(1)	N	C3	H3B	112.(1)
MG	MG	C1E	135.38(7)	MG	C1E	H1EB	105.(1)	C2	C3	H3A	104.(1)
O1	MG	O2	83.50(5)	C2E	C1E	H1EA	106.(1)	C2	C3	H3B	108.(1)
O1	MG	N	76.71(5)	C2E	C1E	H1EB	109.(1)	H3A	C3	H3B	108.(2)
O1	MG	N'	151.02(6)	H1EA	C1E	H1EB	104.(2)	N	C4	C5	110.6(2)
O1	MG	C1E	94.88(7)	C1E	C2E	H2EA	116.(1)	N	C4	H4A	114.(1)
O2	MG	N	108.61(6)	C1E	C2E	H2EB	114.(1)	N	C4	H4B	111.(1)
O2	MG	N'	78.42(5)	C1E	C2E	H2EC	111.(1)	C5	C4	H4A	106.(1)
O2	MG	C1E	120.93(7)	H2EA	C2E	H2EB	105.(2)	C5	C4	H4B	109.(1)
N	MG	N'	87.77(5)	H2EB	C2E	H2EC	109.(2)	H4A	C4	H4B	107.(2)
N	MG	C1E	128.51(8)	H2EB	C2E	H2EC	101.(2)	O2	C5	C4	108.1(2)
N'	MG	C1E	113.82(8)	O1	C1	H1A	112.(1)	O2	C5	H5A	107.(1)
MG	O1	C1	130.8(2)	O1	C1	H1B	104.(1)	O2	C5	H5B	108.(1)
MG	O1	C2	113.2(1)	O1	C1	H1C	109.(1)	C4	C5	H5A	115.(1)
C1	O1	C2	113.6(2)	H1A	C1	H1B	109.(2)	C4	C5	H5B	111.(1)
MG	O2	C5	111.7(1)	H1A	C1	H1C	117.(2)	H5A	C5	H5B	108.(2)
MG	O2	C6	129.9(1)	H1B	C1	H1C	106.(2)	O2	C6	H6A	110.(1)
C5	O2	C6	111.6(2)	O1	C2	C3	106.5(2)	O2	C6	H6B	106.(1)
MG	N	MG	92.23(5)	O1	C2	H2A	104.(1)	O2	C6	H6C	108.(1)
MG	N	C3	104.2(1)	O1	C2	H2B	107.(1)	H6A	C6	H6B	107.(2)
MG	N	C4	119.4(1)	C3	C2	H2A	113.(1)	H6A	C6	H6C	116.(2)
MG	N'	C3	119.6(1)	C3	C2	H2B	112.(1)	H6B	C6	H6C	110.(2)

Numbers in parentheses are estimated standard deviations in the least significant digits.

X-ray Diffraction Analysis of (2-MgEt)₂

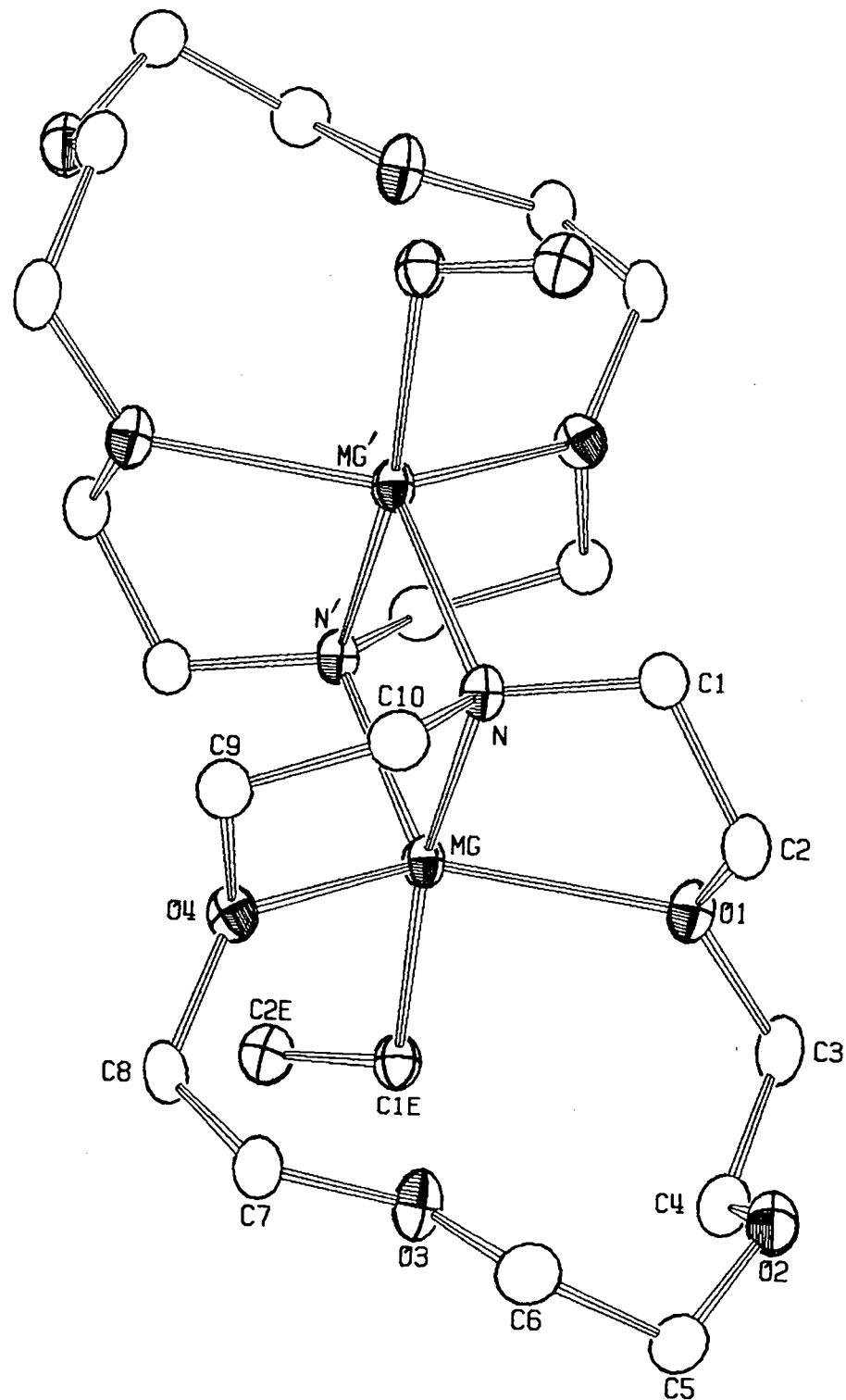


Table of Positional Parameters and Their Estimated Standard Deviations

ATOM	X	Y	Z	B(Å²)
MG	0.4742(1)	0.12666(9)	-0.02833(7)	2.70(2)
O1	0.2054(3)	0.1574(2)	-0.0170(1)	3.63(5)
O2	0.1355(3)	0.3778(2)	0.0667(2)	4.37(6)
O3	0.4932(3)	0.3750(2)	0.1083(1)	4.22(6)
O4	0.6808(3)	0.1772(2)	0.0741(1)	3.33(5)
N	0.4404(3)	0.0228(2)	0.0844(2)	2.87(6)
C1E	0.4783(4)	0.2584(3)	-0.1284(2)	3.20(8)
C2E	0.6059(4)	0.2510(3)	-0.1902(2)	4.13(9)
C1	0.2638(4)	0.0129(3)	0.0917(2)	3.44(8)
C2	0.1716(4)	0.1216(3)	0.0695(2)	3.82(8)
C3	0.1021(4)	0.2484(3)	-0.0568(2)	4.23(9)
C4	0.1631(4)	0.3611(3)	-0.0228(2)	4.27(9)
C5	0.2352(5)	0.4645(3)	0.1131(3)	4.8(1)
C6	0.3943(5)	0.4224(3)	0.1672(3)	4.55(9)
C7	0.6581(4)	0.3560(3)	0.1520(2)	3.97(9)
C8	0.7453(4)	0.2885(3)	0.0912(2)	3.65(8)
C9	0.7029(4)	0.1015(3)	0.1496(2)	3.71(8)
C10	0.5354(4)	0.0673(3)	0.1675(2)	3.58(8)
H1EA	0.492(4)	0.330(3)	-0.096(2)	*****
H1EB	0.372(4)	0.272(3)	-0.166(2)	*****
H2EA	0.594(4)	0.175(3)	-0.229(2)	*****
H2EB	0.708(4)	0.252(3)	-0.158(2)	*****
H2EC	0.605(4)	0.314(3)	-0.238(2)	*****
H1A	0.211(4)	-0.045(3)	0.049(2)	*****
H1B	0.250(4)	-0.014(3)	0.153(2)	*****
H2A	0.211(4)	0.183(3)	0.113(2)	*****
H2B	0.061(4)	0.109(3)	0.062(2)	*****
H3A	0.101(4)	0.244(3)	-0.126(2)	*****
H3B	-0.010(4)	0.234(3)	-0.044(2)	*****
H4A	0.110(4)	0.417(3)	-0.060(2)	*****
H4B	0.281(4)	0.371(3)	-0.025(2)	*****
H5A	0.257(4)	0.529(3)	0.069(2)	*****
H5B	0.185(4)	0.500(3)	0.157(2)	*****
H6A	0.460(4)	0.489(3)	0.200(2)	*****
H6B	0.374(4)	0.360(3)	0.209(2)	*****
H7A	0.718(4)	0.426(3)	0.165(2)	*****
H7B	0.659(4)	0.318(3)	0.211(2)	*****
H8A	0.729(4)	0.324(3)	0.034(2)	*****
H8B	0.867(4)	0.285(3)	0.122(2)	*****
H9A	0.765(4)	0.136(3)	0.205(2)	*****
H9B	0.766(4)	0.034(3)	0.132(2)	*****
H10A	0.555(4)	0.007(3)	0.216(2)	*****
H10B	0.487(4)	0.134(3)	0.196(2)	*****

Starred atoms were refined isotropically.
 Anisotropically refined atoms are given in the form of the
 isotropic equivalent thermal parameter defined as:

$$(4/3) * [a^2*B(1,1) + b^2*B(2,2) + c^2*B(3,3) + ab(\cos \gamma)*B(1,2)
 + ac(\cos \beta)*B(1,3) + bc(\cos \alpha)*B(2,3)]$$

Table of General Temperature Factor Expressions - U's

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
M6	0.0284(5)	0.0435(6)	0.0301(5)	-0.0005(5)	0.0042(4)	0.0018(6)
O1	0.036(1)	0.061(2)	0.043(1)	0.010(1)	0.010(1)	0.005(1)
O2	0.042(1)	0.062(2)	0.066(1)	-0.000(1)	0.021(1)	-0.004(1)
O3	0.036(1)	0.076(2)	0.047(1)	0.011(1)	0.007(1)	-0.004(1)
O4	0.041(1)	0.049(1)	0.034(1)	-0.005(1)	-0.001(1)	0.000(1)
N	0.030(1)	0.048(2)	0.033(1)	0.000(1)	0.010(1)	-0.002(1)
C1E	0.036(2)	0.050(2)	0.036(2)	0.002(2)	0.008(1)	0.006(2)
C2E	0.053(2)	0.061(2)	0.046(2)	0.006(2)	0.017(2)	0.006(2)
C1	0.039(2)	0.048(2)	0.048(2)	-0.004(2)	0.020(1)	0.002(2)
C2	0.036(2)	0.062(2)	0.051(2)	0.000(2)	0.017(1)	0.003(2)
C3	0.033(2)	0.077(3)	0.048(2)	0.010(2)	-0.001(2)	0.005(2)
C4	0.043(2)	0.062(2)	0.056(2)	0.013(2)	0.008(2)	0.013(2)
C5	0.051(2)	0.052(2)	0.082(3)	0.011(2)	0.016(2)	-0.003(2)
C6	0.062(2)	0.057(2)	0.057(2)	0.006(2)	0.017(2)	-0.004(2)
C7	0.036(2)	0.058(2)	0.052(2)	-0.001(2)	-0.003(2)	-0.010(2)
C8	0.029(2)	0.059(2)	0.049(2)	-0.013(2)	0.003(2)	-0.004(2)
C9	0.046(2)	0.054(2)	0.036(2)	-0.001(2)	-0.008(2)	0.002(2)
C10	0.054(2)	0.052(2)	0.029(2)	0.002(2)	0.008(2)	0.001(2)

The form of the anisotropic thermal parameter is:

$$\exp[-2P12Ch2a2U(1,1) + k2b2U(2,2) + 12c2U(3,3) + 2hkabU(1,2) + 2h1acU(1,3) + 2k1bcU(2,3)]$$
 where a,b, and c are reciprocal lattice constants.

Table of Bond Distances in Angstroms

Atom1	Atom2	Distance	Atom1	Atom2	Distance	Atom1	Atom2
=====	=====	=====	=====	=====	=====	=====	=====
M6	M6'	3.138(2)	C1E	C2E	1.527(4)	C5	C6
M6	O1	2.282(2)	C1E	H1EA	0.98(3)	C5	H5A
M6	O4	2.169(2)	C1E	H1EB	0.97(3)	C5	H5B
M6	N	2.158(2)	C2E	H2EA	1.07(3)	C6	H6A
M6	N'	2.139(2)	C2E	H2EB	0.90(3)	C6	H6B
M6	C1E	2.178(3)	C2E	H2EC	1.03(3)	C7	C8
O1	C2	1.445(3)	C1	C2	1.507(4)	C7	H7A
O1	C3	1.440(3)	C1	H1A	0.99(3)	C7	H7B
O2	C4	1.420(4)	C1	H1B	1.01(3)	C8	H8A
O2	C5	1.421(4)	C2	H2A	1.00(3)	C8	H8B
O3	C6	1.424(4)	C2	H2B	0.91(3)	C9	C10
O3	C7	1.419(3)	C3	C4	1.490(5)	C9	H9A
O4	C8	1.432(3)	C3	H3A	1.04(3)	C9	H9B
O4	C9	1.437(3)	C3	H3B	0.99(3)	C10	H10A
N	C1	1.484(3)	C4	H4A	0.93(3)	C10	H10B
N	C10	1.453(3)	C4	H4B	0.99(3)		

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Bond Angles in Degrees

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
=====	=====	=====	=====	=====	=====	=====	=====	=====	=====	=====	=====
MG	MG	O1	102.67(6)	H1EA	C1E	H1EB	99.(2)	C6	C5	H5A	111.(2)
MG	MG	O4	91.21(6)	C1E	C2E	H2EA	112.(2)	C6	C5	H5B	103.(2)
MG	MG	N	42.87(6)	C1E	C2E	H2EB	111.(2)	H5A	C5	H5B	106.(2)
MG	MG	N	43.32(6)	C1E	C2E	H2EC	117.(2)	O3	C6	C5	109.5(3)
MG	MG	C1E	149.35(9)	H2EA	C2E	H2EB	107.(2)	O3	C6	H6A	107.(2)
O1	MG	O4	123.46(8)	H2EA	C2E	H2EC	104.(2)	O3	C6	H6B	106.(2)
O1	MG	N	76.65(7)	H2EB	C2E	H2EC	105.(3)	C5	C6	H6A	110.(2)
O1	MG	N	123.69(8)	N	C1	C2	112.2(2)	C5	C6	H6B	111.(2)
O1	MG	C1E	94.11(9)	N	C1	H1A	109.(2)	H6A	C6	H6B	113.(2)
O4	MG	N	78.06(8)	N	C1	H1B	112.(2)	O3	C7	C8	109.0(2)
O4	MG	N	103.86(8)	C2	C1	H1A	108.(2)	O3	C7	H7A	112.(2)
O4	MG	C1E	100.8(1)	C2	C1	H1B	110.(2)	O3	C7	H7B	110.(2)
N	MG	N	86.19(9)	H1A	C1	H1B	106.(2)	C8	C7	H7A	108.(2)
N	MG	C1E	167.5(1)	O1	C2	C1	106.3(2)	C8	C7	H7B	112.(2)
N	MG	C1E	106.1(1)	O1	C2	H2A	107.(2)	H7A	C7	H7B	106.(3)
MG	O1	C2	111.5(2)	O1	C2	H2B	106.(2)	O4	C8	C7	113.7(2)
MG	O1	C3	126.8(2)	C1	C2	H2A	113.(2)	O4	C8	H8A	105.(2)
C2	O1	C3	113.7(2)	C1	C2	H2B	110.(2)	O4	C8	H8B	110.(2)
C4	O2	C5	113.1(3)	H2A	C2	H2B	114.(3)	C7	C8	H8A	108.(2)
C6	O3	C7	112.4(2)	O1	C3	C4	113.2(3)	C7	C8	H8B	107.(2)
MG	O4	C8	127.1(2)	O1	C3	H3A	106.(2)	H8A	C8	H8B	114.(2)
MG	O4	C9	111.3(2)	O1	C3	H3B	107.(2)	O4	C9	C10	108.7(2)
C8	O4	C9	116.5(2)	C4	C3	H3A	110.(2)	O4	C9	H9A	113.(2)
MG	N	MG	93.81(9)	C4	C3	H3B	111.(2)	O4	C9	H9B	106.(2)
MG	N	C1	111.7(2)	H3A	C3	H3B	110.(2)	C10	C9	H9A	108.(2)
MG	N	C10	110.3(2)	O2	C4	C3	110.6(3)	C10	C9	H9B	111.(2)
MG	N	C1	111.0(2)	O2	C4	H4A	110.(2)	H9A	C9	H9B	110.(2)
MG	N	C10	118.6(2)	O2	C4	H4B	110.(2)	N	C10	C9	108.2(2)
C1	N	C10	110.4(2)	C3	C4	H4A	110.(2)	N	C10	H10A	111.(2)
MG	C1E	C2E	118.5(2)	C3	C4	H4B	112.(2)	N	C10	H10B	117.(2)
MG	C1E	H1EA	107.(2)	H4A	C4	H4B	105.(3)	C9	C10	H10A	106.(2)
MG	C1E	H1EB	113.(2)	O2	C5	C6	113.5(3)	C9	C10	H10B	108.(2)
C2E	C1E	H1EA	109.(2)	O2	C5	H5A	111.(2)	H10A	C10	H10B	106.(2)
C2E	C1E	H1EB	108.(2)	O2	C5	H5B	112.(2)				

Numbers in parentheses are estimated standard deviations in the least significant digits.

X-ray Diffraction Analysis of 2-MgOEt(Et₂Mg)₂.

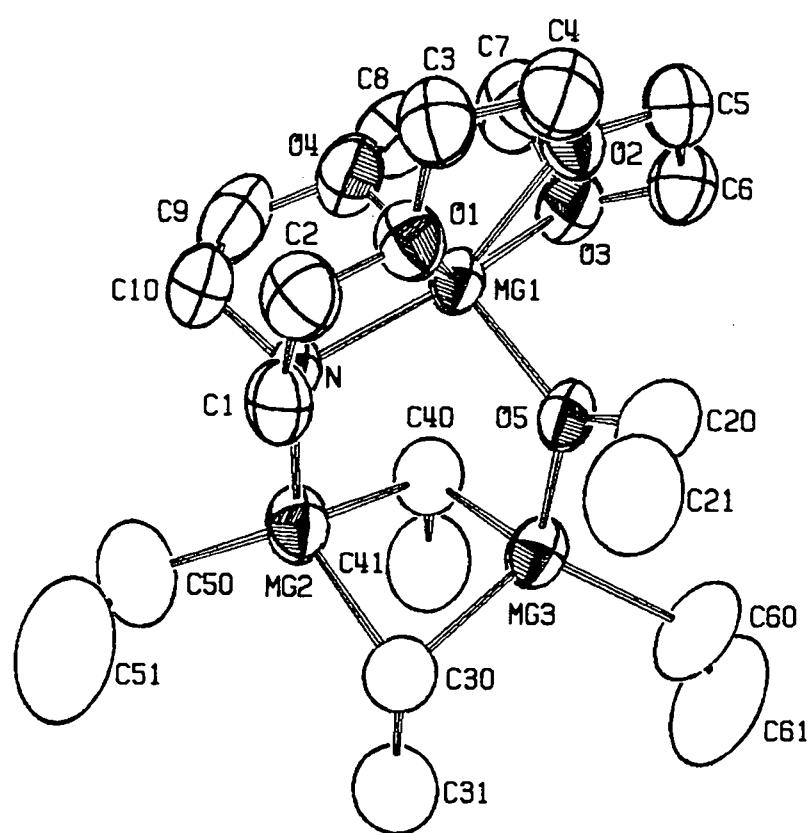


Table of Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	B(A2)
MG1	0.2411(1)	0.1515(2)	0.07275(9)	3.60(4)
MG2	0.3304(1)	-0.1275(2)	0.0229(1)	4.93(5)
MG3	0.2011(1)	-0.0040(2)	-0.1089(1)	4.54(5)
O1	0.3411(2)	0.2970(3)	0.1223(2)	5.0(1)
O2	0.1841(2)	0.3342(3)	0.0921(2)	5.0(1)
O3	0.0965(2)	0.1202(3)	0.0499(2)	4.9(1)
O4	0.2325(2)	0.0413(4)	0.1756(2)	5.7(1)
O5	0.2083(2)	0.1626(3)	-0.0478(2)	4.05(9)
N	0.3536(2)	0.0318(4)	0.1077(2)	4.1(1)
C1	0.4214(3)	0.1193(6)	0.1034(3)	5.7(2)
C2	0.4287(3)	0.2508(6)	0.1481(3)	6.3(2)
C3	0.3283(3)	0.4025(6)	0.1695(3)	5.9(2)
C4	0.2378(4)	0.4481(6)	0.1235(3)	6.7(2)
C5	0.0925(3)	0.3583(6)	0.0547(3)	6.1(2)
C6	0.0510(3)	0.2344(6)	0.0078(3)	5.8(2)
C7	0.0858(3)	0.0922(7)	0.1259(3)	7.1(2)
C8	0.1502(3)	-0.0079(6)	0.1706(3)	6.9(2)
C9	0.3000(4)	-0.0556(7)	0.2091(3)	7.7(2)
C10	0.3780(4)	-0.0099(7)	0.1957(3)	6.9(2)
C20	0.1972(4)	0.2813(6)	-0.0909(3)	7.0(2)
C21	0.2711(5)	0.3277(7)	-0.1079(4)	10.4(3)
C30	0.3380(3)	-0.0544(6)	-0.0994(3)	5.1(2)
C31	0.3317(4)	-0.1656(6)	-0.1595(4)	7.5(2)
C40	0.1813(3)	-0.1639(5)	-0.0248(3)	5.3(2)
C41	0.1528(4)	-0.2882(6)	-0.0755(4)	7.7(2)
C50	0.4021(4)	-0.3004(7)	0.0751(4)	9.3(2)
C51	0.4792(7)	-0.325(1)	0.0732(6)	17.2(4)
C60	0.1017(4)	0.0112(7)	-0.2317(4)	8.4(2)
C61	0.0576(5)	-0.0887(9)	-0.2865(4)	13.6(3)
H1A	0.472(2)	0.072(4)	0.122(2)	*****
H1B	0.413(2)	0.135(4)	0.049(2)	*****
H2A	0.449(2)	0.232(4)	0.211(2)	*****
H2B	0.459(2)	0.306(4)	0.130(2)	*****
H3A	0.371(2)	0.463(4)	0.172(2)	*****
H3B	0.337(2)	0.356(4)	0.222(2)	*****
H4A	0.233(2)	0.489(4)	0.078(2)	*****
H4B	0.218(2)	0.492(4)	0.157(2)	*****
H5A	0.084(2)	0.418(4)	0.020(2)	*****
HSB	0.075(2)	0.360(4)	0.099(2)	*****
H6A	0.056(2)	0.236(4)	-0.040(2)	*****
H6B	-0.010(2)	0.231(4)	-0.001(2)	*****
H7A	0.101(2)	0.169(4)	0.161(2)	*****
H7B	0.031(2)	0.065(4)	0.114(2)	*****

H8A	0.153(2)	-0.018(4)	0.224(2)	*****
H8B	0.138(2)	-0.097(4)	0.142(2)	*****
H9A	0.320(2)	-0.061(4)	0.271(2)	*****
H9B	0.277(2)	-0.148(4)	0.175(2)	*****
H10A	0.416(2)	-0.071(4)	0.208(2)	*****
H10B	0.375(2)	0.062(4)	0.241(2)	*****
H20A	0.187(2)	0.361(4)	-0.064(2)	*****
H20B	0.158(2)	0.280(4)	-0.146(2)	*****
H21A	0.255	0.417	-0.136	*****
H21B	0.277	0.267	-0.148	*****
H21C	0.318	0.334	-0.058	*****
H30A	0.394(2)	-0.031(4)	-0.064(2)	*****
H30B	0.329(2)	0.028(4)	-0.131(2)	*****
H31A	0.276(2)	-0.184(4)	-0.200(2)	*****
H31B	0.368(2)	-0.164(4)	-0.188(2)	*****
H31C	0.346(2)	-0.236(4)	-0.130(2)	*****
H40A	0.190(2)	-0.193(4)	0.026(2)	*****
H40B	0.130(2)	-0.111(4)	-0.029(2)	*****
H41A	0.198(2)	-0.355(4)	-0.074(2)	*****
H41B	0.130(2)	-0.274(4)	-0.125(2)	*****
H41C	0.113(2)	-0.354(4)	-0.067(2)	*****
H50A	0.412(2)	-0.310(4)	0.130(2)	*****
H50B	0.412(2)	-0.424(4)	0.025(2)	*****
H51A	0.509	-0.406	0.106	*****
H51B	0.516	-0.248	0.098	*****
H51C	0.469	-0.334	0.016	*****
H60A	0.119(2)	0.068(4)	-0.251(2)	*****
H60B	0.021(2)	0.026(4)	-0.246(2)	*****
H61A	0.016	-0.060	-0.342	*****
H61B	0.024	-0.145	-0.262	*****
H61C	0.099	-0.150	-0.296	*****

Starred atoms were refined isotropically.
 Anisotropically refined atoms are given in the form of the
 isotropic equivalent thermal parameter defined as:

$$(4/3) * [a^2*B(1,1) + b^2*B(2,2) + c^2*B(3,3) + ab(\cos \gamma)*B(1,2) + ac(\cos \beta)*B(1,3) + bc(\cos \alpha)*B(2,3)]$$

Table of General Temperature Factor Expressions - U's

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
MG1	0.0438(8)	0.050(1)	0.0408(8)	-0.0001(9)	0.0140(6)	0.0022(9)
MG2	0.056(1)	0.063(1)	0.065(1)	0.008(1)	0.0199(8)	-0.002(1)
MG3	0.055(1)	0.068(1)	0.0468(9)	-0.004(1)	0.0168(7)	-0.002(1)
O1	0.053(2)	0.063(2)	0.071(2)	-0.005(2)	0.021(1)	-0.023(2)
O2	0.052(2)	0.057(2)	0.076(2)	0.004(2)	0.019(2)	-0.009(2)
O3	0.060(2)	0.077(3)	0.053(2)	-0.005(2)	0.026(1)	0.005(2)
O4	0.073(2)	0.089(3)	0.060(2)	0.011(2)	0.031(1)	0.025(2)
O5	0.060(2)	0.049(2)	0.043(2)	0.006(2)	0.019(1)	0.016(2)
N	0.042(2)	0.064(3)	0.043(2)	0.003(2)	0.010(2)	0.003(2)
C1	0.045(3)	0.093(4)	0.072(3)	0.009(3)	0.017(2)	-0.006(4)
C2	0.065(3)	0.089(4)	0.086(4)	-0.017(4)	0.029(3)	-0.025(4)
C3	0.073(4)	0.066(4)	0.083(4)	-0.008(3)	0.027(3)	-0.014(3)
C4	0.105(4)	0.067(4)	0.087(4)	0.003(4)	0.045(3)	-0.013(3)
C5	0.062(3)	0.097(5)	0.074(3)	0.027(3)	0.030(2)	0.006(4)
C6	0.056(3)	0.095(5)	0.064(3)	0.003(3)	0.019(3)	-0.007(3)
C7	0.064(3)	0.121(5)	0.090(4)	-0.021(4)	0.035(3)	0.000(4)
C8	0.105(4)	0.103(5)	0.074(3)	-0.023(4)	0.056(2)	0.001(3)
C9	0.120(5)	0.103(5)	0.056(3)	0.006(4)	0.021(3)	0.027(4)
C10	0.082(4)	0.107(5)	0.060(3)	0.046(4)	0.016(3)	0.009(4)
C20	0.108(4)	0.097(5)	0.069(3)	-0.007(4)	0.046(3)	-0.005(4)
C21	0.176(6)	0.102(6)	0.135(5)	0.014(5)	0.079(4)	0.033(4)
C30	0.063(3)	0.072(4)	0.064(3)	-0.008(3)	0.031(2)	-0.019(3)
C31	0.069(3)	0.126(6)	0.093(4)	-0.005(4)	0.037(3)	-0.036(4)
C40	0.066(3)	0.070(4)	0.068(3)	-0.004(3)	0.030(2)	-0.006(3)
C41	0.111(4)	0.075(4)	0.104(4)	-0.019(4)	0.040(3)	-0.010(4)
C50	0.098(4)	0.138(6)	0.128(5)	0.057(4)	0.057(3)	0.039(5)
C51	0.209(9)	0.24(1)	0.178(7)	0.076(8)	0.045(6)	0.108(7)
C60	0.115(5)	0.111(5)	0.056(4)	-0.056(4)	-0.007(3)	-0.020(4)
C61	0.152(6)	0.219(8)	0.108(5)	-0.051(6)	0.013(5)	0.071(5)

The form of the anisotropic thermal parameter is:

$$\exp[-2\pi i 2(ha_2U(1,1) + kb_2U(2,2) + 12c_2U(3,3) + 2hka_bU(1,2) + 2hla_cU(1,3) + 2kla_cU(2,3))] \text{ where } a, b, \text{ and } c \text{ are reciprocal lattice constants.}$$

Table of Bond Distances in Angstroms

Atom1	Atom2	Distance	Atom1	Atom2	Distance	Atom1	Atom2	Distance
=====	=====	=====	=====	=====	=====	=====	=====	=====
MG1	O1	2.130(3)	C1	H1A	0.91(4)	C21	H21A	1.011(6)
MG1	O2	2.159(3)	C1	H1B	0.91(4)	C21	H21B	0.955(7)
MG1	O3	2.304(3)	C2	H2A	1.03(4)	C21	H21C	0.915(7)
MG1	O4	2.149(3)	C2	H2B	0.88(4)	C30	C31	1.504(8)
MG1	O5	1.938(3)	C3	C4	1.473(8)	C30	H30A	0.93(4)
MG1	N	2.107(3)	C3	H3A	0.93(4)	C30	H30B	0.98(4)
MG2	MG3	2.741(2)	C3	H3B	0.98(4)	C31	H31A	0.93(4)
MG2	N	2.106(4)	C4	H4A	0.87(4)	C31	H31B	0.92(4)
MG2	C30	2.288(5)	C4	H4B	0.89(4)	C31	H31C	0.85(4)
MG2	C40	2.316(5)	C5	C6	1.503(8)	C40	C41	1.496(8)
MG2	C50	2.107(7)	C5	H5A	0.83(4)	C40	H40A	0.89(4)
MG3	O5	1.963(3)	C5	H5B	0.92(4)	C40	H40B	0.99(4)
MG3	C30	2.277(5)	C6	H6A	0.87(4)	C41	H41A	1.00(4)
MG3	C40	2.281(5)	C6	H6B	0.97(4)	C41	H41B	0.80(4)
MG3	C60	2.121(6)	C7	C6	1.455(9)	C41	H41C	0.98(4)
O1	C2	1.426(6)	C7	H7A	0.95(4)	C50	C51	1.321(10)
O1	C3	1.407(6)	C7	H7B	0.90(4)	C50	H50A	0.90(4)
O2	C4	1.426(7)	C8	H8A	0.90(4)	C51	H51A	1.009(8)
O2	C5	1.423(6)	C8	H8B	1.01(4)	C51	H51B	0.972(12)
O3	C6	1.412(6)	C9	C10	1.479(8)	C51	H51C	0.934(11)
O3	C7	1.421(7)	C9	H9A	1.00(4)	C60	C61	1.380(10)
O4	C8	1.428(6)	C9	H9B	1.09(4)	C60	H60A	0.78(4)
O4	C9	1.428(7)	C10	H10A	0.85(4)	C60	H60B	1.28(4)
O5	C20	1.384(7)	C10	H10B	1.09(4)	C61	H61A	0.979(7)
N	C1	1.458(6)	C20	C21	1.451(8)	C61	H61B	1.000(7)
N	C10	1.473(6)	C20	H20A	0.98(4)	C61	H61C	0.994(10)
C1	C2	1.516(8)	C20	H20B	0.91(4)			

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Bond Angles in Degrees

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
=====	=====	=====	=====	=====	=====	=====	=====	=====	=====	=====	=====
O1	MG1	O2	70.7(1)	H1A	C1	H1E	101.(4)	C20	C21	H21A	107.3(7)
O1	MG1	O3	139.6(1)	O1	C2	C1	105.1(4)	C20	C21	H21B	105.8(7)
O1	MG1	O4	108.7(1)	O1	C2	H2A	105.(2)	C20	C21	H21C	108.2(6)
O1	MG1	O5	103.7(1)	O1	C2	H2E	110.(3)	H21A	C21	H21B	107.9(6)
O1	MG1	N	79.3(1)	C1	C2	H2A	108.(2)	H21A	C21	H21C	111.4(7)
O2	MG1	O3	69.5(1)	C1	C2	H2E	108.(3)	H21B	C21	H21C	115.8(8)
O2	MG1	O4	97.9(1)	H2A	C2	H2E	120.(4)	MG2	C30	MG3	73.8(2)
O2	MG1	O5	99.3(1)	O1	C3	C4	106.2(5)	MG2	C30	C31	112.5(4)
O2	MG1	N	147.2(1)	O1	C3	H3A	104.(3)	MG2	C30	H30A	83.(3)
O3	MG1	O4	70.4(1)	O1	C3	H3E	100.(3)	MG2	C30	H30E	138.(2)
O3	MG1	O5	89.4(1)	C4	C3	H3A	115.(3)	MG3	C30	C31	108.7(4)
O3	MG1	N	136.2(2)	C4	C3	H3E	113.(2)	MG3	C30	H30A	135.(3)
O4	MG1	O5	146.8(2)	H3A	C3	H3E	116.(4)	MG3	C30	H30E	81.(3)
O4	MG1	N	78.9(2)	O2	C4	C3	108.0(5)	C31	C30	H30A	115.(3)
O5	MG1	N	100.6(1)	O2	C4	H4A	103.(3)	C31	C30	H30E	107.(2)
MG3	MG2	N	95.8(1)	O2	C4	H4E	109.(3)	H30A	C30	H30E	93.(3)
MG3	MG2	C30	52.9(1)	C3	C4	H4A	110.(3)	C30	C31	H31A	117.(3)
MG3	MG2	C40	52.8(1)	C3	C4	H4E	111.(3)	C30	C31	H31B	118.(3)
MG3	MG2	C50	149.3(3)	H4A	C4	H4E	116.(5)	C30	C31	H31C	107.(4)
N	MG2	C30	109.6(2)	O2	C5	C6	106.6(5)	H31A	C31	H31B	106.(4)
N	MG2	C40	104.3(2)	O2	C5	H5A	108.(3)	H31A	C31	H31C	104.(5)
N	MG2	C50	114.6(3)	O2	C5	H5E	105.(3)	H31B	C31	H31C	104.(5)
C30	MG2	C40	99.4(2)	C6	C5	H5A	108.(3)	MG2	C40	MG3	73.2(2)
C30	MG2	C50	115.8(3)	C6	C5	H5E	104.(3)	MG2	C40	C41	113.0(4)
C40	MG2	C50	111.6(3)	H5A	C5	H5E	125.(5)	MG2	C40	H40A	88.(3)
MG2	MG3	O5	94.6(1)	O3	C6	C5	111.2(4)	MG2	C40	H40E	136.(2)
MG2	MG3	C30	53.3(1)	O3	C6	H6A	106.(3)	MG3	C40	C41	108.2(4)
MG2	MG3	C40	54.0(1)	O3	C6	H6E	111.(3)	MG3	C40	H40A	150.(3)
MG2	MG3	C60	155.8(2)	C5	C6	H6A	108.(3)	MG3	C40	H40E	85.(2)

05	MG3	C30	107.8(2)	C5	C6	H6B	110.(3)	C41	C40	H40A	101.(3)
05	MG3	C40	104.9(2)	H6A	C6	H6B	110.(4)	C41	C40	H40B	110.(2)
05	MG3	C60	109.5(2)	03	C7	C6	107.1(5)	H40A	C40	H40B	93.(4)
C30	MG3	C40	100.8(2)	03	C7	H7A	109.(3)	C40	C41	H41A	119.(3)
C30	MG3	C60	117.0(3)	03	C7	H7B	110.(3)	C40	C41	H41B	113.(4)
C40	MG3	C60	115.9(2)	C8	C7	H7A	106.(3)	C40	C41	H41C	124.(3)
MG1	O1	C2	116.2(3)	C8	C7	H7B	113.(3)	H41A	C41	H41B	100.(5)
MG1	O1	C3	118.9(3)	H7A	C7	H7B	113.(4)	H41A	C41	H41C	95.(4)
C2	O1	C3	115.9(4)	04	C8	C7	106.3(5)	H41B	C41	H41C	102.(5)
MG1	O2	C4	120.0(3)	04	C8	H8A	108.(3)	MG2	C50	C51	122.4(7)
MG1	O2	C5	122.3(4)	04	C8	H8B	109.(2)	MG2	C50	H50A	112.(3)
C4	O2	C5	115.7(5)	C7	C8	H8A	111.(3)	C51	C50	H50A	104.(3)
MG1	O3	C6	106.7(3)	C7	C8	H8B	113.(2)	C50	C51	H51A	113.(1)
MG1	O3	C7	111.8(3)	H8A	C8	H8B	109.(4)	C50	C51	H51B	106.(1)
C6	O3	C7	113.7(5)	04	C9	C10	108.8(5)	C50	C51	H51C	105.7(9)
MG1	O4	C8	120.3(3)	04	C9	H9A	111.(2)	H51A	C51	H51B	108.0(9)
MG1	O4	C9	113.0(3)	04	C9	H9B	108.(2)	H51A	C51	H51C	111.(1)
C8	O4	C9	111.7(5)	C10	C9	H9A	104.(2)	H51B	C51	H51C	112.(1)
MG1	O5	MG3	117.4(2)	C10	C9	H9B	109.(2)	MG3	C60	C61	128.9(6)
MG1	O5	C20	123.4(3)	H9A	C9	H9B	116.(3)	MG3	C60	H60A	102.(4)
MG3	O5	C20	119.0(3)	N	C10	C9	110.4(5)	MG3	C60	H60B	123.(2)
MG1	N	MG2	109.8(2)	N	C10	H10A	110.(3)	C61	C60	H60A	115.(4)
MG1	N	C1	104.9(3)	N	C10	H10B	119.(2)	C61	C60	H60B	74.(2)
MG1	N	C10	108.2(3)	C9	C10	H10A	111.(3)	H60A	C60	H60B	112.(5)
MG2	N	C1	111.1(3)	C9	C10	H10B	79.(2)	C60	C61	H61A	115.5(8)
MG2	N	C10	113.7(3)	H10A	C10	H10B	123.(4)	C60	C61	H61B	110.8(8)
C1	N	C10	108.8(4)	05	C20	C21	116.4(6)	C60	C61	H61C	110.7(8)
N	C1	C2	114.4(5)	05	C20	H20A	117.(3)	H61A	C61	H61B	106.8(7)
N	C1	H1A	107.(3)	05	C20	H20B	116.(3)	H61A	C61	H61C	107.2(8)
N	C1	H1B	111.(3)	C21	C20	H20A	99.(3)	H61B	C61	H61C	105.2(8)
C2	C1	H1A	113.(3)	C21	C20	H20B	96.(3)				
C2	C1	H1B	109.(3)	H20A	C20	H20B	109.(4)				

Numbers in parentheses are estimated standard deviations in the least significant digits.