

## THE STRUCTURAL PROPERTIES OF DIMETHYLBIS(QUINUCLIDINE)-MAGNESIUM\*

J. TONEY AND G. D. STUCKY

*Department of Chemistry and Chemical Engineering and Materials Research Laboratory, University of Illinois, Urbana, Illinois, 61801 (U.S.A.)*

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### SUMMARY

A crystalline product,  $(\text{CH}_3)_2\text{Mg} \cdot 2 \text{NC}_7\text{H}_{13}$ , has been obtained from the reaction of dimethyl magnesium with 1-azabicyclo[2.2.2]octane (quinuclidine) in a benzene/hexane solution. The crystal structure was determined from three-dimensional X-ray data, collected by counter methods, and refined by a least-squares technique to a final *R* factor, based on *F*, of 9.4% for the 1460 observed reflections. The material crystallizes in the space group  $C_{2h}^5-P2_1/c$  of the monoclinic system in a cell with dimensions  $a = 12.147(2) \text{ \AA}$ ,  $b = 12.661(4) \text{ \AA}$  and  $c = 12.016(5) \text{ \AA}$ ,  $\beta = 108.94(18)^\circ$ : the density of  $1.07 \text{ g/cm}^3$  calculated for four formula units in the unit cell agrees with the observed density of  $1.08 \pm 0.1 \text{ g/cm}^3$ . The monomeric  $(\text{CH}_3)_2\text{Mg} \cdot 2 \text{NC}_7\text{H}_{13}$  molecule possesses no crystallographic symmetry. The coordination about the Mg atom is distorted tetrahedral with two quinuclidine and two methyl groups bonded to the magnesium atom. The magnesium-carbon distances are 2.163(9) and 2.224(8)  $\text{ \AA}$ , and the magnesium-nitrogen distances are 2.231(6) and 2.247(6)  $\text{ \AA}$ . The C-Mg-C angle is  $129.0(3)^\circ$  and the M-Mg-N angle is  $108.2(2)^\circ$ .

### INTRODUCTION

The reactions of Group II dialkyl compounds with Lewis bases have received considerable attention in recent years<sup>1</sup>. Secondary amines have been found to yield polymeric  $(\text{RMgNR}'_2)_x$  compounds unless the organic groups attached to the donor atom are relatively large<sup>2</sup>. A recent X-ray investigation has shown  $[(\text{CH}_3)_2\text{N}(\text{CH}_2)_2\text{N}(\text{CH}_3)\text{MgCH}_3]_2$  to be a dimer with nitrogen atoms in the bridging and terminal positions<sup>3</sup>. Neutral Lewis bases such as diethyl ether reportedly yield monomeric tri-co-ordinate complexes with diethylmagnesium<sup>4</sup> although  $(\text{C}_6\text{H}_5\text{C}\equiv\text{C})_2\text{Mg} \cdot 4\text{THF}$  is a six co-ordinate monomer in the solid state<sup>5</sup>.

With sufficiently bulky neutral bases, the effects of non-bonded repulsions could be removed by the formation of polymeric species through bridging solvent or alkyl groups, or by the formation of three co-ordinate monomeric species. An investigation of the reaction product of dimethylmagnesium with 1-azabicyclo[2.2.2]-octane was undertaken in order to study the type of solvation and stereochemistry

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around the magnesium atom in this compound. The structural properties of the crystalline material obtained from dimethylberyllium and quinuclidine are currently being studied by Professor J. L. Atwood of the University of Alabama, and will be reported in a subsequent publication.

#### EXPERIMENTAL

Dimethylbis(quinuclidine)magnesium was prepared by reacting dimethylmagnesium, suspended in hexane, with quinuclidine which had previously been dissolved in benzene. The benzene/hexane solution was heated on a hot plate at 72° for 30 min. Upon cooling the solution, transparent crystals appeared at the bottom of the reaction vessel. The entire reaction was carried out in a glove box in a dry, oxygen free, argon atmosphere. A number of crystals were selected for examination and individually loaded and sealed into soft glass capillaries in the glove box.

X-ray precession photographs of one of the crystalline samples showed extinctions  $h0l$  ( $l=2N+1$ ) and  $0k0$  ( $k=2N+1$ ) which uniquely determined the space group as  $P2_1/c$ . The preliminary cell constants obtained from these photographs gave a calculated density of 1.07 g/cm<sup>3</sup> versus a density of  $1.08 \pm 0.1$  g/cm<sup>3</sup> measured by the flotation method in a mixture of chlorobenzene and benzene.

Data were collected with a Picker four-circle diffractometer controlled by a PDP-8I computer. A take-off angle of 2.0° was used. The crystal was mounted with the  $c^*$  axis parallel to the length of the capillary. A 0.0005 inch nickel foil filter was placed at the opening of the scintillation counter detector. Three linearly independent reflections were accurately centered by the FACS-I<sup>6</sup> "Centering" subprogram and used to generate the angular settings for other reflections in the "Data Collection" subprogram. Two cycles of least-squares refinement of  $(\sin \theta/\lambda)^2$  values for 12 reflections in the FACS-I "Least-Squares" subprogram gave the following lattice constants:  $a=12.147(2)$  Å,  $b=12.661(4)$  Å,  $c=12.016(5)$  Å,  $\beta=108.94(18)^\circ$ . Cu-K $\alpha$  radiation was used to collect two symmetry equivalent forms of data to a maximum  $2\theta$  value of 100.0°. All peaks were scanned for 1.4° at a scan rate of 1.0°/min using the  $\theta$ - $2\theta$  scan technique. The stationary crystal-stationary counter technique was used to obtain 20 sec background counts on either side of the diffraction peak. Attenuators were automatically inserted into the diffracted beam for intensities greater than 10,000 counts/sec. A total of 2000 independent reflections were measured and 560 were found to be unobserved with  $I_0 < 3\sigma$  where  $I_0$  = observed intensity and  $\sigma = (\text{total counts} + \text{background})^{1/2}$ . A standard peak, which was measured periodically, showed a decrease in intensity of less than 2.2% during the entire collection of both data forms. The data were corrected for Lorentz and polarization effects and unobserved intensities were assigned using a local program TPRD<sup>7</sup>. The crystal had dimensions of 0.42 × 0.45 × 45 mm and a linear absorption coefficient of 7.95 cm<sup>-1</sup> for Cu-K $\alpha$  radiation. The maximum and minimum transmission factors were found to differ by less than 3%, and absorption corrections were not applied to these data. Equivalent sets of data were averaged before the solution and refinement of the structure.

#### SOLUTION AND REFINEMENT

The co-ordinates of the magnesium atom were located by inspection of the

O, V,  $\frac{1}{2}$  and U,  $\frac{1}{2}$ , W regions of a sharpened Patterson map. A three dimensional electron density map with phases based upon the magnesium atom was calculated and gave the co-ordinates of the two nitrogen atoms and fourteen peaks, which were assigned the weights of carbon atoms. Inclusion of these seventeen atoms into a structure factor calculation and subsequent least squares refinement of the scale factor lowered the *R* factor to 39%. The co-ordinates of all the carbon atoms of the quinuclidine rings were located in five refinement cycles. Only three of the originally assigned carbon atoms were found to be false peaks. Least-squares refinement of the positional parameters of the nineteen non-hydrogen atoms yielded an *R* value of 25%. The positional and isotropic temperature parameters were then refined to an *R* factor of 17%. Inclusion of anisotropic thermal parameters for the 19 non-hydrogen atoms followed by full-matrix least-squares refinement (observed reflections only) gave a final unweighted *R* value of 9.4%, where  $R = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$  and a final weighted value of 10.4%. The least squares treatment minimizes the function  $\sum w \cdot (F_o - F_c)^2$  where  $w = 1/(\sigma \cdot |F_o|)^2$ . The weights, *w*, are defined according to the following relation for counter data:

$$\sigma \cdot (F_o) = \frac{k \cdot F}{2I} \cdot \left[ T_c + \left( \frac{t_p}{t_b} \right)^2 \cdot (\beta_1 + \beta_2) + (n \cdot I)^2 \right]^{\frac{1}{2}}$$

in which *k* = scale factor, *F* = structure factor, *I* = observed intensity, *T<sub>c</sub>* = integrated peak count, *t<sub>p</sub>* = time of peak scan,  $\beta_1$  and  $\beta_2$  = background counts before and after (2 $\theta$ ) each Bragg reflection, *t<sub>b</sub>* = total background count time (*t<sub>b1</sub>* + *t<sub>b2</sub>*) and *n* = 0.02.

The final shift/error ratios of the positional and thermal parameters for the final least squares cycle were less than one. The value obtained for  $[\sum w \cdot (F_o - F_c)^2]^{\frac{1}{2}} / NO - NV$  was 1.38 where *NO* = number of observations and *NV* = number of variables. No attempts were made to locate the hydrogen atoms since the primary interest in

TABLE I

INTERATOMIC BOND LENGTHS FOR (CH<sub>3</sub>)<sub>2</sub>Mg · 2NC<sub>7</sub>H<sub>13</sub>

Atoms	Distance (Å) <sup>a</sup>	Atoms	Distance (Å) <sup>a</sup>
Mg-C(15)	2.163(9) <sup>a</sup>	Mg-N(1)	2.231(6)
Mg-C(16)	2.224(8)	Mg-N(2)	2.247(6)
N(1)-C(1)	1.502(9)	C(6)-C(7)	1.536(12)
N(1)-C(3)	1.506(9)	N(2)-C(10)	1.487(11)
N(1)-C(5)	1.485(10)	N(2)-C(8)	1.501(11)
C(1)-C(2)	1.544(11)	N(2)-C(12)	1.506(10)
C(3)-C(4)	1.556(12)	C(8)-C(9)	1.577(13)
C(5)-C(6)	1.590(12)	C(10)-C(11)	1.602(14)
C(2)-C(7)	1.570(12)	C(12)-C(13)	1.546(12)
C(4)-C(7)	1.534(12)	C(9)-C(14)	1.562(15)
C(11)-C(14)	1.515(14)	C(13)-C(14)	1.564(14)
<i>Non-bonded distances</i>			
C(15)-C(16)	3.960(12)	N(1)-N(2)	3.628(8)
C(15)-N(1)	3.478(10)	N(2)-C(15)	3.504(10)
C(16)-N(1)	3.553(9)	N(2)-C(16)	3.494(10)

<sup>a</sup> Values in parentheses here and in succeeding tables are estimated standard deviations in the least significant digits.

TABLE 2

INTERATOMIC BOND ANGLES FOR  $(\text{CH}_3)_2\text{Mg} \cdot 2\text{NC}_7\text{H}_{13}$ 

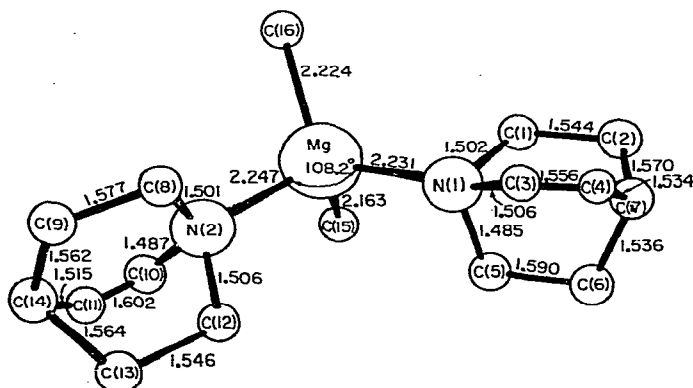
Atoms	Angle (°)	Atoms	Angle (°)
C(15)-Mg-C(16)	129.0(3)	N(1)-Mg-N(2)	108.2(2)
C(16)-Mg-N(1)	105.8(3)	C(16)-Mg-N(2)	102.8(2)
C(15)-Mg-N(1)	104.7(3)	C(15)-Mg-N(2)	105.2(3)
Mg-N(1)-C(1)	113.1(3)	Mg-N(2)-C(8)	111.9(3)
Mg-N(1)-C(3)	103.5(3)	Mg-N(2)-C(10)	115.6(3)
Mg-N(1)-C(5)	112.6(3)	Mg-N(2)-C(12)	100.7(3)
N(1)-C(1)-C(2)	111.5(2)	N(2)-C(8)-C(9)	111.3.(2)
N(1)-C(3)-C(4)	111.4(2)	N(2)-C(10)-C(11)	110.2(2)
N(1)-C(5)-C(6)	111.4(2)	N(2)-C(12)-C(13)	112.5(2)
C(1)-C(2)-C(7)	108.8(2)	C(8)-C(9)-C(14)	107.7(3)
C(3)-C(4)-C(7)	108.9(2)	C(10)-C(11)-C(14)	108.3(3)
C(5)-C(6)-C(7)	107.9(2)	C(12)-C(13)-C(14)	107.6(2)
C(1)-N(1)-C(3)	107.9(3)	C(8)-N(2)-C(10)	111.6(4)
C(1)-N(1)-C(5)	110.8(3)	C(8)-N(2)-C(12)	106.7(3)
C(3)-N(1)-C(5)	108.5(3)	C(10)-N(2)-C(12)	109.4(4)
C(2)-C(7)-C(4)	106.4(2)	C(11)-C(14)-C(13)	110.7(2)
C(2)-C(7)-C(6)	110.5(2)	C(9)-C(14)-C(11)	109.5(3)
C(4)-C(7)-C(6)	109.6(2)	C(9)-C(14)-C(13)	107.6(3)

this structure was concerned with establishing the geometry around the magnesium atom. The scattering factors used were those described in ref. 3.

The final bond distances and angles, computed with program ORFFE<sup>8</sup>, are listed in Tables 1 and 2. The final positional and anisotropic thermal parameters are given in Tables 3 and 4. Table 5 lists the observed and calculated structure factors for the observed reflections.

## DISCUSSION OF THE STRUCTURE

A view of  $(\text{CH}_3)_2\text{Mg} \cdot 2\text{NC}_7\text{H}_{13}$  is shown in Fig. 1. The molecule is a four co-ordinate monomer containing two methyl and two quinuclidine groups bonded to

Fig. 1. Molecular structure of  $(\text{CH}_3)_2\text{Mg} \cdot 2\text{NC}_7\text{H}_{13}$ .

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TABLE 3

POSITIONAL PARAMETERS FOR  $(\text{CH}_3)_2\text{Mg} \cdot 2\text{NC}_7\text{H}_{13}$ 

Atoms	X	Y	Z
Mg	0.2554(2)	0.0746(1)	0.2302(2)
C(15)	0.1814(7)	-0.0346(6)	0.0855(8)
C(16)	0.2978(6)	0.0487(6)	0.4234(6)
N(1)	0.1363(5)	0.2143(5)	0.1848(5)
N(2)	0.4289(5)	0.1229(5)	0.2178(5)
C(1)	0.1013(8)	0.2455(7)	0.0567(7)
C(2)	0.0071(9)	0.3329(8)	0.0252(8)
C(3)	0.0284(7)	0.1762(7)	0.2074(8)
C(4)	-0.0671(7)	0.2637(8)	0.1798(9)
C(5)	0.1846(7)	0.3062(7)	0.2630(8)
C(6)	0.0924(8)	0.4000(7)	0.2377(9)
C(7)	-0.0199(7)	0.3638(7)	0.1399(8)
C(8)	0.4957(8)	0.1940(9)	0.3180(10)
C(9)	0.6261(8)	0.2086(9)	0.3230(12)
C(10)	0.4247(8)	0.1659(10)	0.1013(9)
C(11)	0.5547(9)	0.1883(11)	0.1017(11)
C(12)	0.4919(8)	0.0184(7)	0.2388(10)
C(13)	0.6169(8)	0.0271(8)	0.2332(9)
C(14)	0.6408(8)	0.1464(9)	0.2166(10)

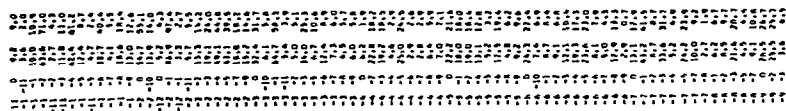
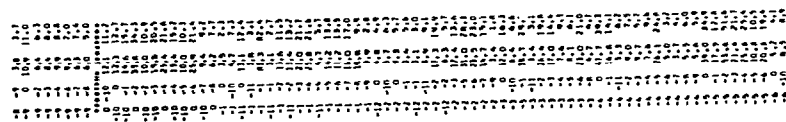
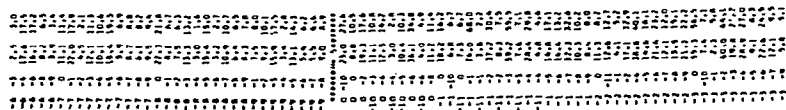
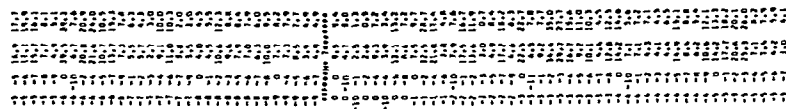
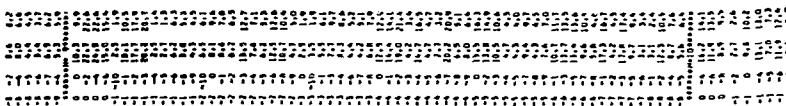
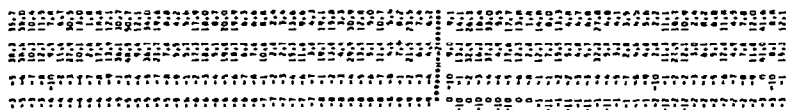
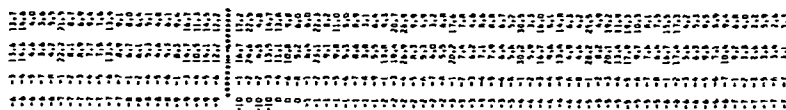
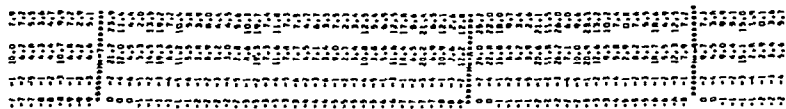
TABLE 4

ANISOTROPIC TEMPERATURE FACTORS ( $\times 10$ ) FOR  $(\text{CH}_3)_2\text{Mg} \cdot 2\text{NC}_7\text{H}_{13}$ <sup>a</sup>

Atom	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
Mg	71(2)	55(1)	91(2)	-3(2)	15(2)	2(2)
C(15)	103(9)	58(7)	157(11)	1(6)	-10(8)	-3(7)
C(16)	67(7)	94(7)	70(7)	-8(6)	19(5)	25(6)
N(1)	714(5)	55(5)	86(6)	-2(4)	31(5)	-4(5)
N(2)	68(5)	57(4)	92(6)	3(4)	22(4)	-2(4)
C(1)	135(9)	92(8)	80(8)	50(7)	48(7)	31(6)
C(2)	159(11)	118(10)	120(10)	68(8)	74(9)	36(8)
C(3)	88(8)	85(7)	151(10)	-10(6)	65(7)	15(7)
C(4)	90(8)	116(9)	170(12)	17(7)	71(8)	27(8)
C(5)	95(8)	58(7)	157(11)	-2(6)	24(7)	-39(7)
C(6)	99(9)	83(8)	184(12)	20(6)	34(8)	-30(8)
C(7)	91(8)	84(7)	135(10)	19(6)	49(7)	11(7)
C(8)	86(8)	142(11)	219(15)	-53(8)	58(9)	-98(10)
C(9)	94(9)	130(11)	281(19)	-32(8)	71(11)	-65(12)
C(10)	123(10)	207(14)	142(12)	37(10)	66(9)	98(11)
C(11)	120(10)	232(17)	193(15)	48(10)	83(10)	113(13)
C(12)	91(8)	67(7)	198(13)	23(6)	45(8)	24(7)
C(13)	102(9)	97(8)	196(13)	26(7)	66(9)	29(9)
C(14)	91(9)	123(10)	198(14)	11(8)	68(9)	29(10)

<sup>a</sup> Anisotropic temperature factors are of the form  $\exp(\beta_{11} \cdot h^2 + \beta_{22} \cdot k^2 + \beta_{33} \cdot l^2 + 2\beta_{12} \cdot h \cdot k + 2\beta_{13} \cdot h \cdot l + 2\beta_{23} \cdot k \cdot l)$ .





the magnesium atom. The distance between magnesium atoms in symmetry related units precludes the possibility of bonding between the units. Busing and Levy<sup>9</sup> have derived models for the correction of bond distances for thermal motion by assuming that the atoms vibrate independently or by assuming, in the case of a heavy atom bonded to a light atom or a light atom bonded to a group of rigidly held light atoms, that the lighter atom "rides" on the heavier atom. In  $(\text{CH}_3)_2\text{Mg} \cdot 2\text{NC}_7\text{H}_{13}$  the methyl carbons are expected to ride on the magnesium atom. The Mg-C(15) and Mg-C(16) interatomic distances corrected for riding motion [2.183(9) Å] and [2.231(8) Å] agree well with the uncorrected values [2.163(8) Å and 2.224(7) Å].

The reason for the small N(1)-Mg-N(2) angle ( $108^\circ$ ) versus the C(15)-Mg-C(16) angle ( $129^\circ$ ) is not immediately obvious. The values obtained for these angles are certainly not those expected a priori from the non-bonded repulsions between two  $\text{N}(\text{CH}_2^-)_3$  molecular fragments compared to the non-bonded repulsions between two  $-\text{CH}_3$  groups. An examination of internuclear distances reveals that the closest carbon-carbon approaches are not those between methylene groups on adjacent quinuclidine molecules (closest approach 3.88 Å) or between the two methyl groups (3.96 Å), but between methyl and methylene carbon atoms (three distances less than 3.7 Å with a closest approach of 3.58 Å). It is conceivable that the methyl groups are forced apart by these interactions. This would also explain the observed difference in Mg-C(15) and Mg-C(16) bond lengths (2.163 Å and 2.224 Å) since the closest methylene carbon approach to C(16) is 3.58 Å while that to C(15) is 3.65 Å. This implies that C(16) is forced away from its preferred co-ordination site by the quinuclidine molecule. The Mg-C(16) distance does appear to be abnormally long. The Mg-C distances in  $\text{C}_2\text{H}_5\text{MgBr} \cdot 2\text{C}_4\text{H}_{10}\text{O}^{10}$ ,  $[\text{C}_2\text{H}_5\text{MgBr} \cdot \text{N}(\text{C}_2\text{H}_5)_3]_2^{11}$ ,  $[\text{C}_2\text{H}_5\text{Mg}_2\text{Cl}_3 \cdot 3\text{C}_4\text{H}_8\text{O}]_2^{12}$ , and  $[(\text{CH}_3)_2\text{N}(\text{CH}_2)_2\text{N}(\text{CH}_3)\text{MgCH}_3]_2^{13}$  are 2.15(2) Å, 2.18(2) Å, 2.14(3) Å, and 2.100(4) Å. For comparison the Mg-C distance found in the electron deficient  $[(\text{CH}_3)_2\text{Mg}]_n^{14,15}$  and  $\text{Mg}[(\text{CH}_3)_4\text{Al}]_2^{16}$  are 2.25(5) Å and 2.21(1) Å. Electronegativity arguments also predict that the magnesium orbitals involved in the more ionic Mg-C bond should have more *s* character, leaving more *p* character in the Mg-N orbitals.

Although the quinuclidine groups apparently interact less than the methyl groups, the tendency of dimethylmagnesium to form a disolvated monomer with quinuclidine can be attributed to the steric factors arising from the relatively large size of the ligand. The quinuclidine groups are not large enough to produce either a monosolvated monomer or a dimer which would utilize methyl bridges and could be compared with  $[\text{C}_2\text{H}_5\text{MgBr} \cdot \text{N}(\text{C}_2\text{H}_5)_3]_2$ . The quinuclidine ligands are too large to permit more ligand groups to bond to the magnesium atom so that the co-ordination number is not increased to five or six as was found for  $(\text{C}_6\text{H}_5\text{C}\equiv\text{C})_2\text{Mg} \cdot 4\text{THF}^5$ .

The quinuclidine molecule can have  $C_{3v-3m}$  symmetry; however, the unco-ordinated molecule crystallizes into a face-centered cubic system with four molecules per unit cell so that the molecule has crystallographic symmetry  $O_h-m3m^*$ . In order to reconcile the trigonal molecular symmetry with the cubic crystal symmetry, it has been suggested that the molecule is rotated spherically about the center of gravity or that there is disorder in the arrangement of the  $\text{CH}_2$  groups in the N- $\text{CH}_2$ - $\text{CH}_2$ -CH plane. In order to clarify the geometry of the quinuclidine molecule, the

\* For a discussion and refs. concerning this molecule see ref. 17.



TABLE 6

DIHEDRAL ANGLE BETWEEN PLANES FOR  $(\text{CH}_3)_2\text{Mg} \cdot 2\text{NC}_7\text{H}_{13}$ 

Planes	Angle ( $^\circ$ )
N(2)-C(8)-C(14) and N(2)-C(9)-C(14)	1.37(81) <sup>a</sup>
N(2)-C(10)-C(14) and N(2)-C(11)-C(14)	3.96(93)
N(2)-C(12)-C(14) and N(2)-C(13)-C(14)	2.55(72)
N(1)-C(1)-C(7) and N(1)-C(2)-C(7)	0.93(66)
N(1)-C(3)-C(7) and N(1)-C(4)-C(7)	0.55(67)
N(1)-C(5)-C(7) and N(1)-C(6)-C(7)	0.66(65)

<sup>a</sup> Numbers in parentheses are estimated standard deviations in the least significant digits.

differences between the dihedral angle formed by the N-X-CH and N-Y-CH planes, where X and Y represent different  $\text{CH}_2$  groups of a bonded  $\text{N-CH}_2\text{-CH}_2\text{-CH}$  group in  $(\text{CH}_3)_2\text{Mg} \cdot 2\text{NC}_7\text{H}_{13}$  were calculated (Table 6). It is interesting to note that the average projected angle of twist about the molecular  $C_3$  axis differs for the two crystallographically independent quinuclidine molecules ( $2.62^\circ$  vs.  $0.71^\circ$ ). Within statistical error, one quinuclidine molecule has  $C_{3v}$  symmetry while the other has  $C_3$ . It would appear that the geometry of the quinuclidine molecules depends on its structural environment. This is consistent with other observations which we have made<sup>18</sup>. The interatomic distances in the quinuclidine molecule can be divided into three groups; bridgehead carbon-ethylene carbon, ethylene carbon-ethylene carbon and nitrogen-carbon. The corresponding average distances are 1.547, 1.569 and 1.501 Å.

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