TABLE III

OBSERVED AND CALCULATED STRUCTURE FACTORS ON A SCALE OF ABSOLUTE VALUES × 10

H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC	H	K	FO	FC
	L = 0			-9	10	351	-350	-3	9	617	-614	-6	9	692	-650	-9	10	349	331	-2	3	1248	1265	-5	6	246	-267	-4	4	507	-509
				-1	11	508	501	-4	9	333	343	-1	10	279	247	-5	11	336	349	-3	3	316	-282	0	7	269	259	0	5	453	-441
-1	2	567	516	-2	11	312	-362	-5	9	537	-572	-2	10	479	-454					-1	4	383	367	-4	7	722	-719	-1	5	1057	1098
-2	3	648	-528	-5	11	396	-392	-7	9	382	369	-5	10	321	-324		L = 4			-2	4	606	-591	-5	7	507	510	-4	5	330	322
-3	3	397	401	-7	11	481	534	0	10	349	337	-6	10	334	350					-3	4	567	-554	-6	7	427	-369	-1	6	408	-390
0	4	882	-834	-8	11	284	-301	-1	10	512	-582	-1	11	389	-323	-1	2	917	-1091	-2	5	326	-351	-1	8	403	372	-4	6	450	-416
-1	4	749	-720	-9	11	341	375	-6	10	760	775	-9	11	388	-428	-2	2	725	816	-3	5	869	833	-2	8	500	-508	-4	7	329	393
-3	4	647	565	-4	12	274	-301	-7	10	537	-587	-10	11	285	149	0	3	903	-916	-5	5	217	241	-4	8	343	337	-2	8	318	255
-1	5	2249	2249	-5	12	366	376	-2	11	505	553	-5	12	486	-438	-1	3	911	957	0	6	235	156	-7	8	369	327	-6	9	542	566
-2	5	486	-457	-8	12	249	-249	-3	11	416	-367					-2	4	1039	-1082	-1	6	403	333	-8	8	484	-484				
-4	5	554	480					-6	11	340	-376		L = 3			-3	4	1157	1207	-3	6	607	590	-3	9	370	355		L = 9		
-5	5	1061	-1003		L = 1			-10	11	314	343					-4	4	220	-203	-4	6	1015	-992	-4	9	341	-322				
0	6	844	782									-1	2	711	869	0	5	292	-264	-2	7	232	-264					-1	2	196	-244
-1	6	827	-788	0	2	321	-312		L = 2			-2	2	449	517	-1	5	755	736	-3	7	582	-566		L = 7			-2	2	166	211
-2	6	325	-292	-1	2	721	-678					0	3	485	-536	-2	5	269	250	-4	7	324	303					0	3	216	225
-3	6	194	212	-1	3	362	308	0	2	740	-666	-1	3	287	362	-3	5	349	353	-5	7	391	403	-1	2	485	558	-1	3	244	249
-4	6	757	-757	-2	3	301	400	-1	3	1134	-1085	-2	3	1067	-1289	-4	5	605	-613	-6	7	377	-380	0	3	309	-316	-2	3	220	182
-5	6	291	290	-3	3	420	-408	-2	3	1799	1696	-1	4	354	-368	-2	6	236	221	-7	7	646	630	-1	3	190	-115	-1	4	348	-370
-1	7	296	-264	0	4	912	853	-3	3	513	421	-2	4	938	1018	-3	6	787	-763	-3	8	742	721	-2	3	481	-503	-2	4	481	-476
-2	7	614	620	-1	4	501	-501	0	4	286	320	0	5	444	407	-5	6	702	704	-5	8	400	-363	-2	4	564	584	-3	4	352	355
-3	7	406	-379	-2	4	912	-823	-1	4	224	207	-3	5	599	-615	-6	6	307	-310	-6	8	475	460	-4	4	306	319	-4	4	502	-508
-4	7	527	559	-3	4	410	421	-2	4	230	204	-1	6	411	-397	-2	7	440	-417	-7	8	591	-559	0	5	701	671	0	5	565	-605
-7	7	661	-658	-2	5	576	557	-3	4	961	-862	-3	6	560	-573	-4	7	968	965	-2	9	413	426	-2	5	325	-283	-2	5	336	364
0	8	414	434	-3	5	160	32	-1	5	1223	-1145	-4	6	813	883	-5	7	713	-691	-3	9	540	-518	-1	6	694	-637	-1	6	624	573
-1	8	295	283	-4	5	225	216	-3	5	198	-274	-6	6	230	257	-6	7	450	438	-4	10	430	420	-2	6	520	440	-2	6	486	-477
-2	8	284	328	-5	5	1284	1191	-4	5	198	-159	0	7	702	754	-1	8	515	-457					-3	6	473	-404	-3	6	326	339
-3	8	579	-594	0	6	220	239	-5	5	424	397	-1	7	349	-326	-2	8	695	686		L = 6			-4	6	378	353	-5	6	273	-331
-4	8	232	251	-1	6	1228	1182	0	6	271	-310	-2	7	401	363	-4	8	528	-565					0	7	321	302	-1	7	292	-222
-5	8	307	-348	-2	6	931	-917	-1	6	475	431	-3	7	543	500	-7	8	628	-626	-1	2	156	202	-5	7	317	-353	-3	9	351	-359
-6	8	359	-350	-3	6	743	699	-2	6	443	420	-5	7	523	-561	-8	8	712	723	-2	2	441	-517	-3	8	321	-251	-5	9	365	-361
-1	9	217	-283	-4	6	410	-372	-4	6	807	696	-6	7	403	391	0	9	258	-245	0	3	614	617	-7	8	378	378				
-2	9	310	-360	-5	6	548	-527	1	7	264	232	-2	8	244	269	-3	9	605	-625	-1	3	750	-804	-3	9	353	390		L = 10		
-4	9	326	380	-1	7	408	-376	-4	7	978	-896	-3	8	642	-693	-4	9	580	620	-2	3	631	669	-5	9	388	370				
-5	9	495	-488	-2	7	377	-360	-5	7	445	416	-5	8	351	354	-7	9	283	313	-2	4	570	561					-1	2	172	-176
-6	9	869	870	-4	7	357	-327	-6	7	356	-342	-6	8	299	-254	-3	10	290	265	-3	4	627	-655		L = 8			-1	3	277	-273
-8	9	247	-271	-5	7	535	515	-7	7	427	375	-7	8	503	544	-4	11	362	-361	0	5	336	283					-2	3	453	467
-9	9	255	267	-7	7	238	212	0	8	616	-590	-2	9	353	-429					-1	5	654	-680	-1	2	206	222	-1	4	224	162
-1	10	444	-390	0	8	241	-246	-1	8	467	414	-3	9	525	582		L = 5			-3	5	471	-439	0	3	250	219	0	5	353	299
-2	10	558	621	-2	8	304	307	-2	8	648	-626	-5	9	241	256					-4	5	227	173	-1	3	248	268	-1	5	642	-646
-5	10	518	492	-3	8	243	255	-3	8	416	328	-8	9	460	-493	0	2	714	748	-1	6	332	303	-2	3	595	-615	-4	6	302	352
-6	10	469	-478	-5	8	199	139	-3	9	309	277	-4	10	370	-370	-1	2	337	-359	-3	6	280	269	-1	4	458	-441	-2	7	316	-213
-7	10	296	-299	-7	8	633	-638	-4	9	261	-262	-6	10	308	-436	-1	3	734	-730	-4	6	389	400	-3	4	327	418	-4	7	350	-316

which two Fe–Fe distances of 2.87 (1) Å subtend angles of 68.9 (2)° at the central tin atom.

Acknowledgments.—We thank both Dr. E. H. Brooks, who supplied the crystals, and Dr. W. A. G. Graham for their continued interest in this work. The financial support of the National Research Council of Canada is gratefully acknowledged.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY AND CHEMICAL ENGINEERING AND
MATERIALS RESEARCH LABORATORY, UNIVERSITY OF ILLINOIS, URBANA, ILLINOIS 61801

Stereochemistry of Polynuclear Compounds of the Main Group Elements. VIII.[1] The Crystal Structure of Bis[2-dimethylaminoethyl(methyl)amino]di(methylmagnesium)[2]

BY V. R. MAGNUSON[3] AND G. D. STUCKY

Received December 16, 1968

The crystal structure of bis[2-dimethylaminoethyl(methyl)amino]di(methylmagnesium), $[(CH_3)_2N(CH_2)_2NCH_3MgCH_3]_2$, has been determined by three-dimensional single-crystal X-ray diffractometer studies. A full-matrix least-squares refinement using 1812 reflections resulted in a final unweighted discrepancy factor of 4.5%. The compound crystallizes as dimers in the triclinic space group $P\bar{1}$, with two molecules in a reduced cell of dimensions a = 7.244 (2) Å, b = 10.387 (3) Å, c = 12.454 (4) Å, α = 82.10 (6)°, β = 88.61 (17)°, and γ = 80.37 (9)°, where $Z = 2$, ρ_{calcd} = 1.04 g/cm³, and ρ_{obsd} = 1.03 g/cm³. The dimers are located on crystallographic centers of inversion and consist of two magnesium atoms bridged by amino groups from two different 2-dimethylaminoethyl(methyl)amino ligands. An amine nitrogen of the chelate and a methyl group occupy the remaining nearly tetrahedral coordination sites on each magnesium atom. The average Mg to bridging N and Mg to terminal N distances are 2.104 ± 0.003 and 2.196 ± 0.003 Å, respectively. Within the four-membered MgNMg'N' ring, the average MgNMg' angle is 88.5 ± 0.1° and the NMgN' angle is 91.5 ± 0.1°.

Introduction

Reagents formed from alkyllithium compounds and various amine derivatives have proven to be extremely useful as intermediates for a wide variety of "metalation" reactions and as catalysts in the telomerization of unsaturated hydrocarbons.[4,5] The corresponding organomagnesium reagents frequently show similar properties although they are usually less reactive and accordingly have been less extensively studied. The stereochemistries of these intermediates are almost without exception unknown, although in the case of

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[1] For Part VII and earlier references, see J. L. Atwood and G. D. Stucky, *J. Am. Chem. Soc.*, **91**, 2538 (1969).

[2] This work was supported by the Advanced Research Projects Agency under Contract SD-131 and the National Science Foundation.

[3] NASA trainee, 1966–1968.

[4] G. G. Eberhardt, *Organometal. Chem. Rev.*, **1**, 491 (1966).

[5] A. W. Langer, Jr., *Trans. N. Y. Acad. Sci.*, 741 (1966).

several group II compounds, Coates[6–8] has formulated the gross structural features on the basis of nuclear magnetic resonance and molecular weight studies.

The reaction product formed by adding N,N,N′-trimethylethylenediamine to a diethyl ether solution of dimethylmagnesium is of particular interest from a stereochemical point of view. First, no crystallographic studies have been made on compounds containing magnesium or aluminum atoms in which each of the bridging electron-deficient groups has been replaced by a three-electron-donor amino group. It is only in the magnesium and aluminum systems that one can hope to make a comparison of the effects on the stereochemistry of one- and three-electron-donor groups. Organoberyllium systems are not amenable to this type of comparison since interligand steric effects predominate in their stereochemistry. Heavy elements of group III such as gallium and indium form dimeric nonelectron-deficient species with three-electron-donor amino groups in the bridging positions but form monomeric alkyls and aryls. In addition, the 2-dimethylaminoethyl(methyl)amino group is unusual in that one nitrogen of the chelating diamine acts as a tertiary amine and the other nitrogen acts as a three-electron-donating bridging group between metal atoms.

The structural properties of several organolithium–amine complexes will be reported in subsequent papers. In this paper the crystal structure of the magnesium derivative bis[2-dimethylaminoethyl(methyl)amino]di(methylmagnesium), $[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$, is reported.

Experimental Section

The compound, $[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$, used in this X-ray investigation was prepared by the dropwise addition of N,N,N′-trimethylethylenediamine to an equimolar amount of dimethylmagnesium in diethyl ether.[8] Sublimation of the white precipitate in a sealed tube at 70° (0.001 mm) over a period of 24–36 hr produced single crystals suitable for X-ray studies. Single crystals of the clear, colorless complex were mounted in thin-walled glass capillaries in a glove box. The crystal used in the analysis was a nearly rectangular box of dimensions 0.50 × 0.23 × 0.27 mm. These dimensions were approximately parallel to the [001], [110], and [$\bar{1}$10] directions. Precession (Mo Kα) photographs showed the crystal system to be triclinic; thus the only possible space groups are P1 and P$\bar{1}$. After alignment of the crystal in a Picker four-circle diffractometer, ten reflections were carefully centered top–bottom and left–right; these reflections formed the basis for a least-squares refinement of cell parameters and the calculation of an orientation matrix which was carried out using program B-101 of Gvildys and Mueller.[9] The takeoff angle used for the determination of the lattice parameters was 1.25°. The reflections were in the range of 62–76° in 2θ and were centered on the Kα_1 component of the incident X-ray beam. The unit cell constants and their corresponding standard deviations as determined by the least-squares refinement for the nonreduced cell are: $a = 10.387$ (3) Å,[10] $b = 12.454$ (12) Å, $c = 7.244$ (6) Å, $\alpha = 91.387$ (5)°, $\beta = 80.375$ (27)°, $\gamma = 97.896$ (10)°, (at 23°, λ(Cu Kα_1) 1.540462 Å).[11] The lattice constants of the reduced primitive triclinic cell derived by a Dalaunay reduction from the nonreduced cell are: $a = 7.244$ (6) Å, $b = 10.387$ (9) Å, $c = 12.454$ (12) Å, $\alpha = 82.104$ (18)°, $\beta = 88.613$ (51)°, $\gamma = 80.375$ (27)°.[12] All results reported here are given in terms of the nonreduced triclinic cell which was used for the structural determination. The reduced primitive cell (unprimed axes) is related to the nonreduced cell (primed axes) by the transformation: $a = -c'$, $b = -a'$, $c = b'$. The Delaunay reduction failed to reveal the presence of any additional lattice symmetry. The measured density of 1.03 ± 0.02 g/cm³ obtained by the flotation method in a mixture of bromobenzene and cyclohexane agrees satisfactorily with the density of 1.04 g/cm³ calculated for two molecules in the unit cell. Thus, no implication of molecular symmetry can be made.

Diffraction data were collected manually on a Picker four-circle X-ray diffractometer using Cu Kα radiation. Cu Kα radiation was selected on the basis of the satisfactorily low absorption coefficient ($\mu = 10.8$ cm^{-1}). The crystal was mounted so that the c^* axis of the nonreduced cell was parallel to the ϕ axis of the four-circle diffractometer and a maximum variation of 1.5% in the integrated intensity of the 002 reflection was observed with respect to rotation around the ϕ axis at $\chi = 90°$. The mosaicity of the crystal was indicated to be satisfactorily low on the basis of narrow source–open counter ω scans through several reflections. A takeoff angle of 1.0° was found to provide 75% of maximum intensity as a function of takeoff angle. Incident beam and diffracted beam collimators of 1.0 and 2.0 mm, respectively, were used in data collection. A 0.0005-in. nickel foil filter was placed between the crystal and the scintillation counter which was mounted 21 cm from the crystal.

All data were collected by the θ–2θ scan technique at a scan rate of 1.0°/min. Reflections with $2\theta \leq 30°$ were scanned for 50 sec while reflections with $2\theta \geq 30°$ were scanned for 60 sec. Stationary counter–stationary crystal background counts of 20 sec were taken at each end of the scan. All scans were recorded on a chart recorder, and, since the diffractometer was operated manually, all peaks could be checked for centering, peak shape, and overlap. The pulse height analyzer was set for approximately an 85% window, centered on the Cu Kα peak. In order to ensure crystal and diffractometer stability, a standard reflection was measured periodically. No systematic drift was observed.

One full form of data to $2\theta_{max} = 100.0°$ was taken. A total of 1821 reflections were measured within this sphere. Background and Lorentz polarization corrections were carried out by the program ACAC according to Guggenberger and Prewitt.[13] Background corrections were made by assuming that the background can be approximated by a straight line between the two points. Of the 1821 independent reflections, 365 were found to be unobserved ($I_{obsd} < 3\sigma_c$ (I) where $\sigma_c = [T_c + 0.25(t_c/t_b)^2 \cdot (B_1 + B_2)]^{1/2}$, T_c is the total integrated counts, t_c/t_b is the ratio of the time spent counting the peak intensity to the time spent counting the background intensities, and B_1 and B_2 are background counts. These reflections were each assigned an intensity equal to the standard deviation of the background intensity in their respective locations. Nine peaks for which $I_{obsd} < (I_b - 3\sigma_c)$ were rejected. It was found that the range of absorption corrections necessary varied from 1.23 to 1.35 and no absorption or secondary extinction corrections were made.

Solution and Refinement of the Structure

In order to determine whether the space group was P$\bar{1}$ or P1, a statistical test (Table I) was performed on

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(6) G. E. Coates and S. I. E. Green, *J. Chem. Soc.*, 3340 (1962).

(7) G. E. Coates and D. Ridley, *ibid.*, 1870 (1965).

(8) G. E. Coates and J. A. Heslop, *ibid.*, *A*, 26 (1966).

(9) J. Gvildys and M. Mueller, "B-101, Orientation and Angle Setting Program," Argonne National Laboratory, Lemont, Ill.

(10) Numbers in parentheses here and in succeeding discussions are estimated standard deviations in the least significant digits. We have observed in a number of cases that measurement of unit cell constants with two different wavelengths (Cu Kα_1 and Mo Kα_1) and/or two different crystals leads to values which typically differ by three to four standard deviations, suggesting that this is probably a valid estimation of the accuracy of the unit cell parameters for this compound.

(11) J. A. Bearden, *Rev. Mod. Phys.*, **39**, 78 (1967).

(12) L. V. Azaroff and M. J. Buerger, "The Powder Method in X-Ray Crystallography," McGraw-Hill Book Co., Inc., New York, N. Y., 1958, pp 134–136.

(13) L. J. Guggenberger and C. Prewitt, "Program ACAC," E. I. du Pont de Nemours and Co., Wilmington, Del.

TABLE I

STATISTICAL TEST RESULTS FOR $[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$

Quantity	Calcd values	Theoret values	
		Centric	Acentric
$\langle E \rangle_{av}$	0.796	0.798	0.886
$\langle E^2 \rangle_{av}$	1.000	1.000	1.000
$\langle E^2 - 1 \rangle_{av}$	0.974	0.968	0.736
$P^a > 1$	31.41	32.00	37.00
$P > 2$	4.39	5.00	1.80
$P > 3$	0.49	0.30	0.01

[a] Per cent of $|E|$'s.

the full set of data using the program FAME of Dewar and Stone.[14] These results strongly support the centric structure, and it was assumed that the correct space group was centrosymmetric $P\bar{1}$ rather than P1. The positions of two independent magnesium atoms were found from a three-dimensional Patterson map.[15] Since in the space group $P\bar{1}$ each of these Mg atoms lies within 1.5 Å of a center of symmetry, the emphasis shifted from finding one complete dimer to finding half of each of two nonsymmetry-related dimers. The dimers, which each must have $\bar{1}$ symmetry, are located at (0, 0, 0) and ($^1/_2$, $^1/_2$, $^1/_2$); hereafter they will be referred to as dimer I and II, respectively. A three-dimensional electron density calculation with phases based on the Mg positions led to the assignment of parameters of eight N and C atoms. Two additional Fourier syntheses resulted in the determination of the entire set of 18 nonhydrogen atoms.

TABLE II

FINAL POSITIONAL AND THERMAL PARAMETERS FOR NONHYDROGEN ATOMS[a]

Atom	x	y	z	β_{11}[b]	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mg	0.0448 (1)[c]	−0.0892 (1)	−0.1185 (2)	0.0106 (2)	0.0060 (1)	0.0224 (3)	0.0013 (1)	−0.0041 (2)	−0.0005 (1)
N(1)	0.0630 (3)	−0.0379 (2)	0.1557 (4)	0.0126 (4)	0.0074 (3)	0.0239 (3)	0.0018 (3)	−0.0062 (5)	0.0030 (4)
N(2)	0.2577 (3)	−0.0370 (3)	−0.1731 (5)	0.0110 (4)	0.0084 (3)	0.0277 (9)	0.0017 (3)	−0.0019 (5)	−0.0002 (4)
C(1)	−0.0152 (4)	−0.2477 (3)	−0.2047 (6)	0.0153 (6)	0.0081 (4)	0.0375 (14)	0.0007 (4)	−0.0042 (7)	−0.0016 (6)
C(2)	0.0141 (5)	−0.1151 (3)	0.3069 (6)	0.0208 (7)	0.0102 (4)	0.0275 (12)	0.0039 (4)	−0.0048 (9)	0.0022 (6)
C(3)	0.1996 (4)	0.0034 (4)	0.1646 (6)	0.0156 (6)	0.0109 (4)	0.0279 (13)	0.0026 (4)	−0.0087 (8)	−0.0029 (6)
C(4)	0.2724 (4)	0.0446 (4)	−0.0229 (7)	0.0122 (6)	0.0104 (4)	0.0423 (16)	0.0004 (4)	−0.0077 (8)	−0.0040 (7)
C(5)	0.3227 (4)	−0.1315 (4)	−0.1466 (7)	0.0154 (6)	0.0112 (4)	0.0511 (18)	0.0042 (5)	−0.0066 (9)	−0.0020 (7)
C(6)	0.3187 (4)	0.0113 (4)	−0.3541 (7)	0.0154 (6)	0.0138 (5)	0.0424 (16)	0.0010 (5)	−0.0002 (9)	0.0010 (8)
Mg	0.6101 (1)	0.4379 (1)	0.5060 (2)	0.0109 (2)	0.0059 (1)	0.0245 (4)	0.0018 (1)	−0.0047 (2)	0.0001 (2)
N(1)	0.4261 (3)	0.4048 (2)	0.4124 (4)	0.0122 (4)	0.0059 (2)	0.0238 (9)	0.0009 (3)	−0.0042 (5)	−0.0013 (4)
N(2)	0.2433 (3)	0.4785 (2)	0.7133 (5)	0.0113 (4)	0.0079 (3)	0.0283 (9)	0.0014 (3)	−0.0037 (5)	−0.0008 (4)
C(1)	0.6692 (5)	0.3157 (3)	0.6556 (6)	0.0221 (8)	0.0099 (4)	0.0341 (14)	0.0055 (5)	−0.0078 (8)	0.0010 (6)
C(2)	0.4296 (4)	0.3667 (3)	0.2205 (6)	0.0143 (6)	0.0105 (4)	0.0330 (14)	0.0012 (4)	−0.0069 (7)	−0.0021 (6)
C(3)	0.3372 (4)	0.3259 (3)	0.5354 (6)	0.0139 (6)	0.0066 (3)	0.0339 (13)	0.0007 (4)	−0.0025 (7)	−0.0004 (6)
C(4)	0.2972 (4)	0.3742 (3)	0.7256 (6)	0.0129 (5)	0.0073 (4)	0.0324 (13)	0.0005 (4)	−0.0016 (7)	0.0011 (6)
C(5)	0.2163 (4)	0.5268 (4)	0.9008 (6)	0.0147 (6)	0.0114 (4)	0.0315 (13)	0.0014 (4)	−0.0001 (7)	−0.0014 (6)
C(6)	0.1187 (4)	0.4600 (3)	0.6379 (7)	0.0121 (6)	0.0116 (4)	0.0474 (16)	0.0019 (4)	−0.0071 (8)	−0.0028 (7)

[a] The first nine atoms constitute the asymmetric half of dimer I; the last nine, that of dimer II. [b] The form of the anisotropic thermal ellipsoid is given by $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}h)]$. [c] Numbers in parentheses here and in succeeding discussions are estimated standard deviations in the least significant digits.

The structure was refined by using a full-matrix least-squares procedure.[16] In the least-squares refinement, the function minimized was $\Sigma w(|F_o| - |F_c|)^2$ where $|F_o|$ and $|F_c|$ are the observed and calculated structure amplitudes and w is the weight defined as $1/\sigma^2(F_o)$. Weights were determined from the relation

$$\sigma(F) = 0.5K(F/I)[T_c + 0.25(t_c/t_b)^2(B_1 + B_2) + (kI)^2]^{1/2}$$

where K is the scale factor, F is the observed structure factor, I is the observed intensity, $K = 0.02$, and T_c, t_c, t_b, B_1, and B_2 are defined above. The residuals R_1 and R_2 are defined as $R_1 = \Sigma(||F_o| - |F_c||)/\Sigma(|F_o|$ and $R_2 = (\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2)^{1/2}$. Atomic scattering factors for all atoms were those based on the Hartree–Fock–Slater calculations of Hansen, Herman, Lea, and Skillman.[17]

Several cycles of least-squares refinement on the positional parameters of the 18 symmetry-independent Mg, N, and C atoms resulted in an R_1 of 22% for observed reflections only. Subsequent isotropic refinement of the thermal parameters as well as additional positional parameter refinement yielded R_1 values of 14.2% including both observed and unobserved reflections and 12.0% including observed reflections only. Anisotropic refinement of the thermal parameters gave an R_1 value of 9.6% and an R_2 value of 12.6%, both including observed data only. By using a difference Fourier map, the positions of all of the hydrogen atoms were located and included in all subsequent refinements. The chemical reasonableness of the hydrogen atom positions included was confirmed by the program Hygen,[18] which generates atomic positions based on known molecular angles and distances. When the 32 hydrogen atoms were included in the refinement (see below), R_1, observed data only, dropped from 9.6 to 8.6%.

The structure was refined to convergence with all nonhydrogen atoms anisotropic and all hydrogen atom temperature factors fixed at 4.0 Å^2. Final positional and anisotropic thermal parameters for all nonhydrogen atoms are given in Table II. Refined positional parameters for the hydrogen atoms are given in Table III. Final R factors for the refined structure for both un-

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(15) W. G. Sly, D. P. Shoemaker, and J. H. van den Hende, "ERFR-3, a Three Dimensional Fourier Program," private communication, 1964.

(16) W. R. Busing, K. O. Martin, and H. A. Levy, ORFLS, "A Fortran Crystallographic Least-Squares Program," Report No. ORNL-TM-305, U. S. Atomic Energy Commission, Oak Ridge National Laboratory, Oak Ridge, Tenn. 1962.

(17) H. P. Hansen, F. Herman, J. D. Lea, and S. Skillman, *Acta Cryst.*, **17**, 1040 (1964).

(18) F. Ross and G. Stucky, private communication, 1967.

Table III

Final Hydrogen Atom Positions[a]

Atom	x	y	z
C(1)H(1)	−0.1034 (36)	−0.2611 (28)	−0.2190 (48)
C(1)H(2)	0.0341 (35)	−0.2628 (28)	−0.3120 (48)
C(1)H(3)	−0.0065 (35)	−0.2940 (27)	−0.1097 (48)
C(2)H(1)	−0.0772 (36)	−0.1464 (29)	0.2941 (50)
C(2)H(2)	0.0647 (36)	−0.1768 (29)	0.2980 (49)
C(2)H(3)	0.0155 (35)	−0.0830 (29)	0.4300 (50)
C(3)H(1)	0.2173 (35)	0.0609 (30)	−0.2533 (50)
C(3)H(2)	0.2595 (35)	−0.0540 (30)	0.1952 (49)
C(4)H(1)	0.3713 (35)	0.0681 (28)	−0.0283 (48)
C(4)H(2)	0.2372 (36)	−0.1100 (29)	−0.0554 (50)
C(5)H(1)	0.2790 (37)	−0.1676 (29)	−0.0287 (52)
C(5)H(2)	0.3062 (36)	−0.1835 (28)	−0.2542 (51)
C(5)H(3)	0.4167 (36)	−0.1062 (29)	−0.1466 (50)
C(6)H(1)	0.2731 (35)	0.0734 (30)	−0.3804 (49)
C(6)H(2)	0.4144 (37)	0.0382 (29)	−0.3606 (49)
C(6)H(3)	0.3043 (35)	−0.0464 (29)	−0.4505 (49)
C(1)H(1)	0.7546 (34)	0.3335 (27)	0.6801 (47)
C(1)H(2)	0.6078 (36)	0.3026 (27)	0.7699 (50)
C(1)H(3)	0.6593 (34)	0.2468 (28)	0.6041 (49)
C(2)H(1)	0.4799 (36)	0.4242 (30)	0.1276 (49)
C(2)H(2)	0.4679 (36)	0.3010 (30)	0.1947 (49)
C(2)H(3)	0.3462 (37)	0.3526 (29)	0.1854 (49)
C(3)H(1)	0.3845 (35)	0.2626 (29)	0.5506 (48)
C(3)H(2)	0.2557 (36)	0.2984 (29)	0.4784 (51)
C(4)H(1)	0.2312 (35)	0.3250 (28)	0.8022 (47)
C(4)H(2)	0.3759 (36)	0.3915 (29)	0.7878 (49)
C(5)H(1)	0.2954 (37)	0.5375 (28)	0.9566 (49)
C(5)H(2)	0.1830 (35)	0.5974 (30)	0.8891 (50)
C(5)H(3)	0.1430 (35)	0.4767 (30)	0.9900 (50)
C(6)H(1)	0.1288 (34)	0.4326 (29)	0.5122 (50)
C(6)H(2)	0.0502 (37)	0.4087 (30)	0.7163 (51)
C(6)H(3)	0.0827 (35)	0.5281 (30)	0.6329 (50)

[a] The first 16 atoms constitute the asymmetric half of dimer I; the last 16, that of dimer II.

observed and observed data were $R_1 = 6.4$ and $R_2 = 6.5\%$. Final R factors excluding unobserved reflections were $R_1 = 4.5$ and $R_2 = 5.0\%$. The error of an observation of unit weight, which is given by $[\Sigma w(|F_o| - |F_c|)^2/(\mathrm{NO} - \mathrm{NV})]^{1/2}$ where NO is the number of observations and NV is the number of variables, was 1.84. It was noted that larger reflections probably received too small a weight based on the weighting scheme described above with $k = 0.02$. On this basis, it was felt that for Cu data unit weights would have been just as acceptable. The final calculated and observed structure factors are listed in Table IV.

Discussion of the Structure

$[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$ crystallizes in the space group P$\bar{1}$ with two molecules per unit cell. The crystalline complex consists of two crystallographically independent dimers of nearly identical molecular configuration. The basic structural feature of each molecular unit is a centrosymmetric four-membered heterocyclic ring system consisting of two Mg atoms and two amino nitrogen atoms of the parent ligands (Figures 1 and 2). In addition to the planar four-membered heterocyclic ring, there is a puckered five-membered ring system made up of a Mg atom and two N and two C atoms from the chelating diamine. As seen in Figures 1 and 2, the local symmetry of each Mg atom is tetrahedral with the Mg atom coordinated to one terminal methyl group and the second nitrogen of the chelating diamine as well as to the two bridging nitrogen atoms. The packing of the two independent dimers in the cell is shown in Figure 3.

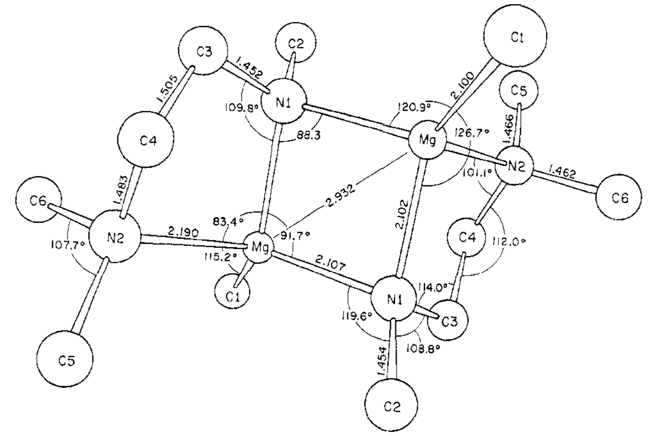

Figure 1.—Molecular configuration of $[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$ for the dimer at the origin (dimer I). Hydrogen atoms are omitted for clarity.

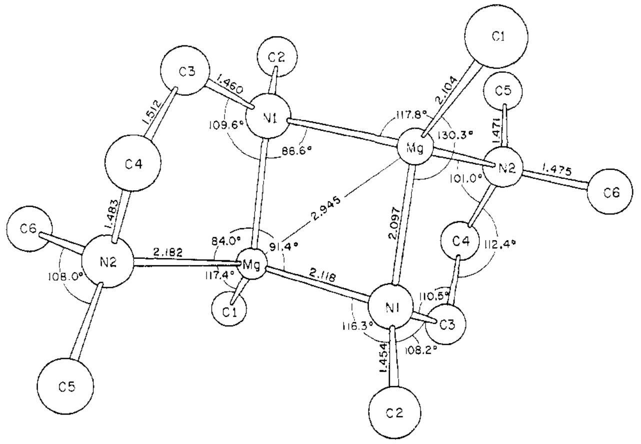

Figure 2.—Molecular configuration of $[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$ for the dimer at $(^1/_2, ^1/_2, ^1/_2)$ (dimer II). Hydrogen atoms are omitted for clarity.

The terminal Mg–C(1) bond lengths of 2.100 (4) Å and 2.104 (4) Å for dimers I and II, respectively, are shorter than those reported for $C_2H_5MgBr \cdot N(C_2H_5)_3$,[19] $C_6H_5MgBr \cdot 2C_4H_{10}O$,[20] and $C_2H_5MgBr \cdot 2C_4H_{10}O$[21] which are 2.18 (2), 2.2 (1), and 2.15 (2) Å, respectively.[22] The sum of Pauling's tetrahedral covalent radii for Mg and C is 2.14 Å, which is also slightly longer.

The average Mg–N(1)–Mg′[23] and N(1)–Mg–N(1)′ angles are 88.30 (12) and 91.70 (12)°, respectively. The Mg–N(1)–Mg′ bridging angle agrees quite well with the average bridging angles in organometallic complexes containing NR_2 groups in the bridging position. Structures containing various bridging groups

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(19) J. Toney and G. Stucky, *Chem. Commun.*, 1168 (1967).

(20) G. Stucky and R. E. Rundle, *J. Am. Chem. Soc.*, **86**, 4825 (1964).

(21) L. J. Guggenberger and R. E. Rundle, *ibid.*, **86**, 5344 (1964).

(22) All bond lengths and angles were calculated using the Busing–Levy Program ORFFE: W. R. Busing, K. O. Martin, and H. A. Levy, "ORFFE, a Fortran Crystallographic Function and Error Program," Report No. ORNL-TM-306, U. S. Atomic Energy Commission, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1964.

(23) An atom designated with a prime is related by a center of symmetry to the unprimed atom.

TABLE IV

OBSERVED AND CALCULATED STRUCTURE AMPLITUDES[a]

H	K	OBS	CAL
******L= 0*******			
1	0	6.0	5.9
2	0	22.3	22.7
3	0	62.7	65.5
4	0	16.0	15.6
5	0	11.0	11.1
6	0	14.6	14.0
7	0	1.1	.9*
8	0	2.4	1.9
9	0	4.2	3.8
0	1	1.8	1.9
1	1	49.7	54.3
1	-1	54.8	58.7
2	1	10.2	9.7
3	1	28.3	28.5
3	-1	27.8	27.3
4	1	46.6	47.4
4	-1	19.9	19.6
5	1	13.9	14.7
5	-1	14.3	14.7
6	1	20.3	20.5
6	-1	3.7	4.4
7	1	1.2	1.8*
7	-1	7.1	7.1
8	1	7.9	7.1
8	-1	13.1	12.7
9	1	3.6	3.1
9	-1	6.5	6.8
10	-1	4.6	4.5
0	2	75.5	85.7
1	2	37.2	38.6
1	-2	24.4	23.5
2	2	29.5	29.7
2	-2	34.9	35.0
3	2	12.3	11.5
3	-2	32.1	32.4
4	2	26.7	27.1
4	-2	13.8	14.3
5	2	26.5	25.4
5	-2	16.7	17.1
6	2	22.2	22.6
6	-2	19.3	19.3
7	2	10.3	10.3
7	-2	19.5	19.8
8	-2	1.1	1.2*
8	2	5.7	5.1
9	-2	1.7	7.8*
9	2	5.0	4.8
10	-2	6.3	6.7
0	3	31.3	30.7
1	3	19.1	19.8
1	-3	26.1	26.4
2	3	3.5	3.4
2	-3	27.3	27.2
3	3	32.1	31.3
3	-3	3.5	3.2
4	3	21.4	19.9
4	-3	16.9	16.5
5	3	1.0	.1*
5	-3	20.6	19.7
6	3	28.1	27.4
6	-3	25.8	26.0
7	3	6.0	5.8
7	-3	3.2	2.6
8	3	1.1	1.2*
8	-3	16.8	16.6
9	3	4.3	4.1
9	-3	5.3	5.3
10	-3	1.0	.4*
0	4	3.2	2.9
1	4	13.7	12.5
1	-4	22.1	22.0
2	4	5.5	5.3
2	-4	61.7	62.7
3	4	5.2	5.0
3	-4	27.7	27.1
4	4	21.2	20.3
4	-4	6.8	7.3
5	4	18.4	17.6
5	-4	20.0	20.0
6	4	4.4	3.2
6	-4	2.9	2.5
7	4	3.6	2.7
7	-4	15.7	15.4
8	4	8.2	8.1
8	-4	7.8	7.8
9	4	4.1	4.5
9	-4	3.0	2.6
0	5	9.4	9.2
1	5	17.4	17.2
1	-5	38.4	37.3
2	5	15.9	17.6
2	-5	7.4	6.9
3	5	3.1	3.0
3	-5	30.8	30.5
4	5	10.6	10.2
4	-5	11.1	11.3
5	5	6.3	5.6
5	-5	5.3	5.0
6	5	5.3	6.0
6	-5	21.4	21.4
7	5	4.5	4.1
7	-5	11.8	10.9
8	5	2.1	1.3
8	-5	5.9	6.7
9	-5	1.1	.0*
0	6	10.9	10.7
1	6	20.9	21.9
1	-6	3.0	2.6
2	6	7.7	7.5
2	-6	67.5	68.7
3	6	17.5	17.4
4	6	3.8	3.8
5	6	9.2	8.8
5	-6	20.0	19.6
6	6	2.5	1.6
6	-6	11.5	11.1
7	6	5.0	5.2
7	-6	1.1	.1*
8	6	4.6	4.9
8	-6	3.7	4.0
9	-6	4.0	3.8
0	7	4.1	3.9
1	7	11.0	10.4
1	-7	20.1	19.9
2	7	10.5	10.1
2	-7	10.1	10.3
3	7	3.1	2.4
3	-7	3.3	3.0
4	7	19.8	19.6
4	-7	8.8	8.6
5	7	7.5	7.2
5	-7	5.4	5.0
6	7	1.1	.8*
6	-7	2.9	2.9
7	7	3.6	3.7
7	-7	5.2	5.5
0	8	4.5	4.8
1	8	9.3	8.9
2	8	1.1	1.2*
2	-8	2.4	1.8
3	8	10.7	10.6
3	-8	5.8	5.4
4	8	8.8	8.6
4	-8	13.5	13.2
5	8	6.4	6.5
5	-8	4.5	4.3
6	8	1.0	.4*
6	-8	12.6	12.4
7	-8	11.0	11.5
8	-8	1.0	.2*
0	9	9.6	9.4
1	9	6.3	5.8
1	-9	4.1	3.9
2	9	2.4	2.8
2	-9	5.0	4.9
3	9	1.1	.8*
3	-9	14.3	14.0
4	9	10.9	11.6
4	-9	4.0	3.5
5	9	1.2	2.0*
5	-9	10.8	10.4
6	-9	10.0	10.1
7	-9	1.2	1.9*
0	10	1.1	1.8*
1	10	10.2	9.9
1	-10	2.5	2.4
2	10	2.3	2.3
2	-10	11.0	11.1
4	10	1.0	1.1*
4	-10	6.2	6.2
5	-10	2.5	2.0
6	-10	5.1	5.0
0	11	3.0	2.5
1	11	1.1	1.1*
2	-11	1.1	.5*
2	11	9.9	9.7
4	-11	3.9	4.0
5	-11	3.4	3.3
0	12	1.1	1.3*
1	-12	2.3	2.0
2	-12	8.5	8.8
3	-12	4.3	4.1
******L= 1*******			
0	0	4.8	4.8
1	0	71.7	85.1
2	0	6.4	6.5
3	0	44.0	46.5
4	0	64.0	65.1
5	0	3.6	3.0
6	0	5.5	5.4
7	0	8.4	7.9
8	0	6.3	5.9
9	0	2.4	2.2
10	0	4.5	4.7
0	1	57.9	61.5
1	1	4.7	4.1
-1	1	9.7	9.0
1	-1	21.6	21.4
2	-1	17.9	17.9
-2	1	18.1	17.3
2	1	51.5	52.4
-3	1	6.3	6.3
3	-1	25.1	25.5
3	1	37.0	36.2
4	1	4.3	2.4
-4	1	29.0	27.9
4	-1	6.5	5.9
5	1	4.4	4.0
-5	1	7.3	7.9
5	-1	11.1	11.6
6	1	17.9	17.9
-6	1	7.6	7.7
6	-1	21.9	21.2
7	1	10.1	8.5
-7	1	13.3	13.2
7	-1	1.1	1.1*
8	1	9.7	9.4
8	-1	7.7	7.2
-8	1	1.1	.6*
9	1	7.0	7.7
-9	1	8.1	8.3
9	-1	1.3	7.6*
10	-1	4.1	4.1
0	2	1.2	1.9
1	-2	46.1	47.4
1	2	44.4	45.5
-1	2	22.3	21.8
2	-2	53.0	52.8
-2	2	19.5	18.9
2	2	.8	.7*
3	2	11.6	11.5
-3	2	20.3	20.6
3	-2	9.4	10.0
4	2	38.7	36.7
4	-2	20.5	20.2
-4	2	3.3	3.6
5	2	16.6	15.9
-5	2	5.5	5.3
5	-2	17.1	16.9
6	2	7.5	8.0
-6	2	12.5	12.5
6	-2	12.0	11.5
7	2	4.6	4.5
7	-2	15.0	15.4
-7	2	1.1	.4*
8	2	2.4	1.2
-8	2	11.3	10.5
8	-2	11.3	11.2
9	2	9.5	9.1
-9	2	4.0	3.9
9	-2	1.1	1.3*
10	-2	1.1	1.5*
0	3	2.7	1.9
1	-3	39.5	38.4
-1	3	51.2	51.1
1	3	20.4	19.6
2	3	27.7	26.8
2	-3	25.3	25.6
-2	3	.9	1.0*
3	3	6.1	6.7
3	-3	33.7	33.8
-3	3	.9	.6*
4	-3	7.9	7.4
4	3	22.1	23.4
-4	3	13.9	14.0
5	-3	11.1	10.4
5	3	10.8	10.4
-5	3	7.8	6.8
6	-3	21.3	21.6
6	3	12.5	11.5
-6	3	1.0	.4*
-7	3	7.7	7.8
7	-3	16.0	16.2
7	3	16.3	16.4
-8	3	7.7	7.6
8	-3	5.5	5.9
8	3	13.8	12.9
9	-3	5.1	6.9
9	3	2.0	1.5
-9	3	4.2	4.6
10	-3	10.6	10.9
0	4	4.5	4.2
1	4	5.6	5.7
-1	4	18.5	18.2
1	-4	12.1	12.3
2	4	24.1	24.3
-2	4	13.0	13.0
2	-4	.9	.1*
-3	4	15.3	15.8
3	-4	42.1	41.2
3	4	13.1	13.1
4	4	6.8	7.1
-4	4	6.2	6.6
4	-4	14.0	12.4
5	-4	10.9	10.9
5	4	6.5	6.9
-5	4	1.0	.6*
6	-4	35.2	34.7
6	4	10.6	10.7
-6	4	7.0	7.1
7	-4	6.5	6.5
7	4	7.8	7.7
-7	4	12.1	12.2
8	-4	3.1	2.7
-8	4	6.1	5.5
8	4	1.1	.3*
9	4	5.4	5.1
9	-4	1.1	1.1*
-9	4	1.0	.4*
10	-4	4.2	3.9
0	5	17.6	17.3
1	5	20.1	19.8
-1	5	5.7	5.2
1	-5	10.5	11.2
2	-5	52.4	51.5
-2	5	26.0	24.9
2	5	.9	.5*
3	5	24.1	23.9
-3	5	1.6	1.7
3	-5	3.0	2.5
4	-5	21.9	22.0
-4	5	17.1	16.9
4	5	1.0	1.9*
-5	5	7.5	8.6
5	-5	28.2	27.7
5	5	12.2	12.0
-6	5	12.2	12.5
6	-5	3.8	3.4
6	5	10.5	10.0
7	-5	6.3	6.3
-7	5	3.1	5.3
7	5	1.3	1.3*
8	-5	8.3	8.3
-8	5	4.8	5.1
8	5	1.2	2.0*
-9	5	3.5	3.9
9	-5	1.1	1.2*
0	6	4.2	4.1
1	6	9.2	9.5
-1	6	14.8	13.8
1	-6	15.9	15.7
-2	6	15.2	15.1
2	6	15.8	16.0
2	-6	1.0	1.3*
3	-6	37.1	36.8
-3	6	7.0	8.1
3	6	1.0	.7*
-4	6	3.1	2.9
4	-6	6.7	6.8
4	6	17.4	14.1
5	-6	18.7	17.7
-5	6	6.2	5.9
5	6	1.1	.7*
-6	6	2.5	2.1
6	-6	24.8	25.3
-7	6	7.9	8.5
7	-6	7.4	6.9
7	6	7.5	7.4
-8	6	3.5	3.7
8	-6	6.4	6.2
8	6	1.1	1.4*
-9	6	2.9	2.8
9	-6	1.4	3.4*
0	7	2.8	1.7
1	7	4.4	3.8
-1	7	15.9	16.2
1	-7	8.6	7.6
2	7	4.3	4.2
2	-7	32.9	32.5
-2	7	1.0	.5*
-3	7	3.3	4.3
3	-7	2.0	2.9
3	7	8.5	7.9
4	-7	8.3	9.3
-4	7	4.0	3.4
5	-7	12.5	13.2
5	7	14.5	13.9
-5	7	1.1	.4*
-6	7	13.4	12.3
6	-7	5.8	6.6
6	7	2.5	2.6
7	-7	1.3	2.6*
-7	7	1.2	.2*
8	-7	3.7	5.3
-8	7	1.2	2.5*
9	-7	2.5	3.0
0	8	11.4	11.7
-1	8	2.4	3.2
1	-8	11.4	11.5
1	8	1.1	.4*
-2	8	21.7	22.0
2	-8	13.5	13.5
2	8	1.1	.6*
-3	8	12.0	12.4
3	-8	6.5	6.9
3	8	3.4	4.5
-4	8	12.9	12.9
4	-8	2.4	2.1
4	8	11.4	11.4
-5	8	13.7	12.8
5	-8	5.5	5.3
-6	8	8.3	8.2
6	8	3.8	4.1
6	-8	1.1	.1*
-7	8	4.1	3.2
7	-8	5.7	5.9
8	-8	7.3	7.7
-8	8	1.0	.7*
0	9	5.3	5.0
-1	9	11.6	11.8
1	-9	6.2	4.1
1	9	12.8	11.6
2	-9	2.8	2.3
2	9	3.9	4.1
-2	9	8.5	9.0
3	9	2.5	1.6
-3	9	8.8	8.7
3	-9	3.6	3.8
-4	9	8.3	9.0
4	-9	9.2	9.2
4	9	4.7	5.0
5	-9	5.6	5.2
5	9	6.8	6.8
-5	9	1.2	1.0*
6	-9	6.7	6.2
-6	9	1.1	.9*
7	-9	7.5	8.3
-7	9	1.2	2.6*
0	10	5.5	5.3
-1	10	10.7	10.2
1	-10	1.1	.6*
1	10	1.1	1.2*
-2	10	9.2	9.0
2	-10	2.7	2.6
2	10	12.6	12.8
3	-10	6.9	6.2
3	10	4.4	4.3
-3	10	1.4	3.4*
-4	10	11.2	11.7
4	-10	1.1	.4*
4	10	.9	.0*
-5	10	5.6	5.6
5	-10	8.6	8.3
-6	10	2.2	2.4
6	-10	7.1	7.3
7	-10	1.2	.8*
0	11	6.3	6.4
1	11	11.4	11.0
1	-11	4.9	4.9
-1	11	1.1	.2*
-2	11	8.2	8.1
2	-11	9.1	9.3
2	11	1.1	2.9*
-3	11	4.4	4.2
3	11	10.9	10.7
3	-11	1.1	.7*
4	-11	8.3	7.8
-4	11	1.1	1.2*
5	-11	3.3	3.8
-5	11	1.1	1.3*
-1	12	10.2	10.4
1	-12	5.0	6.2
-2	12	1.3	2.8*
2	-12	1.2	1.9*
3	-12	8.9	9.0
******L= 2*******			
0	0	21.4	21.1
1	0	19.3	19.4
2	0	16.5	16.1
2	0	16.1	16.1
3	0	3.9	3.6
4	0	25.2	23.7
4	0	20.8	23.7
5	0	21.7	20.8
6	0	6.4	6.9
6	0	8.8	6.9
7	0	2.8	3.3
8	0	13.1	11.9
8	0	13.1	11.9
9	0	4.2	3.6
10	0	4.1	4.4
0	1	52.3	57.7
1	1	9.4	9.8
1	-1	10.7	10.8
-1	1	5.4	5.7
-2	1	21.1	20.3
2	-1	1.7	.9
2	1	4.2	4.6
3	1	17.4	17.1
-3	1	30.0	31.5
3	-1	14.5	14.9
4	-1	46.8	46.9
4	1	24.1	23.5
-5	1	22.7	21.4
5	1	.9	1.0*
5	-1	.9	12.9*
6	1	8.5	7.8
6	-1	2.4	4.0
-6	1	6.1	6.2
7	1	2.6	2.8
7	-1	6.0	5.7
-7	1	6.9	6.5
8	1	9.1	8.5
9	-1	2.3	1.0
-8	1	8.6	8.0
9	1	10.4	10.3
9	-1	13.4	13.9
-9	1	3.5	3.6
10	-1	3.8	3.2
0	2	4.4	4.0
-1	2	9.9	10.6
1	-2	15.1	15.8
1	2	45.3	46.8
2	2	6.0	5.6
-2	2	9.9	10.5
2	-2	19.1	18.7
3	2	7.2	7.6
3	-2	24.6	24.7
-3	2	1.6	.3
4	2	22.7	22.4
-4	2	13.5	12.8
4	-2	10.0	9.0
5	-2	32.8	33.9
5	2	4.1	5.3
-5	2	1.9	1.9
6	2	5.2	4.9
6	-2	6.3	6.6
-6	2	1.1	1.1*
7	2	10.4	10.1
7	-2	1.1	.7*
-7	2	1.1	3.5*
8	-2	10.6	10.8
-8	2	5.6	5.3
8	2	1.2	6.6*
9	-2	4.9	4.4
9	2	2.7	2.5
-9	2	3.2	3.6
10	-2	1.7	1.3*
0	3	32.6	33.4
1	3	8.2	7.9
-1	3	10.4	9.5
1	-3	5.9	5.8
2	3	15.6	15.0
-2	3	15.4	14.7
2	-3	18.3	18.8
3	-3	4.5	3.9
3	3	2.4	2.1
-3	3	8.4	8.1
4	3	10.3	10.8
-4	3	5.0	4.7
4	-3	17.0	18.3
5	-3	1.9	.8
5	3	10.1	10.4
-5	3	1.1	1.3*
6	-3	11.7	11.9
-6	3	2.7	2.3
6	3	1.1	2.1*
7	-3	6.7	6.7
7	3	11.5	11.8
-7	3	14.8	14.3
8	-3	2.6	1.6
8	3	5.6	5.1
-8	3	1.1	1.0*
9	-3	6.5	6.8
9	3	6.9	7.1
-9	3	3.1	2.6
10	-3	4.3	4.6
0	4	14.5	14.9
1	4	43.2	44.4
-1	4	35.5	36.0
1	-4	.9	.9*
2	-4	15.1	15.4
2	4	4.4	4.9
-2	4	17.7	17.3
3	4	25.8	26.2
-3	4	5.5	5.1
3	-4	5.2	5.2
4	-4	5.7	5.9
4	4	21.8	21.8
-4	4	3.6	5.1
5	4	11.8	13.3
5	-4	8.0	8.0
-5	4	1.0	.4*
6	4	14.1	13.7
-6	4	17.3	17.5
6	-4	6.2	6.1
7	-4	14.1	13.9
7	4	4.1	4.0
-7	4	1.1	.5*
8	-4	5.6	5.7
8	4	3.2	4.1
-8	4	6.1	5.5
9	4	2.2	1.6
9	-4	1.1	.4*
0	5	4.4	4.4
1	5	15.4	15.9
-1	5	4.0	3.8
1	-5	13.7	13.5
2	5	8.0	8.3
-2	5	24.7	23.3
2	-5	4.0	3.3
3	-5	20.8	20.4
3	5	10.8	10.4
-3	5	.9	.1*
4	5	22.5	22.5
-4	5	9.6	10.0
4	-5	9.9	10.1
******L= 3*******			
-9	3	2.2	3.1
5	-5	15.9	15.7
5	5	11.1	11.0
-5	5	12.4	12.2
6	-5	31.9	32.4
6	5	1.1	1.6*
-6	5	1.1	.9*
7	-5	7.3	7.5
7	5	12.0	12.3
-7	5	13.8	13.6
8	-5	9.8	9.7
8	5	2.5	2.6
-8	5	1.1	.6*
9	-5	2.8	2.5
0	6	5.3	4.9
-1	6	35.2	34.9
1	-6	1.0	2.4*
1	6	.9	1.8*
2	6	8.6	8.3
-2	6	10.0	10.2
2	-6	7.8	7.3
3	6	5.7	5.6
-3	6	16.8	17.4
3	-6	17.3	17.2
4	-6	2.1	1.9
-4	6	4.2	4.0
4	6	1.1	1.2*
-5	6	7.4	6.9
5	-6	11.9	11.7
5	6	17.2	17.3
6	-6	6.0	5.6
6	6	4.2	3.8
-6	6	19.0	18.9
7	-6	12.5	12.8
-7	6	5.2	5.1
7	6	1.1	1.0*
8	6	7.2	7.0
-8	6	2.9	2.2
8	-6	1.1	1.2*
9	-6	4.0	4.5
0	7	10.8	10.6
-1	7	7.9	8.5
1	-7	25.7	25.6
1	7	1.0	.1*
2	-7	26.1	26.3
2	7	6.3	5.9
-2	7	27.2	27.0
3	-7	10.9	10.7
3	7	11.7	12.0
-3	7	9.5	9.6
-4	7	10.4	10.5
4	-7	4.5	5.2
4	7	14.0	13.9
5	-7	2.0	2.4
-5	7	11.3	11.1
5	7	1.1	1.4*
6	-7	21.1	21.3
6	7	5.4	5.4
-6	7	2.4	1.6
-7	7	6.5	6.7
7	-7	5.0	5.0
7	7	1.1	7.1*
8	-7	5.3	6.7
9	-7	1.0	.3*
0	8	3.1	2.8
1	8	4.8	4.9
-1	8	26.4	26.2
1	-8	1.2	2.2*
2	8	10.5	10.4
-2	8	9.8	9.4
2	-8	11.0	11.3
3	-8	15.2	15.7
-3	8	8.1	8.2
3	8	1.2	1.4*
4	8	4.8	4.8
-4	8	3.9	3.9
4	-8	1.1	.4*
5	-8	6.0	5.9
5	8	10.9	11.2
-5	8	4.5	5.1
6	-8	3.6	3.5
6	8	2.8	3.2
-6	8	1.1	.1*
7	-8	4.8	4.2
-7	8	5.8	5.4
8	-8	2.1	1.4
0	9	7.5	7.2
1	9	2.4	3.6
1	-9	19.8	19.5
-1	9	1.3	1.8*
2	9	9.3	8.6
-2	9	8.0	8.7
2	-9	8.7	8.4
-3	9	8.1	8.2
3	9	9.9	9.5
3	-9	1.1	4.0*
-4	9	9.8	9.7
4	-9	1.1	.8*
4	9	1.3	1.9*
5	-9	6.4	6.4
5	9	4.4	4.0
-5	9	1.1	1.4*
-6	9	2.8	2.3
6	-9	1.1	.6*
7	-9	2.8	3.5
0	10	2.1	1.0
-1	10	13.8	13.9
1	10	5.4	5.6
1	-10	1.3	4.1*
2	10	9.3	8.6
-2	10	5.7	5.7
2	-10	2.3	2.1
-3	10	17.0	17.8
3	-10	3.6	4.1
3	10	11.0	10.8
-4	10	5.0	5.2
4	-10	11.5	11.0
4	10	1.6	4.6*
5	-10	4.6	4.7
-5	10	2.1	1.9
6	-10	4.2	4.9
0	11	2.1	1.6
-1	11	6.0	5.7
1	11	1.1	1.1*
1	-11	1.1	.7*
2	11	12.5	12.4
-2	11	2.9	3.1
2	-11	1.0	.0*
3	-11	12.6	12.0
-3	11	1.4	3.2*
-4	11	8.4	8.4
4	-11	4.7	5.0
5	-11	5.8	6.2
******L= 3*******			
0	0	29.5	28.7
1	0	9.0	9.6
2	0	4.1	3.6
3	0	.9	1.7*
4	0	15.9	16.2
5	0	16.5	15.9
6	0	14.1	14.4
7	0	18.6	17.7
8	0	3.9	4.3
9	0	10.9	10.7
0	1	15.3	14.7
1	1	26.1	24.8
1	-1	9.1	8.6
-1	1	38.2	37.7
2	1	7.3	7.1
2	-1	3.1	2.4
-2	1	12.6	11.6
3	1	5.9	5.9
-3	1	3.8	3.9
3	-1	13.9	13.5
4	1	12.6	11.0
-4	1	22.1	22.6
4	-1	.9	.6*
5	1	18.0	18.0
5	-1	28.5	28.3
-5	1	9.1	7.8
6	1	6.6	5.9
6	-1	7.9	7.0
-6	1	3.8	4.6
7	-1	4.7	4.7
7	1	12.1	12.3
-7	1	2.2	2.4
8	-1	15.4	14.7
8	1	12.5	11.6
-8	1	3.1	3.0
9	-1	3.2	3.2
9	1	1.2	2.6*
0	2	14.1	14.0
1	-2	26.8	26.8
1	2	20.1	19.8
-1	2	4.6	3.6
2	2	12.0	11.6
-2	2	22.8	20.6
2	-2	20.2	19.5
3	2	8.5	8.2
3	-2	3.2	2.6
-3	2	7.6	7.5
4	-2	23.8	23.8
4	2	3.8	3.3
-4	2	13.9	13.0
5	2	20.2	20.1
5	-2	3.6	2.7
-5	2	3.1	1.9
6	2	3.5	4.0
6	-2	9.7	9.5
-6	2	1.1	1.0*
7	-2	19.5	19.8
7	2	3.6	4.6
-7	2	12.1	11.2
8	2	6.1	5.8
8	-2	1.1	.8*
-8	2	1.0	.4*
9	-2	6.7	7.0
9	2	5.9	6.3
0	3	34.7	32.8
1	3	50.1	51.5
-1	3	5.8	5.3
1	-3	21.2	22.3
2	3	11.1	11.3
-2	3	8.2	9.2
2	-3	7.0	7.0
3	3	11.8	11.7
3	-3	33.0	33.6
-3	3	9.2	9.0
4	3	29.1	29.0
-4	3	3.4	2.4
4	-3	9.3	9.1
5	-3	10.2	10.3
5	3	14.1	13.7
6	3	6.4	6.2
6	-3	16.6	17.1
-6	3	11.6	11.7
7	3	5.3	4.3
7	-3	3.3	4.1
-7	3	1.1	.4*
8	3	3.2	2.9
-8	3	6.8	6.8
8	-3	1.1	1.3*
9	-3	2.1	.5
9	3	3.1	2.6
0	4	8.1	6.8
1	4	13.9	13.4
-1	4	26.8	26.9
1	-4	3.3	2.8
2	-4	22.6	23.0
2	4	32.9	31.9
-2	4	8.5	8.2
3	-4	4.4	3.8
3	4	5.2	4.9
-3	4	4.3	4.9
4	4	18.8	19.2
-4	4	8.2	7.4
4	-4	1.0	1.0*
5	-4	5.0	5.5
5	4	25.5	25.1
-5	4	11.7	11.4
6	-4	7.7	7.5
6	4	5.3	5.3
-6	4	1.1	.6*
7	-4	4.3	4.8
7	4	6.2	5.3
-7	4	1.1	6.3*
8	-4	2.1	.3
8	4	3.6	3.6
-8	4	1.0	.7*
9	-4	5.0	4.8
0	5	5.5	4.7
1	5	23.5	23.4
-1	5	33.2	33.1
1	-5	13.1	12.7
2	5	11.6	12.4
-2	5	2.4	3.0
2	-5	4.5	4.7
3	5	8.0	7.6
3	-5	2.2	1.6
-3	5	24.3	24.1
4	5	7.0	6.7
-4	5	6.0	5.7
4	-5	1.0	.5*
5	-5	5.1	5.0
5	5	20.1	20.2
-5	5	1.2	2.0*
6	-5	2.2	2.7
6	5	5.0	4.2
-6	5	12.9	13.1
7	-5	17.6	17.4
7	5	5.1	5.1
-7	5	1.0	.0*
8	-5	6.3	7.1
8	5	7.7	8.0
9	-5	2.5	2.6
0	6	16.9	18.1
1	6	4.7	4.7
-1	6	5.3	5.7
1	-6	2.7	2.4
2	6	7.8	7.2
2	-6	2.3	1.5
-2	6	20.4	20.5
3	6	18.4	18.7
3	-6	1.0	.6*
-3	6	1.1	.5*
4	-6	8.9	8.9
4	6	12.5	12.6
-4	6	18.4	18.5
5	6	5.7	5.4
-5	6	15.3	15.7
5	-6	1.1	.3*
6	6	1.2	5.6*
-6	6	1.2	2.8*
7	-6	3.3	3.2
7	6	7.1	6.9
-7	6	3.1	3.2
8	-6	1.0	1.0*
9	-6	8.6	8.6
0	7	3.8	3.1
-1	7	14.1	16.2
1	-7	13.4	13.4
1	7	1.1	2.0*
2	7	9.8	10.3
-2	7	14.1	13.7
2	-7	2.2	2.2
3	-7	13.7	13.0
-3	7	14.1	13.8
3	7	1.1	.9*
4	-7	6.7	7.1
4	7	5.8	5.2
-4	7	1.1	1.0*
5	-7	3.9	3.9
5	7	16.1	15.5
-5	7	1.2	1.7*
-6	7	5.5	4.1
6	7	2.2	1.3
6	-7	1.2	1.2*
7	-7	11.9	11.6
-7	7	3.0	2.9
8	-7	1.0	.5*
0	8	11.4	11.0
-1	8	9.6	10.0
1	-8	12.5	12.0
1	8	1.2	.8*
2	8	6.0	5.9
2	-8	2.6	2.1
-2	8	1.1	1.4*
3	8	13.4	13.1
-3	8	2.9	2.4
3	-8	2.6	2.5
4	-8	12.6	11.6
-4	8	6.1	6.4
4	8	1.2	2.3*
5	-8	4.1	3.9
-5	8	3.7	3.5
5	8	1.0	2.9*
6	-8	6.8	6.1
6	8	2.0	.2
-6	8	6.9	7.1
7	-8	2.7	2.6
8	-8	3.2	3.7
0	9	2.5	1.6
1	-9	10.5	10.0
1	9	7.1	6.8
2	-9	9.5	9.6
2	9	10.8	10.7
-2	9	10.9	11.0
3	-9	10.6	10.3
3	9	8.9	8.1
-3	9	8.5	8.5
-4	9	3.5	3.4
4	-9	11.2	11.8
5	-9	7.6	7.7
-5	9	5.3	5.1
6	-9	2.0	1.3
7	-9	1.2	1.5*
0	10	9.4	9.1
1	-10	6.4	6.6
-1	10	1.1	.1*
1	10	1.5	3.2*
2	-10	2.7	1.6
2	10	5.6	5.5
-2	10	8.2	8.7
3	-10	4.6	5.8
3	10	4.8	4.5
-3	10	7.4	8.1
4	-10	4.2	4.2
-4	10	6.0	5.8
5	-10	4.4	4.4
6	-10	3.2	3.9
0	11	5.8	6.5
-1	11	1.0	.1*
1	-11	1.1	1.1*
2	-11	2.3	1.9
3	-11	1.1	.8*
******L= 4*******			
0	0	6.9	6.6
1	0	10.5	10.1
2	0	5.2	5.0
3	0	8.2	8.2
4	0	13.4	13.7
5	0	19.3	18.4
-5	0	1.2	2.3*
6	0	1.1	.7*
7	0	6.0	5.8
8	0	13.2	13.2
9	0	5.3	5.1
0	1	15.4	14.8
1	1	17.4	15.6
-1	1	12.5	12.3
1	-1	.9	1.4*
2	1	2.1	.6
-2	1	5.8	5.8
2	-1	.9	.3*
3	1	8.9	9.4
3	-1	18.7	18.2
-3	1	14.5	14.2
4	1	2.4	2.5
4	-1	16.2	16.7
-4	1	7.8	8.2
5	1	15.7	15.8
5	-1	8.2	8.6
-5	1	4.3	4.6
6	-1	9.4	10.2
6	1	7.1	6.9
-6	1	1.1	1.1*
7	1	9.1	8.5
7	-1	14.8	15.0
-7	1	6.1	5.5
8	1	1.1	.7*
8	-1	1.1	1.0*
9	-1	5.4	5.3
0	2	10.0	9.9
-1	2	17.1	16.7
1	-2	2.3	2.2
1	2	1.1	2.2*
2	2	12.1	11.2
-2	2	7.3	6.7
2	-2	1.0	.9*
3	2	10.4	10.7
3	-2	15.8	15.8
-3	2	15.9	16.1
4	-2	3.3	3.7
4	2	9.4	8.5
-4	2	1.1	.1*
5	-2	11.5	11.7
-5	2	7.8	8.2
5	2	1.1	1.6*
6	-2	7.6	9.4
6	2	4.7	4.3
-6	2	5.6	5.5
7	-2	4.3	3.8
7	2	1.2	1.8*
-7	2	1.0	.8*
8	-2	6.7	7.1
8	2	2.1	2.0
9	-2	3.4	2.5
0	3	3.3	3.2
1	3	14.3	13.6
1	-3	5.4	5.5
-1	3	1.1	.4*
2	3	19.3	18.2
2	-3	23.2	23.0
3	3	9.6	10.0
-3	3	12.1	12.1
3	-3	1.0	.7*
4	3	12.5	12.2
4	-3	12.0	11.8
-4	3	13.7	12.8
5	3	17.5	17.0
5	-3	11.8	11.5
-5	3	5.4	6.0
6	3	3.9	3.9
6	-3	6.9	7.7
-6	3	1.2	1.6*
7	-3	13.9	14.3
-7	3	7.8	6.9
7	3	1.1	1.5*
8	-3	1.0	1.4*
9	3	1.0	.6*
9	-3	1.1	1.4*
0	4	1.0	.1*
1	4	8.9	8.5
1	-4	25.5	25.9
-1	4	8.2	7.9
2	-4	11.8	11.5
2	4	12.9	12.0
-2	4	11.8	11.5
3	-4	17.6	17.0
3	4	16.0	15.9
-3	4	1.4	19.5*
4	4	2.0	1.5
4	-4	12.0	12.5
-4	4	7.2	7.1
5	-4	5.8	6.0
5	4	3.7	3.9
-5	4	5.5	5.6
6	-4	12.7	13.0
6	4	3.1	3.0
-6	4	1.1	1.6*
7	4	4.6	4.3
7	-4	1.2	1.1*
-7	4	1.1	1.1*
8	-4	2.6	1.9
8	4	5.7	4.9
9	-4	1.0	.9*
0	5	8.3	8.2
1	5	13.4	13.5
1	-5	5.8	6.1
-1	5	1.1	1.2*
2	5	17.9	18.0
-2	5	19.2	19.5
2	-5	21.9	22.5
3	5	19.9	19.6
3	-5	7.8	8.3
-3	5	5.0	4.7
4	5	2.6	2.4
-4	5	6.6	6.8
4	-5	1.0	.5*
5	5	11.2	11.2
5	-5	9.9	9.9
6	-5	1.2	1.6*
5	5	1.2	2.0*
-6	5	1.1	1.2*
7	-5	5.3	5.9
7	5	1.1	.2*
8	-5	6.1	6.5
0	6	4.1	4.6
1	-6	17.6	17.1
-1	6	5.1	4.9
1	6	1.0	2.6*
2	-6	12.7	12.1
2	6	10.7	10.5
-2	6	4.3	4.6
3	-6	4.4	3.4
3	6	9.5	9.4
-3	6	9.8	10.1
4	6	3.9	4.0
4	-6	2.9	3.4
-4	6	7.1	7.2
5	-6	3.3	3.4
5	6	1.1	2.1*
-5	6	1.1	1.4*
6	-6	2.7	2.6
-6	6	3.6	2.8
6	6	1.3	2.4*
7	-6	10.4	10.5
8	-6	2.9	2.1
0	7	1.1	.8*
1	-7	3.8	4.1
1	7	1.2	1.2*
-1	7	1.5	3.4*
2	-7	15.3	15.1
2	7	1.2	1.0*
-2	7	1.1	.1*
3	7	9.3	9.6
-3	7	5.8	5.8
3	-7	1.2	1.5*
4	-7	11.0	10.9
-4	7	3.0	1.6
4	7	1.2	1.5*
5	-7	9.6	8.7
-5	7	3.6	3.7
5	7	1.1	.4*
6	-7	5.1	4.7
6	7	1.1	.1*
7	-7	4.5	4.2
0	8	1.1	.3*
1	8	9.4	9.8
-1	8	2.6	2.7
1	-8	10.5	10.8
2	-8	2.8	2.7
2	8	1.1	.3*
-2	8	1.1	1.4*
3	-8	7.7	7.8
3	8	1.1	.6*
-3	8	1.3	1.7*
4	-8	6.0	5.4
4	8	5.7	5.7
5	-8	11.5	10.9
5	8	1.1	1.3*
6	-8	3.9	3.6
7	-8	2.2	2.5
0	9	4.7	5.0
1	-9	4.7	4.0
1	9	6.9	7.5
-1	9	1.1	.9*
2	-9	2.2	1.2
2	9	7.8	7.0
-2	9	5.1	5.8
3	-9	4.2	4.1
3	9	4.4	4.4
-3	9	1.1	1.7*
4	-9	16.6	16.1
5	-9	4.4	3.8
6	-9	4.1	4.1
0	10	8.0	7.5
1	-10	1.2	2.1*
2	-10	3.0	2.4
3	-10	10.0	9.9
4	-10	1.1	1.0*
******L= 5*******			
0	0	11.5	11.4
1	0	3.3	3.3
2	0	2.6	2.4
3	0	16.5	17.4
4	0	3.2	3.3
5	0	8.6	8.9
6	0	1.1	.2*
7	0	8.0	7.9
8	0	5.6	6.0
0	1	9.9	9.4
1	1	13.5	13.6
-1	1	2.5	2.2
1	-1	9.6	9.1
2	1	12.4	12.3
2	-1	13.2	13.4
-2	1	19.1	18.2
3	-1	11.5	11.9
-3	1	4.6	4.4
3	1	1.2	1.5*
4	-1	11.4	10.6
4	1	6.7	6.3
-4	1	4.4	4.1
5	1	3.2	2.9
5	-1	7.8	7.8
-5	1	4.1	4.9
6	1	7.3	7.5
6	-1	1.1	.9*
-6	1	1.1	1.0*
7	1	6.8	7.6
7	-1	3.8	3.5
8	-1	10.0	9.8
8	1	1.1	2.3*
1	-2	6.9	6.7
1	2	1.1	1.0*
-1	2	1.1	.2*
2	2	8.0	6.9
2	-2	9.1	9.6
-2	2	2.5	2.8
3	2	1.1	1.1*
3	-2	12.1	11.0
-3	2	3.2	4.0
4	-2	9.1	9.7
4	2	14.1	13.0
-4	2	11.9	12.0
5	-2	1.1	.5*
5	2	8.1	7.5
-5	2	4.1	3.2
6	2	1.1	.1*
-6	2	1.0	.4*
6	-2	5.3	6.3
7	-2	9.4	9.9
7	2	3.4	3.4
8	-2	1.1	1.4*
0	3	18.1	16.9
-1	3	1.4	3.2*
1	-3	9.0	8.3
2	3	3.8	3.7
2	-3	4.3	4.5
-2	3	8.4	8.8
3	-3	13.5	13.1
3	3	4.8	6.3
-3	3	12.0	11.5
4	3	12.9	12.6
4	-3	3.8	3.4
-4	3	2.2	1.1
5	3	1.1	.3*
5	-3	7.3	6.9
-5	3	7.7	8.0
6	3	8.3	9.8
6	-3	4.9	4.9
7	-3	1.1	3.0*
7	3	1.2	2.3*
8	-3	5.0	5.3
0	4	7.6	7.4
1	4	18.7	17.1
-1	4	5.4	5.3
1	-4	3.5	4.4
-2	4	9.0	9.5
2	-4	17.2	16.8
-3	4	1.1	.5*
3	4	15.1	14.8
3	-4	7.0	7.1
4	4	1.3	3.1*
4	-4	7.7	7.4
-4	4	10.6	10.4
-5	4	1.1	1.0*
5	-4	9.1	9.0
5	4	2.4	3.0
6	-4	8.5	8.1
6	4	8.1	8.9
7	-4	8.1	7.8
7	4	2.1	2.8
8	-4	2.7	1.6
0	5	11.6	11.1
1	5	1.2	1.0*
-1	5	7.0	7.6
1	-5	11.9	11.3
2	5	12.5	12.4
2	-5	7.3	7.2
-2	5	6.2	6.8
3	-5	12.5	12.8
-3	5	4.7	4.6
4	-5	7.2	7.5
4	5	9.8	10.5
-4	5	5.3	5.4
5	-5	1.1	.9*
-5	5	1.0	1.2*
5	5	10.2	10.2
6	-5	9.6	9.3
6	5	4.8	5.0
7	-5	2.0	.9
0	6	1.3	2.2*
1	-6	3.1	3.7
1	6	9.6	9.9
-1	6	3.2	4.2
2	6	1.1	.1*
-2	6	1.1	.4*
2	-6	11.5	11.5
-3	6	1.2	2.0*
3	6	7.7	6.7
3	-5	5.3	4.6
4	-6	4.4	4.2
-4	6	2.7	2.8
5	-6	8.2	8.3
5	6	2.0	1.0
6	-6	2.5	2.5
7	-6	1.1	.9*
0	7	2.7	1.9
1	7	1.1	1.2*
-1	7	1.2	1.4*
1	-7	6.9	7.0
2	7	1.2	1.7*
2	-7	3.1	3.1
-2	7	8.0	7.8
3	-7	1.1	.3*
3	7	1.3	1.2*
-3	7	5.3	5.6
4	-7	1.2	1.3*
4	7	6.5	7.0
5	-7	11.6	10.9
6	-7	2.9	2.8
0	8	2.1	1.5
1	-8	1.2	1.7*
1	8	5.2	4.4
-1	8	5.5	5.5
2	8	6.4	5.7
-2	8	5.2	5.7
2	-8	4.8	4.9
3	-8	1.2	1.8*
4	-8	13.1	13.0
5	-8	4.0	4.1
2	-9	1.1	1.9*
******L= 6*******			
0	0	7.8	8.3
1	0	4.1	4.6
2	0	2.5	3.1
3	0	2.8	2.6
-4	0	1.1	.2*
4	0	8.0	7.4
5	0	3.5	3.5
6	0	3.3	4.2
0	1	1.3	2.7*
1	-1	1.2	.8*
1	1	12.1	11.7
-1	1	2.1	1.7
2	-1	1.3	2.5*
2	1	12.2	11.3
-2	1	3.0	2.6
-3	1	1.2	1.1*
3	1	7.6	7.0
3	-1	11.3	11.4
4	1	3.5	4.0
4	-1	5.3	5.1
-4	1	6.6	7.0
5	-1	1.1	.9*
5	1	2.9	2.7
6	1	4.1	4.8
6	-1	5.3	4.9
0	2	13.5	13.3
-1	2	1.1	.9*
1	-2	1.5	3.6*
1	2	2.4	2.9
-2	2	1.2	1.9*
2	-2	11.5	11.8
2	2	4.8	4.4
3	2	1.2	1.9*
3	-2	4.2	4.6
-3	2	7.4	6.8
4	2	1.2	1.7*
4	-2	4.3	4.4
-4	2	3.7	3.3
5	-2	1.0	1.5*
5	2	1.1	1.6*
6	-2	1.1	1.5*
6	2	8.3	7.9
0	3	5.7	6.3
1	-3	1.8	6.6*
1	3	11.5	14.0
-1	3	2.4	2.9
2	-3	1.3	1.6*
2	3	3.8	5.2
-2	3	2.3	2.5
-3	3	1.1	.2*
3	3	3.4	3.6
3	-3	11.5	11.1
4	3	4.8	3.9
4	-3	4.8	4.7
5	-3	1.2	.8*
5	3	1.1	2.2*
6	-3	1.2	1.6*
0	4	9.4	9.3
1	4	1.1	2.7*
-1	4	1.1	1.2*
1	-4	1.3	1.1*
-2	4	1.3	2.4*
2	4	7.3	7.7
2	-4	8.5	8.4
3	4	2.1	1.8
3	-4	5.0	5.8
-3	4	3.2	3.5
4	-4	2.1	1.7
4	4	9.5	8.9
5	-4	1.0	1.0*
5	4	4.0	4.5
6	-4	2.1	2.3
0	5	1.5	3.7*
1	5	1.4	7.8*
-1	5	1.2	2.0*
1	-5	2.3	2.6
2	-5	1.3	2.2*
-2	5	1.0	.1*
2	5	2.5	2.6
3	5	4.1	5.1
4	5	3.6	4.4
4	-5	5.9	5.4
5	-5	6.0	5.5
0	6	1.0	.4*
1	6	1.1	.2*
-1	6	1.0	.9*
1	-6	1.1	1.3*
2	-6	1.1	.1*
2	6	5.5	5.4
3	-6	3.9	4.1
4	-6	3.5	4.0
2	-7	1.1	.2*
******L= 7*******			
0	0	1.1	1.3*
1	0	1.5	4.1*
2	0	1.4	3.7*
3	0	3.5	3.5
0	1	9.5	9.2
1	-1	1.2	2.0*
1	1	2.5	2.1
2	1	5.0	5.3
2	-1	4.1	4.4
3	1	1.1	.8*
3	-1	4.9	4.4
1	2	11.5	11.3
1	-2	4.5	6.6
2	2	1.0	.7*
2	-2	1.3	2.7*
3	-2	7.0	8.2
2	-3	9.9	10.1
******L=-1*******			
1	0	7.5	6.9
2	0	9.3	9.5
3	0	20.8	20.0
4	0	12.6	12.7
5	0	10.6	10.3
6	0	3.5	3.0
7	0	2.6	2.1
8	0	7.7	7.1
9	0	5.8	5.9
0	1	69.8	75.0
1	1	14.0	14.0
2	1	59.9	60.7
3	1	3.7	3.6
4	1	2.7	2.9
5	1	14.8	14.8
6	1	11.2	11.7
7	1	3.0	2.5
8	1	5.4	5.1
9	1	7.3	7.4
0	2	27.7	27.9
1	2	11.0	11.1
2	2	40.4	41.8
3	2	39.4	37.9
4	2	19.4	19.0
5	2	18.0	18.5
6	2	17.4	16.8
7	2	4.3	4.1
8	2	1.1	.8*
8	2	1.1	.8*
9	2	3.9	5.1
0	3	3.3	3.3
1	3	15.9	15.9
2	3	35.6	34.7
3	3	3.6	3.2
4	3	1.0	.6*
4	3	1.0	.6*
5	3	25.0	25.0
6	3	8.2	8.4
7	3	8.6	8.7
8	3	1.1	3.1*
8	3	1.1	3.1*
9	3	4.6	4.8
0	4	5.7	5.7
1	4	12.0	11.6
2	4	2.3	2.5
3	4	13.9	13.7
4	4	28.9	29.1
5	4	3.3	2.8
6	4	11.4	11.4
7	4	8.8	8.5
8	4	8.5	8.5
0	5	7.8	7.8
1	5	12.8	11.8
2	5	18.9	19.3
3	5	7.3	6.5
4	5	8.5	8.3
5	5	13.9	14.7
6	5	11.9	11.9
7	5	5.0	5.0
8	5	1.1	1.5*
0	6	5.5	5.5
1	6	20.1	20.5
2	6	5.1	5.6
3	6	2.6	3.1
4	6	3.8	4.7
5	6	13.3	13.7
6	6	1.1	.9*
7	6	4.1	5.0
0	7	35.1	35.5
1	7	1.3	3.3*
2	7	6.7	7.1
3	7	20.2	19.8
4	7	9.1	9.7
5	7	5.0	5.1
6	7	6.1	6.6
7	7	2.1	1.8
0	8	10.0	10.1
1	8	23.4	22.9
2	8	10.0	10.3
3	8	1.6	3.7*
4	8	15.9	15.8
5	8	3.1	5.5
6	8	2.5	2.0
0	9	15.1	14.5
1	9	1.6	5.1*
2	9	4.9	4.7
3	9	14.8	15.0
4	9	1.2	1.4*
5	9	1.4	2.8*
0	10	1.1	1.1*
1	10	4.3	4.3
2	10	5.9	5.5
3	10	6.4	5.8
4	10	4.4	4.8
0	11	1.2	1.8*
1	11	1.2	2.0*
2	11	3.3	3.6
0	12	1.0	1.4*
******L=-2*******			
1	0	33.0	33.9
2	0	11.3	11.1
3	0	.9	.6*
4	0	23.9	24.3
5	0	5.2	4.9
6	0	10.4	10.0
7	0	5.8	5.5
8	0	2.8	2.4
9	0	6.6	6.6
0	1	23.4	23.9
1	1	13.6	12.7
2	1	16.9	16.3
3	1	17.2	16.7
4	1	1.9	1.0
5	1	16.3	17.1
6	1	7.0	6.9
7	1	2.0	.3
8	1	3.7	3.9
9	1	4.6	4.4
0	2	22.3	22.5
1	2	7.8	7.8
2	2	46.4	45.8
3	2	16.0	16.1
4	2	3.4	3.3
5	2	1.0	.5*
6	2	7.6	7.3
7	2	8.7	9.0
8	2	8.2	8.5
0	3	11.2	11.1
1	3	44.2	43.2
2	3	19.3	18.7
3	3	20.0	19.1
4	3	12.7	12.1
5	3	9.3	9.1
6	3	11.5	12.2
7	3	1.1	.1*
8	3	1.1	1.1*
0	4	11.7	11.3
1	4	20.9	20.9
2	4	25.8	26.7
3	4	24.9	25.5
4	4	5.6	5.9
5	4	4.1	4.4
6	4	1.1	1.5*
7	4	13.9	14.5
8	4	4.0	4.0
0	5	5.5	5.7
1	5	16.2	16.8
2	5	1.0	.1*
3	5	5.3	5.4
4	5	12.9	12.7
5	5	6.7	6.5
6	5	9.6	10.0
7	5	5.4	5.8
0	6	29.9	29.7
1	6	2.6	3.6
2	6	1.0	.4*
3	6	6.0	6.6
4	6	12.4	11.9
5	6	2.3	1.9
6	6	6.1	6.2
7	6	10.8	11.2
0	7	11.3	11.4
1	7	25.9	25.5
2	7	5.7	6.8
3	7	1.1	.1*
4	7	9.9	10.1
5	7	6.3	6.2
6	7	1.1	.4*
0	8	36.3	36.1
1	8	1.5	4.6*
2	8	11.2	10.6
3	8	10.7	11.0
4	8	3.1	2.9
5	8	9.3	9.0
0	9	7.6	7.7
1	9	10.5	10.5
2	9	10.2	11.1
3	9	3.2	3.9
4	9	6.8	7.4
0	10	6.0	6.1
1	10	6.0	5.9
2	10	4.7	4.8
2	10	3.6	4.8
3	10	1.4	3.4*
0	11	1.0	.8*
1	11	1.3	2.3*
******L=-3*******			
1	0	5.0	5.3
2	0	21.0	21.1
3	0	11.5	11.8
4	0	3.4	3.0
6	0	1.1	1.4*
7	0	1.1	.4*
8	0	1.0	.0*
0	1	3.2	4.2
1	1	34.2	33.2
2	1	9.8	10.8
3	1	1.0	1.5*
4	1	16.0	16.3
5	1	3.8	3.7
6	1	2.9	3.1
7	1	1.2	2.2*
8	1	5.1	5.6
0	2	8.4	8.0
1	2	.9	1.3*
2	2	1.0	2.9*
3	2	9.9	9.8
4	2	12.0	12.5
5	2	10.2	10.8
6	2	6.9	6.7
7	2	6.7	6.8
8	2	1.0	.7*
0	3	12.6	13.0
1	3	31.2	31.0
2	3	19.5	19.7
3	3	6.6	6.4
4	3	6.1	6.6
5	3	3.2	2.8
6	3	3.0	2.7
7	3	3.9	4.1
0	4	6.5	5.9
1	4	7.1	6.8
2	4	3.7	4.2
3	4	12.5	12.5
4	4	10.3	10.1
5	4	9.1	9.9
6	4	6.6	6.9
7	4	5.0	4.0
0	5	5.6	5.8
1	5	12.9	12.7
2	5	8.2	9.3
3	5	2.5	3.3
4	5	3.6	4.2
5	5	1.1	1.0*
6	5	1.1	.3*
0	6	7.1	6.8
1	6	5.1	4.8
2	6	10.0	9.6
3	6	13.8	13.1
4	6	6.3	5.9
5	6	1.2	1.0*
6	6	6.6	5.9
0	7	13.8	14.0
1	7	1.1	4.3*
2	7	1.1	.2*
3	7	1.3	2.2*
4	7	7.4	6.8
5	7	1.1	1.1*
1	8	11.8	11.7
2	8	5.6	5.3
3	8	1.2	2.1*
4	8	6.1	5.8
0	9	10.6	10.8
1	9	5.4	5.2
2	9	6.2	6.6
3	9	1.0	.9*
0	10	1.1	.7*
1	10	5.6	5.4
0	11	1.1	1.7*
******L=-4*******			
1	0	29.8	29.3
2	0	9.9	10.0
3	0	4.1	4.4
4	0	2.4	2.5
6	0	3.9	3.7
7	0	3.1	2.8
0	1	36.5	36.2
1	1	1.1	2.1*
2	1	13.6	14.1
3	1	17.3	17.0
4	1	14.0	13.9
5	1	4.0	3.2
6	1	1.1	.0*
7	1	7.9	8.2
0	2	6.8	6.6
1	2	29.8	29.6
2	2	1.1	1.2*
3	2	15.5	14.9
4	2	8.5	7.6
5	2	2.7	2.9
6	2	11.6	11.9
7	2	1.2	2.2*
0	3	10.5	10.4
1	3	9.1	8.7
2	3	9.1	9.3
3	3	2.4	3.2
4	3	10.2	9.6
5	3	9.1	9.0
6	3	2.5	2.2
0	4	1.1	.1*
1	4	7.2	7.9
2	4	13.0	13.0
3	4	1.0	7.6*
4	4	1.3	5.6*
5	4	1.1	.3*
6	4	7.9	7.6
0	5	9.0	9.3
1	5	6.5	5.9
2	5	3.1	2.7
3	5	7.7	7.2
4	5	5.4	5.1
5	5	2.1	2.4
0	6	1.1	.8*
1	6	11.0	10.2
2	6	9.4	9.1
3	6	1.1	.9*
4	6	5.0	5.2
5	6	1.0	1.1*
0	7	7.8	8.1
1	7	3.9	3.5
2	7	12.0	11.5
3	7	8.0	8.2
4	7	1.1	.3*
0	8	5.2	5.1
1	8	7.7	7.3
2	8	4.6	3.6
3	8	2.3	3.0
1	9	6.0	6.2
2	9	5.5	5.6
0	10	7.2	7.0
******L=-5*******			
1	0	5.8	6.3
3	0	2.4	2.0
4	0	1.1	.5*
6	0	1.0	.4*
0	1	1.2	1.9*
1	1	10.3	9.5
2	1	13.2	14.0
3	1	13.4	13.8
4	1	2.5	3.9
5	1	1.1	.0*
0	2	11.6	11.2
1	2	1.2	2.3*
2	2	17.0	17.1
3	2	1.2	1.1*
4	2	6.0	7.1
5	2	9.1	8.7
0	3	5.8	5.9
1	3	10.5	10.9
2	3	4.5	4.2
3	3	15.0	15.0
4	3	1.2	1.8*
5	3	2.5	3.0
0	4	3.1	3.9
1	4	9.5	9.7
2	4	6.4	6.0
3	4	4.8	3.9
4	4	2.9	2.7
0	5	8.8	9.4
1	5	4.1	3.9
2	5	2.9	2.0
3	5	2.6	3.1
4	5	1.1	.5*
0	6	6.6	6.7
1	6	3.1	2.5
2	6	5.4	5.5
3	6	3.1	2.4
1	7	10.2	9.9
2	7	1.1	.7*
0	8	7.1	7.3
1	8	1.0	.2*
******L=-6*******			
2	0	1.1	1.2*
3	0	4.9	4.6
1	1	1.1	.0*
2	1	4.5	4.3
3	1	4.5	5.2
0	2	1.2	1.3*
1	2	3.0	4.1
2	2	6.1	5.3
3	2	6.4	6.6
0	3	1.1	.2*
1	3	1.1	.7*
2	3	12.3	12.8
3	3	3.5	3.3
0	4	1.1	.4*
1	4	5.5	4.8
2	4	3.8	3.4
0	5	1.1	.0*
1	5	1.0	.5*
0	6	1.2	2.2*

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[a] Unobserved reflections are designated with an asterisk.

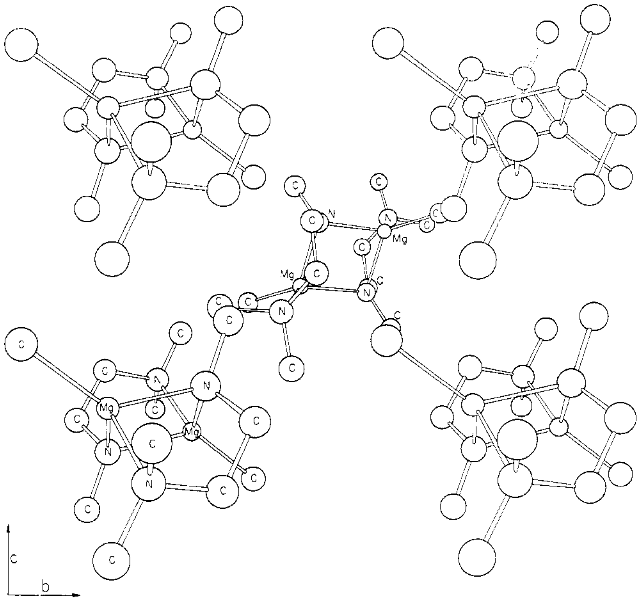

Figure 3.—Molecular packing of the dimers as viewed along the *a* direction. The dimer shown in the center is dimer I with four nearest neighbors of dimer II at $x = {}^1/_2$. The four nearest neighbor dimers at $x = -{}^1/_2$ are not shown.

and the reported bridging angle have been previously tabulated.[24]

Nonequivalent bridging Mg–N(1) bond lengths in dimer I are 2.107 (3) and 2.102 (3) Å, and, in dimer II, they are 2.118 (3) and 2.097 (3) Å. The sum of tetrahedral covalent radii[25] for Mg and N of 2.10 Å agrees quite well with these magnesium–amino nitrogen distances. However, the terminal nitrogen–magnesium (Mg–N(2)) distances of 2.190 (3) and 2.182 (3) Å for dimers I and II, respectively, are significantly longer than Pauling's sum of covalent radii. Also, these distances are longer than that of 2.16 Å found for the magnesium–tertiary amine distance in $C_2H_5MgBr\cdot N(C_2H_5)_3$.[19] In discussions involving bonding in four-membered heterocyclic ring systems such as those found in $[Mg(CH_3)_2]_n$ and $[Al(CH_3)_3]_2$, metal–metal interactions have often been used to help explain the stability of the ring. However, metal–metal bonding in this compound can be considered as nonexistent since the distance between metal atoms is 2.932 (2) and 2.945 (2) Å for dimers I and II, respectively. A complete tabulation of all nonhydrogen atom bond lengths and angles for both dimers I and II is given in Tables V and VI.

One salient feature of the geometry and bonding in the system is the apparent steric strain imposed on the chelating diamine. From Figures 1 and 2, it can be seen that whereas the Mg–N(1)–C(3) angle is nearly tetrahedral, the N(1)–Mg–N(2) angle is severely distorted from the tetrahedral value of 109.47° to an average value for both dimers of 83.7°. Also, the Mg–N(2) bond lengths are somewhat longer (2.182 and 2.190 Å for dimers I and II, respectively) than expected as mentioned previously. The substituted ethylenediamine group appears to function preferentially as a bridging group in the heterocyclic ring systems and secondly as the well-known chelating group through the lone pair on the tertiary amine type of nitrogen. However, in so doing, the diamine nearly retains the preferred tetrahedral coordination for each of its members. Although the diamine is acting as a chelate across tetrahedral Mg coordination sites, it does not appear to have enough length to reach from one Mg coordination site to another. This fact also manifests itself in the average dihedral angle found between the planes formed by Mg–N(1)–Mg′ and C(3)–Mg–C(2) of 88.9° and in the average dihedral angle found between the planes formed by N(1)–Mg–N(1)′ and C(1)–Mg–N(2) of only 72.1°.

Table V

Bond Lengths for Nonhydrogen Atoms for $[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$

Bond	Distance, Å	
	Dimer at (0, 0, 0)	Dimer at (${}^1/_2$, ${}^1/_2$, ${}^1/_2$)
Mg–N(1)	2.107 (3)	2.118 (3)
Mg–N(1)′	2.102 (3)	2.097 (3)
Mg–Mg′	2.932 (2)	2.945 (2)
N(1)–N(1)′	3.021 (6)	3.017 (6)
Mg–N(2)	2.190 (3)	2.182 (3)
Mg–C(1)	2.100 (4)	2.104 (4)
N(1)–C(2)	1.454 (5)	1.454 (5)
N(1)–C(3)	1.452 (5)	1.460 (5)
C(3)–C(4)	1.505 (6)	1.512 (5)
N(2)–C(4)	1.483 (5)	1.483 (5)
N(2)–C(6)	1.462 (5)	1.475 (5)
N(2)–C(5)	1.466 (5)	1.471 (5)

All of the amplitudes of the thermal vibrations of the nonhydrogen atoms are fairly normal. Figure 4 shows the thermal ellipsoids representing the anisotropic thermal motion of the nonhydrogen atoms for dimer I. Most of the atoms exhibit relatively isotropic vibration. Assuming the riding model,[26] the only realistic comparison of bond lengths with and without the effects of thermal motion is that between Mg and C(1). The interatomic distance between Mg and C(1) without

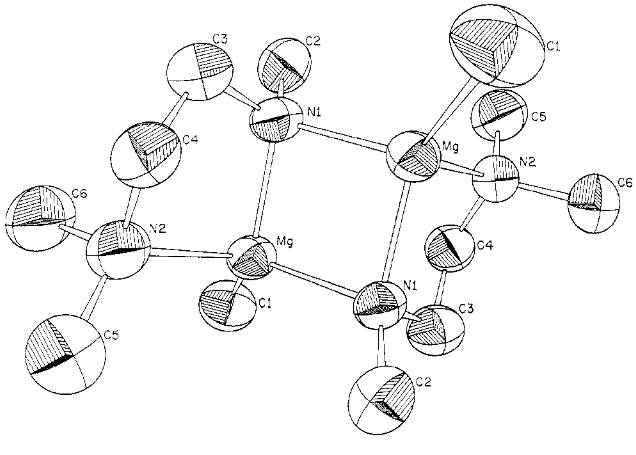

Figure 4.—Perspective view of dimer I of $[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$ with root-mean-square displacements indicated.

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[24] V. R. Magnuson and G. D. Stucky, *J. Am. Chem. Soc.*, **90**, 3269 (1968).

[25] L. Pauling, "The Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960.

[26] W. R. Busing and H. A. Levy, *Acta Cryst.*, **17**, 142 (1964).

TABLE VI

BOND ANGLES FOR NONHYDROGEN ATOMS FOR $[(CH_3)_2N(CH_2)_2N(CH_3)MgCH_3]_2$

Dimer at (0, 0, 0)		Dimer at (1/2, 1/2, 1/2)	
Atoms	Angle, deg	Atoms	Angle, deg
N(2)-Mg-N(1)	83.36 (13)	N(2)-Mg′-N(1)	83.97 (12)
N(2)-Mg-C(1)	115.19 (16)	N(2)′-Mg-C(1)	117.40 (17)
N(2)-Mg-N(1)′	112.02 (13)	N(2)′-MgN(1)	109.80 (14)
N(1)-Mg-C(1)	126.66 (16)	N(1)′-Mg-C(1)	130.26 (16)
N(1)-Mg-N(1)′	91.70 (12)	N(1)′-Mg-N(1)	91.38 (12)
C(1)-Mg′-N(1)	120.86 (16)	C(1)-Mg-N(1)	117.75 (17)
Mg-N(1)-Mg′	88.30 (12)	Mg-N(1)-Mg′	88.62 (12)
Mg-N(1)-C(2)	119.64 (26)	Mg-N(1)-C(2)	116.32 (25)
Mg-N(1)-C(3)	111.03 (25)	Mg-N(1)-C(3)	110.22 (24)
C(2)-N(1)-Mg′	117.89 (26)	C(2)-N(1)-Mg′	122.43 (25)
C(2)-N(1)-C(3)	108.77 (33)	C(2)-N(1)-C(3)	108.22 (25)
C(3)-N(1)-Mg′	109.79 (25)	C(3)-N(1)-Mg′	109.64 (23)
Mg-N(2)-C(5)	107.42 (25)	Mg′-N(2)-C(6)	106.23 (27)
Mg-N(2)-C(6)	119.39 (28)	Mg′-N(2)-C(5)	120.66 (24)
Mg-N(2)-C(4)	101.14 (24)	Mg-N(2)-C(4)	100.98 (23)
C(5)-N(2)-C(6)	107.74 (35)	C(5)-N(2)-C(6)	108.03 (33)
C(6)-N(2)-C(4)	111.61 (35)	C(5)-N(2)-C(4)	109.88 (33)
C(5)-N(2)-C(4)	109.43 (35)	C(6)-N(2)-C(4)	110.75 (32)
C(4)-C(3)-N(1)	111.40 (34)	C(4)-C(3)-N(1)	110.54 (32)
N(2)-C(4)-C(3)	111.95 (36)	N(2)-C(4)-C(3)	112.44 (35)

consideration of thermal motion is 2.100 (4) Å for dimer I and 2.104 (4) Å for dimer II. With the inclusion of thermal motion with the C(1) atom riding on the Mg atom, the interatomic distance is 2.126 (4) Å for dimer I and 2.117 (4) Å for dimer II.

Hydrogen atoms were included in the structure refinement. The average carbon-hydrogen bond length for dimer I is 0.98 (3) Å and, for dimer II, it is also 0.98 (3) Å. Light atom-hydrogen atom distances determined by X-ray analyses are well known to be abnormally short when compared to values obtained by spectroscopic methods and the average values reported above are not inconsistent with the often quoted spectroscopic C-H bond length of 1.1 Å.

CONTRIBUTION FROM LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA 94720

Preparation and Crystal Structures of Dicesium Berkelium Hexachloride and Dicesium Sodium Berkelium Hexachloride[1]

BY L. R. MORSS AND J. FUGER

Received November 25, 1968

Cs_2BkCl_6 and $Cs_2NaBkCl_6$ have been prepared from aqueous solution on the scale of a few micrograms using the isotope ^{249}Bk. The crystal structures of these compounds have been investigated by X-ray powder diffraction. Cs_2BkCl_6 is not isomorphous with Cs_2PuCl_6 and Cs_2CeCl_6, which exhibit trigonal symmetry, but has double the c axis of these compounds, with two molecules per unit cell. The hexagonal lattice parameters (95% confidence) are a = 7.450 (3) Å and c = 12.098 (5) Å. $Cs_2NaBkCl_6$ is face-centered cubic and isomorphous with the corresponding americium compound. The lattice parameter is a = 10.805 (3) Å; the crystal contains four molecules per unit cell.

Because of the comparatively short half-life and presently restricted availability of berkelium (as ^{249}Bk), the preparation and characterization of its compounds present an interesting challenge in inorganic synthesis. If these difficulties can be overcome, basic information can be obtained on the trivalent chemistry of this element and on its even more interesting tetravalent properties.

The frequently observed parallels in berkelium and cerium chemistry focused our interest on a marginally stable compound of tetravalent cerium, Cs_2CeCl_6. We had prepared this compound easily by precipitation from ice-cold concentrated HCl and with considerable difficulty by heating an intimate mixture of $CeCl_3$ and CsCl for several days in several atmospheres pressure of Cl_2. We selected the "wet" preparation as preferable, although it involved more mechanical manipulations. (The major technical difficulty was the quantitative removal of dried, centrifuged precipitate from the bottom of a capillary microcone.)

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(1) This work was performed under the auspices of the United States Atomic Energy Commission.