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THE STRUCTURE OF DIMETHYLBIS(QUINUCLIDINE)BERYLLIUM

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SUMMARY

The structure of $(CH_3)_2Be \cdot 2NC_7H_{13}$ has been determined by single-crystal X-ray diffraction methods. Four monomeric molecules crystallize in a monoclinic unit cell of dimensions $a = 11.82 \pm 0.02$, $b = 12.71 \pm 0.02$, $c = 12.00 \pm 0.02$ Å, and $\beta = 113.1° \pm 0.3°$. The space group is $P2_1/c$. Least-squares refinement resulted in a final R value of 13.2% for the 671 visually observed reflections. A distorted tetrahedral coordination is found about the beryllium atom. The average beryllium–carbon bond distance is 1.83 Å, and the beryllium–nitrogen bond distance is 1.91 Å. The nitrogen–beryllium–nitrogen bond angle is 110.8° compared to the methyl carbon–beryllium–methyl carbon angle of 118.3°.

INTRODUCTION

Dimethylberyllium is well-known to form coordination complexes with monodentate ligands[1]. The stoichiometry of the compounds appears to be a function of the donor properties and steric requirements of the coordinating group. Pyridine reacts with dimethylberyllium to yield a crystalline complex[2] involving two donor molecules, $(CH_3)_2Be \cdot 2NC_5H_5$. With trimethylamine both the 1/1 complex[3], $(CH_3)_2Be \cdot N(CH_3)_3$, and the 1/2 complex[4], $(CH_3)_2Be \cdot 2N(CH_3)_3$, have been reported. In each case, however, structural information on the nature of the bonding of the beryllium atom is lacking.

Toney and Stucky[5] have recently reported the preparation and structure of the 1/2 complex of dimethylmagnesium and 1-azabicyclo[2.2.2]octane: $(CH_3)_2Mg \cdot 2NC_7H_{13}$. About the magnesium atom, certain structural parameters show evidence of large steric interactions between the quinuclidine molecules and the methyl groups. It was therefore of interest to study the analogous beryllium compound in which such interactions must be even larger.

EXPERIMENTAL

Dimethylbis(quinuclidine)beryllium was prepared by reacting dimethylberyllium with quinuclidine (1-azabicyclo[2.2.2]octane) in benzene. The solution was

(continued on p. 20)

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TABLE 1

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	OBS	CAL
******L= 0******			
2	0	76.9	111.4
4	0	19.7	20.8
6	0	24.1	27.4
8	0	12.5	13.8
9	0	14.1	15.5
3	2	72.5	47.5
0	4	31.4	11.8
3	4	4.1	8.4
1	1	52.1	51.1
1	2	64.7	64.4
1	3	15.7	13.1
1	4	12.4	14.1
1	7	15.7	13.1
1	9	22.1	23.5
2	1	51.7	65.4
2	2	62.2	53.8
2	4	26.1	26.2
2	6	11.1	14.1
2	7	14.9	18.1
2	8	11.8	18.0
2	9	11.7	11.5
3	1	21.2	14.0
3	4	54.0	54.6
3	7	11.7	13.2
3	10	16.8	20.8
4	4	15.1	11.8
4	5	5.9	7.5
4	6	17.8	18.1
4	7	8.1	10.9
4	8	15.8	17.6
5	4	16.5	22.1
5	5	10.8	8.2
5	6	20.8	28.8
6	1	20.0	22.5
6	4	11.1	18.4
6	5	20.3	14.3
6	6	28.8	24.9
6	8	18.4	17.4
7	3	16.1	16.1
7	4	10.9	12.5
7	7	11.5	17.2
2	5	3.5	4.8
1	1	3.4	3.7
3	2	5.1	7.4
3	11	7.9	12.5
2	11	7.4	8.5
7	2	4.4	1.8
8	1	11.4	11.4
5	3	37.1	34.6
4	3	33.7	24.9
4	2	20.9	21.5
4	1	31.6	31.1
3	5	11.7	11.2
2	3	32.9	13.2
5	2	8.4	3.5
******L= 1******			
1	10	16.8	11.3
-1	10	17.7	19.5
-2	10	11.1	10.7
-4	10	12.7	10.4
1	1	31.1	27.1
-2	1	11.0	8.4
-3	1	56.9	48.1
3	1	17.3	15.4
-4	1	82.6	85.7
-5	1	56.1	48.2
5	1	15.0	12.6
-6	1	20.6	19.6

H	K	OBS	CAL
-6	5	19.3	18.5
6	5	18.2	11.7
-7	5	23.7	18.5
7	5	10.7	10.4
-8	5	22.1	17.0
-9	5	11.6	11.0
0	6	14.5	11.1
-2	6	15.7	7.4
2	6	25.1	21.6
-4	6	14.4	16.8
-5	6	14.4	17.1
5	6	11.1	15.9
-6	6	14.1	17.1
6	6	11.7	8.8
1	7	3.2	4.7
-1	7	17.1	15.6
-2	7	3.4	3.1
-3	7	7.4	11.7
-4	7	11.4	4.6
4	7	15.2	14.6
-5	7	14.7	12.5
6	7	11.4	10.8
7	7	11.2	10.4
1	8	11.4	15.1
-2	8	16.4	17.2
-3	8	8.4	6.4
-5	8	4.4	5.1
-7	8	13.6	13.4
0	9	9.6	10.1
1	9	7.1	7.1
-3	9	8.0	11.2
-4	9	4.6	14.5
4	9	13.2	14.1
6	9	4.0	8.2
******L= 2******			
-10	0	12.9	9.9
0	10	12.5	12.8
-1	10	12.0	12.4
1	0	30.4	27.5
2	0	26.7	21.5
-3	0	48.3	44.7
-3	10	15.2	17.6
3	-10	11.2	11.2
4	0	40.3	41.9
-5	0	66.7	68.9
5	0	22.3	24.2
-7	0	8.2	4.8
7	0	21.8	19.7
8	0	12.2	9.9
-9	0	18.8	19.7
9	0	15.0	14.9
10	1	6.9	6.8
1	11	9.8	11.2
1	1	43.9	42.6
2	11	12.0	16.4
2	1	21.5	17.4
-3	1	6.6	5.3
3	11	7.4	11.3
-4	1	55.8	52.1
4	1	12.2	14.2
-4	11	9.5	12.8
-5	1	31.4	27.6
5	1	5.8	8.1
-6	1	21.1	19.1
-6	11	7.9	8.1
-7	1	38.7	33.4
-7	11	8.9	7.4
8	1	12.2	12.1
-10	2	12.4	11.0

H	K	OBS	CAL
-1	6	28.0	10.1
2	6	7.3	8.4
3	6	9.3	8.8
-3	6	9.1	8.4
-4	6	9.6	10.3
4	6	13.7	13.5
10	7	7.0	6.8
0	7	17.9	17.6
1	7	6.1	5.5
2	7	14.8	16.8
-2	7	21.4	23.8
-3	7	5.9	6.2
3	7	10.5	9.6
-4	7	10.4	7.9
5	7	9.3	11.7
6	7	11.0	11.7
-7	7	21.7	25.1
-9	7	10.4	13.8
0	8	9.9	11.5
-1	8	12.0	11.6
1	8	9.5	5.3
-2	8	21.2	24.1
2	8	11.5	5.1
-3	8	21.8	23.9
3	8	7.2	10.5
4	8	7.0	6.5
5	8	7.3	10.2
6	8	12.4	14.6
-9	8	7.2	7.1
1	9	13.6	15.1
-1	9	13.4	10.6
2	9	14.6	18.2
-3	9	14.4	14.7
3	9	9.0	10.2
-6	9	7.4	7.0
-9	9	6.0	4.8
******L= 3******			
0	10	10.0	9.0
-1	10	10.2	14.9
1	10	22.0	19.1
-2	10	10.6	11.1
0	1	14.2	13.5
1	1	52.9	50.0
2	1	8.5	7.2
-3	1	8.9	10.0
-4	1	25.8	22.6
4	1	10.0	12.8
-5	1	58.6	55.1
-6	1	23.5	15.0
6	1	13.7	14.1
-7	1	21.2	16.3
-8	1	9.4	5.7
-9	1	13.7	9.9
-10	2	19.4	17.0
0	2	8.2	17.5
-1	2	9.3	17.1
1	2	12.4	4.5
-2	2	20.1	17.7
2	2	8.2	13.5
-3	2	11.9	10.4
3	2	34.8	32.2
-4	2	7.1	9.6
-5	2	31.3	30.6
5	2	17.8	19.5
-6	2	14.0	11.6
6	2	13.0	14.1
-8	2	9.5	8.6
8	2	22.5	23.9
-9	2	16.6	13.4

H	K	OBS	CAL
0	1	7.2	1.6
1	1	6.5	11.7
-2	1	26.0	21.9
2	1	27.8	24.5
-3	1	42.5	41.0
3	1	14.8	14.9
-4	1	7.2	5.5
-8	11	17.0	17.9
-5	1	30.7	31.0
5	1	13.0	14.1
6	1	9.4	7.5
-7	1	27.5	23.6
-8	1	11.1	9.8
0	2	8.5	14.4
1	2	24.9	24.5
-1	2	43.8	40.3
-2	2	38.0	33.5
2	2	30.0	34.1
-3	2	11.1	8.7
3	2	6.9	7.9
-4	2	31.9	29.7
-5	2	27.8	27.7
5	2	8.9	6.8
-6	2	18.9	11.8
6	2	15.5	13.1
-8	2	24.8	25.4
-10	3	17.9	18.1
0	3	20.3	23.6
-1	3	19.0	22.8
2	3	12.3	10.4
-3	3	9.7	5.7
-4	3	8.9	5.9
-5	3	10.9	8.7
5	3	9.1	10.6
-6	3	13.5	10.7
0	4	17.5	19.1
-1	4	8.2	1.2
1	4	19.3	16.0
-2	4	36.6	36.7
-3	4	11.4	14.6
3	4	17.2	21.3
-4	4	29.9	28.6
-5	4	25.6	23.1
5	4	15.0	15.1
-6	4	12.6	13.4
6	4	12.8	9.3
-7	4	8.6	4.6
0	5	11.9	13.8
2	5	7.1	10.9
3	5	5.6	2.5
4	5	11.5	11.8
-5	5	21.6	19.7
-8	5	20.8	20.8
-10	5	13.3	11.6
2	6	12.7	15.6
-2	6	12.5	12.4
3	6	17.2	18.7
-4	6	9.0	7.1
-7	6	9.4	8.2
0	7	9.5	6.2
-1	7	8.3	7.2
1	7	16.6	19.7
2	7	14.3	13.4
-3	7	15.9	16.9
-6	7	13.3	10.9
-7	7	27.4	27.4
0	8	10.2	11.3
-1	8	11.9	11.1
1	8	29.1	28.6
-2	8	17.3	13.3

H	K	OBS	CAL
4	7	13.3	12.1
5	7	13.5	7.2
-6	7	11.2	6.7
2	8	12.9	3.8
1	9	11.7	11.5
-5	9	11.7	10.8
-2	9	11.5	8.5
******L= 4******			
0	0	22.0	22.8
1	0	10.7	5.0
-3	0	24.7	32.2
3	0	16.4	16.9
-4	0	35.4	35.6
4	0	9.5	8.0
-5	0	19.2	17.4
5	0	16.1	14.3
-6	0	13.4	15.7
6	0	13.4	13.4
-8	0	16.8	21.0
0	1	17.6	16.9
-1	1	28.4	30.2
1	1	9.8	6.8
-2	1	17.4	21.8
-4	1	20.9	21.2
5	1	10.5	8.2
-7	1	10.0	8.0
-9	1	23.3	20.5
0	2	33.2	33.2
1	2	7.8	10.2
-2	2	40.3	45.5
2	2	9.1	7.2
-3	2	26.4	18.8
3	2	8.6	6.3
-7	2	12.6	11.7
-8	2	11.1	10.6
0	3	18.3	12.9
1	3	17.1	11.1
-1	3	8.1	9.5
-2	3	10.3	7.9
-3	3	7.7	6.1
3	3	9.1	9.2
4	3	12.4	10.6
-6	3	29.6	32.3
-7	3	10.6	3.1
0	4	16.9	16.3
-1	4	7.7	8.0
4	4	10.4	9.2
-5	4	14.7	11.7
-6	4	22.1	21.3
-7	4	13.0	11.2
-8	4	10.8	9.2
-1	5	13.4	10.8
2	5	16.6	13.7
-4	5	19.3	32.8
-7	5	22.4	16.1
-9	5	15.5	11.6
0	6	8.8	5.9
1	6	13.0	17.4
-4	6	13.2	9.0
-6	6	14.2	8.3
-7	6	24.3	23.6
-8	6	14.6	13.1
-9	6	14.8	11.7
0	7	9.7	8.4
1	7	19.9	19.6
-2	7	11.1	12.0
3	7	12.4	13.8
-3	7	11.5	13.0
-6	7	14.9	10.7

6	1	9.6	10.8
-7	1	14.1	13.0
7	1	14.4	12.9
-8	1	10.2	10.6
2	1	16.8	16.1
8	1	17.5	17.6
-10	2	11.4	11.0
0	2	96.1	107.1
-1	12	4.0	7.1
-1	2	97.8	106.0
1	2	77.2	83.5
-2	12	9.0	10.9
1	12	7.8	7.2
-2	2	23.8	20.4
2	2	20.2	22.0
-3	2	6.9	5.7
3	2	40.4	38.0
-4	2	13.3	15.0
4	-2	7.8	5.9
-5	2	22.8	25.8
5	2	7.9	12.1
-6	2	20.1	19.8
6	2	7.0	8.4
-7	2	19.8	21.4
8	2	21.3	19.9
-9	2	11.1	14.1
-10	3	13.6	13.3
0	3	3.5	1.1
-1	3	52.6	51.2
1	3	16.2	21.3
-2	3	11.3	11.0
2	3	8.6	10.5
-3	3	26.3	18.0
3	3	83.9	85.2
-4	3	15.6	16.7
4	3	24.1	22.9
-5	3	9.8	6.7
5	3	21.5	19.3
-6	3	18.2	11.0
6	3	18.3	16.2
7	3	12.6	9.8
-8	3	20.7	22.9
8	3	21.6	23.8
0	4	27.1	30.3
10	3	16.6	16.9
1	4	30.2	29.5
-1	4	32.2	30.5
-2	4	23.6	23.9
2	4	42.1	43.7
-3	4	18.3	18.0
3	4	10.0	10.8
-4	4	34.5	35.8
4	4	12.9	11.5
5	4	10.4	10.8
6	4	7.5	10.4
-8	4	7.6	6.0
-1	5	3.5	2.0
0	5	9.8	5.6
8	4	8.7	10.6
2	5	55.6	59.2
-2	5	31.5	29.2
-3	5	11.6	11.3
1	5	26.7	26.1
3	5	30.1	26.6
4	5	21.3	23.2
-5	5	13.5	16.4
0	2	9.8	14.2
0	12	11.2	10.4
-1	2	6.1	5.9
1	2	20.1	23.6
-2	2	9.4	16.1
2	2	39.9	35.1
-3	2	11.8	11.4
3	2	15.4	13.0
-3	12	9.3	11.1
-4	2	19.1	19.6
4	2	6.1	8.4
4	12	4.9	12.8
-4	12	9.2	10.2
-5	2	32.4	31.2
-5	12	7.0	8.0
5	2	10.6	12.4
-6	2	21.0	22.4
-7	2	16.0	16.5
7	2	11.4	10.2
-8	2	26.6	22.0
-9	2	13.6	12.9
0	3	21.0	14.5
-1	3	28.4	22.1
1	3	33.6	36.2
-2	3	39.0	37.0
2	3	9.1	5.1
-3	3	45.2	46.6
3	3	8.0	11.6
3	13	6.3	8.1
-4	3	23.4	25.9
4	3	8.6	8.5
-5	3	89.1	87.7
-6	3	5.5	5.0
6	3	6.8	3.9
-7	3	26.3	25.1
7	3	9.4	8.0
8	3	7.9	5.3
9	3	7.1	7.2
-1	4	33.1	29.2
-11	4	7.1	4.6
1	4	13.1	6.5
2	4	18.3	15.1
-3	4	7.5	8.1
3	4	25.9	25.3
-4	4	26.9	22.2
4	4	7.3	8.1
-5	4	18.2	19.5
5	4	18.1	18.5
-6	4	19.8	20.1
6	4	13.2	16.0
-7	4	7.1	9.8
-9	4	15.2	12.2
0	5	39.9	39.2
1	5	16.7	19.4
-2	5	13.3	15.7
-3	5	41.4	43.3
3	5	8.6	7.7
-4	5	22.8	22.6
4	5	11.7	9.6
-5	5	30.6	32.6
5	5	10.7	8.8
-6	5	10.8	9.7
6	5	5.6	2.9
-7	5	6.6	7.8
7	5	12.2	10.5
8	5	7.1	8.0
-9	5	12.3	12.2
0	6	8.7	11.5
0	3	30.4	24.3
-1	3	18.7	15.9
2	3	16.4	20.0
-3	3	16.7	14.8
-4	3	11.9	9.1
4	3	13.5	12.0
-5	3	16.1	14.3
-7	3	24.8	19.7
7	3	21.7	18.4
-8	3	15.7	18.1
0	4	14.6	17.5
1	4	35.8	40.9
2	4	18.1	19.4
-2	4	11.4	8.9
-3	4	24.2	25.1
3	4	25.8	27.0
-4	4	8.2	8.3
5	4	15.7	15.9
6	4	10.9	11.3
-8	4	17.2	16.3
8	4	11.8	13.2
0	5	7.1	8.0
1	5	45.1	40.2
-1	5	21.8	25.4
2	5	7.0	11.5
3	5	12.4	12.1
-4	5	22.1	20.3
4	5	18.4	19.2
-5	5	8.3	10.7
6	5	17.6	16.4
-7	5	26.6	23.5
-8	5	14.8	16.2
-9	5	18.5	11.4
0	6	13.6	17.3
2	6	14.0	14.8
3	6	11.2	8.8
-3	6	15.7	15.4
5	6	22.5	21.7
7	6	10.3	9.6
1	7	9.2	11.5
-1	7	16.9	15.5
-2	7	10.4	9.8
-6	7	15.6	15.6
-9	7	10.8	11.0
0	8	19.8	22.8
-1	8	20.7	19.0
-2	8	9.8	10.8
3	8	10.2	7.4
-4	8	15.6	16.9
5	8	10.8	11.9
-5	8	6.6	5.4
6	8	10.3	12.4
-7	8	14.5	11.5
1	9	9.5	5.2
-2	9	22.0	20.3

******L= 4******

0	10	11.3	12.0
0	0	20.2	23.6
1	10	13.3	14.1
1	0	17.2	10.5
-4	0	11.5	9.4
4	0	14.7	11.0
-5	0	21.5	20.7
-7	0	14.0	8.6
8	0	22.4	25.2
-9	0	10.6	6.1
-10	1	13.2	8.7
-4	8	9.3	7.6
-5	8	12.3	13.1
-6	8	13.0	11.5
-1	9	9.8	11.0
-2	9	15.2	20.7
-3	9	12.4	12.1
-5	9	10.3	9.6

******L= 5******

0	10	11.8	14.0
0	1	8.6	2.5
-1	1	3.3	8.6
1	1	15.6	16.1
-2	1	23.7	23.0
-3	1	19.9	20.3
3	1	14.4	16.3
-5	1	14.8	15.8
5	1	9.6	4.9
-6	1	16.2	20.9
-7	1	14.5	11.4
7	1	13.5	11.8
0	2	9.4	5.7
-1	2	31.7	32.2
-3	2	32.3	30.0
3	2	7.7	5.7
-4	2	4.3	6.6
4	2	8.7	10.8
-5	2	11.9	12.9
6	2	13.1	10.9
-7	2	9.5	0.8
0	3	14.8	12.7
-1	3	16.9	17.9
1	3	19.6	22.5
-2	3	26.3	23.1
2	3	11.0	11.1
-4	3	14.6	15.4
-5	3	15.9	14.6
-6	3	14.0	17.6
-7	3	15.2	18.4
7	3	10.9	13.3
-9	3	19.0	18.1
0	4	32.6	32.7
-1	4	15.0	19.8
1	4	8.6	7.2
-2	4	21.3	20.1
2	4	14.9	13.9
-3	4	7.8	8.0
3	4	20.9	22.1
-4	4	10.2	7.3
5	4	19.3	15.9
0	5	13.2	15.3
1	5	30.4	32.6
3	5	11.4	7.9
4	5	23.2	22.1
-8	5	21.9	20.7
0	6	18.6	17.3
-1	6	9.4	9.1
1	6	24.5	22.2
-2	6	9.3	6.6
2	6	13.1	12.4
5	6	13.4	16.3
-7	6	11.2	8.7
2	7	12.1	9.9
-2	7	26.1	26.8
-3	7	10.2	8.9
3	7	12.8	12.3
-4	7	13.0	7.4
-7	7	10.8	8.5
-8	7	12.5	10.3
0	8	16.0	15.1
1	8	10.7	8.4
-2	8	14.7	11.3
2	8	11.0	9.9
-3	8	14.7	12.4
-7	8	12.5	10.4
-8	8	15.5	12.9

******L= 7******

-1	-10	10.0	8.9
-2	-10	10.0	10.4
0	1	7.0	3.7
1	-1	8.2	7.6
-3	-1	9.1	5.5
-4	-1	3.3	2.1
-5	1	26.8	25.8
-6	1	8.4	10.6
-7	-1	9.3	12.8
-8	1	14.3	12.4
-9	1	7.7	4.5
0	-2	3.5	1.1
-1	-2	40.0	37.0
1	2	19.9	23.5
2	-2	9.5	13.3
-2	-2	2.4	3.8
-3	-2	10.7	11.9
-4	-2	8.0	8.9
-6	2	21.2	20.1
-8	2	13.1	11.6
0	-3	14.7	18.8
-10	3	11.4	12.7
0	-3	18.6	14.8
1	-3	12.5	15.5
-1	-3	12.9	12.0
2	3	20.6	14.5
-2	-3	9.4	12.5
-3	-3	14.7	17.4
-4	-3	7.0	4.2
-5	-3	24.4	28.4
-6	3	22.6	21.9
-7	-3	12.9	12.8
-8	-3	18.1	15.1
-9	3	11.1	11.7
0	-4	30.3	31.1
-7	-4	13.0	15.3
-9	4	11.3	12.7
1	-5	29.6	27.3
-2	-5	6.7	2.3
-4	-5	12.6	7.8
-6	-5	13.7	14.0
-8	-5	9.9	10.6
0	-6	14.4	10.2
-2	-6	7.7	3.7
-3	-6	18.0	15.6
-4	-6	8.9	11.3
-2	-7	19.3	20.3
-3	-7	10.7	14.3
-4	-7	17.1	18.2
-7	-7	11.3	10.4
-3	-8	9.4	11.5
-4	-8	17.2	15.4
-1	-9	11.7	12.4
-2	-9	9.8	9.5
-4	-9	11.6	11.6
-5	-9	9.9	5.5

heated for an hour at 50°. Clear, colorless crystals of the air-sensitive compound were deposited upon cooling. All manipulations were carried out in an inert atmosphere box.

Preliminary Weissenberg (Cu $K\alpha$) and precession (Mo $K\alpha$) photographs of a needle-like crystal of $(CH_3)_2Be \cdot 2NC_7H_{13}$ showed the crystal system to be monoclinic. Systematic absences ($h0l$, $l \neq 2n$; $0k0$, $k \neq 2n$) uniquely determined the space group to be $P2_1/c$. The unit cell parameters are: $a = 11.82 \pm 0.02$ Å; $b = 12.71 \pm 0.02$ Å; $c = 12.00 \pm 0.02$ Å; $\beta = 113.1 \pm 0.3°$; $V = 1658$ Å^3. With $Z = 4$, the calculated density is 1.05 g/cm^3.

Eight layers of multiple-film equi-inclination Weissenberg data ($hk0$ to $hk7$) were obtained with Ni-filtered Cu $K\alpha$ radiation. 671 independent reflections were visually estimated by comparison with a calibrated intensity scale. Individual layers were scaled together by exposure time.

Calculations were made with an IBM 360/50 computer. Lorentz, polarization, and spot size corrections reduced the observed intensities to squared structure factors. The crystal had dimensions of 0.20 × 0.20 × 0.50 mm and a linear absorption coefficient of 4.45 cm^{-1} for Cu $K\alpha$ radiation. The maximum and minimum transmission factors differ by less than 2% and absorption corrections were not applied. Fourier calculations were made with the Argonne program of Gvildys[6]. Full-matrix least-squares refinement was carried out using the Busing and Levy program ORFLS[7]. The function $\Sigma\omega(|F_o|-|F_c|)^2$ was minimized. Neutral atom scattering factors were taken from the compilations of Ibers[8] for hydrogen, beryllium, carbon, and nitrogen. Final bond distances, angles, and errors were computed with the aid of the Busing, Martin, and Levy ORFFE program[9].

STRUCTURE DETERMINATION

The realization of the isomorphism of dimethylbis(quinuclidine)beryllium and dimethylbis(quinuclidine)magnesium allowed the direct solution of the structure.

The fractional coordinates of the non-hydrogen atoms given for the magnesium analogue afforded a discrepancy factor of $R_1 = [\Sigma||F_o| - |F_c||/\Sigma|F_o|] \times 100\% = 42\%$. Several cycles of difference Fourier refinement and least-squares refinement on the scale factors and positional parameters lowered R_1 to 24%. Refinement of isotropic temperature factors followed by positional parameter refinement reduced R_1 to 18.4%. At this point hydrogen atoms were placed on the quinuclidine molecules in calculated positions based on the geometry of the carbon–nitrogen–beryllium skeleton at a distance of 0.95 Å from the associated carbon atoms. After each successive cycle of refinement of atomic coordinates, the hydrogen atom positions were recalculated. This produced a final R_1 value of 13.9% and $R_2 = [\Sigma\omega(|F_o|-|F_c|)^2/\Sigma\omega|F_o|^2]^{\frac{1}{2}} \times 100\%$ of 13.2%. Anisotropic refinement of the structure did not produce significantly different results from the isotropic refinement. The final weighting scheme was an empirical one based on Cruickshank's criterion[10] that data should be weighted so as to make $\omega(|F_o|-|F_c|)^2$ constant over the entire range of $|F_o|$ values. The final cycle of refinement showed no parameter shift greater than 0.04 of one estimated standard deviation. A final difference Fourier map did not unambiguously reveal the location of the hydrogen atoms on the methyl groups and had no other feature greater than 0.5 e/Å^3. The estimated standard deviation for an observation of unit

TABLE 2

FINAL FRACTIONAL COORDINATES AND TEMPERATURE FACTORS

Atom	x/a	y/b	z/c	B
Be	0.2563(16)	0.0852(15)	0.2328(20)	2.45
C(15)	0.1840(14)	−0.0213(14)	0.1219(15)	5.29
C(16)	0.2848(13)	0.0619(11)	0.3901(14)	3.82
N(1)	0.1467(9)	0.2011(8)	0.1801(11)	1.91
N(2)	0.4132(8)	0.1199(8)	0.2304(10)	1.65
C(1)	0.1091(14)	0.2333(12)	0.0560(16)	3.99
C(2)	0.0078(16)	0.3189(15)	0.0114(18)	5.74
C(3)	0.0277(12)	0.1602(11)	0.1925(13)	2.87
C(4)	−0.0695(15)	0.2496(13)	0.1572(16)	4.96
C(5)	0.1850(14)	0.2950(13)	0.2530(16)	4.45
C(6)	0.0849(14)	0.3872(13)	0.2220(15)	4.44
C(7)	−0.0264(13)	0.3492(11)	0.1148(15)	3.44
C(8)	0.4820(16)	0.1955(15)	0.3144(16)	5.69
C(9)	0.6215(16)	0.2036(14)	0.3352(18)	6.27
C(10)	0.4132(21)	0.1473(18)	0.1147(24)	8.94
C(11)	0.5508(20)	0.1749(18)	0.1243(22)	8.88
C(12)	0.4797(14)	0.0209(13)	0.2610(16)	4.96
C(13)	0.6111(14)	0.0273(13)	0.2632(15)	4.44
C(14)	0.6381(13)	0.1381(12)	0.2406(16)	4.05
H1[C(15)]	0.1783	0.0074	0.0454	4.00
H2[C(15)]	0.2412	−0.0784	0.1410	4.00
H3[C(15)]	0.1102	−0.0390	0.1211	4.00
H4[C(16)]	0.3207	0.1221	0.4341	4.00
H5[C(16)]	0.2118	0.0432	0.3975	4.00
H6[C(16)]	0.3428	0.0039	0.4174	4.00
H7[C(1)]	0.1787	0.2588	0.0436	4.00
H8[C(1)]	0.0785	0.1718	0.0069	4.00
H9[C(2)]	−0.0648	0.2912	−0.0501	4.00
H10[C(2)]	0.0354	0.3780	−0.0165	4.00
H11[C(3)]	−0.0013	0.0999	0.1436	4.00
H12[C(3)]	0.0466	0.1410	0.2747	4.00
H13[C(4)]	−0.0949	0.2650	0.2180	4.00
H14[C(4)]	−0.1399	0.2261	0.0862	4.00
H15[C(5)]	0.2574	0.3201	0.2457	4.00
H16[C(5)]	0.2075	0.2724	0.3358	4.00
H17[C(6)]	0.1221	0.4404	0.2088	4.00
H18[C(6)]	0.0685	0.4001	0.2949	4.00
H19[C(7)]	−0.0858	0.4098	0.0958	4.00
H20[C(8)]	0.4459	0.2659	0.2793	4.00
H21[C(8)]	0.4754	0.1886	0.3861	4.00
H22[C(9)]	0.6679	0.1779	0.4109	4.00
H23[C(9)]	0.6433	0.2749	0.3270	4.00
H24[C(10)]	0.3627	0.2114	0.0833	4.00
H25[C(10)]	0.3819	0.0952	0.0553	4.00
H26[C(11)]	0.5640	0.1461	0.0581	4.00
H27[C(11)]	0.5576	0.2528	0.1177	4.00
H28[C(12)]	0.4371	−0.0334	0.2109	4.00
H29[C(12)]	0.4905	0.0033	0.3437	4.00
H30[C(13)]	0.6149	−0.0159	0.1990	4.00
H31[C(13)]	0.6713	0.0040	0.3364	4.00
H32[C(14)]	0.7203	0.1408	0.2426	4.00

TABLE 3

BOND DISTANCES FOR $(CH_3)_2Be\cdot(NC_7H_{13})_2$

Bond	Distance (Å)	Bond	Distance (Å)
Be–C(15)	1.85(3)	Be–N(1)	1.90(2)
Be–C(16)	1.81(3)	Be–N(2)	1.92(2)
N(1)–C(1)	1.44(2)	N(2)–C(8)	1.40(2)
N(1)–C(3)	1.56(2)	N(2)–C(10)	1.43(3)
N(1)–C(5)	1.44(2)	N(2)–C(12)	1.45(2)
C(1)–C(2)	1.55(2)	C(8)–C(9)	1.57(3)
C(3)–C(4)	1.55(2)	C(10–C(11)	1.62(3)
C(5)–C(6)	1.60(2)	C(12)–C(13)	1.55(2)
C(2)–C(7)	1.50(3)	C(9)–C(14)	1.48(3)
C(4)–C(7)	1.52(2)	C(11)–C(14)	1.45(3)
C(6)–C(7)	1.51(2)	C(13)–C(14)	1.49(2)
Nonbonded distances (Å)			
N(1)–N(2)	3.14(1)	N(2)–C(15)	3.08(2)
N(1)–C(15)	2.98(2)	N(2)–C(16)	2.97(2)
N(1)–C(16)	2.99(2)	C(15)–C(16)	3.14(2)
C(15)–C(1)	3.37(2)	C(16)–C(3)	3.28(2)
C(15)–C(3)	3.27(2)	C(16)–C(5)	3.37(2)
C(15)–C(10)	3.48(2)	C(16)–C(8)	3.29(2)
C(15)–C(12)	3.28(2)	C(16)–C(12)	3.29(2)
C(1)–C(10)	3.56(3)		

TABLE 4

BOND ANGLES FOR $(CH_3)_2Be\cdot(NC_7H_{13})_2$

Angle	Degrees (°)	Angle	Degrees (°)
C(15)–Be–C(16)	118.3(1.0)	N(1)–Be–N(2)	110.8(1.2)
C(15)–Be–N(1)	105.3(1.0)	C(16)–Be–N(2)	105.6(0.9)
C(16)–Be–N(1)	107.3(0.7)	C(15)–Be–N(2)	109.4(1.4)
Be–N(1)–C(1)	117.4(1.1)	Be–N(2)–C(8)	116.0(1.2)
Be–N(1)–C(3)	103.5(1.1)	Be–N(2)–C(10)	116.0(1.4)
Be–N(1)–C(5)	115.3(1.2)	Be–N(2)–C(12)	103.3(1.2)
N(1)–C(1)–C(2)	116.2(1.6)	N(2)–C(8)–C(9)	115.9(1.7)
N(1)–C(3)–C(4)	109.2(1.3)	N(2)–C(10)–C(11)	111.4(1.7)
N(1)–C(5)–C(6)	115.6(1.6)	N(2)–C(12)–C(13)	113.6(0.8)
C(1)–C(2)–C(7)	108.6(1.4)	C(8)–C(9)–C(14)	106.7(0.9)
C(3)–C(4)–C(7)	112.9(0.8)	C(10)–C(11)–C(14)	108.3(1.7)
C(5)–C(6)–C(7)	106.3(0.9)	C(12)–C(13)–C(14)	109.3(1.5)
C(1)–N(1)–C(3)	105.7(1.0)	C(8)–N(2)–C(10)	108.0(1.4)
C(1)–N(i)–C(5)	106.7(1.0)	C(8)–N(2)–C(12)	108.0(1.1)
C(3)–N(1)–C(5)	107.4(0.9)	C(10)–N(2)–C(12)	104.5(1.4)
C(2)–C(7)–C(4)	107.2(1.0)	C(11)–C(14)–C(13)	110.9(1.6)
C(2)–C(7)–C(6)	111.0(1.4)	C(9)–C(14)–C(11)	107.3(1.3)
C(4)–C(7)–C(6)	105.9(1.1)	C(9)–C(14)–C(13)	106.8(1.0)

weight after the last cycle of refinement was 2.47. The final calculated and observed structure factors are listed in Table 1. Final atomic parameters and standard deviations are tabulated in Table 2. Interatomic distances, angles, and errors are listed in Tables 3 and 4.

DISCUSSION OF STRUCTURE

The structure consists of monomeric $(CH_3)_2Be \cdot 2NC_7H_{13}$ molecules (Fig. 1) in which the coordination about the beryllium atom is distorted tetrahedral. The average beryllium–carbon bond distance (1.83 Å) is short compared to the beryllium–carbon distance (1.93 Å) in dimethylberyllium[11]. This reflects the difference between a

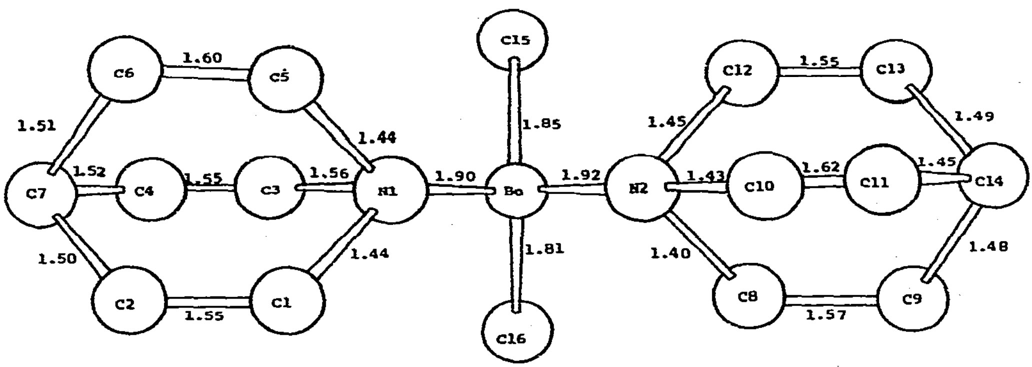

Fig. 1. Structure of dimethylbis(quinuclidine)beryllium.

terminal methyl and a bridge methyl bond length, and is in keeping with the results of structural studies on aluminum compounds[12,13]. The value agrees favorably with the beryllium–carbon length of 1.80 Å reported[14] in $[NaO(C_2H_5)_2] \cdot [(C_2H_5)_4Be_2H_2]$. Both are, however, significantly larger than the 1.70 Å beryllium–carbon length found from an electron diffraction study of di-tert-butyl beryllium[13]. The average beryllium–nitrogen bond length of 1.91 Å is quite long with reference to those found in bis-(dimethylamino)beryllium where[15] the beryllium–nitrogen distance involving both four-coordinate species is 1.78 Å.

The most important structural feature of the molecule is the large C(15)–Be–C(16) bond angle (118°) compared to the N(1)–Be–N(2) bond angle (111°). It is most easily explained in terms of the relatively short beryllium–carbon distance and the resulting demand placed on the methyl–methyl approach. The C(15)–C(16) distance is 3.14 Å with the 118° angle. Methyl–methyl distances of the same magnitude (3.12 Å) are found in bis(dimethylamino)beryllium and are believed to be reason the substance is trimeric rather than polymeric[15]. If the C(15)–Be–C(16) bond angle were to approach 109°, and the Be–C bond length were to remain 1.83 Å, the C(15)–C(16) nonbonded distance would be much smaller than even 3.14 Å. Thus, steric factors alone are sufficient to explain the carbon–beryllium–carbon bond angle.

The same reasoning does not apply to the magnesium analogue (where the carbon–magnesium–carbon bond angle is 129°). Table 5 contains a comparison of

TABLE 5

COMPARISON OF SHORTEST NONBONDED DISTANCES (Å)

Distance	$(CH_3)_2Be \cdot 2NC_7H_{13}$	$(CH_3)_2Mg \cdot 2NC_7H_{13}$
Methyl–methyl	3.14	3.96
Methyl–methylene	3.27	3.58
Methylene–methylene	3.56	3.88

the nonbonded distances in the two compounds. Methyl–methyl repulsions must now be of minor importance. Toney and Stucky propose two possible explanations[5]. The first entails a forcing apart of the methyl groups by interactions with the methylene groups on the quinuclidine molecules. The second is based on a possible difference in *s* and *p* character in the magnesium bonds to carbon and nitrogen. While the methyl–methylene interactions are not severe in the magnesium compound compared to the beryllium compound, they may be a contributory factor. The secondary hybridization argument seems less likely in view of the magnitude of the electronegativity difference in reference to the large deviation of the carbon–magnesium–carbon angle from the tetrahedral value. Certainly, the results of the $(CH_3)_2Be \cdot 2NC_7H_{13}$ structure do not support the idea that electronic effects are of primary importance in determining the geometry about the metal atom. A further consideration that has received no attention is the possibility that the origin of the distortion might be based on interactions between molecules in the lattice. The final explanation of the carbon–magnesium–carbon bond angle will probably be found in a weighted combination of steric, electronic, and lattice effects.

In the quinuclidine molecules the average nitrogen–carbon distance (1.45 Å), carbon–carbon distance (1.57 Å), carbon–terminal carbon distance (1.49 Å), and the symmetry of the cage are not significantly different from those in the magnesium analoque[5].

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