1·841 (4) Å [cf. the mean aryl C−P distance of 1·828 (1) Å for a wide range of structures (Domenicano, Vaciago & Coulson, 1975)], while the C(116)−C(111)−C(112) angle in the phosphido ligand [119·1 (4)°] is only slightly larger than the corresponding angles in the PPh₃ ligand, mean = 118·7° [cf. the mean α angle in the PPh₃ group of 118·5 (1)° (Domenicano et al., 1975)].

The μ₃-P atom lies −1·55 (1) Å from the Ru₃ plane with both the triphenylphosphine P atom and the hydride ligands on the opposite side of this plane at distances of 2·02 (2), 0·86 (7) and 0·82 (7) Å [for P(2), H(1) and H(2) respectively].

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Structure of Bromo(ethyl)[(−)-α-isosparteine]magnesium(II)

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Abstract. MgBr(C₂H₅)(C₁₅H₂₆N₂), C₁₇H₃₁BrMgN₂, $M_r = 367·7$, orthorhombic, $P2_12_12_1$, $a = 10·646$ (4), $b = 14·491$ (4), $c = 11·857$ (3) Å, $Z = 4$, $D_x = 1·34$ Mg m⁻³, $R = 0·074$ for 1235 non-zero reflexions. The Mg atom is tetrahedrally coordinated by the C atom of the ethyl group, the Br atom and the two N atoms of the (−)-α-isosparteine. The distortion from tetrahedral geometry of the Mg atom results from repulsion between (−)-α-isosparteine and the other ligands.

Introduction. During the study of the asymmetric selective polymerization of racemic methacrylates by Grignard reagent–(−)-sparteine complexes, catalyti-cally active EtMgBr–(−)-sparteine and unreactive EtMgBr–(−)-α-isosparteine were isolated as single crystals (Okamoto, Suzuki & Yuki, 1980; Okamoto, Suzuki, Yuki, Kageyama, Miki, Tanaka & Kasai, 1980). A prismatic colourless crystal of EtMgBr–(−)-α-isosparteine, 0·55 × 0·38 × 0·30 mm, was sealed in a glass capillary tube under nitrogen atmosphere. A Rigaku automated four-circle diffractometer was used with graphite-monochromatized Mo Kα radiation ($\lambda = 0·7107$ Å). Systematic absences of $h00$ for $h = 2n + 1$, $0k0$ for $k = 2n + 1$, and $00l$ for $l = 2n + 1$ indicated the space group to be $P2_12_12_1$. Reflexion intensities ($2\theta \leq 50·5°$) were measured by the θ–2θ scan technique, the 2θ scan rate being 4° min⁻¹ and the scan width $\Delta 2\theta = (2·4 + 0·7 \tan \theta)°$. Backgrounds were counted for

Table 1. *Fractional atomic coordinates with e.s.d.'s in parentheses*

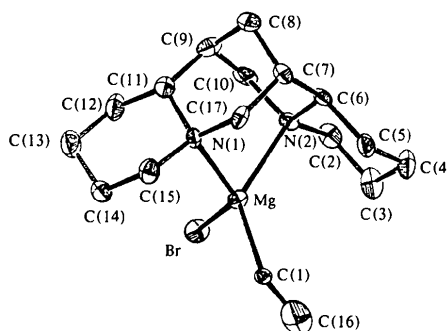|	x	y	z	B_{eq} (Å²)*
Br	0·01848 (17)	0·04953 (11)	0·07701 (14)	5·1
Mg	0·0110 (5)	−0·1001 (3)	0·1827 (4)	3·5
N(1)	−0·1161 (10)	−0·1246 (8)	0·3221 (10)	3·3
N(2)	0·1490 (11)	−0·0749 (8)	0·3167 (10)	3·6
C(1)	−0·0094 (12)	−0·2206 (9)	0·0680 (11)	3·4
C(2)	0·2708 (13)	−0·0437 (12)	0·2696 (13)	4·8
C(3)	0·3221 (16)	−0·1173 (12)	0·1921 (17)	6·4
C(4)	0·3404 (16)	−0·2086 (13)	0·2542 (16)	6·5
C(5)	0·2223 (14)	−0·2362 (10)	0·3099 (15)	4·8
C(6)	0·1693 (13)	−0·1595 (10)	0·3894 (11)	3·8
C(7)	0·0549 (13)	−0·1850 (10)	0·4530 (12)	4·1
C(8)	0·0235 (18)	−0·1097 (12)	0·5375 (13)	5·6
C(9)	−0·0120 (18)	−0·0250 (11)	0·4664 (12)	5·5
C(10)	0·0966 (16)	0·0035 (11)	0·3907 (13)	5·0
C(11)	−0·1341 (14)	−0·0476 (11)	0·4034 (12)	4·6
C(12)	−0·1939 (16)	0·0374 (13)	0·3437 (16)	6·4
C(13)	−0·3178 (17)	0·0107 (14)	0·2885 (16)	6·8
C(14)	−0·3002 (16)	−0·0722 (12)	0·2173 (14)	5·2
C(15)	−0·2410 (15)	−0·1514 (12)	0·2752 (15)	5·2
C(16)	0·0752 (23)	−0·2166 (15)	−0·0211 (25)	11·0
C(17)	−0·0621 (13)	−0·2061 (10)	0·3823 (13)	4·2

* After Hamilton (1959).

Fig. 1. A perspective view of the molecule with the atom numbering. Thermal ellipsoids are drawn at 20% probability level (*ORTEP* II: Johnson, 1976). H atoms are omitted for clarity.
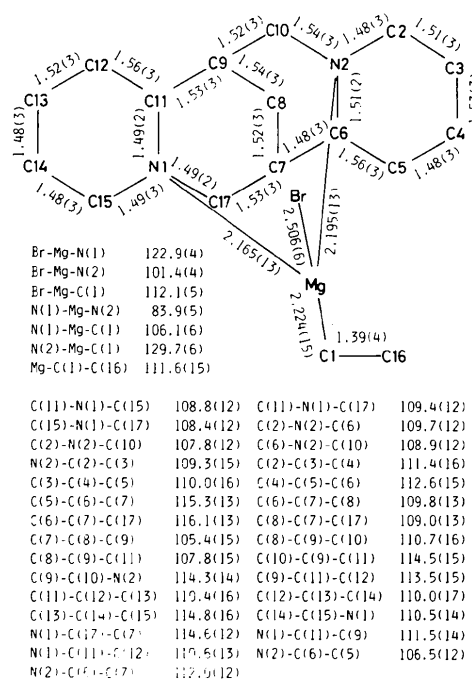

Br-Mg-N(1)   122.9(4)
Br-Mg-N(2)   101.4(4)
Br-Mg-C(1)   112.1(5)
N(1)-Mg-N(2)   83.9(5)
N(1)-Mg-C(1)   106.1(6)
N(2)-Mg-C(1)   129.7(6)
Mg-C(1)-C(16)   111.6(15)

C(11)-N(1)-C(15)	108.8(12)	C(11)-N(1)-C(17)	109.4(12)
C(15)-N(1)-C(17)	108.4(12)	C(2)-N(2)-C(6)	109.7(12)
C(2)-N(2)-C(10)	107.8(12)	C(6)-N(2)-C(10)	108.9(12)
N(2)-C(2)-C(3)	109.3(15)	C(2)-C(3)-C(4)	111.4(16)
C(3)-C(4)-C(5)	110.0(16)	C(4)-C(5)-C(6)	112.6(15)
C(5)-C(6)-C(7)	115.3(13)	C(6)-C(7)-C(8)	109.8(13)
C(6)-C(7)-C(17)	116.1(13)	C(8)-C(7)-C(17)	109.0(13)
C(7)-C(8)-C(9)	105.4(15)	C(8)-C(9)-C(10)	110.7(16)
C(8)-C(9)-C(11)	107.8(15)	C(10)-C(9)-C(11)	114.5(15)
C(9)-C(10)-N(2)	114.3(14)	C(9)-C(11)-C(12)	113.5(15)
C(11)-C(12)-C(13)	110.4(16)	C(12)-C(13)-C(14)	110.0(17)
C(13)-C(14)-C(15)	114.8(16)	C(14)-C(15)-N(1)	110.5(14)
N(1)-C(17)-C(7)	114.6(12)	N(1)-C(11)-C(9)	111.5(14)
N(1)-C(11)-C(12)	110.5(13)	N(2)-C(6)-C(5)	106.5(12)
N(2)-C(11)-C(7)	112.0(12)		

Fig. 2. Selected bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses.

7·5 s at both ends of a scan. Four standard reflexions measured after every 60 reflexions showed less than 5% intensity fluctuation. Usual Lp corrections were applied but absorption and extinction corrections were ignored [μ(Mo $K\alpha$) = 2·41 mm⁻¹]. Of the 1914 reflexions measured 1235 had non-zero intensities [$|F| \geq 3\sigma(F)$]. The structure was solved by the heavy-atom method, and refined anisotropically by the block-diagonal least-squares procedure (*HBLS* V: Ashida, 1973). H atoms were located on a difference Fourier map but some of them could not be refined well. Locations of the H atoms were therefore fixed at the calculated positions (C—H = 1·08 Å and B = 4·0 Å²). In the final cycles of the refinement anomalous dispersion of all the non-hydrogen atoms was considered. The final R value was 0·074 (R_w = 0·078). Atomic scattering factors and anomalous-dispersion coefficients were taken from *International Tables for X-ray Crystallography* (1974). The computations were carried out on an ACOS 700S computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University. The final atomic positional parameters are listed in Table 1.*

Discussion. A perspective view of the molecule with the atom numbering scheme is presented in Fig. 1. Selected

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bond lengths and angles are given in Fig. 2. The special feature of the molecular structure is that the (−)-α-isosparteine moiety has a V-shaped folded-ribbon structure, the Mg atom coordinated to the two N atoms of the (−)-α-isosparteine locates on the V, and the bulky Br atom blocks the upper space of the Mg atom from the substrate. This may cause the low reactivity of the present complex in the polymerization of α-methyl-benzyl methacrylate. The Mg—N bond lengths are equal [2·165 (13) and 2·195 (13) Å]. The N—Mg—N angle [83·9 (5)°] is smaller than the tetrahedral angle because the N atoms are fixed in the rigid (−)-α-isosparteine skeleton. The Mg—C(1) bond length [2·224 (15) Å] is slightly longer than those in the other
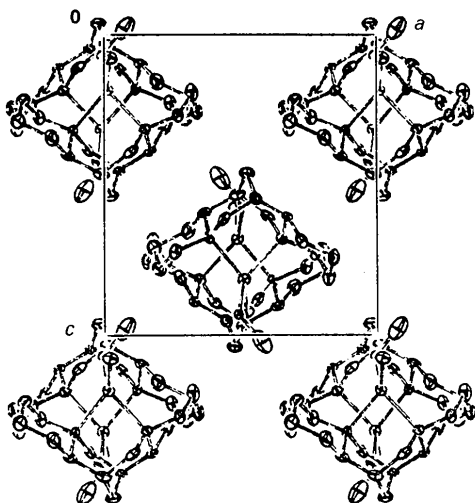
Fig. 3. Molecular packing in a unit cell (*ORTEP* II: Johnson, 1976).

Grignard reagent–ligand systems [2·09–2·15 Å]. This suggests that the Mg–C(1) bond is probably weakened by the coordination of the Mg atom to the two N atoms and also by the steric repulsion between the Et group

and the (—)-α-isosparteine moiety. The dihedral angle between the plane defined by the C(1), Mg, and Br atoms and that by the N(1), Mg, and N(2) atoms is 71·5 (5)°. The Grignard reagent often takes polymeric structures in the crystal but the present complex has a monomeric structure as shown in Fig. 3. The closest intermolecular atomic contact between non-hydrogen atoms is 3·60 (2) Å [C(17)$(x,y,z)\cdots$Br$(-x, -0·5 + y, 0·5 - z)$].

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