Table IV. Selected Interbond Angles (deg) for $[Rh_2(MeN(P(OMe)_2)_2)_4(\mu\text{-}MeO_2CC{=}CCO_2Me)](ClO_4)_2\cdot MeOH$[a]

Rh2–Rh1–P1	86.58 (3)	P6–Rh2–P7	96.79 (5)
Rh2–Rh1–P2	92.39 (3)	P6–Rh2–P8	88.90 (5)
Rh2–Rh1–P3	169.49 (4)	P6–Rh2–C6	159.3 (1)
Rh2–Rh1–P4	108.09 (3)	P7–Rh2–P8	68.58 (5)
Rh2–Rh1–C5	70.30 (8)	P7–Rh2–C6	103.7 (1)
P1–Rh1–P2	94.80 (5)	P8–Rh2–C6	96.3 (1)
P1–Rh1–P3	96.54 (5)	Rh1–P1–N1	117.9 (2)
P1–Rh1–P4	163.71 (5)	Rh1–P2–N2	115.8 (2)
P1–Rh1–C5	91.1 (1)	Rh1–P3–N3	96.8 (2)
P2–Rh1–P3	97.33 (5)	Rh1–P4–N3	93.4 (2)
P2–Rh1–P4	91.70 (5)	Rh2–P5–N1	114.9 (2)
P2–Rh1–C5	161.4 (1)	Rh2–P6–N2	116.4 (2)
P3–Rh1–P4	67.78 (5)	Rh2–P7–N4	94.5 (2)
P3–Rh1–C5	99.6 (1)	Rh2–P8–N4	95.2 (2)
P4–Rh1–C5	87.4 (1)	P1–N1–P5	115.6 (2)
Rh1–Rh2–P5	93.71 (3)	P2–N2–P6	119.2 (3)
Rh1–Rh2–P6	90.21 (4)	P3–N3–P4	102.1 (2)
Rh1–Rh2–P7	163.52 (4)	P7–N4–P8	101.7 (2)
Rh1–Rh2–P8	96.77 (4)	Rh1–C5–C6	108.4 (3)
Rh1–Rh2–C6	69.34 (8)	Rh1–C5–C7	127.8 (4)
P5–Rh2–P6	97.06 (5)	C6–C5–C7	123.8 (5)
P5–Rh2–P7	100.17 (5)	Rh2–C6–C5	111.6 (3)
P5–Rh2–P8	167.92 (5)	Rh2–C6–C8	125.4 (3)
P5–Rh2–C6	81.8 (1)	C5–C6–C8	123.0 (4)

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

The crystal structure of $[Rh_2(PNP)_2(\mu\text{-}PNP)_2(\mu\text{-}DMAD)](ClO_4)_2\cdot MeOH$ consists of discrete bimetallic cations interspersed with perchlorate anions and solvent methanol molecules. There are no unusually short intermolecular contacts. Selected bond distances and interbond angles are presented in Table III and IV, respectively, while additional metrical parameters are included as supplementary material.

Discussion

As can be seen from the figures and the scheme, the binding of DMAD to $[Rh(PNP)_2(\mu\text{-}PNP)_2]^{2+}$ to form the title compound causes one end of each chelating ligand (here P3 and P7) to swing away from the center of the cation by ca. 90°[5] and the metal-metal distance to decrease from 3.2727 (5) Å in the parent to 2.7595 (5) Å in the adduct. The latter distance compares well with that found in $[Rh_2Cl_2(\mu\text{-}HFB)(DPPM)_2]$ (2.7447 (9) Å),[6] and as it is also shorter than the P- -P contacts within each of the two bridging ligands (P1- -P5 = 2.842 (2), P2- -P6 = 2.917 (2) Å), the presence of a Rh–Rh single bond is indicated. The coordination about each metal can be roughly described as octahedral if the position of the second metal is included, but as can be seen from Figure 2 and Table IV, there are significant distortions. The coordination sphere about Rh1 is somewhat more regular than that about Rh2, but in neither instance is any set of three ligating atoms coplanar with the attached metal (Table S8, supplementary material). The major distortions in the coordination spheres of the metals result from constraints imposed by the short "bite" of the chelating ligands and the binding of the olefinic moiety. Smaller distortions are a rotation of the chelating ligand on Rh2 away from P5 so that the Rh1–Rh2–P7 angle is significantly less than the Rh2–Rh1–P3 angle and an opening of the P5–Rh2–P6 angle as compared with P1–Rh1–P2. These latter distortions can be attributed to nonbonded contacts. Because of the different rotational orientations of the carbomethoxy groups, O4 makes contacts of 2.52 and 2.60 Å, respectively, with H813 and H722,[7] but the closest approach of the other with the chelating ligand on Rh1 is 2.88 Å (O2- -H411). Also, one perchlorate ion is situated close to the methoxy groups on P5, P6, and P7 (O1A- -H623 = 2.86 Å, O1A- -H712 = 2.55 Å, O2A- -H511 = 2.62 Å), but the corresponding site on the other side of the cation is not similarly occupied.

As was found for $[Rh_2(CNCMe_3)_4(\mu\text{-}HFB)(DPPM)_2](PF_6)_2$,[8] one side of the cation is twisted relative to the other by ca. 15.51 (5)°[10] although interestingly the angle between the Rh1–Rh2 and C5–C6 vectors is only 6.5 (1)°. Here also it appears likely that the twist is to minimize nonbonded contacts (e.g. O41- -H813 = 2.47 (Å), O82- -H423 = 2.41 Å),[7] as is the case in $[Rh_2(PNP)_2(\mu\text{-}PNP)_2]^{2+}$.

The C5–C6, Rh1–C5, and Rh2–C6 distances (Table III) compare favorably with the corresponding values found in $[Rh_2(CNCMe_3)_4(\mu\text{-}HFB)(DPPM)_2](PF_6)_2$ (1.318 (9), 2.089 (4) Å).[8] The difference in the two Rh–C distances is of only marginal significance ($\Delta/\sigma = 3$). The Rh–P distances vary considerably although all are in the range found previously for the $[Rh_2(PNP)_2(\mu\text{-}PNP)_2]^{2+}$ cation.[3,9] Comparison of corresponding pairs between the two sides of the cation shows close agreement except for Rh1–P4 vs Rh2–P8. The possibility that this arises from an incorrect choice of enantiomer was checked by calculating all Rh–P distances and P–Rh–P angles in the enantiomer that had been rejected. Essentially the same values were found, and we therefore have no obvious explanation for the difference. Within the dimeric cation, all other metrical parameters are unexceptional although there is considerable congestion (Table S7, supplementary material). The perchlorate ions also have normal geometries although showing considerable thermal motion.

The carbonyl adduct of $[Rh_2(PNP)_2(\mu\text{-}PNP)_2]^{2+}$ proved too labile to be isolated. However, the observation of a strong absorption at 1864 cm^{-1} in a CO-saturated solution indicated the presence of a bridging carbonyl. This together with the pale yellow color strongly suggests the carbonyl adduct has a structure similar to that of $[Rh_2(PNP)_2(\mu\text{-}PNP)_2(\mu\text{-}DMAD)]^{2+}$ with a metal–metal bond.

Acknowledgment. Support from the Tulane University Chemistry Department and assistance from Dr. Marie P. Johnson in the data collection are gratefully acknowledged.

Supplementary Material Available: Tables S1–S9, listing bond distances, interbond angles, anisotropic thermal parameters, amplitudes of anisotropic thermal displacement, calculated hydrogen atom positions, torsion angles, selected nonbonded contacts, results of least-squares plane calculations, and full details of data collection and refinement (25 pages); Table S10, listing observed and calculated structure factors (50 pages). Ordering information is given on any current masthead page.

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[5] In $[Rh_2(PNP)_2(\mu\text{-}PNP)_2]^{2+}$, with reference to the numbering scheme of Figure 2, P3 and P7 are approximately trans respectively to P2 and P6.

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[7] The hydrogen atoms are numbered such that the first digit or digits refer to the number of the attached carbon atom, while the final digit identifies the particular hydrogen atom of that methyl group.

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[10] This is the average of the torsional angles P1–Rh1–Rh2–P5 (15.91 (5)°), P2–Rh1–Rh2–P6 (13.49 (5)°), and P4–Rh1–Rh2–P8 (17.14 (5)°).

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Evidence for the Existence of the $[MgCl(THF)_5]^+$ Cation. Crystal Structures of $[MgCl(THF)_5][FeCl_4]\cdot THF$ and $[MgCl(THF)_5][AlCl_4]\cdot THF$

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The formation of $[Mg(THF)_6]^{2+}$, $[Mg_2(\mu\text{-}Cl)_3(THF)_6]^+$, and $[MgCl_4]^{2-}$ ions from $[MgCl_2(THF)_2]$ in tetrahydrofuran (THF) is well documented.[1-3] The conductivity of $[MgCl_2(THF)_2]$ in

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Table I. Crystal Data for $[MgCl(THF)_5]^+[MCl_4]^-\cdot THF$

	M = Al	M = Fe
color	colorless	yellow
cryst dimens, mm	0.7 × 0.5 × 0.9	0.6 × 0.3 × 0.7
space group	$C2/c$	$C2/c$
cell dimens (15 reflcns)	at 301 (2) K	at 288 (1) K
a, Å	13.33 (1)	13.32 (1)
b, Å	24.52 (2)	24.48 (2)
c, Å	10.72 (1)	10.74 (1)
β, deg	90.47 (7)	90.34 (7)
molecules/cell	4	4
vol, Å^3	3504 (6)	3502 (6)
calcd density, g/cm^3	1.25	1.31
measd density, g/cm^3	1.21	1.31
wavelength, Å	0.71069	0.71069
mol wt	661.4	690.1
linear abs coeff, cm^{-1}	4.90	8.72
no. of unique intens	2629	2363
no. with $F > 6.0\sigma(F)$	802	1001
final residuals		
$R(F)$	0.079	0.072
$R_w(F)$	0.071	0.068
max Δ/σ for last cycle	0.3	0.3

THF is low and is equal to 0.05 μS. This is indicative of a small amount of ions in solution and suggests that magnesium dichloride undergoes ionization in tetrahydrofuran.

$$3[MgCl_2(THF)_2] \xrightleftharpoons{\text{THF}} 2[MgCl(THF)_5]^+ + [MgCl_4]^{2-} \qquad (1)$$

The structure of the $MgCl^+$ cation was unknown, but it was frequently postulated, e.g., as an intermediate in reactions with Grignard reagent. However, the equilibrium in (1) could be shifted to the right by an acid[1,2] or a $[NBu_4]^+$ cation.[3] We have now prepared the crystalline salts $[MgCl(THF)_5][MCl_4]\cdot THF$ (M = Fe, Al) in a direct reaction between $FeCl_3$ or $AlCl_3$ and $[MgCl_2(THF)_2]$ in THF. The results of an X-ray diffraction study are presented.

Experimental Section

General Procedures and Materials. All reactions were carried out under N_2 by using the standard Schlenk-tube technique. THF was dried by Na/benzophenone. $[MgCl_2(THF)_2]$ and $AlCl_3(THF)_2$ were obtained by a literature method.[4,5] Commercial $FeCl_3$ and $AlCl_3$ were sublimed before use. ESR spectra were obtained on a Varian ES spectrometer. Solid-state magnetic moments were determined by the Gouy method. IR spectra were obtained by using a Perkin-Elmer 180 spectrometer. Microanalyses were performed at the University of Wrocław.

$[MgCl(THF)_5][FeCl_4]\cdot THF$ (I). $FeCl_3$ (1 g, 6.1 mmol) and $[MgCl_2(THF)_2]$ (2.9 g, 12 mmol) were dissolved separately in 50 and 20 cm^3 of tetrahydrofuran, respectively. The solutions were filtered off and mixed. Immediately, a yellow precipitate settled out and was filtered off, washed with THF (3 × 10 cm^3), and dried in vacuo. Yield: 3.4 g; 90%. Anal. Calcd for $C_{24}H_{48}Cl_5FeMgO_6$: C, 41.77; H, 7.01; Cl, 25.69; Mg, 3.52; Fe, 8.09. Found: C 41.41; H, 6.34; Cl, 25.52; Mg, 3.69; Fe, 8.23.

From the filtrate stored in the refrigerator (in the dark), many-faced yellow crystals settled out. Good quality crystals for X-ray examination were taken directly from this solution.

$[MgCl(THF)_5][AlCl_4]\cdot THF$ (II). $AlCl_3$ (1.2 g, 9.2 mmol) and $[MgCl_2(THF)_2]$ (2.2 g, 9.2 mmol) were dissolved separately in 25 and 50 cm^3 of THF. The solutions were filtered, mixed, and left to stand overnight. Colorless crystals separated. These were filtered off, washed well with n-hexane (3 × 10 cm^3), and dried in vacuo. Yield: 6 g; 83%. Anal. Calcd for $C_{24}H_{48}AlCl_5MgO_6$: Cl, 26.83; Mg, 3.67; Al, 4.08. Found: Cl, 26.68; Mg, 3.56; Al, 4.16.

Crystals for X-ray examination were taken directly from the postreaction mixture.

Crystallography of I and II. Crystals of the title compounds suitable for X-ray study were readily found from material synthesized for other studies. The crystals were cut from bulk species and mounted in capillaries. Weissenberg photographs showed the space group of each compound to be Cc or $C2/c$. The choice of the centrosymmetric $C2/c$ space group was confirmed by the successful solution and refinement of the structures. Characteristics of the data collections on a Syntex $P2_1$ four-circle diffractometer, processing, and refinement are given in Table I. Only unique intensities below $\theta = 24°$ for I and $\theta = 22°$ for II were measured.

Table II. Final Atomic Parameters with Data Presented as Pairs of Values with the Value for the Aluminum Complex Preceding That of the Iron Complex

atom	X/A	Y/B	Z/C	U_{eq}, Å^2
Al	0.5	0.7923 (3)	0.25	0.11
Fe	0.5	0.7932 (2)	0.25	0.11
Mg	0.5	0.3903 (3)	0.25	0.08
	0.5	0.3904 (3)	0.25	0.08
Cl(1)	0.5	0.4880 (2)	0.25	0.12
	0.5	0.4878 (2)	0.25	0.13
Cl(2)	0.5571 (4)	0.8403 (2)	0.3931 (5)	0.22
	0.5609 (4)	0.8424 (2)	0.3967 (4)	0.22
Cl(3)	0.6154 (5)	0.7457 (3)	0.1785 (6)	0.24
	0.6203 (4)	0.7453 (2)	0.1748 (5)	0.22
O(1)	0.5	0.3028 (5)	0.25	0.09
	0.5	0.3019 (5)	0.25	0.09
C(11)	0.4274 (14)	0.2687 (7)	0.1876 (16)	0.14
	0.4226 (12)	0.2675 (7)	0.1905 (14)	0.14
C(12)	0.4472 (15)	0.2128 (7)	0.2310 (24)	0.19
	0.4443 (13)	0.2106 (6)	0.2321 (19)	0.18
O(2)	0.6218 (8)	0.3870 (4)	0.3770 (9)	0.10
	0.6223 (7)	0.3864 (3)	0.3761 (8)	0.10
C(21)	0.6156 (15)	0.3957 (9)	0.5087 (18)	0.16
	0.6166 (12)	0.3971 (8)	0.5053 (15)	0.16
C(22)	0.7189 (18)	0.3948 (12)	0.5540 (20)	0.23
	0.7193 (18)	0.3977 (11)	0.5474 (17)	0.22
C(23)	0.7750 (15)	0.4036 (14)	0.4600 (22)	0.26
	0.7824 (15)	0.3991 (12)	0.4560 (20)	0.24
C(24)	0.7189 (15)	0.4005 (10)	0.3444 (16)	0.19
	0.7219 (14)	0.3981 (9)	0.3430 (14)	0.20
O(3)	0.6012 (7)	0.3862 (5)	0.0965 (8)	0.10
	0.5995 (6)	0.3855 (5)	0.0969 (7)	0.10
C(31)	0.6368 (14)	0.3380 (9)	0.0356 (18)	0.12
	0.6359 (12)	0.3386 (7)	0.0328 (16)	0.10
C(32)	0.6834 (22)	0.3584 (12)	−0.0773 (28)	0.24
	0.6840 (19)	0.3596 (12)	−0.0839 (22)	0.23
C(33)	0.6993 (21)	0.4119 (14)	−0.0701 (29)	0.25
	0.6929 (19)	0.4144 (11)	−0.0679 (26)	0.23
C(34)	0.6430 (18)	0.4321 (9)	0.0306 (20)	0.18
	0.6408 (15)	0.4323 (8)	0.0359 (18)	0.17
O(4)	0.5	−0.0298 (8)	0.25	0.76
	0.5	−0.0283 (8)	0.25	0.73
C(41)	0.5748 (12)	0.0109 (7)	0.2674 (33)	0.27
	0.5769 (10)	0.0124 (7)	0.2529 (30)	0.27
C(42)	0.5353 (23)	0.0590 (7)	0.1953 (24)	0.32
	0.5263 (26)	0.0608 (7)	0.1919 (20)	0.39

The structure of complex I was solved initially by direct methods and then the atomic parameters were used as a basis for the refinement of the structure of the iron and aluminum complexes. All non-H atoms were visible in an E map. All hydrogen atoms were introduced in calculated positions with d(C–H) = 1.08 Å and were not refined. It was assumed that, in both salts, the $[MgCl(THF)_5]^+$ cation and $[MCl_4]^-$ anions and free THF molecules are present in a 1:1:1 ratio. The full-matrix least-squares[6] refinement was completed by using anisotropic thermal parameters for all non-hydrogen atoms. Since the thermal parameters for O and C atoms of the noncoordinated THF molecule were very high and suggested possible disorder, the constraints for C–O and C–C bonds were included on the basis of literature[7] data. Most likely this was the reason for the fact that the THF molecule during the first measurements of compound I was not found.[8] Neutral-atom scattering factors from ref 9 and real and imaginary components of anomalous dispersion were included for all non-H atoms. Absorption and extinction were ignored. The final difference syntheses were essentially featureless; the largest peaks of 0.4 e/Å^3 in both cases were near the noncoordinated

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Table III. Principal Interatomic Distances (Å) and Angles (deg)[a]

	M = Al	M = Fe
Mg–Cl(1)	2.396 (9)	2.384 (9)
Mg–O(1)	2.146 (14)	2.166 (14)
Mg–O(2)	2.112 (10)	2.115 (9)
Mg–O(3)	2.139 (9)	2.121 (8)
M–Cl(2)	2.073 (7)	2.139 (5)
M–Cl(3)	2.069 (8)	2.147 (6)
C–O	1.38 (3)–1.44 (3)	1.41 (3)–1.47 (2)
C–C	1.28 (4)–1.51 (4)	1.30 (4)–1.53 (3)
Cl(1)–Mg–O(2)	92.2 (4)	92.7 (3)
Cl(1)–Mg–O(3)	92.7 (4)	93.2 (4)
O(2)–Mg–O(3)	90.4 (5)	90.6 (4)
O(2)–Mg–O(2′)	175.6 (5)	174.7 (4)
O(3)–Mg–O(3′)	174.6 (5)	173.5 (5)
O(2)–Mg–O(3′)	89.4 (5)	89.1 (4)
Cl(2)–M–Cl(2′)	110.8 (3)	111.5 (3)
Cl(2)–M–Cl(3)	108.6 (3)	107.7 (3)
Cl(2)–M–Cl(3′)	108.0 (3)	108.1 (3)
Cl(3)–M–Cl(3′)	112.9 (4)	113.8 (3)
C–O–C	91 (2)–109 (2)	92 (2)–110 (1)
Mg–O–C	123 (2)–127 (2)	124 (1)–129 (1)

[a] Atoms marked with a prime are related to those at *x*, *y*, *z* by a 2-fold axis at $^1/_2$, *y*, $^1/_4$.

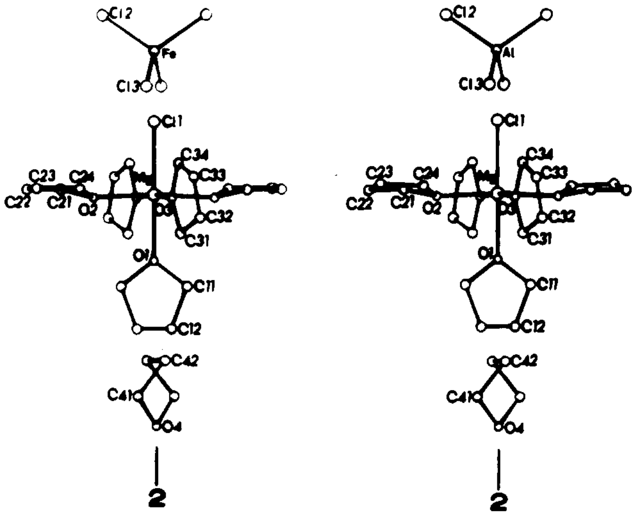

Figure 1. Relationship of the structures of I (left) and II (right) to each other in the crystal along the 2-fold axes of $[MCl_4]^{2-}$, $[MgCl(THF)_5]^+$, and THF molecules.

THF molecule. The results of the X-ray study are given in Tables II and III. All these species are situated on 2-fold axes. Their relationship to each other is shown in Figure 1.

Results and Discussion

The addition of $[MgCl_2(THF)_2]$ to $FeCl_3$ in THF at 1:1 molar ratio gave a yellow air-sensitive compound formulated as $MgFeCl_5(THF)_6$ (I). The new species is paramagnetic, with μ_{eff} ca. 5.8 μ_B at room temperature. The ESR spectrum in the solid and in THF is broad and is typical for Fe^{3+} ions. Its IR spectrum presents the stretching ν_{Fe-Cl} band at 383 cm^{-1} (s), which is typical for the $[FeCl_4]^-$ anion, as well as the symmetric and asymmetric ν_{C-O-C} vibrations of coordinated THF molecules at 838 (vs, br), 927 (vs), and 1038 cm^{-1} (vs). The salt I is sparingly soluble in THF and is light sensitive. The yellow crystals of I were precipitated in the dark. When a solution of I was allowed to stand in the light after 2 days, the colourless crystals $[(THF)_4Mg(\mu\text{-}Cl)_2FeCl_2]$ (Ia) were formed instead. The structure of Ia was published earlier.[10]

$$\mathrm{FeCl_3 + [MgCl_2(THF)_2] + 4THF \rightarrow \underset{I}{[MgCl(THF)_5][FeCl_4]\cdot THF}} \tag{2}$$

In a direct reaction between $AlCl_3$ and $[MgCl_2(THF)_2]$ in THF, the air-sensitive salt $[MgCl(THF)_5][AlCl_4]\cdot THF$ (II) is formed.

$$\mathrm{AlCl_3 + [MgCl_2(THF)_2] + 4THF \rightarrow \underset{II}{[MgCl(THF)_5][AlCl_4]\cdot THF}} \tag{3}$$

The IR spectrum of this species shows, besides the symmetric and asymmetric ν_{C-O-C} vibrations, the characteristic $[AlCl_4]^-$ frequency at 490 cm^{-1} (vs).

The geometries of the $[MgCl(THF)_5]^+$ cations in I and II are presented in Figure 1. The magnesium atoms are six-coordinated. The MgO_5Cl unit forms a pseudooctahedron with the C_2 axis passing through Cl(1), Mg, and O(1). The average Mg–Cl bond length in crystals I and II is 2.39 (1) Å and exceeds the Mg–Cl distance of 2.326 (2) Å in the $[MgCl_4]^{2-}$ anion.[3] This difference is typical in comparing the bond lengths in the octahedron and the tetrahedron. The terminal length of the Mg–Cl bonding in the octahedron has been unknown up to now. The average Mg–O(cis to Cl(1) atom) distance is somewhat shorter than the average Mg–O(1) distance of 2.156 (14) Å. Similar Mg–O bond lengths were earlier observed in the $[Mg(THF)_6]^{2+}$ cation.[2]

The $[FeCl_4]^-$ and $[AlCl_4]^-$ anions have C_2 symmetry. A number of structures that contained those anions have been described earlier. In all cases the $[MCl_4]^-$ units were of the same dimensions.

Our earlier results suggest that reaction 3 is the key to the unusual role played by magnesium dichloride as a support for super-high-activity Ziegler–Natta catalysts for olefin polymerization.[11]

$$\mathrm{AlEt_2Cl + AlCl_3 \rightarrow 2AlEtCl_2} \tag{4}$$

The elimination of $AlCl_3$ from the composition of the catalyst by $MgCl_2$ (eq 3) prevents the formation of $AlEtCl_2$, which poisons the catalysts. $AlEtCl_2$ is formed during the synthesis of the catalyst.[12]

Registry No. I, 119819-00-0; II, 119819-02-2.

Supplementary Material Available: Tables of anisotropic thermal parameters and hydrogen atom parameters for compounds I and II (2 pages); listings of observed and calculated structure factors for compounds I and II (9 pages). Ordering information is given on any current masthead page.

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Open Cluster Configurations of $[1,1\text{-}(PPh_3)_2\text{-}1\text{-}H\text{-}1,2,4\text{-}IrC_2B_8H_{10}]$ and Other Formally Wadian 24-Electron 11-Vertex Species

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Known closed monometallapolyborane cluster compounds that do not contain heteroatoms, but which have C_{2v} 9-vertex,[1] C_{3v} 10-vertex,[2,3] and C_{2v} 11-vertex[3,4] cluster configurations do not conform[5] with the basic Williams–Wade[6,7] cluster-geometry electron-counting formalism. This has engendered discussion of

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