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, $^1/_4$.
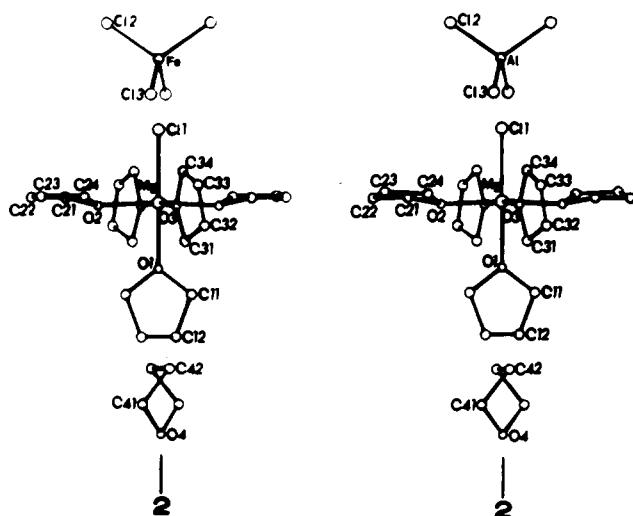

Figure 1. Relationship of the structures of I (left) and II (right) to each other in the crystal along the 2-fold axes of $[MCl_4]^{2-}$, $[MgCl(THF)_5]^+$, and THF molecules.

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 Mg-$FeCl_5(THF)_6$ (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 $[(THF)_4Mg(\mu-Cl)_2FeCl_2]$ (Ia) were formed instead. The structure of Ia was published earlier.[10]

$$FeCl_3 + [MgCl_2(THF)_2] + 4THF \rightarrow$$
$$[MgCl(THF)_5][FeCl_4]\cdot THF \quad (2)$$
$$I$$
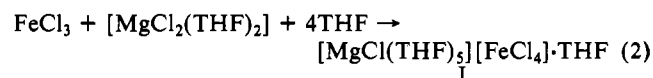
In a direct reaction between $AlCl_3$ and $[MgCl_2(THF)_2]$ in THF, the air-sensitive salt $[MgCl(THF)_5][AlCl_4]\cdot THF$ (II) is formed.

$$AlCl_3 + [MgCl_2(THF)_2] + 4THF \rightarrow$$
$$[MgCl(THF)_5][AlCl_4]\cdot THF \quad (3)$$
$$II$$
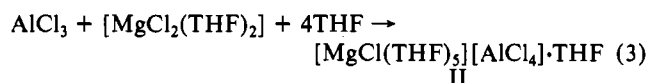
The IR spectrum of this species shows, besides the symmetric and asymmetric ν_{C-O-C} vibrations, the characteristic $[AlCl_4]^-$ frequency at 490 cm^{-1} (vs).

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]

$$AlEt_2Cl + AlCl_3 \rightarrow 2AlEtCl_2 \quad (4)$$

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]

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Open Cluster Configurations of [1,1-(PPh₃)₂-1-H-1,2,4-IrC₂B₈H₁₀] 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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Table I. Crystal Data for [1,1-(PPh₃)₂-1-H-1,2,4-IrC₂B₈H₁₀]

|	
formula	$C_{38}H_{41}B_8IrP_2$
M	838.40
$a/\text{Å}$	12.932 (2)
$b/\text{Å}$	15.565 (2)
$c/\text{Å}$	18.566 (2)
β/deg	90.20 (1)
V/nm^3	3.7373 (9)
Z	4
space group	$P2_1/c$
$D_{calcd}/\text{g cm}^{-3}$	1.50
$F(000)$	1664
μ/cm^{-1}	35.28
scan speeds/deg min⁻¹	2.0–29.3
scan width/deg	2.0 + α-doublet splitting
range/deg	4.0 < 2θ < 50.0
no. of data collected	7025
no. of data obsd	5471 [$I > 2.0\sigma(I)$]
R	0.0302
R_w	0.0285
weighting scheme	$w = [\sigma^2(F_o) + 0.0002(F_o)^2]^{-1}$
no. of variables	415

the bonding in these and related species.[8-12] The question of the bonding is not entirely resolved, and it is important that additional experimental evidence be available to contribute to theoretical deliberations in these areas.

During the course of the bonding discussion, it has been reasonably presumed that the 11-vertex metalla*dicarba*borane [1,1-(PPh₃)₂-1-H-1,2,4-IrC₂B₈H₁₀] is of closed deltahedral configuration (structure I).[10] This hypothesis can readily be tested,

I   **II**
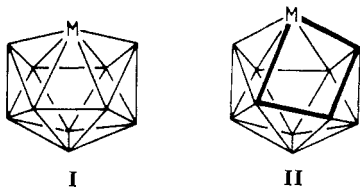
and we now report experimental results to show that, interestingly, this compound is *not* in fact of completely closed deltahedral configuration but that it has a well-defined open face (structure II).

A sample of [1,1-(PPh₃)₂-1-H-1,2,4-IrC₂B₈H₁₀] was prepared from [5,6-C₂B₈H₁₂],[13] 1,8-bis(dimethylamino)naphthalene, and [IrCl(PPh₃)₃] in ether (in a minor variation of the previously published[14] reaction) and purified by chromatography. Measurement and assignment of the cluster ¹¹B and ¹H NMR shielding
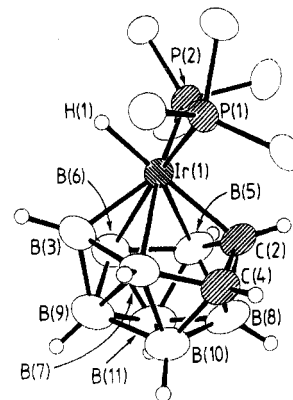
Figure 1. Crystallographically determined molecular structure of [1,1-(PPh₃)₂-1-H-1,2,4-IrC₂B₈H₁₀].[18] Distances (Å) from Ir(1) are as follows: to C(2), 2.192 (7); to B(3), 2.087 (8); to C(4), 2.779 (8); to B(5), 2.342 (8); to B(6), 2.342 (8); to B(7), 2.347 (7); to P(1), 2.345 (3); to P(2), 2.362 (3); to H(1), 1.62 (5). Selected other distances (Å) are as follows: C(2)–C(4), 1.486 (8); C(2)–C(7), 2.579 (10); C(4)–B(7), 1.715 (10); B(7)–B(3), 1.789 (10); B(3)–B(6), 1.765 (11); B(6)–B(5), 1.816 (12); B(5)–C(2), 1.615 (10). Angles (deg) in the Ir(1)C(2)C(4)B(7) open face are as follows: at Ir(1), 69.1 (3); at C(2), 96.3 (4); at C(4), 107.2 (5); at B(7), 84.8 (4). The dihedral angle between the planes B(7)Ir(1)C(2) and B(7)C(4)C(2) is 17.6°.

behavior[15] then suggested to us that the iridadicarbaborane was perhaps not a straightforward closo species. This supposition was confirmed by a single-crystal X-ray diffraction analysis (Figure 1 and Table I),[18] using a crystal grown from dichloromethane–pentane.

It can be seen that a description of the {IrC₂B₈} cluster structure as closed deltahedral (structure I) is rather a severe approximation. There is in fact a very marked distortion from this ideal, in that there is a quadrilateral open face [Ir(1)C(2)C(4)B(7)], with the distance Ir(1)–C(4) being nonbonding at 2.779 (8) Å (structure II).[19] There has previously been no reason to suppose that the cluster structure of this particular compound was anything but straightforward closo, and it had reasonably been assumed to be such.[10,14] The result illustrates that theoretical work in this and closely related areas[10,12] needs to be supported in some detail by experimental observation.

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(15) NMR data (ordered as follows: position, δ(¹¹B)/ppm [δ(¹H)/ppm]): BH(3), +56.1 [+8.54]; BH(9), +10.8 [+5.04]; BH(5), +3.7 [+2.16]; BH(6), −5.4 [+1.44]; BH(11), −10.7 [−0.28]; BH(8), −22.6 [+1.28]; BH(10), −24.2 [+0.04]; BH(7), −44.6 [−2.94]; CH(2), ... [+3.08]; CH(4), ... [+2.38]. δ(¹H):IrH, −6.26 ppm [²J(³¹P–¹H) = 17.4 and 33.0 Hz]. δ(³¹P) (relative to 85% H₃PO₄): +6.4 and −3.9 ppm. [²J(³¹P–³¹P) 12 Hz]. A CDCl₃ solution was used at 291–294 K (¹¹B and ¹H) and 223 K (³¹P); cluster positions were assigned by ¹H–{¹¹B} and [¹H–¹H]–COSY–{¹¹B} experiments.[16] Compare with data[17] for the open-structured compound [2,4-Me₂-1-(η⁶-MeC₆H₄-4-ⁱPr)-1,2,4-RuC₂B₈H₈].
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(18) All crystallographic measurements were made on a Nicolet P3/F diffractometer operating in the ω/2θ scan mode using graphite-monochromatized Mo Kα radiation (λ = 0.710 69 Å) following a standard procedure described in detail elsewhere.[23] The data set was corrected for absorption empirically once the structure had been determined.[24] The structure was solved via standard heavy-atom methods and refined by full-matrix least-squares techniques using the SHELX program system.[25] All non-hydrogen atoms were assigned anisotropic thermal parameters. The phenyl groups were treated as rigid bodies with idealized hexagonal symmetry (C–C = 1.395 Å). Phenyl hydrogen atoms were included in calculated positions (C–H = 1.08 Å) and were assigned to an overall isotropic thermal parameter. The metal hydride and borane hydrogen atoms were located in a Fourier difference map and were freely refined with individual isotropic thermal parameters.
(19) The structure in fact very closely resembles the "isonido" 11-vertex cluster geometry now established for the 11-vertex, formally 24-electron, dimetallaborane [1,2-(η⁵-C₅Me₅)₂-1,2-RhIrB₉H₁₁] (numbered as in Figure 1). This dimetallaundecaborane has a definitive 1,2,4,7 quadrilateral open face as in structure II, but now also with hydrogen bridges on the open face, at Rh(1)–Ir(2) and B(4)–B(7): Nestor, K.; Fontaine, X. L. R.; Greenwood, N. N.; Kennedy, J. D.; Thornton-Pett, M. *J. Chem. Soc., Chem. Commun.*, in press.

In a wider empirical context, the result adds to an awareness that the behavior of the formally closo, formally 24-electron, 11-vertex cluster system may in fact quite regularly deviate from that expected on the basis of the straightforward Williams-Wade[6,7] formalism. Thus, of structurally characterized species, the present compound, the related ruthenadicarbaborane [2,4-Me$_2$-1-(η^6-MeC$_6$H$_4$-p-^{i}Pr)-1,2,4-RuC$_2$B$_8$H$_8$],[17] and the substituted dicarbaborane [2,3-Me$_2$C$_2$B$_9$H$_6$-4,7-(OH)$_2$-10-Br][20] all exhibit very significant deviations from straightforward closo structure, the osmadicarbaborane [(η^6-C$_6$Me$_6$)OsC$_2$Me$_2$B$_8$H$_8$] can adopt a conventional nido structure,[21] and the high fluxionalities of the closo-structured [CB$_{10}$H$_{11}$]$^-$ anion[22] and of [B$_{11}$H$_{11}$]$^{2-}$ itself[23] show that transition states of presumably open structure must be energetically very near to the closed deltahedral configuration for these simpler species also. The open structure reported here in fact resembles that proposed as the intermediate for the series of diamond–square–diamond steps in the "pentagonal belt rotation" mechanism suggested[22] for these fluxionalities.

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Production of an Optically Active Sulfoxide by Use of Colloidally Dispersed Λ-Tris(2,2'-bipyridyl)ruthenium(II) Montmorillonite as a Photosensitizer

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Recently an ion-exchanged adduct of a clay and an optically active metal complex has been used as a chiral adsorbent in optical resolution and asymmetric syntheses, motivated by the finding that some metal chelates were adsorbed stereoregularly on a clay.[1,2] Tris(1,10-phenanthroline)iron(II) ([Fe(phen)$_3$]$^{2+}$), for example, is adsorbed in the form of a racemic pair of its Δ- and Λ-enantiomers. When a pure enantiomer of the same chelate, [Λ-Fe-(phen)$_3$]$^{2+}$, for example, is added, the enantiomer is adsorbed, leaving half of the surface unoccupied.

In a comparison of the surfaces of the clays ion-exchanged with enantiomeric and racemic metal chelates, it was postulated that

an empty space on the former surface would become a stereoselective site for adsorption or chemical reactions on a clay surface. In fact, an ion-exchange adduct of a clay with optically active [Ru(phen)$_3$]$^{2+}$ has been proved to be quite effective in resolving racemic mixtures.[1,2]

As an extension of these results, photoinduced asymmetric reactions have been attempted on an adduct of a clay and a photosensitive metal complex. It was intended to produce an optically active sulfoxide by illuminating a sulfide in the presence of optically active tris(2,2'-bipyridyl)ruthenium(II) [(Ru(bpy)$_3$]$^{2+}$) montmorillonite. As a result, optically active alkyl phenyl sulfoxides were obtained with the optical purity of 15–20% at room temperature.

Experimental Section

The clay used in these studies is sodium montmorillonite, purchased from Kunimine Ind. Co.; the cation-exchange capacity (CEC) is 115 mequiv/100 g. The complex [Ru(bpy)$_3$]Cl$_2$ was prepared and resolved according to the literature.[3,4] Alkyl phenyl sulfides were synthesized by reacting alkyl iodides with thiophenol in alkaline solutions.[5] Water containing ^{18}O (97.1 atom %) was purchased from ISOTECC Inc. The electronic spectra were recorded with a spectrophotometer, UVIDEC-430A (JEOL). The high-performance liquid chromatography (HPLC) was performed with a BIP-1 chromatograph (JEOL) equipped with a UV detector, UVIDEC-100-VI (JEOL). The circular dichroism spectra were measured with a polarimeter, J-20 (JEOL). The m/e of a sulfoxide was determined with a mass spectrometer, GCMS-QP1000 (Shimazu).

A clay–chelate adduct is prepared as follows: 1 g of sodium montmorillonite is dispersed in 100 mL of water. About 90 μL of this clay suspension is added to 10 mL of water. An aqueous solution of [Λ-Ru-(bpy)$_3$]Cl$_2$ (5 × 10^{-7} mol) is then slowly added to it. The mixture is centrifuged and the supernatant solution is discarded.

Oxidation of the sulfide is performed in the following way: a sulfide (1 × 10^{-6} mol) in 5 mL of methanol is mixed with the clay–chelate adduct as prepared above. The mixture is illuminated with a 500-W tungsten lamp under bubbling oxygen gas. The light is focused on the sample solution. The temperature of the sample is kept constant by circulating water or methanol. After some interval, the medium is centrifuged and the supernatant liquid separated. The precipitate is washed with a few milliliters of methanol. The organic components are extracted with dichloromethane from the combined supernatant solution and methanol wash liquid. After evaporation of the dichloromethane, the residue is dissolved in 200 μL of methanol and analyzed by HPLC. The solutions eluting under peaks corresponding to sulfide or sulfoxide are collected and the concentrations of the compounds determined from the absorbances in the UV spectra (ϵ = 6460 at 257 nm for cyclohexyl phenyl sulfide, and ϵ = 4390 at 242 nm for cyclohexyl phenyl sulfoxide). The optical purity of the sulfoxide is estimated by resolving the mixtures into each enantiomer with a liquid column chromatograph of Λ-tris(1,10-phenanthroline)iron(II) montmorillonite. From the amplitude of the circular dichroism (CD) spectrum, Δϵ at 242 nm is determined to be 30 for (R)-cyclohexyl phenyl sulfoxide.

The above procedures were repeated two or three times, using the same clay–chelate adduct as a catalyst. Reproducible results within the experimental errors of 5% of the reaction yields and optical purities were obtained.

Results

(i) Photoinduced Oxidation of a Sulfide in the Presence of [Ru(bpy)$_3$]$^{2+}$. Electronically excited [Ru(bpy)$_3$]$^{2+}$ is known to act as an oxidizing or reducing agent. We first examined how excited [Ru(bpy)$_3$]$^{2+}$ reacts with a sulfide in a methanol–water mixture.

About 1 × 10^{-6} mol of cyclohexyl phenyl sulfide was added to 5 mL of 1:4 (v/v) methanol–water containing 5 × 10^{-5} mol of [Λ-Ru(bpy)$_3$]Cl$_2$. The solution was illuminated with a 500-W tungsten lamp under 1 atm of oxygen for 180 min. When we analyzed the solution, the sulfide was found to be completely converted to the sulfoxide and no sulfone was produced. Oxidation

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