scans approximately three to six times faster than with the conventional single pulse experiment. Additionally, a given signal/noise level can be attained $(E_d)^2$ times faster from polarization transfer than from the single pulse metods. These factors combine to give time saving factors varying from about 30 to 300.

Acknowledgment. This work was supported by the Air Force Office of Scientific Research, Air Force System Command, USAF, under Grant Number AFOSR 78-3570. The United States Government is authorized to reproduce and distribute reprints for Governmental purposes notwithstanding any copyright notation thereon. We thank the National Science Foundation for a predoctoral fellowship to B.J.H. Helpful discussions with Mr. Bruce Adams are also gratefully acknowledged.

Registry No. Me$_4$Si, 75-76-3; Me$_3$SiCl, 75-77-4; Me$_2$SiCl$_2$, 75-78-5; MeSiCl$_3$, 75-79-6; *t*-Bu$_2$SiCl$_2$, 18395-90-9; *t*-Bu$_2$SiF$_2$, 558-63-4; *t*-BuSiCl$_3$, 18171-74-9; *t*-BuMeSiCl$_2$, 18147-18-7; Ph$_2$SiCl$_2$, 80-10-4; Ph$_2$SiH$_2$, 775-12-2; PhSiH$_3$, 694-53-1; Et$_3$SiH, 617-86-7; (EtO)$_3$SiH, 998-30-1; (MeO)$_4$Si, 681-84-5; (HMe$_2$Si)O, 3277-26-7; (Me$_3$SiOMe$_2$Si)$_2$O, 141-62-8; (Me$_2$SiO)$_5$, 541-02-6; *p*-(Me$_3$Si)$_2$C$_6$H$_4$, 13183-70-5; (Me$_3$Si)$_2$C$_2$, 14630-40-1; (Me$_3$Si)$_2$NH, 999-97-3; (Me$_3$Si)$_2$S, 3385-94-2; (Me$_3$Si)$_2$, 1450-14-2; (HMeS$_2$Si)$_2$, 74864-45-2; (*t*-BuMeSi)$_4$, 63357-19-7; (Me$_2$Si)$_6$, 4098-30-0.

Coupling of Iridium-Bound Methylene with Ethylene[†]

David L. Thorn

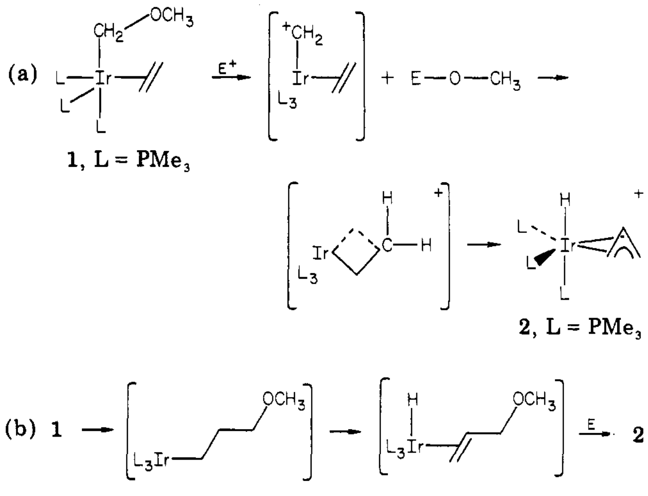
Central Research and Development Department
E.I. du Pont de Nemours and Company
Experimental Station, Wilmington, Delaware 19898

Received December 22, 1981

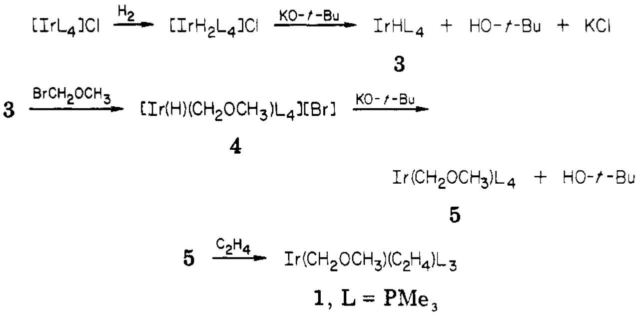
Summary: The (methoxymethyl)ethyleneiridium(I) complex Ir(CH$_2$OCH$_3$)(C$_2$H$_4$)(P(CH$_3$)$_3$)$_3$ (**1**) reacts readily with trimethylsilyl trifluoromethanesulfonate to form the hydrido allyl compound [IrH(C$_3$H$_5$)(P(CH$_3$)$_3$)$_3$][CF$_3$SO$_3$] (**2**). Suspected intermediates include a methylene ethylene complex which rapidly converts to a metallacyclobutane complex and subsequently undergoes β elimination to form **2**.

The currently favored mechanism for the olefin metathesis reaction requires, as one important step, the coupling of an olefin with a transition-metal carbene ligand to form a metallacyclobutane complex.[1] Examples of the intramolecular reaction of a metal-bound olefin with a metal-bound carbene or alkylidene ligand, in well-characterized metal complexes, are however few[2,3] and are generally confined to complexes of the earlier transition metals.[4,5] Methoxymethyl complexes of the later transition metals have been found to be convenient precursors to methylene complexes, which in turn react with olefins to form cyclopropanes.[6,7] Some of these latter reactions are believed to occur by a stepwise process in which the olefin is never directly bonded to the metal in the usual η^2 fashion.[7,8]

Scheme I



Scheme II



$$[\mathrm{IrL_4}]\mathrm{Cl} \xrightarrow{\mathrm{H_2}} [\mathrm{IrH_2L_4}]\mathrm{Cl} \xrightarrow{\mathrm{KO}\text{-}t\text{-}\mathrm{Bu}} \underset{\mathbf{3}}{\mathrm{IrHL_4}} + \mathrm{HO}\text{-}t\text{-}\mathrm{Bu} + \mathrm{KCl}$$

$$\mathbf{3} \xrightarrow{\mathrm{BrCH_2OCH_3}} \underset{\mathbf{4}}{[\mathrm{Ir(H)(CH_2OCH_3)L_4}][\mathrm{Br}]} \xrightarrow{\mathrm{KO}\text{-}t\text{-}\mathrm{Bu}} \underset{\mathbf{5}}{\mathrm{Ir(CH_2OCH_3)L_4}} + \mathrm{HO}\text{-}t\text{-}\mathrm{Bu}$$

$$\mathbf{5} \xrightarrow{\mathrm{C_2H_4}} \mathrm{Ir(CH_2OCH_3)(C_2H_4)L_3}$$

1, L = PMe$_3$

[†] Contribution No. 2981.

(1) Recent reviews include: (a) Haines, R. J.; Leigh, G. J. *Chem. Soc. Rev.* **1975**, *4*, 155–188. (b) Calderon, N.; Ofstead, E. A.; Judy, W. A. *Angew. Chem.* **1976**, *88*, 433–442; *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 401–409. (c) Katz, T. J. *Adv. Organomet. Chem.* **1977**, *16*, 283–317. (d) Rooney, J. J.; Stewart, A. *Catalysis* **1977**, *1*, 277–334. (e) Grubbs, R. H. *Prog. Inorg. Chem.* **1978**, *24*, 1–50. (f) Calderon, N.; Lawrence, J. P.; Ofstead, E. A. *Adv. Organomet. Chem.* **1979**, *17*, 449–492.

(2) Examples of well-characterized mononuclear alkylidene–olefin complexes have been published by: Schultz, A. J.; Brown, R. K.; Williams, J. M.; Schrock, R. R. *J. Am. Chem. Soc.* **1981**, *103*, 169–176. Fellmann, J. D.; Schrock, R. R.; Rupprecht, G. A. *Ibid.* **1981**, *103*, 5752–5758. Heteroatom-substituted, "carbene"–olefin complexes have been reported by: Nesmeyanov, A. N.; Sal'nikova, T. N.; Struchkov, Yu. T.; Andrianov, V. G.; Pogrebnyak, A. A.; Rybin, L. V.; Rybinskaya, M. I. *J. Organomet. Chem.* **1976**, *117*, C16–C20. Mitsudo, T.-A.; Nakanishi, H.; Inubushi, T.; Morishima, I.; Watanabe, Y.; Takegami, Y. *J. Chem. Soc., Chem. Commun.* **1976**, 416–417. Rosenblum, M.; Priester, W. *Ibid.* **1978**, 26–27. Hiraki, K.; Sugino, K. *J. Organomet. Chem.* **1980**, *201*, 469–475. Casey, C. P.; Shusterman, A. J. *J. Mol. Catal.* **1980**, *8*, 1–13. Hiraki, K.; Onishi, M.; Ohnuma, K.; Sugino, K. *J. Organomet. Chem.* **1981**, *216*, 413–419.

(3) A "valence isomer" of a methylene–acetylene adduct of Ti is discussed by: McKinney, R. J.; Tulip, T. H.; Thorn, D. L.; Coolbaugh, T. S.; Tebbe, F. N. *J. Am. Chem. Soc.* **1981**, *103*, 5584–5586. The electronic factors which guide the interconversion of methylene–olefin and metallacyclic complexes are discussed by: Eisenstein, O.; Hoffmann, R.; Rossi, A. R. *Ibid.* **1981**, *103*, 5582–5584.

(4) A number of well-characterized alkylidene complexes have been found to be active catalysts for the olefin metathesis reaction. For leading references see the review articles cited in ref 1. See also: Schrock, R. R. *Acc. Chem. Res.* **1979**, *12*, 98–104. Wengrovius, J. H.; Schrock, R. R.; Churchill, M. R.; Missert, J. R.; Youngs, W. J. *J. Am. Chem. Soc.* **1980**, *102*, 4515–4516. Schrock, R. R.; Rocklage, S.; Wengrovius, J.; Rupprecht, G.; Fellmann, J. *J. Mol. Catal.* **1980**, *8*, 73–83.

(5) Electron-rich olefins dismutate in the presence of rhodium catalysts, and Rh(I) d^8 carbene intermediates have been characterized. Cardin, D. J.; Doyle, M. J.; Lappert, M. F. *J. Chem. Soc., Chem. Commun.* **1972**, 927–928. Hitchcock, P. B.; Lappert, M. F.; Terreros, P.; Wainwright, K. P. *Ibid.* **1980**, 1180–1181.

(6) (a) Jolly, P. W.; Pettit, R. *J. Am. Chem. Soc.* **1966**, *88*, 5044–5045. (b) Green, M. L. H.; Ishaq, M.; Whiteley, R. N. *J. Chem. Soc. A* **1967**, 1508–1515. (c) Davison, A.; Krusell, W. C.; Michaelson, R. C. *J. Organomet. Chem.* **1974**, *72*, C7–C10. (d) Flood, T. C.; DiSanti, F. J.; Miles, D. L. *Inorg. Chem.* **1976**, *15*, 1910–1918. (e) Riley, P. E.; Capshaw, C. E.; Pettit, R.; Davis, R. E. *Ibid.* **1978**, *17*, 408–414. (f) Brookhart, M.; Tucker, J. R.; Flood, T. C.; Jensen, J. *J. Am. Chem. Soc.* **1980**, *102*, 1203–1205.

(7) Other isolable carbene (or alkylidene) complexes which react with olefins include those reported by: (a) Casey, C. P.; Burkhardt, T. J. *J. Am. Chem. Soc.* **1974**, *96*, 7808–7809. (b) Casey, C. P.; Burkhardt, T. J.; Bunnell, C. A.; Calabrese, J. C. *Ibid.* **1977**, *99*, 2127–2134. (c) Casey, C. P.; Polichnowski, S. W. *Ibid.* **1977**, *99*, 6097–6099. (d) Brookhart, M.; Nelson, G. O. *Ibid.* **1977**, *99*, 6099–6101. (e) Casey, C. P.; Polichnowski, S. W.; Shusterman, A. J.; Jones, C. R. *Ibid.* **1979**, *101*, 7282–7292. (f) Brookhart, M.; Tucker, J. R.; Husk, G. R. *Ibid.* **1981**, *103*, 979–981. (g) Brookhart, M.; Humphrey, M. B.; Kratzer, H. J.; Nelson, G. O. *Ibid.* **1980**, *102*, 7802–7803.

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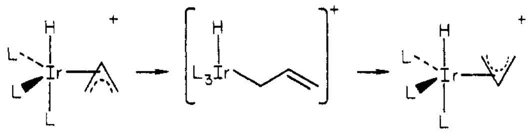
This communication describes the preparation and characterization of a stable methoxymethyl ethylene complex, $Ir(CH_2OCH_3)(C_2H_4)(P(CH_3)_3)_3$ (1).[9] Treatment of compound 1 with electrophilic reagents results in the formation of the cationic hydrido allyl complex $[IrH(C_3H_6)(P(CH_3)_3)_3)_3]^+$ (2). A plausible mechanism for the formation of 2 is illustrated in Scheme Ia. The electrophilic reagent cleaves a C–O bond of the methoxymethyl group, forming (at least incipiently) a reactive methylene ligand.[10] Under the reaction conditions employed here, a bond rapidly forms between the carbon atoms of the methylene ligand and the ethylene moiety. The initial product is the metallacyclobutane complex but this complex rapidly undergoes β elimination, resulting in the formation of the hydrido allyl complex 2.[11]

An alternative mechanism[12] for the conversion of 1 to 2 is shown in Scheme Ib. In this mechanism the initial step is insertion of ethylene into the iridium–methoxymethyl bond, followed by β elimination to form the hydrido olefin complex; subsequent reaction with the electrophilic reagent forms the hydrido allyl complex 2. This mechanism does not require the intermediacy of iridium-bound methylene or metallacyclobutane species. However, a reluctance of ethylene to insert into ordinary iridium–carbon (alkyl) bonds is demonstrated by the stabilities of compound 1 and a related methyl ethylene iridium(I) complex;[10] and the facile reaction of the iridium-bound methoxymethyl group with electrophilic reagents is known.[10] Thus, while the mechanism of Scheme Ib cannot be excluded by the present data, that of Scheme Ia appears to be more likely.

The novel method employed here for the preparation of compound 1 is summarized in Scheme II.[13] Typically, $[Ir(P(CH_3)_3)_4][Cl]$[14] can be converted to compound 1 in about 25% overall yield. Two crucial aspects of this reaction sequence are the facile removal of a "hydrido" hydrogen atom by strong base with concomitant reduction of the metal center and the remarkable stability of hydrido alkyl iridium(III) complexes.[15] The attempted preparation of the rhodium analogues of 5[16] or 1 by this reaction sequence fails, presumably owing to immediate decomposition of the hydrido alkyl analogue of 4.[17]

Scheme III



2 7

Compound 1 is reactive toward a number of electrophilic reagents, including [H-pyridine]$[PF_6]$ and several trimethylsilylating reagents. The cationic hydrido allyl complex 2 has been the only characterizable compound formed in these reactions. The reaction conditions found to be most favorable for the formation of 2 are the addition of a solution of compound 1 in hexamethyldisiloxane to a solution of an equivalent of trimethylsilyl trifluoromethanesulfonate in hexamethyldisiloxane at room temperature, from which analytically pure hydrido allyl complex $[IrH(C_3H_5)(P(CH_3)_3)_3][CF_3SO_3]$ (2) precipitates immediately in 88% yield.[18] The same hydrido allyl cation has been obtained by reacting the iridium(I) tris(trimethylphosphine) allyl compound $Ir(C_3H_5)(P(CH_3)_3)_3$ (6)[19] with HPF_6 in pyridine. Potassium *tert*-butoxide removes H^+ from 2 to restore the neutral allyliridium(I) compound 6.

Compound 2 slowly isomerizes to a second hydrido allyl compound, 7.[20] This second hydrido allyl complex is most likely derived from the first by a "rotation" of the allyl group (see Scheme III), which may occur by a slow $\eta^3 \rightleftharpoons \eta^1$ isomerization mechanism. From the spectral data, the structures of 2 and 7 cannot be unambiguously determined, and the proposed assignments illustrated in Scheme III may be incorrect. However, since compound 2 is believed to be formed directly from the metallacyclobutane complex (see Scheme Ia), the structure shown is reasonable.[11]

The method described in Scheme II unfortunately is not general for preparing other iridium alkyloxyalkyl olefin complexes. Many other alkyl halides react with compound 3 but form undesired products, with little or no hydrido alkyl compound being formed. Also, under the conditions explored in this study, many other olefins (e.g., cyclooctadiene, propene) do not react with compound 5. Thus, continuing efforts to probe certain aspects of this reaction by examining substituent effects have not yet been successful.

The reaction illustrated in Scheme Ia supports the possiblity of facile carbon–carbon bond formation in d^8 olefin–methylene complexes.[5,21] The reverse reaction, the fragmentation of a d^6 metallacyclobutane complex into a d^8 olefin–methylene complex, has been proposed in rearrangement reactions of platinacyclobutane complexes.[22] Experiments are in progress to determine whether these

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(8) Indirect evidence for prior coordination of olefin is discussed by: Casey, C. P.; Cesa, M. C. *Organometallics* **1982**, *1*, 87–94.

(9) ^1H NMR data for 1 (C_6D_6): $P(CH_3)_3$, δ 1.00 (d, $J = 7$ Hz), 1.40 (t, J (eff) = 3.5 Hz), C_2H_4, δ 1.4 (m), $IrCH_2OCH_3$, 3.30 (m), OCH_3, δ 3.28 (s). Anal. Calcd: C, 31.64; H, 7.35. Found: C, 31.25; H, 7.26.

(10) Other methoxymethyliridium complexes form reactive "carbene" compounds. Thorn, D. L.; Tulip, T. H. *J. Am. Chem. Soc.* **1981**, *103*, 5984–5986.

(11) The conversion of metallacyclobutane complexes of iridium into hydrido allyl complexes was first discovered by: Tulip, T. H.; Ibers, J. A. *J. Am. Chem. Soc.* **1978**, *100*, 3252–3254; *Ibid.* **1979**, *101*, 4201–4212. Another cationic hydrido allyl iridium complex was reported by: Howarth, O. W.; McAteer, C. H.; Moore, P.; Morris, G. E. *J. Chem. Soc., Chem. Comm.* **1981**, 506–507. Beta elimination in metallacyclobutane complexes can terminate olefin metathesis catalytic cycles; see ref 4.

(12) We thank a reviewer for noting this mechanistic possiblity.

(13) Full details will be published separately. Satisfactory ^1H NMR spectra have been obtained for all compounds, and satisfactory C and H analyses for 4 and 5. All manipulations were carried out at room temperature in a nitrogen-filled drybox, owing to the air and moisture sensitivity of the iridium compounds and the probable toxicity of bromomethyl methyl ether.

(14) Herskovitz, T.; Guggenberger, L. J. *J. Am. Chem. Soc.* **1976**, *98*, 1615–1616. Herskovitz, T. *Ibid.* **1977**, *99*, 1648–1649. Herskovitz, T. *Inorg. Synth.*, in press.

(15) Thorn, D. L. *J. Am. Chem. Soc.* **1980**, *102*, 7109–7110. Thorn, D. L. *Organometallics* **1982**, *1*, 197–204.

(16) The cobalt analogue of 5 has been mentioned by: Klein, H.-F. *Angew. Chem.* **1980**, *92*, 362–375; *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 362–375.

(17) Thorn, D. L., to be submitted for publication.

(18) ^1H NMR of 2 (py-d_5) Ir–H, δ –16.0 (d, $J = 130$ Hz, of t, $J = 18$ Hz), $P(CH_3)_3$, δ 1.18 (d, $J = 8$ Hz of d, J(H-hydride) = 1 Hz), 1.45 (d, $J = 9.5$ Hz), C_3H_5, δ 1.8 (br m), 2.5, (br d), 5.0 (br m). ^{31}P-decoupled ^1H NMR: C_3H_5, δ 1.75 (d, $J = 5$ Hz), 2.53 (d, $J = 7$ Hz), 5.05 (t of t). Anal. Calcd: C, 25.53; H, 5.44. Found C, 25.59, H, 5.44.

(19) Prepared from $[Ir(P(CH_3)_3)_4][Cl]$ using allylmagnesium bromide. Tulip, T. H., to be submitted for publication.

(20) In pyridine solution the isomerization requires at least 1 week at room temperature or several hours at 65 °C. ^1H NMR data for 7 (py-d_5): Ir–H, δ –14.5 (d, $J = 145.5$ Hz, of t, $J = 21$ Hz), $P(CH_3)_3$, δ 1.05 (d, $J = 7.5$ Hz of d, J (H-hydride) = 1 Hz), 1.52 (d, $J = 10$ Hz), C_3H_5, δ 2.5 (br d), 3.8 (br m). Compound 7 has not yet been obtained analytically pure.

(21) Binuclear methylene and carbyne complexes react with ethylene: Sumner, C. E., Jr.; Riley, P. E.; Davis, R. E.; Pettit, R. *J. Am. Chem. Soc.* **1980**, *102*, 1752–1754. Theopold, K. H.; Bergman, R. G. *Ibid.* **1981**, *103*, 2489–2491. Dyke, A. F.; Guerchais, J. E.; Knox, S. A. R.; Roue, J.; Short, R. L.; Taylor, G. E.; Woodward, P. *J. Chem. Soc., Chem. Commun.* **1981**, 537–538.

(22) Puddephatt, R. J. *Coord. Chem. Rev.* **1980**, *33*, 149–194 and references therein.

or related reactions can be observed in different transition-metal systems.

Registry No. 1, 81360-30-7; 2, 81360-32-9; 6, 81360-33-0; [Ir(P(CH_3)$_3$)$_4$][Cl], 60314-48-9.

Crystal and Molecular Structure of Tribenzylaluminum, a Novel η^1-Arene Coordinated Structure

A. F. M. Maqsudur Rahman, Kaniz F. Siddiqui, and John P. Oliver*

Department of Chemistry, Wayne State University
Detroit, Michigan 48202

Received February 22, 1982

Summary: The molecular and crystal structure of tribenzylaluminium has been determined by single-crystal X-ray diffraction techniques. The compound was found to crystallize in the orthorhombic system, space group *Pbca*, with unit-cell dimensions of a = 9.480 (3) Å, b = 34.248 (11) Å, c = 10.546 (5) Å, d_{calcd} = 1.165 g cm^{-3}, V = 3424 (2) Å^3, and Z = 8. Full-matrix least-squares refinement on 1100 data gave R_F = 5.3% and R_{wF} = 5.6%.

The structures for a wide variety of organoaluminum compounds have been reported and show that these species form bridged dimers in the solid state. The structures of trimethylaluminum,[1] of triphenyl-,[2] dimethyl-μ-phenyl-,[3] and tri-*o*-tolylaluminum,[4] of diisobutyl-μ-(*trans-tert*-butylvinyl)-aluminum,[5] and of tricyclopropylaluminum[6] have all been determined and shown to have symmetrically bridge bonds. Diphenyl-μ-(phenylethyl)-aluminum,[7] and dimethyl-μ-cyclopentadienyl-aluminum, on the other hand, are unsymmetrically bridged. The only monomeric species for which structures have been reported are trimethylaluminum[9] and dimethylcyclopentadienylaluminum,[10] both determined by electron diffraction under conditions which lead to dissociation.

We now wish to report the crystal and molecular structure of tribenylaluminium, a molecule which has a unique structure in the solid state with the Al(CH_2Ph)$_3$ units bound together through strong intermolecular interactions between C(22)′, one of the ortho carbon atoms of the phenyl group on the adjacent molecular unit, and the vacant p orbital of the aluminum atom.

Experimental Data. Tribenzylaluminum was prepared from dibenzylmercury[11] (6.0 g, 0.015 mol) and aluminum powder (4.0 g, 0.15 mol, 8–20 mesh) following the literature procedure.[12] The reactants were placed in a tall cylindrical vessel, equipped with a standard taper joint and stopcock. The vessel was evacuated and 50-60 mL of toluene distilled into it. It was attached to a pressure equalizing system filled with Ar gas to maintain approximate atmospheric pressure. It as then heated in an oil bath (~100 °C) continuously for 3 days. The reaction mixture was worked up by using standard Schlenk techniques, yield ~60% of purified Al(CH_2Ph)$_3$.

Table I. Experimental Data from the X-ray Diffraction Study on Tribenzylaluminum

mol formula	$AlC_{21}H_{21}$
mol wt	300.38
cryst syst	orthorhombic
space group	*Pbca*
cell dimens	
a	9.480 (3) Å
b	34.248 (11) Å
c	10.546 (5) Å
vol	3424 (2) Å^3
Z	8
D_{calcd}	1.165 g cm^{-3}
radiation Mo	K$\bar{\alpha}$ (λ = 0.71069 Å)
monochromator	graphite crystal
reflctns measd	$h, {}^+k, {}^+l$
2θ range	45°
scan type	ω
scan speed	2.0° min^{-1}
bkgd measuremt	the ratio of the background to scan time was 0.5.
std reflctns	3 std reflctns measd every 97 reflctns; no significant deviation from the mean was observed
unique data	2604
unique data with $F^2 \geqslant 2.5\sigma(F_o^2)$	1100
abs coeff, μ	1.082 cm^{-1}
F(000)	1280
max shift/error for last least squares cycle	0.00749
R_F	5.3% where $R_F = \Sigma \mid \lvert F_o \rvert - \lvert F_c \rvert \mid / \Sigma \lvert F_o \rvert$
R_{wF}	5.6% where $R_w = \{\Sigma w(\lvert F_o \rvert - \lvert F_c \rvert)^2 / \Sigma w \lvert F_o \rvert^2\}^{1/2}$

A suitable single crystal for X-ray diffraction studies was grown from a benzene solution and was mounted in a thin-walled glass capillary tube under an Ar atmosphere in a drybox. The tube was plugged and, on removal from the drybox, flame sealed and mounted on a goniometer head. The data were collected on a Syntex P2_1 automated diffractometer with Mo K$\bar{\alpha}$ radiation diffracted from a highly oriented graphite crystal in the bisecting condition with a ω scan. The specific conditions, unit cell, unit dimensions, and other experimental parameters are given in Table I.

Solution and Refinement of the Structure. The crystal was found to be orthorhombic and was assigned to the space group *Pbca* on the basis of systematic absences. The crystallographic data on the unit cell and other pertinent data are collected in Table I. The structure was initially solved by light atom techniques through the use of MULTAN[13] which gave positions for all 22 nonhydrogen atoms. The hydrogen atom positions were calculated by

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[1] Lewis, P. H.; Rundle, R. E. *J. Chem. Phys.* **1953** *21*, 987. Vranka, R. G.; Amma, E. L. *J. Am. Chem. Soc.* **1967**, *89*, 3121. Huffman, J. C. C.; Streib, W. E. *J. Chem. Soc. D* 1971, 911.

[2] Malone, J. F.; McDonald, W. S. *J. Chem. Soc., Dalton Trans.* **1972**, 2646.

[3] Malone, J. F.; McDonald, W. S. *J. Chem. Soc., Dalton Trans.* **1972**, 2649.

[4] Barber, M.; Liptak, D.; Oliver, J. P., submitted for publication in *Organometallics*.

[5] Albright, M. J., Butler, W. M.; Anderson, T. J.; Glick, M. D.; Oliver, J. P. *J. Am. Chem. Soc.* **1976**, *98*, 3995.

[6] Ilsley, W. H.; Glick, M. D.; Oliver, J. P.; Moore, J. W. *Inorg. Chem.* **1980**, *19*, 3572.

[7] Stucky, G. D.; McPherson, A. M.; Rhine, W. E.; Eisch, J. J.; Considine, J. L. *J. Am. Chem. Soc.* **1974**, *96*, 1941.

[8] Teclé, B.; Corfield, P. W. R.; Oliver, J. P. *Inorg. Chem.* **1982**, *21*, 458.

[9] Almenningen, A.; Halvorsen, S.; Haaland, A. *Acta Chem. Scand.* **1971**, *25*, 1937.

[10] Drew, D. A.; Haaland, A. *Acta Chem. Scand.* **1973**, *27*, 3735.

[11] Nesmeyonov, A. V.; Borisov, A. E.; Novikova, N. V. *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk* **1959**, 1216.

[12] Eisch, J. J.; Biedermann, J.-M. *J. Organomet. Chem.* **1971**, *30*, 167.

[13] Germain, G.; Main, P.; Wolfson, M. M. *Acta Crystallogr., Sect. B* **1970**, *B26*, 274.

0276-7333/82/2301-0881$01.25/0