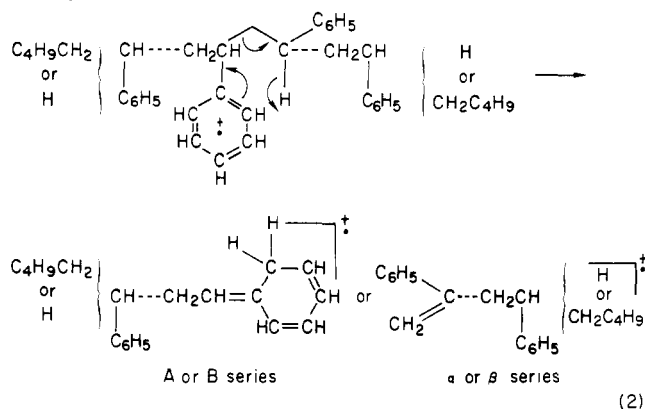


Figure 1. Fragment ion intensities in the MIKE spectrum of the m/z 2556.6 $[M]^+$ ion plotted against the bond numbers of the backbone bonds broken in their formation.

heating current (ehc),⁷ disappeared if the ehc was lowered below that threshold, and reappeared if the ehc was raised again. Thresholds were higher for $[M]^+$ ions of higher masses.⁸ Above threshold, MIKE spectra were not sensitive to ehc. Peak intensities in the MIKE spectrum of the m/z 2556.6 $[M]^+$ ion are plotted against the bond numbers [see (1)] of the ruptured bonds (Figure 1). The peaks fall into four series (A, B, α , and β) with nominal masses given by $m_A = n104 + 58$, $m_B = n104 + 92$, $m_\alpha = n104$, and $m_\beta = n104 + 70$. Mechanism 2 accounts for all four series of fragment ions.



The effects of ehc on the MIKE spectra indicate that the internal energy E of an $[M]^+$ ion stems from thermal energy of its neutral precursor.¹⁰ The thresholds are taken to be the ehc's above which there are detectable numbers of $[M]^+$ ions possessing internal energy E appropriate for decomposition within the experimental time window (20–200 μ s). The shift of the threshold to higher ehc as the masses of the $[M]^+$ ions increase is attributed to the slower rise of the rate constant $k(E)$ with E as the number of internal degrees of freedom increases,¹¹ i.e., a kinetic effect akin to kinetic shift.¹²

Consider the B series of fragment ions and compare rupture at bond 5 with rupture at bond 25 in the m/z 2556.6 $[M]^+$ ion. The fragment ion from rupture at bond 5 is the longer of the two fragment ions by 10 repeat units, and the neutral product from

rupture at bond 5 is the shorter of the two neutrals by the same amount. The fragment ion from rupture at bond 5 will, therefore, have a lower heat of formation than the fragment ion from rupture at bond 25, whereas the neutral from rupture at bond 5 will have a higher heat of formation than its counterpart from rupture at bond 25. We propose that, because of charge delocalization, the heats of formation of the ions depend more strongly upon size than the heats of formation of the neutrals.¹³ In which case, the difference between the heats of formation of the two fragment ions will be greater than the difference between the heats of formation of the two neutral products, and the sum of the heats of formation of the products of bond-5 rupture must be lower than that of the products of bond-25 rupture. We propose that it is this difference in the values of the combined heats of formation of products, reflected in a difference in the critical energies, which is responsible for the fragment ion from bond-5 rupture being much more intense than that from bond-25 rupture.^{14,15} The reversal in the pattern of charge retention (β series replacing B series, for example), once the midpoint of the $[M]^+$ chain is passed, supports our proposal that the size of the ionic fragment is the factor directing fragmentation. RRKM calculations^{11a,12c} indicate that an energy difference of about 10 kJ mol⁻¹ could account for this observed difference in intensities, provided the critical energies for decomposition did not exceed about 100 kJ mol⁻¹. The hypothesis proposed implies that the charge is effectively delocalized in the polystyrene $[M]^+$ ion, since ionization could occur at any of the aromatic nuclei.

Acknowledgment. We are pleased to acknowledge financial support under the Australian Research Grants Scheme.

Registry No. Polystyrene radical cation, 76582-08-6.

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(14) The argument extends to fragment ions between bonds 5 and 29 and to the other series. The reduced intensity of bond-3 and absence of bond-1 fragment ions are attributed to high heats of formation of small neutrals.

(15) Expressed differently, rupture at bond 5 is favored because the ionization energy of the neutral precursor $C_7H_7(CH_2CH(C_6H_5))_2H$ of the bond-5 fragment ion is lower than that of the neutral precursor $C_7H_7(CH_2CH(C_6H_5))_{11}H$ of the bond-25 fragment ion, cf. Stevenson's rule.¹²

Photoinduced α -Hydrogen Elimination of an Iridium(III) Dialkyl: Formation of an Isolable Iridium Methylidene

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Although α -elimination from transition-metal alkyls³ might be expected to be a general route to alkylidene derivatives,⁴ such reactivity has only been well documented for the early metals.⁵ In fact, there are only two reports of α -elimination involving a group 8–10 metal alkyl: the isolation⁶ of $[Ir=C(CH_2CH_2P(t-$

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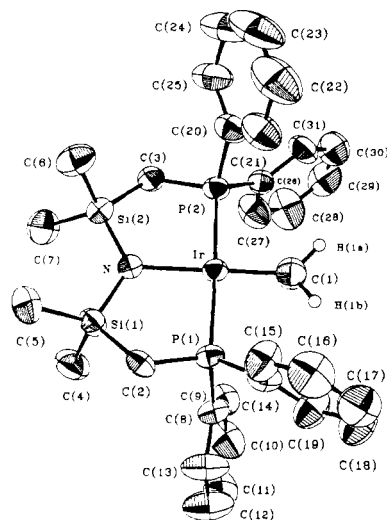
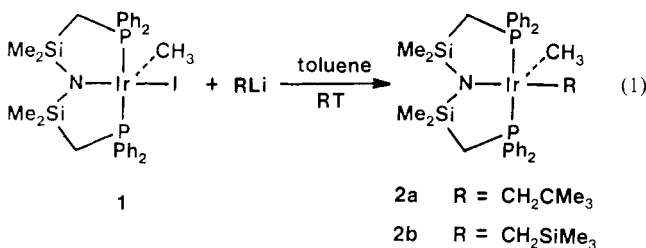


Figure 1. ORTEP diagram and labeling scheme for $\text{Ir}(\text{CH}_2)[\text{N}(\text{SiMe}_2\text{CH}_2\text{PPh}_2)_2]$. Selected bond lengths (Å) and bond angles (deg): Ir–C(1), 1.868 (9); Ir–N, 2.080 (5); Ir–P(1), 2.286 (2); Ir–P(2), 2.290 (2); C(1)–H(1a), 1.00 (9); C(1)–H(1b), 1.01 (8); Ir–C(1)–H(1a), 132 (5); Ir–C(1)–H(1b), 123 (5); H(1a)–C(1)–H(1b), 99 (7).

$\text{Bu})_2)_2\text{Cl}]$ from the thermolysis of $[\text{IrCH}(\text{CH}_2\text{CH}_2\text{P}(t\text{-Bu})_2)_2(\text{H})\text{Cl}]$ and the inference⁷ of an alkylidene species in a platinum metallacycle/alkene rearrangement. Until now, transformations of simple hydrocarbyl derivatives of the group 8–10 metals have resulted in no isolable or even spectroscopically observable alkylidene derivatives, presumably owing to available decomposition routes to more stable species.⁸ We wish to report the photo-synthesis and X-ray crystal structure of an isolable iridium methylidene⁹ complex, which, on the basis of labeling experiments and product analysis, appears to form via an α -hydrogen elimination sequence.

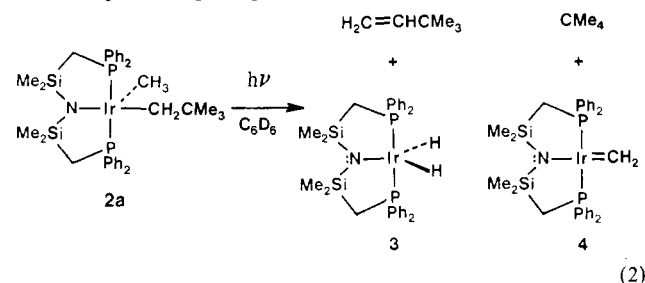
The reaction of organolithium reagents with the square-pyramidal iridium(III) methyl iodide derivative¹⁰ **1** is a facile process, at least for those alkyl groups that have no β -hydrogens. In particular, reaction of $\text{Ir}(\text{CH}_3)\text{I}[\text{N}(\text{SiMe}_2\text{CH}_2\text{PPh}_2)_2]$ with neopentyl lithium at room temperature in toluene results in a rapid color change from deep green to cranberry red. Recrystallization from hexanes at -30°C results in purple-red crystals¹¹ of **2a** in >80% isolated yields (eq 1). A similar reaction obtains for **1** with [(tri-



methylsilyl)methyl]lithium, resulting in deep purple crystals¹¹ of **2b**. To our knowledge, these are the first examples of stable five-coordinate iridium dialkyls.

Both of the iridium dialkyl complexes **2a** and **2b** are thermally stable¹² even at temperatures above 80°C for extended periods

of time. However, exposure of a C_6D_6 solution of **2a**, sealed under N_2 , to sunlight (or a 275-W sunlamp) results in an approximately 55:45 mixture of two iridium species: a five-coordinate iridium dihydride **3**¹³ as the major product and a new complex¹¹ **4**, the first example of a square-planar iridium methylidene (eq 2). Also



produced in this reaction are *tert*-butylethylene and neopentane in the approximate ratio of 55:45, respectively (by ^1H NMR and GC/MS).

Diagnostic of an iridium-bound methylidene unit in **4** is a downfield triplet in the ^1H NMR at 16.3 ppm ($^3J_{\text{P}} = 14.5$ Hz). The $^{13}\text{C}\{^1\text{H}\}$ NMR of 93% ^{13}C -enriched **4**, made by photochemical decomposition of 93% ^{13}C -enriched **2a**, has a triplet at 200.1 ppm ($^2J_{\text{P}} = 11$ Hz) for the methylidene carbon; in the proton-coupled ^{13}C NMR, this resonance is further split into a triplet of triplets ($^1J_{\text{H}} = 136$ Hz). Also produced from the ^{13}C -enriched starting material **2a** is $^{13}\text{CH}_2=\text{CHCMe}_3$ (93% ^{13}C by ^1H NMR). The deuterated starting material, $\text{Ir}(\text{CD}_3)\text{CH}_2\text{CMe}_3[\text{N}(\text{SiMe}_2\text{CH}_2\text{PPh}_2)_2]$ (99.5% ^2H enriched), was also prepared and the fate of the deuterium label was checked by both ^1H and ^2H NMR. The only products observed in the ^2H NMR after photolysis¹⁴ were $\text{Ir}(\text{CD}_2)[\text{N}(\text{SiMe}_2\text{CH}_2\text{PPh}_2)_2]$ and CH_2DCMe_3 in a 2:1 deuterium ratio, as well as $\text{Ir}(\text{H})\text{D}[\text{N}(\text{SiMe}_2\text{CH}_2\text{PPh}_2)_2]$ and $\text{CD}_2=\text{CHCMe}_3$ in a deuterium ratio of 1:2; the ^1H NMR analysis complements the above results. When the photolysis of **2a** is periodically monitored by $^{31}\text{P}\{^1\text{H}\}$ NMR, only singlets due to the starting dialkyl **2a**, the dihydride **3**, and the iridium methylidene **4** can be observed at any time.

Single-crystal X-ray analysis¹⁵ of purple crystals of **4** indicates that the $\text{Ir}=\text{CH}_2$ unit is coplanar with the square plane of the complex.¹⁶ The Ir–C(1) distance of 1.869 (9) Å is nearly identical to the iridium–carbon bond length of 1.872 (7) Å found¹⁸ in the octahedral iridium(III) dichlorocarbene complex $\text{IrCl}_3(\text{CCl}_2)(\text{PPh}_3)_2$.

On the basis of the ^{13}C and ^2H labeling studies outlined above, it is apparent that the iridium methylidene complex arises from the coordinatively unsaturated dialkyl **2a** via an α -elimination sequence. We will speculate on the details of this process and report the reactivity of the iridium methylidene in future publications.

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(14) This experiment was performed after approximately 60% conversion of reactant **2a**; if this mixture is allowed to sit, a certain amount of scrambling occurs which is currently under investigation.

(15) Crystals of $\text{Ir}(\text{CH}_2)[\text{N}(\text{SiMe}_2\text{CH}_2\text{PPh}_2)_2]$ are triclinic, $a = 11.404$ (2) Å, $b = 15.358$ Å, $c = 11.097$ (2) Å, $\alpha = 86.62$ (2)°, $\beta = 105.53$ (1)°, $\gamma = 95.77$ (1)°, $Z = 2$, space group $P\bar{1}$. The structure was solved by conventional heavy-atom methods and was refined by full-matrix least-squares procedures to $R = 0.040$ and $R_w = 0.047$ for 5861 independent absorption-corrected reflections with $I \geq 3\sigma(I)$ collected at 22°C with Mo $K\alpha$ radiation on an Enraf-Nonius CAD4-F diffractometer. The methylidene hydrogen atoms were refined and the remaining hydrogen atoms were fixed in idealized positions.

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and to Professor James Trotter for the use of the diffractometer and structure-solving programs.

Supplementary Material Available: Experimental details for the preparation of $\text{Ir}(\text{CH}_2)[\text{N}(\text{SiMe}_2\text{CH}_2\text{PPh}_2)_2]$, ^1H NMR and microanalytic data for all new compounds, tables of final positional and isotropic thermal parameters, calculated hydrogen coordinates, final anisotropic thermal parameters, bond lengths, bond angles, intraannular torsion angles, torsion angles, and structure factor amplitudes (observed and calculated) (47 pages). Ordering information is given on any current masthead page.

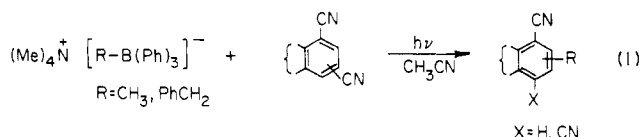
Photoalkylation of Dicyanoarenes with Alkyltriphenylborate Salts

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The promising synthetic utility of light-induced single-electron-transfer (SET) reactions has been partly realized in the generation of stabilized radicals by oxidation of olefins,¹ amines,² silanes,³ or stannanes⁴ with following loss of an electrophilic group. Herein we introduce a new class of photooxidizable reagents, alkyltriphenylborates, that enable the facile transfer of an alkyl group to an electron acceptor. Irradiation of acetonitrile solutions of dicyanoarenes and either methyl- or benzyltriphenylborate gives good yields of alkylcyanoarenes, eq 1.



The preparation of methyl- (1) or benzyltriphenylborate (2) proceeds smoothly from the alkylolithium and triphenylboron.⁵ The borates are easily isolated as their crystalline tetramethylammonium salts.⁶ These salts are stable in air and protic solvents and are soluble in acetonitrile.

The electrochemical oxidation of tetra-*n*-butylborate is reported to generate the butyl radical.⁷ The cyclic voltammograms of borates 1 and 2 exhibit irreversible one-electron oxidation waves at ca. 1.11 and 0.85 V vs. SCE, respectively. We suspect that cleavage of the alkyl carbon-boron bond⁸ follows oxidation of 1 or 2.

The fluorescence of 1,4-dicyanonaphthalene (DCN) is quenched by borate 2 with a bimolecular rate constant at the diffusion limit ($1.6 \times 10^{10} \text{ M}^{-1} \text{ s}^{-1}$ in CH_3CN). This finding is not unexpected since the free energy of electron transfer from 2 to $\text{DCN}^{\bullet+}$ is exothermic by more than 30 kcal/mol.⁹ The generation of $\text{DCN}^{\bullet-}$ in this reaction is confirmed by measurement of its characteristic absorption spectrum using laser flash photolysis techniques.¹⁰

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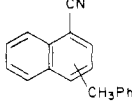
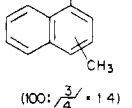
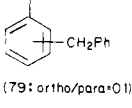
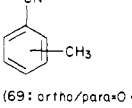
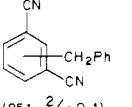
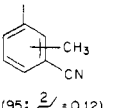
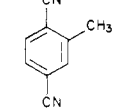
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Table I. Product Distribution of Photoreaction between Borate (1 or 2) and Dicyanoarene (DCN, *p*-DCB, or *m*-DCB)

$[\text{R}-\text{BPh}_3]^-$ $[\text{N}(\text{CH}_3)_4]^+$	Ar(CN) ₂	conv, % ^a	product, % ^{b,c}
R = PhCH ₂	DCN	78	 (95: 3/4 = 0.6)
R = CH ₃	DCN	65	 (100: 3/4 = 1.4)
R = PhCH ₂	<i>p</i> -DCB	38	 (79: ortho/para = 0.1)
R = CH ₃	<i>p</i> -DCB	21	 (69: ortho/para = 0.4)
R = PhCH ₂	<i>m</i> -DCB	59	 (85: 2/4 = 0.1)
R = CH ₃	<i>m</i> -DCB	71	 (95: 2/4 = 0.12)  (27)

^a Conversions are based on loss of borates for DCN and on loss of dicyanoarenes for *p*-DCB and *m*-DCB. ^b The yields are determined by gas chromatography except for 2 and *p*-DCB where the products were isolated. ^c A low yield of toluene is obtained from all reactions of 2.

Irradiation (350 nm) of DCN (11.2 mM) in deoxygenated acetonitrile containing methylborate 1 (8.3 mM) gives two methylated cyanonaphthalenes in essentially quantitative yield, Table I. The methylcyanonaphthalenes were isolated by chromatography and identified as 3- and 4-methyl-1-cyanonaphthalene in a ratio of 7:5 by comparison with authentic material independently prepared.¹¹ Similarly, photolysis of DCN in acetonitrile solution containing benzylborate 2 gives a 95% yield of 3- and 4-benzyl-1-cyanonaphthalene¹³ in this case, however, the former isomer is the minor product. A low yield of toluene is also observed in this reaction.

The issue is slightly more complex when *m*- or *p*-dicyanobenzene (*m*-DCB, *p*-DCB) is photolyzed in place of DCN. Irradiation (Hg arc, Pyrex) of *p*-DCB in the presence of methylborate 1 gives

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