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LIGAND EXCHANGE REACTIONS WITH η^6-ARENE-η^5-CYCLOPENTADIENYLIRON CATIONS UNDER THERMAL OR PHOTOCHEMICAL CONDITIONS

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Summary

Reactions of various η^6-arene-η^5-cyclopentadienyliron or substituted cyclopentadienyliron cations with trimethyl, triethyl or triphenyl phosphite under either thermal or photochemical conditions all resulted in the replacement of the arene ligand with three phosphite ligands to give η^6-tris(trimethyl, triethyl or triphenyl phosphite)-η^5-cyclopentadienyliron or substituted cyclopentadienyliron cations. The yields of the phosphite complexes were higher from photolysis than from the analogous thermolysis. Photolysis of the η^6-chlorobenzene-η^5-cyclopentadienyliron cation (IX) carried out in the presence of a more basic or more electron-rich aromatic ligand resulted in the exchange of the chlorobenzene of IX with the more basic arene, thus providing synthetic routes to cyclopentadienyliron complexes that may be difficult to prepare by other means. New complexes synthesized in this way are the η^6-2-phenylethyl tosylate-η^5-cyclopentadienyliron cation and the $CpFe^+$ complexes of thiophene, 2-methylthiophene, 3-methylthiophene and 2,5-dimethylthiophene.

Introduction

Ligand exchange reactions between ferrocene and arenes, carried out in the presence of $AlCl_3/Al$, as originally reported by Nesmeyanov and coworkers [1,2], have been extensively utilized in preparations of η^6-arene-η^5-cyclopentadienyliron cations [3]. We wish to report the use of the η^6-chlorobenzene-η^5-cyclopentadienyliron cation as reactant in photochemically induced ligand exchanges with arenes that are more electron rich than chlorobenzene as an alternative method for the preparation of cyclopentadienyliron complexes of such arenes. Leading to the choice of the chlorobenzene complex as substrate, we investigated the replacement of the arene ligand in η^6-arene-η^5-cyclopentadienyliron cations with phosphite ligands and this work is also described.

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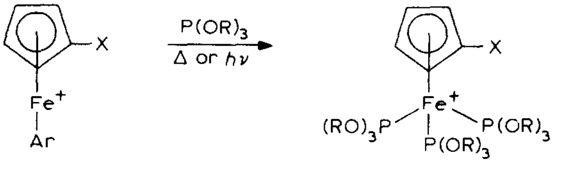
Results and discussion

Gill and Mann [4,5] have investigated the photolysis of the hexafluorophosphate salt of the η^6-p-xylene-η^5-cyclopentadienyliron cation, carried out in the presence of 6- or 2-electron donor ligands. Replacement of the arene ring by one 6-electron or three 2-electron ligands was observed and synthetic applications of such reactions have been pointed out. Among the compounds synthesized were the hexafluorophosphates of the η^6-tris(trimethyl, triethyl and triphenyl phosphite)-η^5-cyclopentadienyliron cations, $[CpFe(P(OCH_3)_3)_3]^+$ (Ia) [5], $[CpFe(P(OC_2H_5)_3)_3]^+$ (Ib) [5], and $[CpFe(P(OC_6H_5)_3)_3]^+$ (Ic) [4]. We have found that similar ring replacements or ligand exchanges also took place under thermal conditions at 130–140°C [6]. When the hexafluorophosphate of an η^6-arene-η^5-cyclopentadienyliron cation was heated in an excess of triethyl phosphite, the product obtained was Ib and the same product, Ib, was formed regardless of whether the arene ligand in the $[CpFeArene]^+$ cation was nitrobenzene, *o*-, *m*- or *p*-nitrotoluene, aniline or *o*-, *m*- or *p*-toluidine [6]. Similarly, reaction of η^6-nitrobenzene or aniline-η^5-methylcyclopentadienyliron hexafluorophosphate with $P(OC_2H_5)_3$ gave the η^6-tris(triethyl phosphite)-η^5-methylcyclopentadienyliron cation (IIb) [6]. In the present work, we have extended these studies by investigating the effects of substituents on the Cp ring in the arene replacement reactions under both thermal and photochemical conditions.

Gill and Mann [5] have pointed out that the photochemical efficiency of the ring dissociation, and hence its replacement, may be related to the basicity of the arene ring, and as the ring becomes more basic, the ease of its replacement decreases. In the reactions of η^6-arene-η^5-cyclopentadienyliron cations (to be called the arene complex) with trialkyl phosphites, the ligand that was replaced or has undergone exchange was the arene ring, giving rise to η^6-tris(trialkyl phosphite)-η^5-cyclopentadienyliron cations (to be called the phosphite complex). It is of interest to find out if electron-withdrawing substituents on the Cp ring might be able to decrease its electron density or basicity sufficiently to allow for a replacement or exchange of the substituted Cp rather than the arene ligand. Reactions of $P(OR)_3$, $R = CH_3$, C_2H_5 or C_6H_5, with $[ArFeCp(X)]^+$, $X = H$, CH_3, C_2H_5, CH_3CO, Cl or COOR and Ar = benzene or various substituted benzenes, were carried out under both thermal and photochemical conditions. In all cases, phosphite complexes (Ia, Ib, Ic to VIa, VIb, Vc) were obtained, as shown in eq. 1, regardless of whether the substituent X on the Cp ring was electron releasing or electron withdrawing.



(1)

(Ia, Ib, Ic – VIa, VIb, VIc)
(I to VI, $X = H$, CH_3, C_2H_5, CH_3CO,
Cl, and COOR, respectively; a, b, and c, $R = CH_3$, C_2H_5, C_6H_5,
respectively)

Although no replacement of the Cp ring was observed, the work did give rise to phosphite complexes Ia, Ib, Ic to VIa, VIb, VIc. Moreover, the ester group in VIa, VIb, VIc could be hydrolyzed to give the η^6-tris(trimethyl, triethyl and triphenyl phosphite)-η^5-carboxycyclopentadienyliron cations (VIIa, VIIb, VIIc). Thus 21 phosphite complexes have been prepared, only 4 of which, Ia, Ib, Ic and IIb, have previously been reported [4–6]. It was also found that in all the preparations of the phosphite complexes given by eq. 1, the yield derived from a photolysis was higher than that derived from the corresponding thermal reaction. Some data illustrating these differences are given in Table 1.

In their work on the photolysis of the η^6-*p*-xylene-η^5-cyclopentadienyliron cation (VIII), Gill and Mann [5] also observed the replacement of *p*-xylene with cycloheptatriene, cyclooctatetraene and 2,2-paracyclophane, while similar exchanges of the *p*-xylene ligand in VIII with various metacyclophanes have been studied by Swann and Boekelheide [7]. In the present work, further ligand exchange studies were carried out utilizing the η^6-chlorobenzene-η^5-cyclopentadienyliron cation IX as a reactant. The choice of IX with an electron withdrawing chloro substituent on the arene ring should allow for a wider variety of exchange reactions with more basic arene ligands. Photochemical conditions were employed in these studies since it was found earlier that photolysis gave higher yields than thermolysis in the preparation of the various phosphite complexes. Thus the photolysis of IX in the presence of an excess of benzene or toluene gave the η^6-benzene-η^5-cyclopentadienyliron or η^6-toluene-η^5-cyclopentadienyliron cation in yields of 80 or 85%, respectively, while no exchange was observed in a similar treatment of IX with nitrobenzene. Interestingly, photolysis of IX in CH_2Cl_2 containing an excess of tetralin or 9,10-dihydroanthracene also resulted in ligand exchange to give the known η^6-tetralin-η^5-cyclopentadienyliron or η^6-9,10-dihydroanthracene-η^5-cyclopentadienyliron cation [8], while similar photolysis in the presence of naphthalene or anthracene failed to give any

TABLE 1

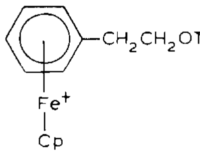
SOME DATA ON THE YIELDS OF PHOSPHITE COMPLEXES FROM LIGAND EXCHANGE REACTIONS BETWEEN ARENE COMPLEXES AND TRIMETHYL, TRIETHYL OR TRIPHENYL PHOSPHITE UNDER THERMAL OR PHOTOCHEMICAL CONDITIONS

Arene [a]	Yield (%)					
	Ia [b]		IIb [c]		VIc [d]	
	Δ	hν	Δ	hν	Δ	hν
Benzene	40	82	41	80	26	67
p-Xylene	41	77	44	78	29	64
p-Chlorotoluene	43	69	43	77	31	59
m-Chlorotoluene	44	68	37	77	27	60
Aniline	46	72	60 [e]	79	30	68
o-Toluidine	41	70	42	78	26	61
m-Toluidine	40	68	43	75	27	62
p-Toluidine	47	74	46	80	31	62
Nitrobenzene	48	71	36 [e]	75	–	–

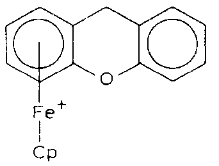
[a] Arene in the hexafluorophosphate salt of an arene complex, $[Cp(X)FeAr]^+$ PF_6^-. [b] $[CpFe(P(OCH_3)_3)_3]^+$, isolated as its hexafluorophosphate, from reaction of various $[CpFeAr]^+$ PF_6^- with $P(OCH_3)_3$. [c] $[Cp(CH_3)Fe(P(OC_2H_5)_3)_3]^+$, isolated as its hexafluorophosphate, from reaction of various $[Cp(CH_3)FeAr]^+$ PF_6^- with $P(OC_2H_5)_3$. [d] $[Cp(COOC_6H_5)Fe(P(OC_6H_5)_3)_3]^+$, isolated as its hexafluorophosphate, from reaction of various $[Cp(COOC_6H_5)FeAr]^+$ PF_6^- with $P(OC_6H_5)_3$. [e] From ref. 6.

ligand exchange. This difference possibly may be rationalized by considering that tetralin and 9,10-dihydroanthracene are similar to a benzene ring with two saturated, electron-donating *o*-substituents while naphthalene and anthracene do not have such saturated substituents; moreover, complexation of one of the aromatic rings of naphthalene or anthracene may result in the loss of aromatic resonance in the adjacent ring in these fused aromatic hydrocarbons.

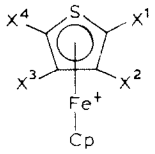
The arene complexes described above from the exchange of the chlorobenzene ligand of IX with other arenes are known compounds that have previously been prepared from ligand exchange reactions between ferrocene and various arenes carried out in the presence of $AlCl_3$ and Al powder [3]. However, the present method generally gave higher yields, and furthermore, it may also provide an alternative route to cyclopentadienyliron complexes that may not be readily prepared by the Nesmeyanov procedure [1,2]. One such example is the preparation of the η^6-2-phenylethyl tosylate-η^5-cyclopentadienyliron cation (X). Extensive solvolytic studies on 2-phenylethyl tosylate, especially in relation to 1,2-phenyl shifts in the 2-phenylethyl cation, have been reported [9]. It would be of interest to investigate the possibility of similar 1,2-shifts in X, with the phenyl group of 2-phenylethyl tosylate complexed to the cyclopentadienyliron moiety. The preparation of X via ligand exchange between ferrocene and 2-phenylethyl tosylate should be avoided since $AlCl_3$ may interact with the tosylate function. 2-Phenyl[1-^{14}C]ethyl tosylate has been prepared by the $LiAlH_4$ reduction of labeled phenylacetic acid to the alcohol followed by conversion to the tosylate [10]. However, an attempted analogous reduction of the η^6-phenylacetic acid-η^5-cyclopentadienyliron cation with $LiAlH_4$ failed to give the desired product because of complications arising from the addition of hydride ion to the complexed phenyl ring. Utilizing the presently developed method of photochemically induced ligand exchanges between arenes, it was found that photolysis of IX in CH_2Cl_2 containing two molar equivalents of 2-phenylethyl tosylate readily gave a 70% yield of X which was isolated as its hexafluorophosphate.



(X)



(XI)



(XII)

(a: $X^1 = X^2 = X^3 = X^4 = H$;
b: $X^1 = CH_3$, $X^2 = X^3 = X^4 = H$
c: $X^2 = CH_3$, $X^1 = X^3 = X^4 = H$
d: $X^1 = X^4 = CH_3$, $X^2 = X^3 = H$
e: $X^1 = X^2 = X^3 = X^4 = CH_3$)

Cyclopentadienyliron complexes of heterocyclic systems have been prepared from ligand exchanges between ferrocene and the heterocyclic compounds [11,12]. Usually, the complexation involves bonding of the Fe atom to a carbocyclic ring in the heterocyclic system such as in the η^6-xanthene-η^5-cyclopentadienyliron cation (XI). Direct bonding of the Fe atom to a ring containing the heteroatom is rather rare, and an example is the η^5-tetramethylthiophene-η^5-cyclopentadienyliron cation (XIIe) obtained from ligand exchange between ferrocene and tetramethylthiophene [13].

TABLE 2

YIELDS AND ^1H NMR DATA FOR THE CYCLOPENTADIENYLIRON COMPLEXES OF 2-PHENYLETHYL TOSYLATE (X), THIOPHENE (XIIa), 2-METHYLTHIOPHENE (XIIb), 3-METHYLTHIOPHENE (XIIc) AND 2,5-DIMETHYLTHIOPHENE (XIId)

Complex [a]	Yield (%)	δ (acetone-d_6) (ppm for TMS)		
		Cp	Complexed ring	Others
X	70	5.23(s,5H)	6.40(s,5H)	2.42(s, 3H, CH_3) 3.25(t, 2H, C(2) CH_2) 4.43(t, 2H, C(1) CH_2) 7.30–7.80(two d, 4H, OTs)
XIIa	75	5.20(s, 5H)	6.38(d, 2H, α-H's) 6.54(t, 2H, β-H's)	
XIIb	70	5.13(s, 5H)	6.20–6.50(m, 3H)	2.57(s, 3H, CH_3)
XIIc	75	5.17(s, 5H)	6.23–6.60(m, 3H)	2.57(s, 3H, CH_3)
XIId	85	5.16(s, 5H)	6.30(s, 2H)	2.53(s, 6H, CH_3)

[a] Isolated as the hexafluorophosphate. No melting point or decomposition temperature for any of the complexes was recorded because such data are not reproducible. The heating of a FeCp complex of an arene would result in the liberation of the arene ligand and pyrolytic sublimation has actually been employed as a method of regenerating the free arenes from such complexes [18,19].

The unsubstituted thiophene complex, however, has not been prepared. In order to test further the usefulness of the present method of ligand exchange, we have found that photolysis of IX in CH_2Cl_2 in the presence of 2-3 equivalents of thiophene, 2-methylthiophene, 3-methylthiophene or 2,5-dimethylthiophene all gave the expected complex, XIIa, XIIb, XIIc, or XIId, respectively. The yields and ^1H NMR data for X and XIIa, XIIb, XIIc, XIId are summarized in Table 2. The successful preparations of these complexes thus demonstrate that the photochemically induced ligand exchange with IX as a reactant is capable of giving wide synthetic applications.

Experimental

The arene complexes

The η^6-arene-η^5-cyclopentadienyliron or substituted cyclopentadienyliron cations required for the ligand exchange reactions with trimethyl, triethyl or triphenyl phosphite were prepared as their hexafluorophosphate salts from ligand exchanges between the arene and ferrocene or 1,1′-disubstituted ferrocene by the method of Nesmeyanov [1–3]. For these reactions, ferrocene, 1,1′-dimethyl-, 1,1′-diacetyl- and 1,1′-dicarboxy-ferrocenes were obtained commercially. 1,1′-Diethylferrocene was prepared by the Clemmensen reduction [14] of 1,1′-diacetylferrocene, while 1,1′-dichloroferrocene was obtained by converting ferrocene to 1,1′-dilithioferrocene followed by treatment of the latter with a tetrahydrofuran solution of 1,2-dichlorotetrafluoroethane [15,16].

The phosphite complexes (Ia, Ib, Ic to VIIa, VIIb, VIIc)

The reactions of various arene complexes with trimethyl, triethyl, or triphenyl phosphite under thermal conditions were carried out as previously described [6].

Each hexafluorophosphate of an arene complex was treated with 4 equivalents of the phosphite at 130–140°C under N_2 for 18 h. The excess phosphite was then removed by distillation under reduced pressure. The solid residue was dissolved in acetone and chromatographed through an alumina column (5 × 30 cm, packed with Fisher F-20 alumina). Elution with a 1/1 mixture of acetone/CH_2Cl_2 gave the hexafluorophosphates of the various phosphite complexes, Ia, Ib, Ic to VIa, VIb, VIc.

Under photochemical conditions, each hexafluorophosphate of an arene complex (2.0 mmol) was dissolved in 50 ml of CH_2Cl_2 in a 6 × 60 cm pyrex tube and the solution was deoxygenated by bubbling N_2 through it for 15 min. Trimethyl, triethyl, or triphenyl phosphite (5.0 equiv.) was then added. The reaction tube was attached to a reflux condenser fitted with a $CaCl_2$ tube, placed in the Rayonet photochemical reactor equipped with 3000 A lamps and then irradiated at ambient temperature for 5 h. The resulting solution was concentrated by the removal of most of the solvent under reduced pressure, and upon addition of hexane, the hexafluorophosphate of the phosphite complex precipitated.

The above procedure was used in the photochemical preparation of phosphite complexes Ia, Ib, Ic to Va, Vb, Vc. In the case of VIa, VIb, VIc, the arene complexes used as reactants were obtained from treatment of the η^6-arene-η^5-carboxycyclopentadienyliron hexafluorophosphates with $P(OCH_3)_3$, $P(OC_2H_5)_3$ or $P(OC_6H_5)_3$ thus converting the carboxyl group to the corresponding ester before irradiation. This conversion to the ester was necessary because of the low solubility of the carboxy-containing arene complexes in CH_2Cl_2. Basic hydrolysis of VIa, VIb, VIc regenerated the carboxyl group, giving rise to phosphite complexes VIIa, VIIb, VIIc.

The ^1H NMR spectra of all of these phosphite complexes are in agreement with their assigned structures. For example, in agreement with Gill and Mann [5], the ^1H NMR spectrum of $[CpFeP(OCH_3)_3]^+$ PF_6^- (Ia-PF_6) showed a quartet for the Cp protons at 4.84 ppm due to coupling with the 3 equivalent P nuclei, with the 27 trimethyl phosphite protons appearing as a complex multiplet at 3.7–3.9 ppm. As another illustration, in the ^1H NMR spectrum of $[Cp(COCH_3)Fe(P(OC_2H_5)_3)_3]^+$ PF_6^- (IVb-PF_6), the Cp protons appear as two 2-proton multiplets at 4.9–5.1 and 5.4–5.6 ppm, with the acetyl substituent on the Cp ring giving a 3-proton singlet at 1.9 ppm, while the triethyl phosphite ligands gave a 27-proton triplet at 1.5 ppm for the CH_3 groups and a complex 18-proton multiplet at 4.2–4.5 ppm for the CH_2 groups. The C and H analyses for these phosphite complexes are summarized in Table 3.

Ligand exchange reactions with the η^6-chlorobenzene-η^5-cyclopentadienyliron cation (IX)

A solution of 1.0 mmol of the hexafluorophosphate of IX [17] and 2–3 mmol of arene or thiophene in 20 ml of CH_2Cl_2 was deoxygenated with bubbling N_2 and then irradiated in a Rayonet photochemical reactor for 5 h. The resulting material was filtered and the solvent was removed under reduced pressure. The residue was washed several times with ether followed by decantation to remove the unreacted free ligand. The washed solid was redissolved in acetone, purified by passage through an alumina column, and upon addition of ether to the acetone solution, the hexafluorophosphate of the ligand exchange product precipitated. For the new ligand exchange products, X and XIIa, XIIb, XIIc, XIId, the ^1H NMR data are

TABLE 3

ANALYTICAL DATA FOR THE PHOSPHITE COMPLEXES

Complex	Analysis (Calcd. (Found) (%))	
	C	H
Ia, $C_{14}H_{32}O_9FeP_4F_6$	26.35	5.05
	(26.45)	(5.03)
Ib, $C_{23}H_{50}O_9FeP_4F_6$	36.14	6.59
	(36.83)	(6.94)
Ic, $C_{59}H_{50}O_3FeP_4F_6$	59.21	4.21
	(58.85)	(4.42)
IIa, $C_{15}H_{34}O_9FeP_4F_6$	27.62	5.25
	(27.48)	(5.10)
IIb, $C_{24}H_{52}O_9FeP_4F_6$	37.03	6.73
	(36.72)	(6.81)
IIc, $C_{60}H_{52}O_9FeP_4F_6$	59.51	4.33
	(59.41)	(4.22)
IIIa, $C_{16}H_{36}O_9FeP_4F_6$	28.84	5.45
	(28.78)	(5.59)
IIIb, $C_{25}H_{54}O_9FeP_4F_6$	37.89	6.87
	(37.93)	(6.78)
IIIc, $C_{61}H_{54}O_9FeP_4F_6$	59.81	4.44
	(60.01)	(4.46)
IVa, $C_{16}H_{34}O_{10}FeP_4F_6$	28.25	5.04
	(28.35)	(5.19)
IVb, $C_{25}H_{52}O_{10}FeP_4F_6$	37.23	6.50
	(37.30)	(6.57)
IVc, $C_{61}H_{52}O_{10}FeP_4F_6$	59.14	4.23
	(59.20)	(4.30)
Va, $C_{14}H_{31}O_9ClFeP_4F_6$	25.00	4.65
	(25.10)	(4.71)
Vb, $C_{23}H_{49}O_9ClFeP_4F_6$	34.58	6.18
	(34.75)	(6.24)
Vc, $C_{59}H_{49}O_9ClFeP_4F_6$	57.55	4.01
	(57.71)	(4.15)
VIa, $C_{16}H_{34}O_{11}FeP_4F_6$	27.60	4.92
	(28.00)	(5.04)
VIb, $C_{26}H_{54}O_{11}FeP_4F_6$	37.33	6.51
	(37.41)	(6.61)
VIc, $C_{66}H_{54}O_{11}FeP_4F_6$	60.19	4.13
	(60.79)	(4.29)
VIIa, $C_{15}H_{32}O_{11}FeP_4F_6$	26.41	4.73
	(26.12)	(4.78)
VIIb, $C_{24}H_{50}O_{11}FeP_4F_6$	35.66	6.23
	(36.00)	(6.27)
VIIc, $C_{60}H_{50}O_{11}FeP_4F_6$	58.08	4.06
	(58.40)	(4.14)

summarized in Table 2 and the C and H analyses are in agreement with their structures. As examples of the analytical data, for X-PF_6, Anal. Found: C, 44.33; H, 4.05. $C_{20}H_{21}O_3SFePF_6$ calcd.: C, 44.30; H, 3.90%. For XIIa-PF_6, Anal. Found: C, 35.34; H, 3.36. $C_{11}H_{13}SFePF_6$ calcd.: C, 34.94; H, 3.46%.

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Reference

1 A.N. Nesmeyanov, N.A. Vol'kenau, and I.N. Bolesova, Dokl. Akad. Nauk SSSR, 149 (1963) 615.
2 A.N. Nesmeyanov, N.A. Vol'kenau, and I.N. Bolesova, Tetrahedron Lett., (1963) 1725.
3 R.G. Sutherland, J. Organomet. Chem. Library, 3 (1977) 311.
4 T.P. Gill and K.R. Mann, Inorg. Chem., 19 (1980) 3007.
5 T.P. Gill and K.R. Mann, J. Organomet. Chem., 216 (1981) 65.
6 C.C. Lee, U.S. Gill, M. Iqbal, C.I. Azogu, and R.G. Sutherland, J. Organomet. Chem., 231 (1982) 151.
7 R.T. Swann and V. Boekelheide, J. Organomet. Chem., 231 (1982) 143.
8 R.G. Sutherland, S.C. Chen, J. Pannekoek, and C.C. Lee, J. Organomet. Chem., 101 (1975) 221.
9 C.J. Lancelot, D.J. Cram, and P.v.R. Schleyer, in G.A. Olah and P.v.R. Schleyer (Eds.), Carbonium Ions, Vol. III, Wiley-Interscience, New York, 1972, p. 1347–1483.
10 C.C. Lee, G.P. Slator, and J.W.T. Spinks, Can. J. Chem., 35 (1957) 1417.
11 J.F. Helling and W.A. Hendrickson, J. Organomet. Chem., 141 (1977) 99.
12 C.C. Lee, B.R. Steele, and R.G. Sutherland, J. Organomet. Chem., 186 (1980) 265.
13 P. Bachmann and H. Singer, Z. Naturforsch. B, 31 (1976) 525.
14 M.D. Rausch, M. Vogel, and H. Rosenberg, J. Org. Chem., 22 (1957) 903.
15 M.D. Rausch and D.J. Ciappenelli, J. Organomet. Chem., 10 (1967) 127.
16 R.F. Korar and M.D. Rausch, Organomet. Chem. Syn., 1 (1971) 173.
17 I.U. Khand, P.L. Pauson, and W.E. Watts, J. Chem. Soc. C, (1968) 2261.
18 R.G. Sutherland, J. Pannekoek, and C.C. Lee, Can. J. Chem., 56 (1978) 1782.
19 R.G. Sutherland, A. Piorko, U.S. Gill, and C.C. Lee, J. Heterocyclic Chem., 19 (1982) 801.