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CORRELATION OF ^{31}P CHEMICAL SHIFTS AND CARBONYL STRETCHING FORCE CONSTANTS WITH SUBSTITUENT CONSTANTS FOR A SERIES OF RING-SUBSTITUTED ARENE CHROMIUM DICARBONYL TRIPHENYLPHOSPHINE COMPLEXES

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Abstract—Fourteen mono-substituted arene chromium tricarbonyl and arene chromium dicarbonyl triphenylphosphine complexes have been prepared and characterized by IR and NMR spectroscopy. Carbonyl stretching force constants for all compounds have been calculated. Correlations of stretching forces constants of arene chromium tricarbonyl complexes with a series of substituent constants agree well with those reported in earlier studies. Similarly, both the stretching force constants and ^{31}P chemical shifts of arene chromium dicarbonyl triphenylphosphine complexes have been compared with the same series of substituent constants and were found to exhibit trends similar to those previously observed.

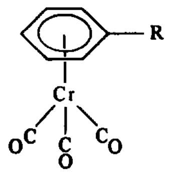
In our previous communications[1,2] we have demonstrated that the Taft substituent constants, σ_I and σ_R^0, and the carbonyl force constants of arene $Cr(CO)_2L$ and $CpMn(CO)_2L$ complexes vary in a regular way depending on the π acidity of the ligand L, where L is a phosphine or phosphite.

Several workers have investigated the mechanism of transmission of electron density from arene rings to a complexed metal atom or its ligands. A variety of physical parameters have been used to probe the changes in metal electron density which result from the introduction of substituents onto an arene ring. For example, carbonyl stretching frequencies and force constants, and ^{13}C NMR chemical shifts of the metal carbonyls have been measured for broad series of compounds. Senoff has exhaustively reviewed the literature on this subject up to 1979.[3] Neuse has presented a particularly thorough study of the changes in the carbonyl stretching frequencies and force constants for 28 arene chromium tricarbonyl compounds which were correlated with a set of selected substituent parameters.[4]

We have been concerned with the synthesis of a variety of arene chromium dicarbonyl phosphine compounds and have noted that the ^{31}P NMR chemical shifts appeared to be sensitive to substituents on the arene rings. A similar qualitative observation has been made by Setkina *et al.*[5] In order to determine the nature of this relationship, we have prepared a series of arene chromium dicarbonyl triphenylphosphine complexes and measured the ^{1}H and ^{31}P NMR chemical shifts as well as the carbonyl stretching frequencies for all compounds. The correlations between ^{31}P NMR chemical shift, carbonyl force constants and the same set of substituent constants examined by Neuse has been determined and is discussed in this communication.

RESULTS AND DISCUSSION

Fourteen mono-substituted arene chromium tricarbonyl (I) and arene chromium dicarbonyl triphenylphosphine (II) derivatives were prepared and their NMR and IR spectra recorded. In most cases it was possible to prepare good yields of the arene chromium tricarbonyl derivative by refluxing the arene with chromium hexacarbonyl in a mixture of



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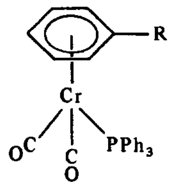

II

butyl ether and tetrahydrofuran as described by Top and Jaouen.[6] However, in the case of benzaldehyde and acetophenone, only low yields of the desired chromium complexes could be obtained. It was found that conversion of benzaldehyde and acetophenone to their dioxolane derivatives followed by metallation in the usual way gave excellent yields of the corresponding dioxolane chromium tricarbonyl derivatives which could be easily hydrolysed to the corresponding aldehyde and ketone complexes. Drefahl *et al.* adopted a similar strategy by using the diethylacetal of benzaldehyde as an intermediate in the preparation of benzaldehyde chromium tricarbonyl.[7]

Triphenylphosphine derivatives of arene chromium tricarbonyl compounds are readily prepared by photolysis in benzene.[8] Photolysis of the dioxolane chromium tricarbonyl derivatives with triphenylphosphine gave the corresponding triphenylphosphine complexes in good yield. Hydrolysis of the dioxolanes cleanly gave the aldehyde and ketone derivatives.

IR spectra were recorded for all compounds using dichloromethane as a solvent. Carbonyl stretching frequencies and stretching force constants for all compounds are recorded in Table 1. Stretching force constants for the carbonyl groups of both the arene chromium tricarbonyl and arene chromium dicarbonyl triphenylphosphine complexes were computed by using the approximations developed by Cotton and Kraihanzel.[9] IR stretching force constants for the arene chromium tricarbonyl derivatives agree very well with those reported by Neuse for the same solvent and are slightly lower than those reported by van Meurs *et al.* for isooctane solutions.[10]

Substitution of a triphenylphosphine for a carbonyl group resulted in a shift of the carbonyl stretching frequencies to lower energy, consistent with the reduced π acidity of the triphenylphosphine relative to carbonyl. The reduction in force constant upon introduction of the phosphine is highly uniform, averaging 0.879 mdyne A^{-1} with a standard deviation of about 4.3%. Carbonyl stretching frequencies and force constants are presented in Table 1.

^1H, ^{19}F and ^{31}P NMR spectra, as appropriate, were recorded for these compounds and were consistent with reported values. ^{31}P NMR chemical shifts are reported in Table 1. Other spectral parameters for new compounds are presented in the Experimental. Arene ring proton chemical shifts are affected only slightly when phosphine is substituted for carbonyl, while the chemical shifts of substituent methyl groups (Me, *t*-Bu, OMe, NMe_2, $SiMe_3$) are shifted downfield. ^{19}F NMR chemical shifts of the trifluoromethyl group also move to lower field [99.94 ppm for $CF_3C_6H_5Cr(CO)_3$ to 100.90 for $CF_3C_6H_5Cr(CO)_2P(C_6H_5)_3$] upon introduction of the phosphine.

Fletcher and McGlinchey have reported the ^{19}F chemical shifts of a series of *m*- and *p*-substituted fluorobenzene chromium tricarbonyl derivatives.[11] The effect of these substituents upon the chemical shifts of the fluorine was correlated using Swain–Lupton field and resonance parameters with elec-

Table 1. IR[a] and ^{31}P[b] spectral data and force constants[c]

	$XC_6H_5Cr(CO)_3$				$XC_6H_5Cr(CO)_2P(C_6H_5)_3$				
X	*A*	*E*	*k*	k_i	A_1	B_1	*k*	k_i	^{31}P
H	1973	1892	14.88	0.4218	1892	1833	14.19	0.4394	90.93
CH_3	1971	1892	14.87	0.4208	1887	1828	13.94	0.4448	91.68
$C(CH_3)_3$	1966	1887	14.79	0.4092	1883	1824	13.88	0.4466	91.90
OCH_3	1972	1892	14.87	0.4158	1886	1827	13.92	0.4385	91.90
$N(CH_3)_2$	1955	1867	14.53	0.4510	1869	1806	13.64	0.4671	93.47
$Si(CH_3)_3$	1968	1891	14.84	0.3985	1888	1833	13.98	0.4094	91.30
F	1982	1907	15.08	0.3897	1903	1847	14.19	0.4411	88.93
Cl	1981	1907	15.07	0.3847	1904	1845	14.22	0.4162	88.54
Br	1972	1897	14.92	0.3944	1892	1835	14.03	0.4320	88.31
$COCH_3$	1983	1913	15.15	0.3675	1908	1854	14.29	0.4108	86.36
CHO	1988	1919	15.23	0.3605	1917	1861	14.41	0.4269	84.85
CO_2CH_3	1983	1913	15.15	0.3675	1908	1854	14.29	0.4108	86.65
C_6H_5	1970	1893	14.87	0.4021	1889	1834	14.00	0.4157	90.58
CF_3	1988	1914	15.18	0.3908	1914	1860	14.38	0.4117	85.88

[a] Solvent CH_2Cl_2, carbonyl stretching frequencies in cm^{-1}.
[b] 85% H_3PO_4 reference, $CDCl_3$ solvent.
[c] Force constants in mdyn $Å^{-1}$.

tron donating groups being found to cause an upfield shift of the ^{19}F resonance. In this work, the upfield shift of the ^{19}F resonances resulting from substitution of a phosphine for a carbonyl [29.50 ppm for $FC_6H_5Cr(CO)_3$ and 22.55 ppm for $FC_6H_5Cr(CO)_2P(C_6H_5)_3$] is found to be roughly equivalent to the effect of placing a strongly resonance donating group, such as a methoxy or an amine, in a *para* position relative to the fluorine.

^{31}P chemical shifts were recorded and were found to be dependent upon the ring substituents as was suggested by our qualitative observations. Electron donating ring substituents cause a downfield shift of the phosphine resonance while electron withdrawing groups shift the resonance upfield. ^{31}P chemical shifts show a good correlation ($r = 0.946$) with the carbonyl stretching force constants indicating that ring substituents are exerting a proportional effect on both groups.

Following Neuse, we have examined the correlations of k(CO) arene chromium tricarbonyl, k(CO) arene chromium dicarbonyl triphenylphosphine and ^{31}P chemical shifts with several sets of substituent constants, Table 2, to determine the nature of the interaction between the metal ligands and the ring substituents. The six sets of substituent constants which were examined include σ^n, σ_p^0, σ_m, σ_p, σ_I and σ_R^0. The σ^n and σ_p^0 series are closely related and measure the effects of arene substituents on reaction centres which are isolated from the arene ring by a methylene group.[12,13] By isolating the arene ring, direct conjugation of the substituent with the reaction centre is precluded so that the observed constants reflect the total electron density changes in the ring resulting from the substituent. The σ_m and σ_p constants are traditional Hammett constants as measured by Brown and are based on the ionization of substituted benzoic acids.[14] Direct conjugation of *para* substituents such as amine and methoxy with the reaction centre will result in "exalted" substituent constants for these groups. σ_I and σ_R^0 constants have been developed by Taft and others to separate the inductive and resonance contributions of substituents.[15–17] As in the case of the σ^n and σ_p^0 constants described above, these values should be free of direct conjugation effects.

The two sets of stretching force constants and the set of ^{31}P chemical shifts were examined for correlations with all six sets of substituent constants. The resulting correlations, Table 3, substantially parallel those reported by Neuse and show that these three sets of data are best correlated by those constants which measure both the inductive and resonance contributions, i.e. σ^n and σ_p^0, of the substituents. The poor correlations for σ_I and σ_R^0 constants confirm that the substituent effect is not a simple inductive or resonance phenomenon. k(CO) arene chromium tricarbonyl and k(CO) arene chromium dicarbonyl triphenylphosphine were found to have almost identical slopes when compared to the σ_n and σ_p^0 constants. The aldehyde derivatives were found to yield data which were very far out of line with the remaining values. It is not clear whether the failure of aldehyde to conform is due to solvent effects or to intermolecular interactions between the aldehyde and the chromium atom.

The correlations presented in this work and in our previous paper illustrate the remarkably uniform interrelationships between the physical constants of these arene chromium tricarbonyl and

Table 2. Substituent constants

X	σ^n	σ_p^0	σ_m	σ_p	σ_I	σ_R^0
H	0.00	0.00	0.00	0.00	0.00	0.00
CH_3	−0.10	−0.124	−0.069	−0.170	−0.08	−0.146
$C(CH_3)_3$	−0.14	−0.174	−0.10	−0.197	—	−0.13
OCH_3	−0.09	−0.100	0.115	−0.268	0.25	−0.43
$N(CH_3)_2$	−0.24	−0.48	—	−0.83	0.10	−0.54
$Si(CH_3)_3$	—	—	−0.04	−0.07	—	0.00
F	0.18	0.212	0.337	0.062	0.52	−0.32
Cl	0.29	0.281	0.371	0.227	0.47	−0.18
Br	0.30	0.296	0.391	0.232	0.44	−0.16
$COCH_3$	0.502	0.502	0.376	0.502	0.28	0.19
CHO	0.43	0.53	—	—	0.25	—
CO_2CH_3	0.46	0.46	0.37[a]	0.45[a]	0.31[a]	0.15
C_6H_5	0.06	0.039	0.06	−0.01	0.08	−0.093
CF_3	0.53	0.505	0.43	0.54	0.41	0.13

[a] Literature values are for $CO_2C_2H_5$.

Table 3. Summary of linear regression analyses[a]

(a) Comparison of $XC_6H_5Cr(CO)_3$ carbonyl force constants with various substituent constants and $XC_6H_5Cr(CO)_2(C_6H_5)_3P$ carbonyl force constants

Sub. Const. Set	Slope (*m*)	Intercept (*b*)	Correlation
σ^n	1.24	−18.39	0.908
σ_p^0	0.596	14.88	0.959
σ_m	1.31	−19.42	0.883
σ_p	1.93	−28.75	0.943
σ_I	0.539	−7.82	0.553
σ_R^0	0.801	−12.08	0.668
k(CO)	0.924	0.236	0.901
$k(\mathrm{CO})^b$	1.112	−2.55	0.988

(b) Comparison of $XC_6H_5Cr(CO)_2(C_6H_5)_3P$ carbonyl force constants with various substituent constants

Sub. Const. Set	Slope (*m*)	Intercept (*b*)	Correlation
σ^n	1.17	−16.33	0.890
σ^{nb}	1.18	−16.40	0.934
σ_p^0	0.576	13.78	0.895
σ_p^{0b}	0.664	−13.99	0.963
σ_m	1.06	−14.71	0.853
σ_p	1.73	−24.31	0.953
σ_I	0.564	−7.69	0.599
σ_R^0	0.762	−10.84	0.713

(c) Comparison of ^{31}P chemical shifts with various substituent constants and carbonyl force constants

Sub. Const. Set	Slope (*m*)	Intercept (*b*)	Correlation
σ^n	−0.105	9.56	0.934
$\sigma_n{}^b$	−0.107	9.73	0.996
σ_p^0	−0.144	10.3	0.979
σ_p^{0b}	−1.22	11.02	0.985
σ_m	−0.084	7.69	0.908
σ_p	−0.145	13.0	0.947
σ_I	−0.0521	4.91	0.646
σ_R^0	−0.0652	5.72	0.730
k(CO)	−7.96	21.20	0.946
$k(\mathrm{CO})^b$	−7.97	21.21	0.947

[a] Data sets calculated without aldehyde derivative included.

[b] Data are presented for the relationship: $y = mx+b$. Linear regression calculations were performed using standard statistical methods. The correlation coefficient R is reflective of "goodness-of-fit" where $R = 1.0$ for a perfect straight line.

arene chromium dicarbonyl triphenylphosphine compounds. Using the carbonyl force constants as a reference, it would appear that the contributions of the phosphine and ring substituents can be treated as separate, additive quantities so that

$$\delta k(\mathrm{CO}) = \delta k(\mathrm{substituent}) + \delta k(\mathrm{ligand}).$$

If this simple relationship holds for other sets of organometallic compounds, then it might be possible to determine the δ contributions for a series of ligands and substituents on different metal complexes and use these δ values as a measure of the sensitivity of a metal complex to electron donation or withdrawal.

EXPERIMENTAL

IR spectra were recorded on a Perkin–Elmer Model 1750 FT/IR spectrophotometer. NMR spectra were recorded on a Varian Associates FT80A NMR spectrometer. HPLC analysis of all compounds and reaction mixtures was conducted on a GOW-MAC Model 080-20 HPLC using a silica gel column and 3/7 THF/petroleum ether as the elutant.

Chromium hexacarbonyl was purchased from Strem. Dioxobenzaldehyde and dioxoacetophenone were prepared by literature procedures.[18] Substituted arene chromium tricarbonyl and arene chromium dicarbonyl triphenylphosphine complexes were generally prepared by literature procedures, $XC_6H_5Cr(CO)_3$: X = H,[19] CH_3,[19] CH_3O,[19] CH_3CO,[19] CH_3O_2C,[19] $(CH_3)_2N$,[19] $(CH_3)_3C$,[19] C_6H_5,[20] FC_6H_4,[21,22] ClC_6H_4,[19,22] BrC_6H_4,[23] CHO,[7] $(CH_3)_3Si$,[24] CF_3.[24] $XC_6H_5Cr(CO)_2P(C_6H_5)_3$: X = H,[5], CH_3,[25] CH_3O,[5] CH_3CO,[26] CH_3O_2C,[27] $(CH_3)_2N$,[5] $(CH_3)_3C$,[25] C_6H_5.[5] Spectral and physical properties of these compounds were compared with those reported in the literature and were consistent in all cases.

Exceptions to these procedures are described below. Photolysis reactions were conducted in an Ace doubly-jacketed photolysis vessel with a quartz inner vessel using a Hanovia medium-pressure mercury lamp. Microanalysis was conducted by Galbraith Laboratories of Knoxville, TN. All reactions were conducted under nitrogen.

Synthesis of dioxolane benzeldehyde chromium tricarbonyl

A 500 cm^3 round bottom flask outfitted with a spin bar was charged with $Cr(CO)_6$, 8.80 g (40.0 mmol) and dioxobenzeldehyde, 7.0 g (46.7 mmol). The flask was flushed with nitrogen and dibutylether (260 cm^3) and THF (30 cm^3) were added under nitrogen. The flask was outfitted with a reflux condenser and the reaction mixture refluxed for two days. The reaction mixture was filtered, reduced in volume to about 50 cm^3 and 50 cm^3 of petroleum ether was added to precipitate 9.0 g of dioxobenzaldehyde chromium tricarbonyl as a yellow powder, m.p. 70–73°C. IR: (CH_2Cl_2) 1973, 1896. ^{1}H NMR: $(CDCl_3)$ 5.56–5.22 (m, aromatic protons), 4.17–3.98 (m, ethylene protons). Yield: 79%. Found: C, 50.2; H, 3.8. Calc. for $C_{12}H_{10}O_5Cr$: C, 49.7; H, 3.4%.

The known benzaldehyde chromium tricarbonyl can be formed from dioxobenzaldehyde chromium tricarbonyl in quantitative yield by brief reaction of the former with *p*-toluenesulphonic acid in acetone. The reaction mixture was stripped of solvent and the resulting oily residue was chromatographed on alumina using methylene chloride as an eluant. Benzaldehyde chromium tricarbonyl is recovered as a red oil.

Synthesis of dioxoacetophenone chromium tricarbonyl

In a reaction analogous to that described above for dioxobenzaldehyde, $Cr(CO)_6$, 4.40 g (20 mmol) and dioxoacetobenzene, 4.0 g (24.3 mmol), were reacted in butyl ether/THF. After removal of the reaction solvent the blue–yellow residue was taken up in hot benzene and filtered through Celite. The benzene solution was concentrated to about 5 cm^3 and petroleum ether added to precipitate 1.6 g of dioxoacetobenzene chromium tricarbonyl as a yellow crystalline solid, m.p. 87–89°C. Yield: 27%. ^{1}H NMR: $(CDCl_3)$ 5.72–5.41 (m, 5H), 4.08 (s, 4H), 1.60 (s, 3H). Found: C, 51.9; H, 4.4. Calc. for $C_{13}H_{12}O_5Cr$: C, 51.3; H, 3.9%.

Synthesis of arene chromium dicarbonyl triphenylphoshine derivatives

In a typical reaction, 0.5 g of arene chromium tricarbonyl was photolysed overnight with a 10% molar excess of triphenylphosphine in benzene (200 cm^3) at 12°C. Photolyses were conducted in an Ace Hanovia reaction vessel equipped with a water-jacketed quartz cold finger and a water-jacketed reaction vessel. Nitrogen was bubbled through the reaction mixture throughout the photolysis. After photolysis the reaction mixture was reduced in volume to about 10 cm^3 and filtered through Celite. Addition of petroleum ether gave the desired products in 40–50% yield. Products could be further purified by recrystallization from benzene.

Data for new compounds

Fluorobenzene. Orange crystalline solid, m.p. 188–190°C. Yield: 83%. ^{1}H NMR: $(CDCl_3)$ 7.59–6.98 (m, 15H), 4.26–3.77 (m, 5H). ^{19}F NMR: $(CDCl_3)$ 22.56 (m). Found: C, 67.2; H, 4.7; P, 7.1. Calc. for $C_{26}H_{20}FCrO_2P$: C, 66.9; H, 4.3; P, 6.6%.

Chlorobenzene. Yellow–orange crystals, m.p. 186–190°C. Yield: 72%. ^{1}H NMR: $(CDCl_3)$ 7.58–6.99 (m, 15H), 4.44–4.13 (m, 5H). Found: C, 64.7; H, 4.6; P, 6.8. Calc. for $C_{26}H_{20}ClCrO_2P$: C, 64.7; H, 4.1; P. 6.4%.

Bromobenzene. Orange plates, m.p. 168–170°C. ^{1}H NMR: $(CDCl_3)$ 7.57–6.99 (m, 15H), 4.45 (m, 2H), 4.05 (m, 3H). Found: C, 59.0; H, 3.7; P, 5.7. Calc. for $C_{26}H_{20}BrCrO_2P$: C, 59.2; H, 3.8; P, 5.9%.

Trifluoromethylbenzene. Orange crystals, m.p. 195–198°C. Yield: 38%. ^{1}H NMR: ($CDCl_3$) 7.50–7.00 (m, 15H), 4.71 (m, 2H), 4.06 (m, 3H). ^{19}F NMR: ($CDCl_3$) 100.90 (m). Found: C, 62.8; H, 4.2; P, 6.2. Calc. for $C_{27}H_{20}F_3CrO_2P$: C, 62.8; H, 3.9; P, 6.0%.

Dioxolanebenzaldehyde. Orange crystals, m.p. 135–137°C. IR: ($CHCl_3$) 1901, 1835. ^{1}H NMR: ($CDCl_3$) 7.63–7.00 (m, 15H), 5.65 (s, 1H), 4.89 (d, 2H), 4.25 (m, 3H), 3.50 (m, 4H). ^{31}P NMR: ($CDCl_3$) 90.07 ppm. Found: C, 66.5; H, 5.0; P, 5.2. Calc. for $C_{29}H_{25}CrO_4P$: C, 66.9; H, 4.8; P, 5.9%.

Benzaldehyde. Orange crystals, m.p. 134–137°C. IR: (CH_2Cl_2) 1917, 1861, 1437 (CHO). ^{1}H NMR: ($CDCl_3$) 9.04 (s, 1H), 7.49–7.03 (m, 15H), 5.09 (d, 2H), 4.30 (t, 1H), 4.09 (t, 2H). Found: C, 68.1; H, 4.6. Calc. for $C_{27}H_{21}CrO_3P$: C, 68.1; H, 4.4%.

Dioxolaneacetophenone. Orange crystals, m.p. 128–131°C. IR: ($CHCl_3$) 1893, 1833. ^{1}H NMR: (C_6D_6) 7.65–7.02 (m, 15H), 5.26 (m, 5H), 4.18–3.99 (m, 4H), 1.64 (s, 3H). ^{31}P NMR: ($CDCl_3$) 90.75 ppm. Found: C, 67.4; H, 5.3; P, 5.1. Calc. for $C_{30}H_{27}CrO_4P$: C, 66.9; H, 5.0; P, 5.8%.

Trimethylsilylbenzene. Orange crystals, m.p. 178–182°C, ^{1}H NMR: ($CDCl_3$) 7.64–7.06 (m, 15H), 4.83 (d, 2H), 4.23 (m, 3H), 0.29 (s, 9H). Yield: 59%. Found: C, 66.3; H, 6.0; P, 6.1. Calc. for $C_{29}H_{29}SiCrO_2P$: C, 66.9; H, 5.6; P, 6.0%.

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REFERENCES

1. T. E. Bitterwolf, *Polyhedron* 1983, **2**, 675.
2. T. E. Bitterwolf, *Polyhedron* 1988, **7**, 409.
3. C. V. Senoff, *Coord. Chem. Rev.* 1980, **32**, 111.
4. E. W. Neuse, *J. Organometal. Chem.* 1975, **99**, 287.
5. L. A. Fedorov, P. V. Petrovskii, E. I. Fedin, G. A. Panosyan, A. A. Tsoi, N. K. Baranetskaya and V. N. Setkina, *J. Organometal. Chem.* 1979, **182**, 499.
6. S. Top and G. Jaouen, *J. Organometal. Chem.* 1979, **182**, 381.
7. G. Drefahl, H.-H. Horhold and K. Kuhne, *Chem. Ber.* 1965, **98**, 1826.
8. W. Strohmeier and H. Hellmann, *Chem. Ber.* 1963, **96**, 2859.
9. F. A. Cotton and C. S. Kraihanzel, *J. Am. Chem. Soc.* 1962, **84**, 4432.
10. F. van Meurs, J. M. A. Baas and H. van Bekkum, *J. Organometal. Chem.* 1976, **113**, 353.
11. J. L. Fletcher and M. J. McGlinchey, *Can. J. Chem.* 1975, **53**, 1525.
12. Y. Yukawa, Y. Tsuno and M. Sawada, *Bull. Chem. Soc. Japan* 1972, **45**, 1198.
13. A. J. Hoefnagel and B. M. Wepster, *J. Am. Chem. Soc.* 1973, **95**, 5357.
14. D. H. McDaniel and H. C. Brown, *J. Org. Chem.* 1958, **23**, 420.
15. R. W. Taft, Jr., N. C. Deno and P. S. Skell, *Ann. Rev. Phys. Chem.* 1958, **9**, 287.
16. R. W. Taft, E. Price, I. R. Fox, I. C. Lewis, K. K. Andersen and G. T. Davis, *J. Am. Chem. Soc.* 1963, **85**, 709.
17. R. W. Taft, E. Price, I. R. Fox, I. C. Lewis, K. K. Andersen and G. T. Davis, *J. Am. Chem. Soc.* 1963, **85**, 3146.
18. T. W. Greene, *Protective Groups in Organic Synthesis*, p. 124. Wiley, New York (1981).
19. B. Nicholls and M. C. Whiting, *Proc. Chem. Soc.* 1958, 152; B. Nicholls and M. C. Whiting, *J. Chem. Soc.* 1959, 551.
20. E. O. Fischer, N. Kriebitzsch and R. D. Fischer, *Chem. Ber.* 1959, **92**, 3214.
21. R. D. Fischer, *Chem. Ber.* 1960, **93**, 165.
22. A. C. Knipe, S. J. McGuinness and W. E. Watts, *J. Chem. Soc., Perkin II* 1981, 193.
23. K. Ofele, *Chem. Ber.* 1966, **99**, 1732.
24. F. van Meurs, J. M. van der Toorn and H. van Beekum, *J. Organometal. Chem.* 1976, **113**, 341.
25. W. R. Jackson, C. F. Pincombe, I. D. Rae, D. Rash and B. Wilkinson, *Aust. J. Chem.* 1976, **29**, 2431.
26. B. Boyer, G. Lamaty, B. Caro and G. Jaouen, *J. Organometal. Chem.* 1981, **216**, C51.
27. G. Jaouen and R. Dabard, *J. Organometal. Chem.* 1973, **61**, C36; G. Jaouen and R. Dabard, *J. Organometal. Chem.* 1974, **72**, 377.