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Preliminary communication

Preparation of an optically active and other organosilicon derivatives of π-cyclopentadienyliron dicarbonyl

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SUMMARY

The preparation and some properties of the compounds (π-Cp)Fe(CO)$_2$SiR$_3$ (R$_3$ = Me$_2$Ph, MePh$_2$, Ph$_3$, Ph$_2$(CH=CH$_2$) or MePh(1-Np)), optically active (–)-(π-Cp)Fe(CO)$_2$[SiMePh(1-Np)] and (π-Cp)Fe(CO)(PPh$_3$)SiPh$_3$ are reported.

Compounds containing silicon to transition metal bonds are currently of interest[1], but few possessing an optically active silicon atom have been isolated[2–4].

The new compounds (π-Cp)Fe(CO)$_2$SiR$_3$ (I) (R$_3$ = Me$_2$Ph, MePh$_2$, Ph$_3$, Ph$_2$(CH=CH$_2$) or MePh(1-Np)) were obtained in 60–80% yield by reaction of [(π-Cp)Fe(CO)$_2$]$^-$Na$^+$ with R$_3$SiCl in THF. The complexes are yellow to orange crystalline solids and the solutions are air sensitive. No pure products were isolated from the reactions with R$_3$SiCl (R$_3$ = (OMen)Ph(1-Np), (H)Ph(1-Np), (CH=CH$_2$)Ph(1-Np) or Ph$_2$(CH$_2$CH=CH$_2$)), although in some cases oils were obtained which decomposed on attempted distillation or chromatography. It seems that the (1-Np) group has a destabilising effect on the compounds, sometimes precluding isolation. The compound (π-Cp)Fe(CO)$_2$SiMe$_2$(H) has previously been prepared[5].

UV irradiation of a dilute solution of compound I (R$_3$ = Ph$_3$) in n-hexane in the presence of PPh$_3$ gave (π-Cp)Fe(CO)(PPh$_3$)SiPh$_3$ (II). However photolytic irradiation of complex I (R$_3$ = Ph$_2$(CH=CH$_2$)) resulted in decomposition. The compound (π-Cp)Fe(CO)(PPh$_3$)SiMe$_3$ has previously been isolated[6].

Some chemistry of compound I (R$_3$ = Ph$_2$(CH=CH$_2$)) has been studied and is similar to that of (π-Cp)Fe(CO)$_2$SiMe$_3$ [7]. Complex I did not react with the reagents EtO$^-$, NaBH$_4$, KHF$_2$, H$_2$O/dioxane or a solution of anhydrous HCl in pentane, however the Si–Fe bond is cleaved by chlorine to give Ph$_2$(CH=CH$_2$)SiCl. The ^{1}H NMR spectrum of a solution of I in CCl$_4$ shows a band of multiplicity 12 assignable to the vinyl group.

From (+)-MePh(1-Np)SiCl ($[\alpha]_D^{25}$ +6.3°; *c* 0.84 (cyclohexane)), analytically pure yellow crystals of (–)-(π-Cp)Fe(CO)$_2$SiMePh(1-Np) ($[\alpha]_D^{25}$ –25.5°; *c* 0.67 (cyclohexane); m.p. 108.5–110.5°) were obtained. The racemic compound melts at 124–126° (see Table 1). Studies are in progress on the optically active compound and its reactions.

All new compounds have been fully characterised by IR and ^{1}H NMR spectroscopy, analyses, and in some cases by mass spectrometry.

TABLE I

Compound	*R$_3$*	*Colour*	*m.p. (°C)*	*Yield (%)*	*ν(CO) (cm^{-1})* *(cyclohexane)*
I	Me$_2$Ph	Yellow	68–68.5	72	1998, 1947
I	MePh$_2$	Yellow	103.5–105.5	78	2002, 1951
I	Ph$_3$	Yellow	164–165.5	60	2002, 1952
I	Ph$_2$(CH=CH$_2$)	Yellow	114–114.5	68	2000, 1949
I	MePh(1-Np)	Yellow	124–126	64	2000, 1950
II		Orange	199 (dec.)	92	1916

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