rearrangement of eq 3 as a function of K gave (5) and (6).

$$k_1 + k_2K_A[H^+]^{-1} = k_{eq}K(K[X^-] + 1)^{-1} \qquad (5)$$

$$k_{-1} + k_{-2}K_A'[H^+]^{-1} = k_{eq}(K[X^-] + 1)^{-1} \qquad (6)$$

The plots of $k_{eq}K(K[X^-] + 1)^{-1}$ and $k_{eq}(K[X^-] + 1)^{-1}$ against $[H^+]^{-1}$ were linear as shown in Figures 1 and 2 and gave the values $k_1 = 0.10$ (NCS^-) and 0.20 M^{-1} s^{-1} (N_3^-), $k_2K_A = 42 \times 10^{-8}$ (NCS^-) and 3.8×10^{-8} s^{-1} (N_3^-), $k_{-1} = 0.024$ (NCS^-) and 0.001 s^{-1} (N_3^-), and $k_{-2}K_A' = 52 \times 10^{-8}$ (NCS^-) and 1.1×10^{-8} M s^{-1} (N_3^-).

Discussion

Although there are two replaceable aquo ligands in the complex $[Cr(salen)(H_2O)_2]^+$, only monosubstitution occurred on the stopped-flow time scale. This is consistent with the widely differing Cr^{III}–OH_2 bond lengths in the complex, viz., 1.923 and 2.085 Å.[7] With the conjugate base of $[Cr(salen)(H_2O)_2]^+$, the observed single-stage kinetics is to be expected because the direct substitution of OH^- by other nucleophiles is considered unlikely in this class of compounds. The observed direct dependence of the forward rates on $[X^-]$ at any $[H^+]$ does not provide compelling evidence for dissociative mechanisms for the reactions of either $Cr(salen)(H_2O)_2^+$ or its conjugate base, though X-ray structure data would suggest dissociative processes. However the similarity of the k_1 terms for the $Cr(salen)(H_2O)_2^+$ reactions with NCS^- (0.10 M^{-1} s^{-1}) and N_3^- (0.20 M^{-1} s^{-1}), after making allowance for the statistical factor for N_3^-,[9] is in keeping with a dissociative I_d-type process with small ion-pairing constants. Assumption of low ion-pairing constants for the reactions of uninegative ions like NCS^- and N_3^- with $Cr(salen)(H_2O)_2^+$ and its conjugate base will be consistent with the low charges on the complex reactants. The reverse rates for the complexes $[Cr(salen)(H_2O)X]$ with $X^- = NCS^-$ (0.024 s^{-1}) and N_3^- (0.001 s^{-1}) and their equilibrium constants [NCS^- (2.2 M^{-1}) and N_3^- (50 M^{-1})] are also in keeping with a dissociative mechanism involving rate-limiting M–OH_2 bond dissociation. On the other hand, magnetic measurements on the conjugate base of $Cr(salen)(H_2O)_2^+$ as a solid indicate a five-coordinate [Cr(salen)OH] structure in the ground state.[7] If the conjugate base were to be six-coordinate in solution and the mechanism involved rate-limiting loss of the sixth ligand, viz., water, the two similarly charged anions NCS^- and N_3^- should have similar k_2K_A terms. The values obtained for k_2K_A (4.2×10^{-7} M s^{-1} for NCS^- and 3.8×10^{-8} M s^{-1} for N_3^-) were markedly different. On the other hand, if the conjugate base were to be a five-coordinate [Cr(salen)OH] complex in solution also, the nucleophiles such as N_3^- and NCS^- may undergo bimolecular addition reactions to give six-coordinate products with large variations in the k_2 term. In light of the recent reports on the interesting features of complexes with a five-coördinate structure,[10] further work is in progress to distinguish between the two possible mechanisms, viz., nucleophilic substitution and bimolecular addition for the reactions of the conjugate base of $[Cr(salen)(H_2O)_2]^+$.

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Registry No. $Cr(salen)(H_2O)_2^+$, 47248-17-9; NCS^-, 302-04-5; N_3^-, 14343-69-2.

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Comparisons of Ir–Cl Stretching Frequencies and ^{31}P Chemical Shifts in *trans*-$IrCl[P(C_6H_5)_3]_2L$ Complexes

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Many techniques have been used to probe the trans and, recently, cis (bond-weakening) influences of ligands in transition-metal complexes.[1] For square-planar Pt(II) complexes in particular extensive direct (structural)[2,3] and indirect (spectroscopic) information has been amassed. These indirect indicators include Pt–halide and –hydride stretching frequencies,[4,5] ^{195}Pt–^{31}P and –^{1}H coupling constants,[6–8] and ^{195}Pt and ^{1}H chemical shifts.[8,9] Although analogous square-planar Ir(I) complexes have attracted much attention,[10] trans-influence data for these compounds are scarce. Many of the spectroscopic indicators available in Pt(II) systems do not apply to Ir(I) complexes. In the absence of structural results, of which there are only a few reports, we have been forced to use the Ir–Cl stretching frequency as our sole indicator of trans influence in complexes of the type *trans*-$IrCl(PPh_3)_2L$ [Ph = C_6H_5; L = N_2Ar^+, $N_2C_5X_4$ (Ar = aryl; X = Cl, Br)].[11,12] In an effort to find additional trans-influence probes for these complexes, we have examined their ^{31}P NMR chemical shifts. We reasoned that within a closely related series of complexes differences in this parameter will arise predominantly from variations in the inductive-shielding contribution of the metal. These shieldings should reflect relative metal-centered electron densities which, although not themselves direct measures of the trans influences of the ligands, will be closely related as both are functions of the effective L → M charge transfer. The obvious ease with which the ^{31}P NMR method can be applied to complexes difficult or impossible to isolate or stable only at low temperature and even to mixtures of complexes makes it a very attractive alternative to infrared spectroscopy which generally requires isolable, relatively pure solids. Here we describe the results of our comparisons of Ir–Cl stretching frequencies and ^{31}P chemical shifts for a series of complexes, *trans*-$IrCl(PPh_3)_2L$.

Experimental Section

All procedures were carried out under inert atmosphere. The complexes *trans*-$IrCl(PPh_3)_2L$, where L = CO,[13] CS,[14] N_2,[15] C_2H_4,[16] PPh_3,[17] NO^+,[18] N_2Ar^+,[11] and $N_2C_5Cl_4$,[12] were prepared by literature

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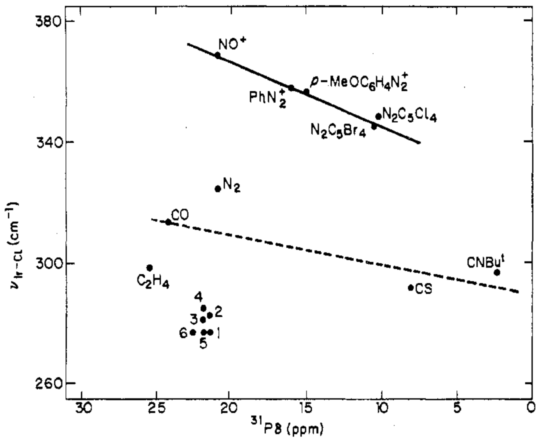


Figure 1. Comparison of the ^{31}P chemical shifts and Ir–Cl stretching frequencies for the complexes *trans*-IrCl(PPh$_3$)$_2$L. The numbered points (see Table I) correspond to the complexes in which L = phosphorus ligand.

methods. The NO$^+$ and N$_2$Ar$^+$ complexes were PF$_6^-$ salts. Those complexes involving *t*-BuNC or phosphorus ligands other than PPh$_3$, i.e., where L = P(p-MeC$_6$H$_4$)$_3$, P(p-FC$_6$H$_4$)$_3$, P(p-MeOC$_6$H$_4$)$_3$, P(O-i-Pr)$_3$, or PMe$_2$Ph (*t*-Bu = *tert*-butyl, Me = CH$_3$, *i*-Pr = isopropyl), were synthesized by addition of the appropriate ligand to a benzene solution of *trans*-IrCl(N$_2$)(PPh$_3$)$_2$ followed by precipitation with hexane. The complex in which L = N$_2$C$_5$Br$_4$[19] was prepared in a manner analogous to that employed for the L = N$_2$C$_5$Cl$_4$ complex.

The ^{31}P NMR spectra were recorded with the use of a Varian CFT-20 or JEOL FX90Q spectrometer, operating at 32.199 and 36.20 MHz, respectively. The spectra were obtained at ambient temperature and are referenced with respect to external 85% H$_3$PO$_4$, with positive chemical shift downfield. Dried and degassed chloroform-d_1 was used as solvent except for the L = phosphorus-ligand samples, in which benzene-d_6 was substituted. Infrared spectra were recorded in the 600–200-cm^{-1} region as Nujol mulls between polyethene disks with use of a Perkin-Elmer 283 spectrometer.

The ^{31}P NMR spectra of those complexes for which L ≠ phosphorus ligand consisted of a single peak. Where L = phosphorus ligand, the spectra were all of first-order AB$_2$ type, each exhibiting a 1:1 doublet and a binomial triplet, arising from the equivalent, mutually trans PPh$_3$ ligands and the unique phosphorus ligand, respectively.

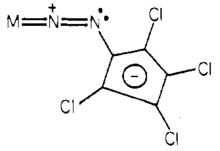
Results and Discussion

The observed stretching frequencies and chemical shifts are listed in Table I and are displayed graphically in Figure 1. As expected, the Ir–Cl stretching frequency (ν_{Ir-Cl}) is sensitive to the nature of the trans ligand and the trends are in general agreement with previously reported series.[1] The order of increasing energy and of decreasing trans influence, phosphorus ligands > olefin ≥ isocyanide ≥ CO, approximates that found in Pt(II)[1] and Ir(III)[20] complexes. Note the lack of discrimination between various L = phosphorus-ligand complexes. The trans influence of the thiocarbonyl ligand appears to be comparable with that of the isocyanide and ethylene ligands and significantly higher than that of carbonyl. This difference may arise from the enhanced σ-donating propensity of the CS ligand, as compared with that of CO.[21] The value for the complex when L = N$_2$ suggests a trans influence weaker than that of CO and is consistent with the lower σ donation of the dinitrogen ligand.[22] Those complexes which involve the diazo, diazonium(1+), and nitrosyl(1+) ligands have ν_{Ir-Cl} values at high energies, indicating substantially diminished trans influences as compared with those of the ligands discussed above. In the N$_2$Ar$^+$ and NO$^+$ complexes, the polarization is such that net electron density is withdrawn from the iridium center toward L, thereby enhancing the Ir–Cl interaction. We have previously noted metal–chlorine bond length decreases trans to both NO$^+$ and N$_2$Ar$^+$ ligands,[23] and the ordering is that which we observe here, with NO$^+$ being the greater trans bond enhancer. We have also shown that the N$_2$C$_5$Cl$_4$ ligand (A) can mimic the aryldiazonium ligand (B) by binding to a metal center in a charge-separated fashion.[12] Substantial negative charge is localized in the pseudo-aromatic C$_5$ ring, as judged by ring C–C bond distances, thereby causing the N$_2$ portion of the ligand to become electron poor. Thus the metal-bound end of the diazo ligand closely resembles that of the N$_2$Ar$^+$ system. As above, electron flow toward the positively charged N$_2$ moiety from the metal center strengthens the Ir–Cl bond. The partial positive charge localized at the ligating nitrogen atom increases in the series N$_2$C$_5$X$_4$, N$_2$Ar$^+$, and NO$^+$, and this is consistent with the trend in ν_{Ir-Cl} values observed.

Table I. ^{31}P Chemical Shifts and Ir–Cl Stretching Frequencies for *trans*-IrCl(PPh$_3$)$_2$L Complexes

L	^{31}P chem shifts, δ^a	Ir–Cl str freq, ν, cm^{-1}
t-BuNC	3.2	297
CS	8.0	292
N$_2$C$_5$Cl$_4$	10.1	348
N$_2$C$_5$Br$_4$	10.3	345
p-MeOC$_6$H$_4$N$_2^+$	15.0	357
PhN$_2^+$	16.0	358
N$_2$	20.9	325
NO$^+$	20.9	369
P(p-FC$_6$H$_4$)$_3$ (1)^b	21.4	276
P(O-i-Pr)$_3$ (2)	21.4	283
P(p-MeC$_6$H$_4$)$_3$ (3)	21.9	282
P(p-MeOC$_6$H$_4$)$_3$ (4)	21.9	285
PMe$_2$Ph (5)	21.9	276
PPh$_3$ (6)	22.6	277
CO	24.1	314
C$_2$H$_4$	25.5	298

^{a} Downfield from external 85% H$_3$PO$_4$. ^{b} The numbers assigned here for the L = phosphorus-ligand complexes correspond to those used in Figure 1.



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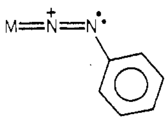

B

Sufficient structural data are not available for iridium(I) complexes to allow us to corroborate fully our proposed trans-influence series. We are aware of only four structural investigations of complexes of the type *trans*-IrCl(PAr$_3$)$_2$L: Ar = Ph, L = C$_2$H$_4$,[24] N$_2$Ph$^+$,[25] and N$_2$C$_5$Cl$_4$;[12] Ar = 2-CH$_3$C$_6$H$_5$, L = CO.[26] The Ir–Cl bond lengths in the three PPh$_3$ complexes are in accord with the trans influences indicated by our vibrational data. The Ir–Cl bond distances in the C$_2$H$_4$ and CO complexes, 2.371 (2) and 2.43 (1) Å, respectively, are reversed with respect to the ordering which we have observed, but the long value found for the CO complex may be a result of the steric effects of the bulky ortho methyl groups and/or from a disorder of the Cl and CO ligands.

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The ^{31}P chemical shift of the mutually trans phosphorus nuclei is also sensitive to the nature of L. The observed shifts appear to be correlated with the donor/acceptor properties of L in the expected manner. Thus $\delta_{C_2H_4}$ and δ_{CO}, shifts for the ethylene and carbonyl complexes, respectively, are at lower field than $\delta_{t\text{-BuNC}}$. The π-acceptor strengths of the ethylene and carbon monoxide ligands reduce the metal-centered electron densities and thereby deshield the ^{31}P nuclei relative to those of the L = isocyanide complex. The isocyanide ligand is predominantly a σ donor in complexes containing a metal of oxidation state +1 or greater.[27] This is consistent with the relatively high-field value of $\delta_{t\text{-BuNC}}$. A similar screening effect for isocyanide ligands has been reported in ^{13}C NMR studies.[28] The relative deshielding in the nitrosyl complex is also consistent with the electron-withdrawing strength of NO^+, as described above and documented previously in terms of the π-acceptor strength of the ligand.[23,29] The trend to higher field of the ^{31}P chemical shifts in the series L = NO^+, N_2Ar^+, and $N_2C_5X_4$[11] arises from the same sources responsible for the ν_{Ir-Cl} ordering. Decreased electron demand results in increased metal-centered electron density and thus in increased shielding of the ^{31}P nuclei. The dinitrogen ligand is a weaker π acceptor than CO.[22] The relatively low-field value of δ_{N_2} may arise from compensation by the poor donor ability of the ligand, causing a net electron withdrawal similar to that of the nitrosyl ligand. Of interest is the high-field value of δ_{CS}. It has been suggested that while CS is only a slightly better σ donor than CO, it is a much better π acceptor.[21] We would thus expect that δ_{CS} be greater than δ_{CO}. The unexpected screening in the thiocarbonyl complex may arise from an interaction involving occupied orbitals of the CS ligand.

The results for the complexes in which L = phosphorus ligand are disappointing. As for the stretching frequencies, there is no significant difference between any of the chemical shifts. This lack of discrimination is unexpected in light of the substantial substituent effects discussed by Tolman.[30] Note also the low-field shifts for the complexes in which L = phosphorus ligand. The high trans influence of these ligands indicates that they are strong σ donors.[2a] In accord with the results described above, a substantial π-acceptor capacity would be required to produce the observed low-field shifts. However, Mason and Meek[2a] have suggested that π interactions contribute minimally to M–P bonding. Clarification of these ambiguities must await further study. It is necessary to recognize that our ambiguous NMR results for the complexes in which L = phosphorus ligand may be a result of the substitution of benzene-d_6 for chloroform-d_1 as solvent and that in these systems steric effects may have a substantial impact not present with the nonphosphorus ligands. Finally, recent discussions of the structural cis influence in Pt(II) complexes have indicated that this parameter is largely a function of π-acceptor strength. In the majority of complexes described above, the π-acceptor strength of L appears to be correlated with the ^{31}P chemical shift of the phosphorus nuclei cis to L.

There is a striking correlation between our IR and NMR results within closely related sets of complexes. We find that stretching frequency and chemical shift data for the closely related NO^+, N_2Ar^+, and $N_2C_5X_4$ complexes are linearly related (Figure 1, solid line, linear correlation coefficient = 0.99). An analogous correlation (Figure 1, dashed line, linear correlation coefficient = 0.91) is present for the carbon-ligand complexes, L = CO, CS, and *t*-BuNC, but additional spectroscopic data for similar carbonyl-like ligand complexes are needed. Thus, information on trans influence may be obtained from ^{31}P NMR chemical shifts for at least two types of Ir(I) complexes. It is of interest to extend this technique to other types of ligands, metals, and geometries to determine general applicability in structural investigations of solution species.

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Registry No. *trans*-$IrCl(PPh_3)_2$(CN-*t*-Bu), 74331-50-3; *trans*-$IrCl(PPh_3)_2(CS)$, 30106-92-4; *trans*-$IrCl(PPh_3)_2(N_2C_5Cl_4)$, 66973-33-9; *trans*-$IrCl(PPh_3)_2(N_2C_5Br_4)$, 73396-30-2; *trans*-$IrCl(PPh_3)_2$(*p*-$MeOC_6H_4N_2^+$), 74331-51-4; *trans*-$IrCl(PPh_3)_2(PhN_2^+)$, 74331-52-5; *trans*-$IrCl(PPh_3)_2(N_2)$, 74331-53-6; *trans*-$IrCl(PPh_3)_2(NO^+)$, 25246-36-0; *trans*-$IrCl(PPh_3)_2$(P(*p*-$FC_6H_4)_3$), 74331-54-7; *trans*-$IrCl(PPh_3)_2$(P(O-*i*-$Pr)_3$), 74331-55-8; *trans*-$IrCl(PPh_3)_2$(P(*p*-$MeC_6H_4)_3$), 74331-56-9; *trans*-$IrCl(PPh_3)_2$(P(*p*-$MeOC_6H_4)_3$), 74331-57-0; *trans*-$IrCl(PPh_3)_2(PMe_2Ph)$, 74331-58-1; *trans*-$IrCl(PPh_3)_3$, 16070-58-9; *trans*-$IrCl(PPh_3)_2(CO)$, 15318-31-7; *trans*-$IrCl(PPh_3)_2(C_2H_4)$, 32761-43-6.

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Synthesis and Properties of Borohydride Derivatives

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The remarkable stability and selectivity of sodium cyanoborohydride, $NaBH_3CN$, induced by the strong electron-withdrawing cyanide group, establish it as one of the more important hydride reagents.[1,2] However, due to its toxicity, sodium cyanoborohydride has found limited industrial application. Recent research activities at Ventron have been concerned with the development of new hydride reducing agents. In this note, we want to report some interesting properties of a related derivative, sodium (thiocyanato)borohydride,[3] $NaBH_3SCN$, and a new synthetic route of $NaBH_3CN$.

Sodium (Thiocyanato)borohydride

Sodium (thiocyanato)borohydride was prepared according to the procedure described in the literature,[3] by passing B_2H_6 into anhydrous NaSCN in ethylene glycol–dimethyl ether (monoglyme). Dioxane was then added to the filtered solution to precipitate the product, $NaBH_3SCN\cdot 2$(dioxane). The solid is soluble in tetrahydrofuran (THF), water, and ethanol. The infrared spectrum shows B–H stretching at 2290, 2330, and 2380 cm^{-1}. Both SCN (2180 cm^{-1} strong) and NCS (2080 cm^{-1} weak) groups are observed, indicating the presence of two isomers. The ^{11}B nuclear magnetic resonance spectrum was found[3] to be consistent with a monosubstituted BH_4^-.

It is interesting to note that a different result was obtained by using $BH_3\cdot THF$ as reactant in THF. Thus, when $BH_3\cdot THF$ was titrated into a suspension of NaSCN in THF, a clear solution was obtained at the point when the molar ratio of [$BH_3\cdot THF$] to [NaSCN] was ~0.65. The infrared spectrum of this solution was identical with that of $NaBH_3SCN\cdot 2$(dioxane) in the B–H and SCN stretching regions. A white

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