mixture is nearly linear (for large DMSO concentration) as would be required by the mechanism in eq 3 when it is recognized that $k_{-1}$ is a second-order rate constant.

It has recently been unfashionable to suggest that the intermediate of five-coordination might be energetically favored in the absence of electronically esoteric ligands to stabilize it.[19] However, we would rest our case for giving serious consideration to the possibility that the intermediate is "relatively stable" in this system on the convergence of three lines of evidence which are probably worth recapitulating. First, the nmr exchange studies in mixed solvents require some sort of intermediate. Second, the complex formation rates in pure DMSO cannot be readily fitted into the scheme that works for aqueous systems. Third, the effect of adding $CH_3NO_2$ affects complex formation rates as would be predicted if there were a five-coordinate intermediate. No one of these lines of evidence is convincing alone. Together, they add up to sufficient circumstantial evidence to warrent serious consideration of the pathway in eq 3. Perhaps, the reason that the intermediate arises in DMSO is steric. Hindrance has been shown to favor five-coordination.[20]

With respect to the energetic objection to the intermediate, Tobe's warning is relevant:[21] ". . . attacking (the intermediate) on energetic grounds (is) . . .harking back to the arguments used against Dr. Ingold in the thirties when he proposed the now accepted unimolecular mechanism for certain substitution reactions at tetrahedral carbon."

**Acknowledgment.**—We are indebted to the National Research Council of Canada for financial support. C. H. L. acknowledges an Alfred P. Sloan research fellowship for 1968–1970.

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[19] See ref 15, pp 132–135.

[20] P. Paoletti and M. Ciampolini, *Inorg. Chem.*, **6**, 64 (1967).

[21] M. L. Tobe, *Advan. Chem. Ser.*, **No. 49**, 29 (1965).

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# A New Three-Coordinate Complex of Nickel(0). Tris(tri-*o*-tolyl phosphite)nickel

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The preparation and properties of coordinatively unsaturated tris(tri-*o*-tolyl phosphite)nickel(0) and coordinatively saturated tetrakis(tri-*o*-tolyl phosphite)nickel(0) are described. Spectrophotometric measurements give a ligand-association equilibrium constant of $15 \pm 2\ M^{-1}$ in benzene at 28°. The temperature dependence of the equilibrium gives $\Delta H° = -13 \pm 1.5$ kcal/mol and $\Delta S° = -37 \pm 4$ eu. Facile reactions of the three-coordinate complex with tri-*o*-tolyl phosphite, CO, and $C_2H_4$ are described.

## Introduction

Since the discovery of $Ni[PCl_3]_4$ in 1951 by Irvine and Wilkinson,[1] a variety of zerovalent nickel complexes with phosphorus ligands have been described.[2] In most cases the complexes have been four-coordinate.[3] Two- and three-coordinate phosphine complexes $Ni[P(C_6H_{11})_3]_2$[4] and $Ni[P(C_6H_5)_3]_3$[5] have been reported. A two-coordinate phosphite complex, $Ni[P(O\text{-}o\text{-}C_6H_4CH_5)_3]_2$, has been claimed in a recent patent.[6] Claims of three-coordinate phosphite complexes $Ni[P(OC_2H_5)_3]_3$ based on molecular weights[7] and $Ni[P(OC_6H_5)_3]_3$ based on elemental analysis[8] have been made but are probably erroneous, as shown by recent spectrophotometric and $^{31}P$ nmr studies of the tetrakis complexes.[3] The preparation of material formulated as $Ni[P(O\text{-}p\text{-}C_6H_4Cl)_3]_3$ has recently been reported.[9,10]

We wish to report a detailed account of the preparation and properties of a three-coordinate phosphite complex of Ni(0), tris(tri-*o*-tolyl phosphite)nickel. Spectrophotometric data are presented which permit determination of the ligand-association equilibrium constant to form the tetrakis-phosphite complex, whose preparation is also described. Measurements of $K$ in benzene at 10, 30, 50, and 70° were made to determine $\Delta H°$ and $\Delta S°$. Finally, rapid reactions of the coordinatively unsaturated tris complex with CO and $C_2H_4$ will be described.

## Experimental Section

Because of the sensitivity of these complexes to oxygen, all synthetic operations were carried out in a nitrogen atmosphere with degassed solvents. Special precautions were taken in the spectrophotometric experiments. Reagent grade benzene solvent was thoroughly deoxygenated by three cycles of freezing,

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[1] J. W. Irvine, Jr., and G. Wilkinson, *Science*, **113**, 742 (1951).

[2] References to the preparation of a number of these are given in ref 3.

[3] C. A. Tolman, *J. Amer. Chem. Soc.*, **92**, 2956 (1970).

[4] (a) P. W. Jolly and K. Jonas, *Angew. Chem., Int. Ed. Engl.*, **7**, 731 (1968); (b) K. Jonas and G. Wilke, *ibid.*, **8**, 519 (1969).

[5] Molecular weight evidence for $Ni[P(C_6H_5)_3]_3$ was given by P. Heimbach, *Angew. Chem.*, **76**, 586 (1964).

[6] M. A. McCall and H. W. Coover, British Patent 1,146,074 (1969).

[7] R. S. Vinal and L. T. Reynolds, *Inorg. Chem.*, **3**, 1062 (1964).

[8] N. von Kutepow, H. Seibt, and F. Meier, U. S. Patent 3,346,608 (1967).

[9] J. J. Levison and S. D. Robinson, *J. Chem. Soc. A*, 96 (1970).

[10] The formation of this tris complex under the conditions reported is surprising in view of our studies of zero-valent nickel compounds. More of our results will be reported in detail later.

pumping, and thawing. Solutions were prepared in a HE-43-2 drybox manufactured by Vacuum Atmospheres, Inc., under conditions where 1.5 *M* diethylzinc in heptane did not smoke (less than 5 ppm of $O_2$).

**Tris(tri-*o*-tolyl phosphite)nickel,** $Ni[P(O\text{-}o\text{-}C_6H_4CH_3)_3]_3$, was prepared by the gradual addition of sodium borohydride[11] to a stirred solution of 2.0 g of $Ni(NO_3)_2 \cdot 6H_2O$ (7 mmol) and 10.0 g of tri-*o*-tolyl phosphite[12] (32 mmol) in 50 ml of anhydrous acetonitrile. Gas evolution ceased after addition of 2 g of $NaBH_4$ (0.05 mol). An additional 1 g was added and the solution was stirred for 2 hr. Filtration gave a pale yellow solution which was concentrated under reduced pressure to a red grease. The grease was dispersed in 50 ml of benzene and the resulting mixture was filtered. The benzene was evaporated from the filtrate under reduced pressure. The residue was treated with 75 ml of hexane and the resulting mixture was filtered. Washing with hexane and drying gave the product as a red powder, 3.3 g (42% yield), dec pt 125–150°, the temperature depending on the rate of heating.

The infrared spectrum of tris(tri-*o*-tolyl phosphite)nickel (Nujol) in the region of 4000–600 $cm^{-1}$ was the same as that of the ligand. Nmr data are given in the text. The cryoscopic molecular weight in benzene was 1143 (calcd for the tris complex 1116).[13] *Anal.* Calcd for $C_{63}H_{63}O_9P_3Ni$: C, 67.81; H, 5.69; P, 8.32; Ni, 5.26; O, 12.90. Found: C, 67.87; H, 5.82; P, 8.06; Ni, 5.50; O, 13.21.

The complex is readily soluble in benzene or toluene to give red-orange solutions which are stable for hours at room temperature. The complex is rapidly destroyed by halogenated solvents such as chloroform or carbon tetrachloride.

**Tetrakis(tri-*o*-tolyl phosphite)nickel,** $Ni[P(O\text{-}o\text{-}C_6H_4CH_3)_3]_4$, was prepared by the addition of 0.5 g of tri-*o*-tolyl phosphite ligand to a fresh filtered solution of 1.5 g of tris(tri-*o*-tolyl phosphite)nickel in 15 ml of benzene. Removal of the solvent under reduced pressure gave a pale amber grease. Addition of methanol gave a gray powder. Washing with methanol and pentane gave the product as an off-white powder, 1.5 g (75% yield), turning red and melting at 90–105°. *Anal.* Calcd for $C_{84}H_{84}O_{12}P_4Ni$: C, 68.72; H, 5.77; Ni, 4.00. Found: C, 68.43; H, 5.77; Ni, 3.83.

The complex dissolved readily in benzene or toluene to give red-orange solutions.

Melting points were determined in evacuated, sealed capillaries on a Mel-Temp apparatus and are uncorrected.

Proton nmr spectra were determined in 90% $C_6D_6$–10% tetramethylsilane (TMS) using a Varian HA-100 at ambient temperature (29 ± 1°). The TMS was added as an internal reference and lock signal.

Optical spectra of solutions of $NiL_3$ were recorded on a Cary 14 spectrophotometer using quartz cells made by the Optical Cell Co., Inc. The cells were modified by the addition of an 8-mm o.d. neck extension for serum caps, through which additions of ligand could be made by syringe without admission of air.

Beer's law was confirmed for solutions of $Ni[P(O\text{-}o\text{-}C_6H_4CH_3)_3]_3$, over a 100-fold concentration range by running spectra at concentrations of $1.89 \times 10^{-2}$, $1.89 \times 10^{-3}$, and $1.89 \times 10^{-4}$ *M* in cells of 0.1–10 mm so that the product of concentration and path length was held constant. The spectrum of the red complex (benzene) showed an absorption maximum at 407 mμ ($\epsilon$ $4.5 \times 10^3$ $cm^{-1}$ $M^{-1}$) and a shoulder at 450 mμ ($\epsilon$ $3.5 \times 10^3$). The ligand association equilibrium constant was determined by running spectra after additions of neat $P(O\text{-}o\text{-}C_6H_4CH_3)_3$ by syringe to the 0.1-mm cell containing 1 ml of $1.89 \times 10^{-2}$ *M* $NiL_3$. A gastight 100-μl Hamilton syringe was used with a Hamilton automatic repeating dispenser, which permitted injections of 2-μl volumes with a precision of ±0.1 μl. The temperature of the Cary 14 sample compartment during these measurements was 28°.

The temperature dependence of the equilibrium constant was determined in a 1-mm thermostated cell[14] at 10, 30, 50, and 70°. The temperature in the center of the cell was determined using a 36-gauge copper–constantan thermocouple with an ice reference junction. Temperatures are accurate to ±0.5°. Spectra of at least four solutions were run at each temperature. Each solution contained $2 \times 10^{-3}$ *M* $NiL_3$ and variable added ligand concentration in the range of 0.007–1.5 *M*, with higher concentrations at higher temperatures where formation of the four-coordinate complex was less favorable. Great care was required in loading the thermostated cell by syringe to prevent any air from entering the highly oxygen-sensitive solution.

## Results and Discussion

The tris(tri-*o*-tolyl phosphite)nickel reported here (abbreviated $NiL_3$) is red and three-coordinate. The elemental analysis is consistent with the composition $NiL_3$. For complexes with ligands of this molecular weight (352), however, a change in coordination number by 1 makes rather little change in the per cent composition. A more definitive analytical result is the molecular weight (calcd, 1116; found, 1143), which shows that three phosphites are attached to nickel in the red complex.

The $NiL_3$ complex dissolves in benzene to give a red-orange solution, whose proton nmr spectrum (Figure 1a) shows resonances of the ligand protons in a 1:3:3 ratio assignable to the *ortho* proton, the unresolved *meta* and *para* protons, and the *o*-methyl protons, respectively. Injection of additional ligand by syringe quickly changes the color to orange, and the *ortho* proton resonances indicate the presence of three kinds of ligands.[15] In addition to resonances of $NiL_3$, now diminished in intensity, resonances appear in the positions of free L and in new positions; the latter are assigned to $NiL_4$. Proton chemical shifts of various species are given in Table I. Further addition of ligand caused the resonances of free L and of $NiL_4$ to increase in intensity at the expense of $NiL_3$. Figure 1b shows the spectrum obtained after addition of 20 μl of L (0.064 mmol) to 0.07 g of $NiL_3$ (0.063 mmol) in 0.3 ml of benzene solution. The same spectrum was obtained with a benzene solution of the $NiL_4$ complex.

TABLE I

PROTON NMR SPECTRA OF L = TRI-*o*-TOLYL PHOSPHITE AND ITS COMPLEXES OF Ni(0) IN $C_6D_6$ ($\tau$ (TMS))

| Species | *o*-H | *m*- and *p*-H | *o*-$CH_3$ |
|---|---|---|---|
| L | 2.68 | 3.07 | 7.89 |
| $NiL_3$ | 2.48 | 3.12 | 8.04 |
| $NiL_4$ | 2.26 | 3.24 | 8.04 |

Disappearance of $NiL_3$ and appearance of $NiL_4$ and free L as L was added was also shown by $^{31}P$ spectra in toluene. Sharp resonances of the three species were observed at −128.3, −129.0, and −130.0 ppm (85%

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[11] The general method of preparing nickel(0) phosphite complexes by sodium borohydride reduction of $Ni(NO_3)_2 \cdot 6H_2O$ in acetonitrile solution in the presence of phosphite ligands was described to us by Dr. William C. Drinkard of the Plastics Department, E. I. du Pont de Nemours and Co.

[12] E. N. Walsh, *J. Amer. Chem. Soc.*, **81**, 3023 (1959).

[13] Unless extreme care was used to exclude air from the solvent and the freezing point apparatus, greatly reduced apparent molecular weights were obtained.

[14] The cell and the technique for loading and cleaning were described by C. A. Tolman, *J. Amer. Chem. Soc.*, **92**, 4217 (1970).

[15] The rapidity of the color change indicates that the reaction is quite fast on a chemical time scale. The observation of separate sharp resonances for bound and free ligands, however, indicates that the exchange reaction is slow on an nmr time scale.

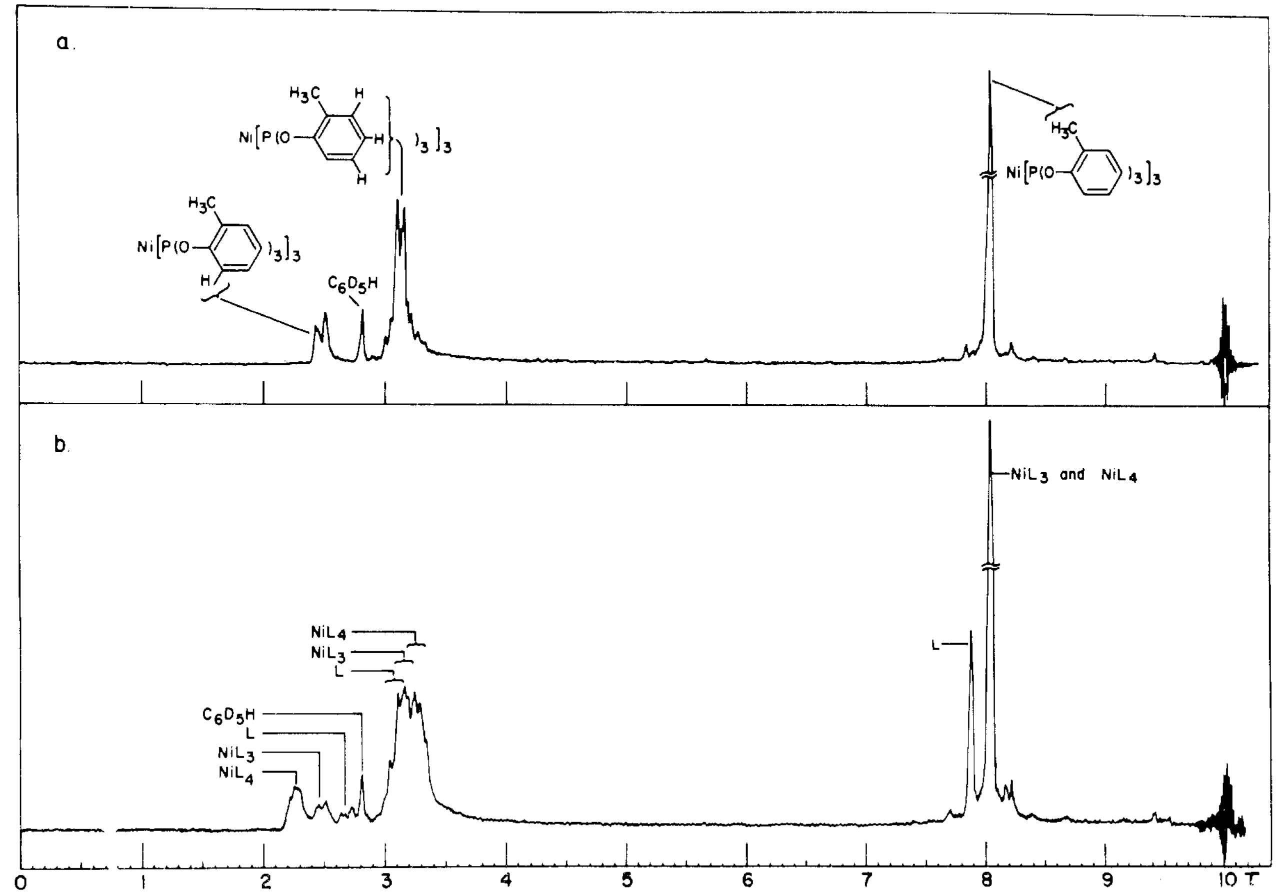


Figure 1.—(a) The 100-Mc proton nmr spectrum of 0.07 g of Ni[P(O-*o*-$C_6H_4CH_3$)$_3$]$_3$ (0.063 mmol) in 0.3 ml of $C_6D_6$ solution. (b) Spectrum after addition of 20 μl of P(O-*o*-$C_6H_4CH_3$)$_3$ (0.064 mmol).

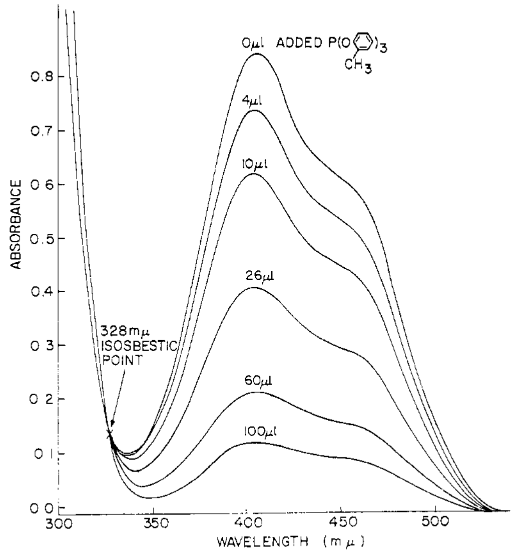


Figure 2.—Optical spectra for addition of P(O-*o*-$C_6H_4CH_3$)$_3$ to 1 ml of $1.89 \times 10^{-2}$ *M* Ni[P(O-*o*-$C_6H_4CH_3$)$_3$]$_3$ in benzene in a 0.1-mm cell at 28°. A 0.1-mm cell of benzene was used in the reference beam.

$H_3PO_4$), respectively.[3] Identical $^{31}P$ spectra were obtained from a solution of $NiL_3$ to which 1 equiv of L had been added and from the orange toluene solution obtained by dissolving the $NiL_4$ complex.

The nmr results are consistent with a ligand-association equilibrium in solution as indicated by reaction 1.

$$NiL_3 + L \overset{K}{\rightleftharpoons} NiL_4 \qquad (1)$$

This interpretation was confirmed and a value for *K* was determined by spectrophotometric experiments in which neat L was added to a red-orange benzene solution of $NiL_3$. The color changed as rapidly as the ligand was mixed in, successive additions causing a change from red-orange to orange to yellow. Selected spectra are shown in Figure 2. The existence of an isosbestic point at 328 mμ indicates that the reaction involves conversion of one chromophore ($NiL_3$) to another ($NiL_4$), as indicated by eq 1. The absorbance at the 407-mμ maximum was used to determine an associative equilibrium constant at ambient temperature of $K = 15 \pm 2$ $M^{-1}$ as shown in Table II.

The value of *K* determined in a 1-mm thermostated cell diminished with increasing temperature as shown below.

| Temp, °C | *K*, $M^{-1}$ | Temp, °C | *K*, $M^{-1}$ |
|---|---|---|---|
| 10 | 97 ± 12 | 50 | 4.5 ± 0.6 |
| 30 | 16 ± 2 | 70 | 1.4 ± 0.4 |

A plot of log *K* *vs.* 1/*T* gave a straight line from whose slope and intercept values of $\Delta H° = -13 \pm 1.5$ kcal/mol and $\Delta S° = -37 \pm 5$ eu were determined. Formation of a nickel–phosphorus bond as shown in eq 1 is exothermic to the extent of 13 kcal/mol. The large negative entropy change for the reaction is quite reasonable for a ligand-association process.

TABLE II

ADDITION OF TRI-*o*-TOLYL PHOSPHITE TO 1.0 ML OF $1.89 \times 10^{-2}$ *M* $\mathrm{NiL_3}$ IN BENZENE AT 28° (0.1-MM CELL)

| Added L[a] μl | Added L[a] mmol $\times 10^2$ | $10^2$[L],[b] *M* | *A*(407)[c] | $10^2$[$\mathrm{NLi_3}$],[d] *M* | $10^2$[$\mathrm{NiL_4}$],[e] *M* | *K*,[f] $M^{-1}$ |
|---|---|---|---|---|---|---|
| 0 | 0.00 | 0.00 | 0.84 | 1.89 | 0.00 | . . . |
| 2 | 0.64 | 0.50 | 0.78 | 1.75 | 0.14 | 16.0 |
| 4 | 1.27 | 1.03 | 0.735 | 1.65 | 0.24 | 14.2 |
| 6 | 1.91 | 1.57 | 0.69 | 1.55 | 0.33 | 13.6 |
| 10 | 3.19 | 2.67 | 0.62 | 1.39 | 0.49 | 13.2 |
| 16 | 5.10 | 4.32 | 0.51 | 1.15 | 0.71 | 14.3 |
| 26 | 8.28 | 7.15 | 0.41 | 0.92 | 0.92 | 14.0 |
| 40 | 12.7 | 11.0 | 0.30 | 0.67 | 1.15 | 15.6 |
| 60 | 19.1 | 16.7 | 0.22 | 0.50 | 1.28 | 15.3 |
| 100 | 31.9 | 27.6 | 0.125 | 0.28 | 1.44 | 18.7 |
| ∞ | | | 0.00 | | | Av 15 ± 2 |

[a] The molar volume of L is 314 ml/mol. [b] $[\mathrm{L}] = \{(\text{mmol of L}) - (1 + v)[\mathrm{NiL_4}]\}/(1 + v)$ where $v$ is the volume of L added in milliliters. [c] The absorbance at 407 mμ. [d] $[\mathrm{NiL_3}] = (A(407)/0.84)(1.89 \times 10^{-2})\ M$. [e] $[\mathrm{NiL_4}] = \{(1.89 \times 10^{-2} - (1 + v)[\mathrm{NiL_3}]\}/(1 + v)\ M$. [f] $K = [\mathrm{NiL_4}]/[\mathrm{L}][\mathrm{NiL_3}]\ M^{-1}$.

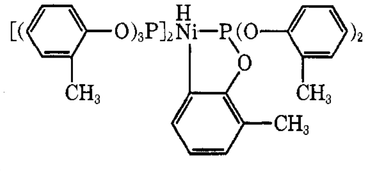
The absence of a two-coordinate $\mathrm{NiL_2}$ complex in solution *via* reaction 2 is most conclusively demonstrated by the adherence of the $\mathrm{NiL_3}$ solutions to Beer's law.[16]

$$\mathrm{NiL_3} \overset{K'}{\rightleftharpoons} \mathrm{NiL_2} + \mathrm{L} \qquad (2)$$

Absence of $\mathrm{NiL_3}$ dissociation is also shown by the appearance of a single type of ligand in $^1$H or $^{31}$P nmr spectra of $\mathrm{NiL_3}$.[17] In view of a lack of detectable dissociation of $\mathrm{NiL_3}$ *via* eq 2, McCall and Coover's[6] claim of preparing a solution containing a substantial concentration of bis(tri-*o*-tolyl phosphite)nickel is highly questionable, especially since they carried out their preparation in the presence of *excess* phosphite. Unfortunately, they did not give enough experimental data to indicate the basis of their choice of coordination number.

The location of the phosphorus atoms about the nickel in the $\mathrm{NiL_3}$ complex is not known, but they probably lie at the corners of an equilateral triangle, with the metal in the center, similar to the structure which has been determined by X-ray diffraction for $\mathrm{Pt[P(C_6H_5)_3]_3}$.[18]

Because of the recent demonstration that aryl phosphite ligands can undergo intramolecular oxidative addition reactions with low-valent metals,[19] we also considered structures such as the nickel(II) hydride complex



Any significant amount of this material can be ruled out from the nmr spectra. The $^{31}$P spectrum of $\mathrm{NiL_3}$ has only a single sharp resonance,[20] rather than the two expected for the "hooked" complex. The proton spectrum of $\mathrm{NiL_3}$ also shows that only one type of ligand is present. Furthermore, searches above τ 10 failed to give evidence for a metal hydride resonance.

The participation of tris-phosphite complexes $\mathrm{Ni[P(OC_2H_5)_3]_3}$[21] and $\mathrm{Ni[P(O\text{-}\mathit{p}\text{-}C_6H_4OCH_3)_3]_3}$[22] as steady-state intermediates in ligand substitution reactions of the tetrakis complexes has been proposed on the basis of kinetic studies. The reactions were SN1, the slow rate-determining dissociation of a ligand to give $\mathrm{NiL_3}$ being followed by a much more rapid addition of the attacking ligand to the coordinatively unsaturated tris-phosphite complex. Spectroscopic experiments[3] have not shown detectable concentrations of $\mathrm{NiL_3}$ species in solutions of $\mathrm{Ni[P(OC_2H_5)_3]_4}$ or $\mathrm{Ni[P(O\text{-}\mathit{p}\text{-}C_6H_4OCH_3)_3]_4}$.

The isolability of a tris-phosphite complex, in the case of $\mathrm{P(O\text{-}\mathit{o}\text{-}C_6H_4CH_3)_3}$, but not with $\mathrm{P(O\text{-}\mathit{p}\text{-}C_6H_4OCH_3)}$ or $\mathrm{P(OC_2H_5)_3}$, can be understood on the basis of steric effects. The first two ligands are electronically indistinguishable, based on carbonyl stretching frequencies of $\mathrm{Ni(CO)_3L}$ complexes,[23] so that an electronic effect is clearly not responsible. Ligand cone angles for these ligands are 165, 121, and 109°, respectively.[24]

The large steric bulk of tri-*o*-tolyl phosphite destabilizes the tetrakis complex to the point that $\mathrm{NiL_3}$ becomes isolable. The existence of coordinatively unsaturated phosphine complexes $\mathrm{Ni[P(C_6H_{11})_3]_2}$[4] and $\mathrm{Ni[P(C_6H_5)_3]_3}$[5] can also be understood in terms of steric effects; the two ligands have cone angles[24] of 179 and 145°, respectively.

In contrast to the reaction rates of the four-coordinate complexes $\mathrm{Ni[P(OC_2H_5)_3]_4}$[21] and $\mathrm{Ni[P(O\text{-}\mathit{p}\text{-}C_6H_4OCH_3)_3]_4}$,[22] the reactions of our $\mathrm{NiL_3}$ complex are very fast. The reactions are so fast because it is unnecessary to lose a ligand to make a coordination position available. Addition of L, CO, or $\mathrm{C_2H_4}$ to solutions of the tris complex caused the red color to disappear as soon as the attacking ligand was mixed in. The carbonyl complex $\mathrm{NiL_2(CO)_2}$ was identified by the very strong $A_1$ and $B_1$ carbonyl bands at 2045 and 1988 cm$^{-1}$ after CO was bubbled through a toluene solution of $\mathrm{NiL_3}$ for 5 min.[25] The rapid reaction of ethylene with $\mathrm{NiL_3}$ gives the complex $\mathrm{(C_2H_4)NiL_2}$,[26] which will be described in a subsequent publication.

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(16) Assuming that a 10% dissociation of $\mathrm{NiL_3}$ would have been detectable in the $1.89 \times 10^{-4}$ *M* solution, an upper limit of about $2 \times 10^{-6}$ *M* can be set on *K′*.

(17) This interpretation of the nmr data for $\mathrm{NiL_3}$ requires that exchange of bound and free L in solutions of $\mathrm{NiL_3}$ be slow by nmr. This criterion is met in our system.

(18) V. Albano, P. L. Bellon, and V. Scatturin, *Chem. Commun.*, 507 (1966).

(19) G. W. Parshall, W. H. Knoth, and R. A. Schunn, *J. Amer. Chem. Soc.*, **91**, 4990 (1969).

(20) The presence of a hydrogen on Ni would also be expected to give rise to a P–H spin–spin coupling, which was not observed.

(21) M. Meier, F. Basolo, and R. G. Pearson, *Inorg. Chem.*, **8**, 795 (1969). Their kinetic results indicate that $\mathrm{Ni[P(OC_2H_5)_3]_4}$ is not extensively dissociated in solution.

(22) R. D. Cramer, private communication.

(23) C. A. Tolman, *J. Amer. Chem. Soc.*, **92**, 2953 (1970).

(24) Ligand cone angles as a quantitative measure of steric effects of ligands in complexes were described in ref 3.

(25) The same spectrum was obtained by the reaction of $\mathrm{Ni(CO)_4}$ with excess $\mathrm{P(O\text{-}\mathit{o}\text{-}C_6H_4CH_3)_3}$ as described in ref 3.

(26) Briefly reported by G. Wilke, *Angew. Chem., Int. Ed. Engl.*, **2**, 105 (1963).