$P(C_6H_5)_3$, 603-35-0; $P(OC_6H_5)_3$, 101-02-0; $P(n\text{-}C_4H_9)_3$, 998-40-3; $PH(C_6H_5)_2$, 829-85-6; $P(C_2H_5)(C_6H_5)_2$, 607-01-2; $P(C_6H_{11})_3$, 2622-14-2; $As(C_6H_5)_3$, 603-32-7; $P(OCH_3)_3$, 121-45-9.

Supplementary Material Available. Table I, a listing of reagent concentrations, experimental conditions, and observed rate constants, will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D. C. 20036. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche, refering to code number INORG-74-1944.

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Synthesis and Properties of Cobalt(I) Compounds. I. Triethyl Phosphite Complexes

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The preparation and properties of $[CoL_5]Cl$, $[CoClL_3]$, $[CoL_5][B(C_6H_5)_4]$, $[CoL_5][CoCl_3]$, and $[CoL_2B(C_6H_5)_4]$ are described (L = triethyl phosphite). The reaction of cobalt(II) chloride and triethyl phosphite in the presence of triethylamine gave $[CoL_5]Cl$. It appears that two products previously formulated as $[CoClL_4]$ and $[CoClL_3]$ are respectively $[CoL_5]Cl$ and a salt $[CoL_5]_{n-2}[CoCl_n]^{2-n}$. In the complex $[CoL_2B(C_6H_5)_4]$, one of the phenyl groups σ bonded to boron appears to be π bonded to cobalt.

The simple phosphite complexes of cobalt(I), CoX(phosphite)$_n$, which have been reported are, with one exception, complexes of trimethyl phosphite or of sterically constrained polycyclic phosphites with nitrate, perchlorate, or tetraphenylborate counterions.[1] The exception is work by Vol'pin and Kolomnikov[2] who report the preparation of CoCl(triethyl phosphite)$_4$ and CoCl(triethyl phosphite)$_3$. Because of the potential for interesting catalytic properties in these complexes, we have investigated their report. Our study suggests that they did indeed obtain cobalt(I) complexes with phosphite ligands but that the products are derivatives of the $[CoL_5]^+$ ion (L = triethyl phosphite).

Although the experiments in ref 2 are reported in insufficient detail to be repeated with confidence, we have carried out procedures like those described and have obtained products with similar properties. We isolated a yellow compound with moderate water stability and a peak at *ca.* 390 nm in the visible spectrum as reported for the $[CoClL_4]$ formulation.[2] We also isolated a green, water-sensitive compound with a peak at *ca.* 390 nm as reported[2] for $[CoClL_3]$. The work described below shows that our yellow compound is $[CoL_5]Cl$ and that our green compound is $[CoL_5][CoCl_3]$. It is likely that the yellow compound in the earlier work was also $[CoL_5]Cl$ and that the green compound was $[CoL_5][CoCl_3]$ or $[CoL_5]_2[CoCl_4]$. We have prepared $[CoClL_3]$ and its properties, including its ease of conversion to $[CoL_5]Cl$, indicate that it is unlikely that $[CoClL_4]$ was a product of the reaction described by Vol'pin and Kolomnikov.

The Reaction of $CoCl_2$ with Triethyl Phosphite

Vol'pin and Kolomnikov treated $CoCl_2 \cdot 6H_2O$ in ethanol with triethyl phosphite and triethylamine to prepare their yellow and green compounds. We have used dry $CoCl_2$ in place of the hydrate so that the amount of water in the mixtures could be controlled more easily. We have found that the rate of the reaction is quite sensitive to the presence of water. Under nominally anhydrous conditions the reaction is at best very slow. The reaction mixture remains dark blue-purple and clear for at least 24 hr at room temperature. Addition of small amounts of water, up to *ca.* 3% by volume, produced increasingly rapidly a transition to a cloudy green liquid followed by transition to a pale yellow liquid containing a large amount of dark flocculent precipitate. The green compound was obtained by interrupting the sequence when the liquid had become dark green and isolating the product. The yellow compound was obtained easily by waiting until the green color had faded to pale yellow. Figure 1 shows the 550–750-nm region of the spectrum at *ca.* 0.1 *M* for the green compound isolated from the green reaction mixture, for a green compound obtained by adding cobalt(II) chloride to the yellow compound, and for mixtures of tetraethylammonium chloride and cobalt(II) chloride. It is clear that both green compounds contain the same chromophore as is obtained by adding ionic chloride to cobalt(II) chloride in a 1:1 ratio. It can also be seen that there is a clear qualitative difference between the spectrum for 3:1 chloride:cobalt(II) and 4:1 chloride:cobalt(II). (The spectra of these systems are known to be dependent on the nature and concentration of the anions.[3]) Meakin and Jesson[4] have shown by computer simulation of the low-temperature (*ca.* −130°) ^{31}P nmr spectrum that our yellow compound contains the trigonal bipyramidal $[CoL_5]^+$ cation. The *ca.* 390-nm peak observed in the spectrum of our yellow compound and in that described by Vol'pin is very similar to that reported for $\{Co[P(OCH_3)_3]_5\}ClO_4$.[1d] Preparation of a salt, $\{Co[P(OCH_3)_3]_5\}_2[Co(NO_3)_4]$, from $\{Co[P(OCH_3)_3]_5\}NO_3$ and cobalt(II) nitrate has also been described.[1d]

It is thus clear that, in the synthesis procedure, $[CoL_5]Cl$ is the primary species produced and that interruption of the procedure before complete consumption of the cobalt(II) chloride can permit the isolation of the green salts containing $[CoL_5]^+$ and a chlorocobaltate anion. In principle either

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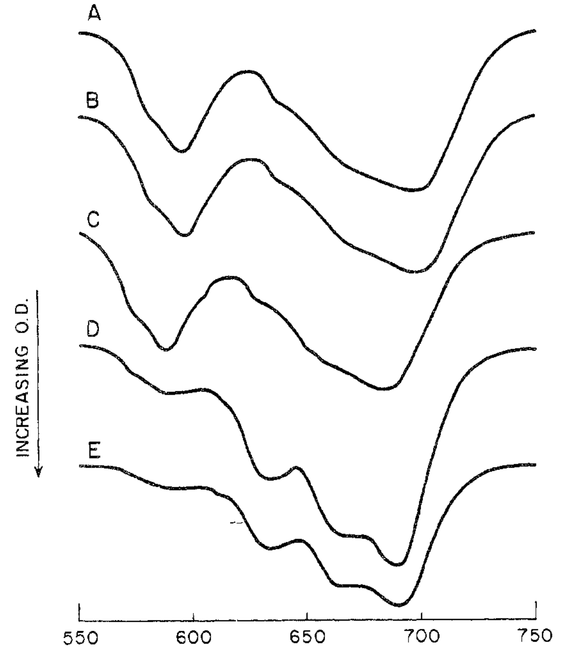

Figure 1. Cobalt concentrations were *ca.* 0.1 *M* in a 0.10-mm path length in acetonitrile: (A) green compound from $CoCl_2$, L, and triethylamine; (B) $[CoL_5][CoCl_3]$; (C) Et_4NCl–$CoCl_2$, 1:1, (D) Et_4NCl–$CoCl_2$, 2:1; (E) $[CoL_5]Cl$–$CoCl_2$, 2:1.

$[CoL_5][CoCl_3]$ or $[CoL_5]_2[CoCl_4]$ could be isolated depending on the details. The $[CoL_5][CoCl_3]$ compound is insoluble in benzene while $[CoL_5]Cl$ is benzene soluble. A solid can be obtained which gives the 550–750-nm absorption (Figure 1) indicative of 4:1 chloride:cobalt(II). This can then be converted to material with a 3:1 ratio by washing with benzene. Although the simple trichlorocobaltate(II), $K[CoCl_3]$, has been reported,[5] it is probable that, in our case, the cobalt(II) remains four coordinate with the fourth site occupied by solvent. The green solids always show weak infrared peaks near 3300 cm^{-1} assignable to hydroxyl functions.

If our formulation of Vol'pin and Kolomnikov's green product is correct, it is apparent that their observed magnetic moment ($\mu_c = 2.74$) is fortuitous in its agreement with that predicted for a tetrahedral d^8 cobalt(I) ion. Instead, the observed moment is that associated with the tetrahedral d^7 cobalt(II) in the anion somewhat diluted by the presence of a diamagnetic cation.

Preparation and Properties of $[CoClL_3]$

Authentic $[CoClL_3]$ was prepared by heating $[CoL_5]Cl$ a little above its melting point under vacuum. About 40% weight loss occurred and large pale brown crystals were obtained from the residue by crystallization from pentane at *ca.* $-35°$. This material has properties consistent with the $[CoClL_3]$ formulation. It is paramagnetic with μ_{eff} of 3.15 as estimated by the Evans[6] nmr method. This value is in good agreement with those reported for $[CoCl(PR_3)_3]$ compounds[7] and suggests that it is a tetrahedral complex. (The $[Co(Ph_2PCH_2CH_2PPh_2)_2]X$ compounds are diamagnetic and are presumably square planar or square pyramidal complexes.[8])

The assignment of the $[CoClL_3]$ formulation to this brown compound is encouraged by the observation that addition of 2 equiv of triethyl phosphite to an acetonitrile solution regenerates $[CoL_5]Cl$. Similarly, addition of triethyl phosphite to an ethanol solution followed by addition of sodium tetraphenylborate gave immediate precipitation of $[CoL_5][B(C_6H_5)_4]$. However, addition of only 1 equiv of triethyl phosphite to an acetonitrile solution of $[CoClL_3]$ produced a solution with a visible spectrum which was nearly a composite of the spectra of $[CoL_5]Cl$ and $[CoClL_3]$ indicating that $[CoClL_4]$ is not a favored species under these circumstances. Addition of triethyl phosphite to hexane or benzene solutions of $[CoClL_3]$ produced very little color change. It was possible to obtain a moderate rate of reaction of $[CoClL_3]$ with an excess of L by using as solvent 2:1 by volume tetrahydrofuran–ethanol or ether–ethanol. It was then possible to demonstrate that the rate of production of $[CoL_5]Cl$ was dependent on the concentration of L (with constant initial $[CoClL_3]$ concentrations). It was also possible to demonstrate that in the 2:1 tetrahydrofuran–ethanol solvent the rate of formation of $[CoL_5]Cl$ was retarded by *ca.* 0.3 *M* LiCl. The dependence of the rate on the concentration of L makes it unlikely that simple dissociation, $[CoClL_3] \rightleftharpoons [CoL_3]^+ + Cl^-$, is the rate-determining step. However, the strong dependence of the rate on dielectric constant and the retardation under some circumstances by added chloride do suggest that dissociation of chloride is important in the reaction. The following scheme is consistent with the qualitative observations above including the preparation of $[L_3CoCl]$ from $[L_5Co]Cl$.

$$\begin{tikzcd}
L + [CoClL_3] \arrow[r, phantom, "\rightleftharpoons"] & {[CoClL_4]} \arrow[r, phantom, "\rightleftharpoons"] & {[CoL_4]^+ + Cl^-} \arrow[d, shift left, "+L"] \\
 & & {[CoL_5]^+} \arrow[u, shift left, "-L"]
\end{tikzcd}$$

It would be reasonable to expect the relative values of the rate constants and the concentrations of the intermediates to depend on the details of any specific situation. Verification and elaboration of the scheme will require quantitative rate data.

Preparation of $[CoL_2B(C_6H_5)_4]$

Addition of sodium tetraphenylborate to an ethanol solution of $[CoClL_3]$ without prior addition of triethyl phosphite produced a precipitate only slowly, and examination of this material established that it was a mixture of $[CoL_5][B(C_6H_5)_4]$ and an entirely new cobalt compound, $[CoL_2B(C_6H_5)_4]$. This formulation is consistent with the spectral data and the elemental analyses. This compound, like the analogous rhodium compound $[RhL_2B(C_6H_5)_4]$ (L = trimethyl phosphite)[9] probably has one of the phenyl groups of the tetraphenylborate moiety π bonded to the metal (see Figure 3). The methylene regions of the ^1H nmr spectra of triethyl phosphite, $[CoL_5]^+$, and $[CoL_2B(C_6H_5)_4]$ are presented in Figure 2. The complex pattern observed for $[CoL_2(BC_6H_5)_4]$ was identical at 60 and 100 MHz. It is interesting to note that the methyl groups in $C_5H_5Co[P(OCH_3)_3]_2$ are also apparently coupled to both phosphorus nuclei.[10] These are

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[6] D. F. Evans, *J. Chem. Soc., London*, 2003 (1959).

[7] (a) M. Aresta, M. Rossi, and A. Sacco, *Inorg. Chim. Acta*, 3, 227 (1969); (b) L. Sacconi and S. Midollini, *J. Chem. Soc., Dalton Trans.*, 1213 (1972); (c) G. N. La Mar, E. O. Sherman, and G. A. Fuchs, *J. Coord. Chem.*, 1, 289 (1971).

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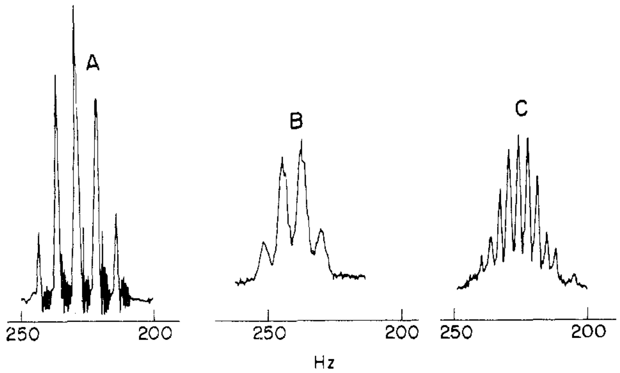

Figure 2. ^1H nmr spectra of the methylene protons of free and complexed triethyl phosphite. Peak positions are in hertz from internal tetramethylsilane at 60 MHz: (A) triethyl phosphite, (B) $[CoL_5][B(C_6H_5)_4]$-$DCCl_3$, (C) $[CoL_2B(C_6H_5)_4]$-$DCCl_3$.

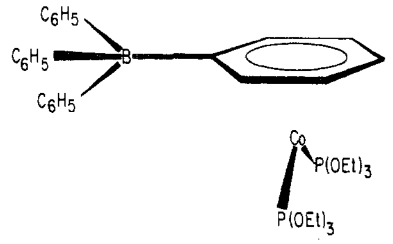

Figure 3. Possible structure of $[CoL_2B(C_6H_5)_4]$.

Table I. Elemental Analyses

	C	H	Co	Cl
$[CoL_5]Cl$ found	38.96	8.00	6.46	4.04
$C_{30}H_{75}O_{15}P_5CoCl$	38.94	8.17	6.37	3.83
$[CoL_5][CoCl_3]$ found	34.12	7.36	11.65	10.57
$C_{30}H_{75}O_{15}P_5Co_2Cl_3$	34.15	7.17	11.17	10.08
$[CoL_5][B(C_6H_5)_4]$ found	53.37	8.00	5.01	
$C_{54}H_{95}O_{15}P_5CoB$	53.65	7.92	4.89	
$[CoL_2B(C_6H_5)_4]$ found	60.60	7.23	8.52	
$C_{36}H_{50}O_6P_2CoB$	60.86	7.09	8.29	
$[CoClL_3]$ found	36.15	7.35	10.06	6.33
$C_{18}H_{45}O_9P_3CoCl$	36.46	7.65	9.94	5.98

Table II. Electronic (Visible) Spectra

Compd	Solvent	Absorption nm (ϵ)
$[CoL_5]Cl$	CH_3CN	386 (1.0×10^3)
$[CoL_5][B(C_6H_5)_4]$	CH_3CN	390 (1.0×10^3)
$\{Co[P(OCH_3)_3]_5\}[B(C_6H_5)_4]$[a]	CH_3CN	380 (1.1×10^3)
$\{Co[P(OCH_3)_3]_5\}ClO_4$[b]	CH_2Cl_2	382 (1.0×10^3)
$[CoL_5][CoCl_3]$	CH_3CN	392 (9.4×10^2) 588 (4.0×10^2) 660 sh (4.4×10^2) 685 (5.5×10^2)
$[CoClL_3]$	Hexane	437 (38) 774 (1.9×10^2)
$[CoL_2B(C_6H_5)_4]$	CH_3CN	415 (9.8×10^2)

[a] Sample prepared from $Co(C_8H_{12})(C_8H_{13})$, $(CH_3O)_3P$, and HCl.[13]
[b] Value reported in ref 1d.

apparently part of a growing list of first row transition metal complexes which exhibit virtual coupling.[11]

Experimental Section

Commercially available materials were used without special preparation. Melting points are uncorrected. The nmr spectra were obtained with Varian 60- and 100-MHz instruments and a Bruker 90-MHz instrument modified for Fourier transform operation. Elemental analyses, electronic spectra (determined on a Cary 17), and proton nmr data are reported in Tables I-III.

All experiments were carried out in the absence of air. A nitrogen-filled glove box was used in most cases. No quantitative air sensitivity data were obtained. However, $[CoClL_3]$ seemed to be quite air sensitive while solid $[CoL_5][B(C_6H_5)_4]$ was much more resistant.

Pentakis(triethyl phosphite)cobalt(I) Chloride. A solution of 28 ml of triethyl phosphite, 600 ml of anhydrous ethanol, 10 ml of water, and 7.8 g of anhydrous cobalt(II) chloride was prepared in a nitrogen atmosphere. A solution of 16 ml of triethylamine in 200 ml of ethanol was added and the mixture was left overnight at room temperature. The precipitate was removed by filtration through Celite and the pale yellow filtrate was concentrated to dryness under vacuum. The residue was washed free of yellow color with ether leaving the amine hydrochloride behind. The filtrate was concentrated to dryness under vacuum. The residue was crystallized from diethyl ether or ether-pentane mixtures to give 17.5 g of yellow crystals of pentakis(triethyl phosphite)cobalt(I) chloride, mp 75-77° dec.

This material was also prepared by reaction of 2 mol equiv of triethyl phosphite with $[CoClL_3]$. To a solution of 1 g of $[CoCl[P(OEt)_3]_3]$ in 10 ml of acetonitrile was added 0.70 ml of triethyl phosphite. After 1 hr at room temperature the volatiles were removed under vacuum and the residue was crystallized from an ether-hexane mixture to give 1.5 g of yellow crystals. This material was recrystallized from benzene-hexane to give 1.3 g of yellow powder. The infrared (Nujol), the 60-MHz ^1H nmr, and the visible spectra were the same as those of the material prepared from $(EtO)_3P$, $CoCl_2$, and Et_3N.

Tris(triethyl phosphite)cobalt(I) Chloride. A 15-g sample of $[CoL_5]Cl$ was warmed in an evacuated flask with a 90° oil bath for *ca.* 0.5 hr. The weight loss was 5.8 g or 39%. The weight loss in five separate experiments was 40 ± 2%.

The volatile material was recovered from the cold trap and shown to be substantially triethyl phosphite by comparison with the infrared and ^1H nmr spectra of authentic material as well as by vpc. Both ir and vpc showed the presence of a few per cent ethanol.

After cooling to room temperature the brown oily residue crystallized. It was recrystallized from pentane at −35° to give 6.6 g of large brown crystals, mp 50-51°.

An attempt to record the ^1H nmr spectrum showed that the only signal was a broad peak *ca.* 390 Hz downfield from internal tetramethylsilane, but the silane was shifted relative to external silane. An approximate χ_m at 314°K was then obtained by the Evans[6] method, $\chi_m \cong 3630 \times 10^{-6}$ emu. A correction of -389×10^{-6} emu for the diamagnetism of the ligands gave $\chi_m{}' \cong 4019 \times 10^{-6}$ emu or $\mu_{eff} \cong 3.15$ BM. This is in good agreement with the values reported for tetrahedral cobalt(I) chloride with arylphosphine ligands.[7]

Pentakis(triethyl phosphite)cobalt(I) Tetraphenylborate from $[CoL_5]Cl$. A solution of 2.0 g of $[CoL_5]Cl$ in 20 ml of ethanol was mixed with a solution of 0.8 g of sodium tetraphenylborate in 10 ml of ethanol. A yellow precipitate formed immediately. It was collected and washed with ethanol to give 2.1 g of product. This was crystallized from methylene chloride-pentane-ether, mp 148-152° dec. It can also be crystallized from ether or from acetone at −35°.

From $[CoClL_3]$. A solution of 0.12 g of $[CoClL_3]$ in 0.5 ml of ethanol was prepared and 0.078 ml triethyl phosphite was added. The red color almost completely disappeared leaving a yellow amber solution. A solution of 75 mg of sodium tetraphenylborate in 0.5 ml of ethanol was added. A yellow precipitate formed immediately. The solid was collected, washed, and dried: 0.22 g; mp *ca.* 150° dec, which was undepressed by mixing with material prepared above. Also the ir (Nujol), ^1H nmr, and visible spectra matched those of material prepared as described above.

Bis(triethyl phosphite)cobalt(I) Tetraphenylborate. To a solution of 8 g of $[CoClL_3]$ in 25 ml of anhydrous ethanol was added a solution of 5 g of sodium tetraphenylborate in 50 ml of ethanol. There was no immediate formation of precipitate. After 6 hr at room temperature, brown solid had formed. The mixture was cooled to −35° and the solid (9.1 g) was collected. The ^1H nmr spectrum of the crude material had an apparent lopsided "quartet" at *ca.* 1.2 ppm, a complex multiplet at *ca.* 3.9 ppm, small broad peaks at 5.15 and 5.75 ppm, and peaks at *ca.* 7.1 and 7.6 ppm. The ratio of the area for the ethyl groups to that of the aromatic protons was 2.20. In two similar experiments with both relatively more and less precipitate per gram of starting material the alkyl area to aromatic area ratio was essentially the same. This result is in good accord with a ratio of three phosphite ligands to each tetraphenylborate unit in the crude product. Careful fractional crystallization of the crude prod-

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Table III. ^{1}H Nmr Spectra[a]

Compd	Solvent	CH_2	CH_3	C_6H_5	Area ratio
$[CoL_5]Cl$	D_3CCN	Broad quartet, δ 4.05, area 105	Triplet, δ 1.25, $J = 7$, area 160		
$[CoL_5][B(C_6H_5)_4]$	$DCCl_3$	Broad quartet, δ 4.0, area 134	Triplet, δ 1.22, $J = 7$, area 195	Mult δ 6.8–7.6, area 90	Alkyl/arom = 3.7
$[CoL_5][CoCl_3]$	D_3CCN	Broad quartet, δ 4.15, area 129	Triplet, δ 1.28, $J = 7$, area 195		
$[CoL_5][CoCl_4]$	D_3CCN	Broad quartet, δ 4.12, area 55	Triplet, δ 1.27, $J = 7$, area 80		
$[CoL_2B(C_6H_5)_4]$	$DCCl_3$	Symmetrical, 9-line pattern, δ 3.8, $J = 3.5$, area 110, identical patterns at 60 or 100 MHz	Triplet, δ 1.15, $J = 7$, area 175	Mult 5.15, area 16 Mult 5.75, area 16 Mult 6.8–7.6, area 156	Alkyl/arom = 1.5

[a] The chemical shifts, δ, are in ppm from internal tetramethylsilane. The apparent coupling constants, J, are in Hz.

uct from ether gave several grams of yellow crystalline product which was shown by its low-temperature ^{31}P nmr, ^{1}H nmr, and ir spectra to be identical with $[CoL_5][B(C_6H_5)_4]$ prepared directly from $[CoL_5]Cl$. Also isolated was 3.0 g of dark red crystalline $[CoL_2B(C_6H_5)_4]$, mp 131–134° dec. A superposition of the ^{1}H nmr spectra of $[CoL_5]B(C_6H_5)_4$ and $[CoL_2B(C_6H_5)_4]$ in a *ca.* 1:2 ratio reproduced the spectrum of the crude mixture.

Pentakis(triethyl phosphite)cobalt(I) Trichlorocobaltate. A nitrogen-sparged solution of 2.5 ml of water and 14 ml of triethyl phosphite in 300 ml of anhydrous ethanol was used to dissolve 3.9 g of $CoCl_2$, and a solution of 8 ml of triethylamine in 100 ml of ethanol was added. After standing overnight at room temperature, the mixture was filtered through Celite and the dark green filtrate was concentrated under vacuum to an oil. Two *ca.* 10-ml portions of benzene were added and then evaporated under vacuum. The residue was stirred with *ca.* 20 ml of benzene and the supernatant was decanted. The remaining benzene was removed under vacuum and the residue was crystallized from ether at *ca.* −35° to give 0.85 g of dark green crystals, mp 137–139° dec.

This procedure was not very reproducible. Material with the same melting point, appearance, and visible spectrum was obtained from $Co[P(OEt)_3]_5Cl$ and $CoCl_2$ under anhydrous conditions as described below.

A solution was prepared from 3.6 g of $[CoL_5]Cl$, 0.52 g of $CoCl_2$, and 40 ml of ethanol. The ethanol was removed under vacuum and the residue was crystallized twice by dissolving the solid in a solution of 2 ml of ethanol and 10 ml of ether, filtering, adding 100 ml of ether, and cooling the solution to −35°: 2.5 g of dark green crystals; mp 137–139° dec, which was undepressed on mixing with material obtained above.

Infrared Spectra (Nujol). The ir spectra of $[CoClL_3]$, $[CoL_5]Cl$, and $[CoL_5][CoCl_3]$ were virtually the same as that of triethyl phosphite ligand. The spectrum of $[CoL_5][B(C_6H_5)_4]$ was similar to a superposition of triethyl phosphite and sodium tetraphenylborate spectra except for a strong new band at *ca.* 730 cm^{-1} which is also present in $\{Co[P(OCH_3)_3]_5\}[B(C_6H_5)_4]$.[12] The spectrum of $[CoL_2B(C_6H_5)_4]$ is similar to that of $[CoL_5][B(C_6H_5)_4]$ down to *ca.* 850 cm^{-1}, although the former has a new medium intensity band at 1460 cm^{-1}. There are major differences in the 700–850-cm^{-1} regions of the spectra. The strong *ca.* 1250–1300-cm^{-1} P=O band was not evident in any of the spectra.

^{31}P Nmr Spectra. The ^{31}P nmr spectra of $[CoL_5]Cl$, $[CoL_5][CoCl_3]$, and $[CoL_5][B(C_6H_5)_4]$ all were single peaks at room temperature *ca.* 3-ppm downfield from external trimethyl phosphite. On cooling, the spectra broadened and then complex multiplets appeared at *ca.* −120 to −140° which were essentially the same in all these complexes. This pattern has been shown by Meakin and Jesson[4] to be consistent with a trigonal bipyramidal arrangement of the phosphite ligands. The ^{31}P nmr spectrum of $[CoL_2B(C_6H_5)_4]$ showed only one line *ca.* 4 ppm downfield from external trimethyl phosphite down to −140°.

Registry No. $[CoClL_4]$, 15488-42-3; $[CoL_5]Cl$, 51464-44-9; $[CoClL_3]$, 15488-43-4; $[CoL_5][CoCl_3]$, 51464-46-1; $[CoL_5][CoCl_4]$, 51464-47-2; $[CoL_5][B(C_6H_5)_4]$, 51464-48-3; $[CoL_2B(C_6H_5)_4]$, 51464-49-4; $P(OEt)_3$, 122-52-1; $Na[B(C_6H_5)_4]$, 143-66-8.

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(12) Sample supplied by J. P. Jesson.[13]

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