Table II. Positions and Intensities of Visible Bands in $CoCl_2 \cdot 2H_2O$

Position,[a] cm^{-1}	Oscillator strength[b] $f_{a'}$	f_b	f_c
16,400	2.0	7.5	6.5
19,800	26	3.5	37

[a] Centroid at 10 K. [b] Measured at 10 K in units of 10^{-6} (±10%). $f_{a'}$, f_b, and f_c refer to the appropriate polarizations with data taken from *a'b* and *bc* faces only.

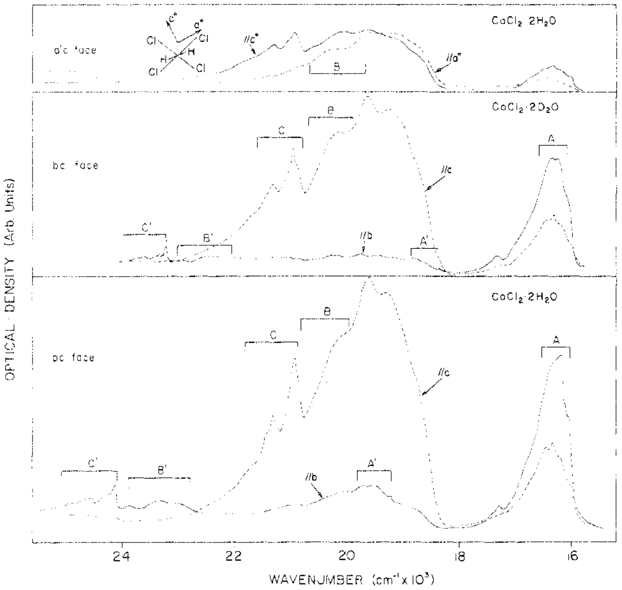

Figure 5. Polarized absorption spectra of $CoCl_2 \cdot 2H_2O$ and $CoCl_2 \cdot 2D_2O$ (10 K) showing details of the anomalous crystal field bands.

appreciable splitting from a consideration of the anomalous crystal field bands, given below.

Crystal Field Parameter. As the $^4B_{2g}$ state cannot be definitely located, we cannot assign a value to *Dq*. If we assume that it is near 14,500 cm^{-1} (see Figure 4), then its separation from 8,200 cm^{-1} (assignment I) gives $Dq = 630$ cm^{-1} while its separation from 6150 cm^{-1} (assignment II) gives $Dq = 835$ cm^{-1}. Using both of these values and a range of positive and/or negative *Ds* and *Dt* values no reasonable fit of the observed bands in the near-infrared and visible regions could be obtained. We conclude that the effect of the latter is so large that discussion of the assignments in terms of a quantitative tetragonal field theory is not warranted.

The Anomalous Bands. As with the spectrum of $CoCl_2 \cdot 6H_2O$ there are bands in the spectrum of $CoCl_2 \cdot 2H_2O$ which can be assigned to the symmetrical water vibration acting as a perturbing vibration which removes the center of symmetry. From a comparison of the various polarized spectra of $CoCl_2 \cdot 2H_2O$ and $CoCl_2 \cdot 2D_2O$ we can assign four bands in this way. The first is in the near-infrared region, near 12,000 cm^{-1} in the spectrum of $CoCl_2 \cdot 2H_2O$, mentioned above. The other three occur in the visible region and they are denoted A', B', and C' in Figure 5. The associated normal crystal field bands are shown in A, B, and C. In each case, for the hydrate, the anomalous bands are 3200 cm^{-1} to higher energy. For the deuterate the shift is about 2300 cm^{-1}.

In every case the anomalous bands occur only in *b* polarization; *i.e.*, they are polarized in the same direction as the symmetrical stretching mode (see Figure 1). The effectiveness of perturbation is greatest for B' and C' (see Figure 5). Now, the state represented by band C must be one or more doublet levels arising, most probably, from the ^2P term. Clearly, this requires spin–orbit mixing with the nearby quartet state (B–B' pair) and, from the similarity of the intensities in bands B' and C', the doublet and quartet states are very strongly mixed.

Although the role of the water vibration in promoting the electronic transition on the cobalt ion appears to be clearly established, the polarizations of the bands A' and B' are not consistent with the expected orbital symmetries of the states A and B. If A is assigned to $^4A_{2g}$ (^4A_g in C_{2h}), then B must come from $^4E_g{}^c$ (^4B_g in C_{2h}) and the a_u symmetrical water stretching mode coupled to each of these states should give rise to opposite polarizations for A' and B', which is contrary to observation.

The presence of more than one excited quartet state under the broad band is shown by the B–B' relationship and the change of polarization in the spectrum of the *a'c* face (see Figure 5). It is not surprising therefore that efforts to fit the band energies using tetragonal symmetry fail.

Registry No. $CoCl_2 \cdot 2D_2O$, 52918-71-5; $CoCl_2 \cdot 2H_2O$, 38722-37-1.

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Bimetallic Iron Carbonyl Derivatives of Bidentate Phosphine Ligands

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In preparing a number of bimetallic derivatives, the photochemical reaction of $Fe(CO)_5$ with a series of bidentate phosphine ligands $(C_6H_5)_2P(CH_2)_nP(C_6H_5)_2$, $n = 1$, 2, 3, and 4, was examined. For the $n = 2$, 3, and 4 phosphines, the course of the reaction is similar and a large scale preparation of the principle product $(C_6H_5)_2P(CH_2)_nP(C_6H_5)_2[Fe(CO)_4]_2$, $n = 2$, 3, and 4, was developed. For the $n = 2$ phosphine smaller scale preparations of the principal product had previously been reported.[1,2]

In the case of the $n = 1$ phosphine, the reaction is more complex and a second major product $(C_6H_5)_2PCH_2P(C_6H_5)_2Fe_2(CO)_7$ is produced. This product alternatively can be prepared photolytically either from $Fe_2(CO)_9$ and the $n = 1$ phosphine or from the $n = 1$ bimetallic derivative. The characterization data for this unique product are compatible with a structure formally derived from an isomer of $Fe_2(CO)_9$ containing only one bridging ligand and four terminal ligands in which the phosphine ligand replaces one terminal carbonyl on each iron atom and serves as a bridging ligand.[3] The length of the methylene bridge of the phosphine ligand is apparently important in the formation of this compound since the $n = 2$, 3, and 4 phosphines do not readily form analogous derivatives.

Table I. Elemental Analyses and Molecular Weight Results

Compd	C		H		P		Fe		O[a]		Mol wt	
	Calcd	Obsd	Calcd	Obsd	Calcd	Obsd	Calcd	Obsd	Calcd	Obsd	Calcd	Obsd
$(C_6H_5)_2P(CH_2)_2P(C_6H_5)_2[Fe(CO)_4]_2$ (I)	55.40	55.94	3.26	3.40	8.41	8.39	15.15	15.22	17.40	17.05	734	720
$(C_6H_5)_2P(CH_2)_3P(C_6H_5)_2[Fe(CO)_4]_2$ (II)	56.20	55.71	3.48	3.73	8.29	8.12	14.95	15.23	17.15	17.21	748	758
$(C_6H_5)_2P(CH_2)_4P(C_6H_5)_2[Fe(CO)_4]_2$ (III)	56.50	55.84	3.67	3.91	8.14	8.09	14.64	14.23	16.80	17.93	762	777
$(C_6H_5)_2PCH_2P(C_6H_5)_2[Fe(CO)_4]_2$ (IV)	55.07	55.04	3.10	3.17	8.58	8.66	15.46	15.39	17.72	17.74	720	718
$(C_6H_5)_2PCH_2P(C_6H_5)_2Fe(CO)_4$ (V)	63.07	62.17	4.02	4.35	11.22	10.93	10.11	10.17	11.57	12.38	552	550
$(C_6H_5)_2PCH_2P(C_6H_5)_2Fe(CO)_3$ VI)	64.02	63.40	4.21	4.52	11.87	11.98	10.68	10.78	9.24	9.32	524	b
$(C_6H_5)_2PCH_2P(C_6H_5)_2Fe_2(CO)_7$ (VII)	55.53	55.20	3.20	3.33	8.95	9.10	16.14	16.26	16.18	16.11	692	684

[a] By difference. [b] Not determined due to air sensitivity.

Experimental Section

The bidendate phosphines were purchased from the Pressure Chemical Co. and iron pentacarbonyl was purchased from Alfa Inorganics. Photochemical reactions were run in a Rayonet photochemical reactor equipped with a magnetic stirrer. Infrared spectra were obtained using a Perkin-Elmer 621 spectrophotometer and molecular weights were obtained using a Hitachi Perkin-Elmer osmometer. The benzene solvent in the osmometer studies was distilled under nitrogen from calcium hydride. The melting points are uncorrected. The pmr spectra were run on a Varian A-60 nmr and chemical shift data are in ppm downfield from tetramethylsilane. Chemical analyses were performed by Schwarzkopf Microanalytical Laboratories.

Preparation of $(C_6H_5)_2P(CH_2)_nP(C_6H_5)_2[Fe(CO)_4]_2$, n = 2–4. The initial preparation of all the derivatives is similar but the workups are slightly different. The following procedure can be scaled up at least fivefold. A solution consisting of benzene (40 ml), 5 mmol of the phosphine, and 70 mmol of $Fe(CO)_5$ (13.7 g, 9.4 ml) was irradiated (350 nm) with stirring under nitrogen for 20 hr. The reaction solution was filtered through Celite to remove solids (principally $Fe_2(CO)_9$) and the yellow filtrate evaporated.

$(C_6H_5)_2P(CH_2)_2P(C_6H_5)_2[Fe(CO)_4]_2$ (I). The residue of the reaction mixture was dissolved under nitrogen in hot 2-propanol (250 ml) and the solution filtered through Celite. Upon cooling the filtrate in an ice bath 2.06 g of yellow product (56% yield) crystallized. A second recrystallization yielded a product pure by thin layer chromatography (tlc) with a melting point 184–185°. The pmr spectrum ($CDCl_3$) of the derivative only contained resonances characteristic of the phosphine ligand, a phenyl multiplet (intensity 5) centered at 7.42 ppm, and a $(CH_2)_2$ multiplet (intensity 2) centered at 2.63 ppm.

$(C_6H_5)_2P(CH_2)_3P(C_6H_5)_2[Fe(CO)_4]_2$ (II). The residue from the reaction was an oil. The oil was filtered through a column of Silicar CC-7 using benzene as the eleuent. Removal of the solvent from the yellow fraction gave a 46% yield of a solid which was principally product. The solid could be recrystallized under nitrogen from 2-propanol. The yield of product (mp 147–148°) pure by tlc was 0.90 g (23%). The pmr spectrum ($CDCl_3$) of the product only contained resonances due to the phosphine ligand, a phenyl multiplet (intensity 10) at 7.41 ppm, and a $(CH_2)_3$ region triplet (intensity 2) at 2.45 (J = 8.0 Hz) and multiplet (intensity 1) at 1.80 ppm.

$(C_6H_5)_2P(CH_2)_4P(C_6H_5)_2[Fe(CO)_4]_2$ (III). The solid residue from the reaction mixture was crystallized under nitrogen from 2-propanol (250 ml). The yield of yellow product (mp 183–184°) pure by tlc was 2.62 g (71%). The pmr spectrum only contained resonances from the phosphine ligand, a phenyl multiplet (intensity 10) at 7.42 ppm, and two multiplets in the $(CH_2)_4$ region (intensity 2) at 2.38 ppm and (intensity 2) at 1.68 ppm.

The Reaction of $Fe(CO)_5$ and $(C_6H_5)_2PCH_2P(C_6H_5)_2$. The photochemical reaction was run with the same amounts as in the case of the other phosphines. However, a red oil resulted upon work-up. The oil was chromatographed on a Silicar CC-7 column (40 × 2.2 cm diameter) and a 68% yield of four products (based on phosphine) was obtained. Careful elution with hexane and then benzene:hexane mixtures down to a 75:25 ratio yields the four products in order of elution: $(C_6H_5)_2PCH_2P(C_6H_5)_2[Fe(CO)_4]_2$ (IV) (2.83 g, 25%, mp 140–142°); $(C_6H_5)_2PCH_2P(C_6H_5)_2Fe(CO)_4$ (V) (1.81 g, 16%, mp 171–173°); $(C_6H_5)_2PCH_2P(C_6H_5)_2Fe(CO)_3$ (VI) (1.29 g, 11%, mp 131–133°); and $(C_6H_5)_2PCH_2P(C_6H_5)Fe_2(CO)_7$ (VII) (1.80 g, 16%, mp 170–171°). The first three products were yellow and the last was red. Care must be taken to effectively separate the last two products and in some instances a second chromatographic separation using benzene:hexane (60:40) was required. All products can be obtained pure by tlc by recrystallization. The first three products can be recrystallized under nitrogen from hot 2-propanol while the red product can be recrystallized from heptane:toluene (60:40). The pmr spectra of three of the compounds were obtained in $CDCl_3$ solvent and the spectra contained only the resonance due to $(C_6H_5)_2PCH_2P(C_6H_5)_2$ ligand with the expected intensities. The chemical shifts (ppm) of the phenyl multiplet and methylene group are as follows: $(C_6H_5)_2PCH_2P(C_6H_5)_2[Fe(CO)_4]_2$ 7.28 (C_6H_5), 3.93 triplet (J = 9 Hz) (CH_2); $(C_6H_5)_2PCH_2P(C_6H_5)_2[Fe(CO)_4]_2$, 7.20 ($C_6H_5$), 3.32 doublet ($J$ = 9 Hz) (CH_2); $(C_6H_5)_2PCH_2Fe_2(CO)_7$, 7.22 ($C_6H_5$), 3.54 triplet ($J$ = 11 Hz) (CH_2). The pmr spectrum of $(C_6H_5)_2PCH_2P(C_6H_5)_2Fe(CO)_3$ was not obtained as decomposition occurred rapidly in the solution in the presence of air.

Discussion

The Experimental Section describes the preparation of the air sensitive bimetallic derivatives according to eq 1. These

$$2Fe(CO)_5 + (C_6H_5)_2P(CH_2)_nP(C_6H_5)_2 \xrightarrow[n = 1,2,3,4]{h\nu} 2CO + (C_6H_5)_2P(CH_2)_nP(C_6H_5)_2[Fe(CO)_4]_2 \quad (1)$$

procedures can be applied to the preparation of I, previously reported,[1,2] as well as to the preparation of the new derivatives II, III, and IV using the bis(diphenylphino)propane, -butane, and -methane ligands, respectively. In the reaction using bis(diphenylphosphino)methane, several other products in addition to III are formed in substantial yield necessitating chromatographic separation. Nevertheless, IV is still the major product.

Table I contains the results of elemental analyses and molecular weight determinations for the iron phosphine derivatives. Table II contains a list of the carbonyl absorptions in the infrared spectra of the derivatives. For compounds I–IV these data indicate an analogous bimetallic composition and structure with one $Fe(CO)_4$ group bonded to each phosphorus atom of the ligand. Derivatives I–IV exhibit four infrared absorptions in the carbonyl stretching region. This is one more absorption than expected for $LFe(CO)_4$ complexes of C_{3v} symmetry. Apparently the symmetry about the metal atom is reduced as had previously been noted for I and other complexes which contain bulky ligands.[4] The reduction in symmetry is apparently greater in complexes containing the bis(diphenylphosphino)methane ligand since the spectra of both IV and V have all bands well resolved. For complexes I–III, the additional band is not well resolved and appears as a slight shoulder.

A detailed examination of the reaction of $Fe(CO)_5$ with $(C_6H_5)_2PCH_2P(C_6H_5)_2$ yielded three minor products V–VII in addition to IV. In compound V, the phosphine utilizes only one phosphorus atom to bind to one $Fe(CO)_4$ group. Compound V and its analogs are probably intermediates in the formation of all the bimetallic derivatives of the phosphines (I–IV). In compound VI, the phosphine acts as a chelating ligand.[5] A similar compound is formed in the reaction of $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ with $Fe(CO)_5$.[4] The red product

Table II. The Carbonyl Stretching Frequencies of the Iron–Phosphine Complexes

Compd	CO stretch (cm^{-1}) in $CHCl_3$ solvent
$(C_6H_5)_2P(CH_2)_2P(C_6H_5)_2[Fe(CO)_4]_2$ (I)	2057 m, 1977 m, 1938 s, 1933 sh
$(C_6H_5)_2P(CH_2)_3P(C_6H_5)_2[Fe(CO)_4]_2$ (II)	2047 m, 1975 m, 1936 s, 1930 sh
$(C_6H_5)_2P(CH_3)_4P(C_6H_5)_3[Fe(CO)_4]_2$ (III)	2045 m, 1973 m, 1935 s, 1928 sh
$(C_6H_5)_2PCH_2P(C_6H_5)_2[Fe(CO)_4]_2$ (IV)	2053 m, 1980 m, 1946 s, 1938 s
$(C_6H_5)_2PCH_2P(C_6H_5)_2Fe(CO)_4$ (V)	2052 m, 1978 m, 1943 s, 1937 s
$(C_6H_5)_2PCH_2P(C_6H_5)_2Fe(CO)_3$ (VI)	1989 s, 1919 m, 1909 s
$(C_6H_5)_2PCH_2P(C_6H_5)_2Fe_2(CO)_7$ (VII)	2049 s, 1994 s, 1982 m, 1951 sh, 1940 m, 1923 s, 1754 m

VII is unique to the reaction with bis(diphenylphosphino)methane. The elemental analysis and molecular weight data are compatible with a composition of two iron atoms, seven carbonyls, and one phosphine ligand. The infrared spectrum of VII is quite complex and contains seven absorptions, six in the terminal and one on the bridging carbonyl region. The mechanism by which VII is formed in the reaction is not clear; however, separate infrared experiments indicate VII can be formed by two different routes. When $Fe_2(CO)_9$ is photolyzed with benzene solutions of $(C_6H_5)_2PCH_2P(C_6H_5)_2$ for 24 hr, VII is produced. The photolysis of IV in benzene solution for 12 hr also produces VII. Probably both of these routes are operative under the reaction conditions. Thermal reaction[2] of $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ with $Fe_2(CO)_9$ did not produce an analog of VII and photolyses of I in benzene over a 6-day period also does not yield an analog of VII.

The structure of VII is not definitively established by either its infrared or pmr spectrum. These spectra are compatible with a structure in which the phosphine is a chelating ligand replacing two terminal carbonyls on one iron atom of $Fe_2(CO)_9$ or its tautomer $(OC)_4Fe(\mu\text{-}CO)Fe(CO)_4$[6] or with a structure in which the phosphine replaces a terminal carbonyl on each iron atom of the tautomer and serves as a bridging ligand. The apparent inability of a better chelating phosphine $(C_6H_5)_2P(CH_2)_2P(C_6H_5)_2$ to form an analog of VII favors the latter structure.[3]

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Registry No. I, 14977-13-0; II, 52843-15-9; III, 52843-16-0; IV, 52843-17-1; V, 52843-12-6; VI, 52843-13-7; VII, 52843-14-8.

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Triarylstibine Complexes of Rhodium(I). II[1]

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Previous investigations have shown that although Rh(I) phosphine complexes are four coordinate, the analogous stibine complexes prefer to be five coordinate in the solid state.[1] Furthermore the reaction of $Rh_2Cl_2(CO)_4$ with PPh_3 resulted in the isolation of $Rh_2Cl_2(CO)_x(L)_{4-x}$ (x = 1–3) dependent on the P/Rh ratio,[2] but the analogous SbR_3 reactions gave no evidence for the existence of such dimeric complexes.[1] Recently[3] the reactions of $Rh_2Cl_2(CO)_x(C_2H_4)_{4-x}$ (x = 1–3) with PR_3 were reported as more suitable routes to complexes of the type $Rh_2Cl_2(CO)_x(L)_{4-x}$ (x = 1–3). As a continuation of our studies on Rh(I) stibine complexes, the study of $Rh_2Cl_2(CO)_x(C_2H_4)_{4-x}$ (x = 1–3) with SbR_3 was undertaken in an attempt to observe dimeric stibine complexes and further compare Rh(I) stibine and phosphine reactions.

Experimental Section

All reactions were carried out under an atmosphere of argon. Solvents were dried and deaerated prior to use. The ligand $(p\text{-}CH_3C_6H_4)_3Sb$[4] and the complexes $Rh_2Cl_2(CO)_4$,[5] $Rh_2Cl_2(C_2H_4)_4$,[6] and $Rh_2Cl_2(CO)_x(C_2H_4)_{4-x}$[3] ($x$ = 1–3) were prepared by literature methods.

Infrared Solution Studies. The reaction of $Rh_2Cl_2(CO)_x(C_2H_4)_{4-x}$ (x = 1–3) with SbR_3 (R = Ph or $p\text{-}CH_3C_6H_4$) was monitored using a Perkin-Elmer 621 ir spectrometer in the 2100–1900-cm^{-1} region during the addition of aliquots of SbR_3 in $CHCl_3$ to 0.05×10^{-3} *M* solutions of the dimers in $CHCl_3$. A portion of the solution was withdrawn by syringe 1 min after addition of the aliquot and the solution spectrum recorded, the portion was then returned and another aliquot added. Figures 1 and 2 include a grating and scale change at 1980 cm^{-1}.

Results and Discussion

As described previously,[3] reaction of 1 mol of $Rh_2Cl_2(CO)_4$ (Y_0) with $^1/_3$, 1, or 3 mol of $Rh_2Cl_2(C_2H_4)_4$ (Y_4) resulted in high concentrations of $Rh_2Cl_2(CO)_3(C_2H_4)$ (Y_1), $Rh_2Cl_2(CO)_2(C_2H_4)_2$ (Y_2), or $Rh_2Cl_2(CO)(C_2H_4)_3$ (Y_3), respectively.

Ethylene was found to be readily displaced from Y_1, Y_2, or Y_3 by phosphines of various basicities, to give phosphine-substituted complexes.[3] The following ir absorptions were observed when the reaction of Y_1, Y_2, or Y_3 with SbR_3 was monitored in $CHCl_3$ solution.

Reaction of $Rh_2Cl_2(CO)_3(C_2H_4)$ (Y_1) with SbR_3 (Figure 1). At Sb/Rh = 0, absorptions due to Y_1 were observed at 2098 and 2038 cm^{-1}. At Sb/Rh = 0.5, absorptions at 2095, 2060, and 2015 cm^{-1} were observed in addition to the absorptions of Y_1. At Sb/Rh = 1.6, absorptions due to Y_1 were absent and absorptions at 2090, 2060, 2015, and 1966 cm^{-1} were present. At an Sb/Rh = 2.5, only the absorption at 1966 cm^{-1} remained.

Reaction of $Rh_2Cl_2(CO)_2(C_2H_4)_2$ (Y_2) with SbR_3 (Figure 2). At Sb/Rh = 0, absorptions due to Y_2 were observed at 2028 and 2031 cm^{-1}. At Sb/Rh = 0.8, absorptions at 2060, 1975, and 1966 cm^{-1} were observed in addition to the absorptions due to Y_2. At Sb/Rh = 1.65, absorptions due to Y_2 were absent while absorptions were present at 2060 and 1966 cm^{-1}. Additional SbR_3 caused the 2060-cm^{-1} absorption to diminish until at Sb/Rh = 2.5 only the absorption at 1966 cm^{-1} remained.

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