difference being the reason for the stereospecificity of the PDTA ligand). Therefore a strong driving force exists for the return to conformation **1**. The rate of return, however, is very slow due to the kinetic inertness of the Rh(III) complexes.

At present, the details of the photochemical processes and the explanation of the photostationary state must await further investigation. It is interesting to note, however, that the photoequilibrium is established in less than 5 min in spite of the inertness of Rh(III) compounds.

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Tungsten Carbonyl Complexes Containing Positively Charged Phosphorus Ligands

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Monoquaternized diamines as ligands have been investigated rather extensively by Quagliano and coworkers.[1] Few studies of monoquaternized diphosphines as ligands have been reported and most of these have been of complexes of transition metal halides which are usually insoluble and were studied in the solid state.[2–6] Only one carbonyl complex containing a monoquaternized diphosphine has been reported.[5]

Since CO stretching frequencies of metal carbonyls are especially sensitive to changes in electronic properties of coordinated ligands, small electronic changes in ligand-donating properties are detectable with ir spectroscopy. For example, it has been shown that the E modes of the carbonyl stretching frequencies of the complexes $(CO)_5WP(C_4H_9)_3$ and $(CO)_5WP(C_6H_5)_3$ are 1934 and 1942 cm^{-1}, respectively.[7] In this study a comparison of CO stretching frequencies and approximate force constants of $(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2$ and $(CO)_5WP(C_6H_5)_2CH_2P(C_6H_5)_2$ with $[(CO)_5WP(C_6H_5)_2CH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][PF_6^-]$ and $[(CO)_5WP(C_6H_5)_2CH_2P^+(C_6H_5)_2CH_3][I^-]$ is made.[8] These complexes are all sufficiently soluble in polar organic solvents to be studied in solution with ir and nmr spectroscopy.

Experimental Section

Proton nmr spectra were recorded with a Varian T-60 spectrometer. All nmr spectra were obtained from saturated deuteriochloroform solutions unless otherwise indicated. TMS was used as a reference in all measurements.

Infrared spectra in the carbonyl region were recorded with a Perkin-Elmer 337 infrared spectrometer. These spectra were expanded with an E.H. Sargent SR recorder and are considered to be accurate to ±2 cm^{-1}. Polystyrene was used as a frequency standard and chloroform was used as a solvent for each measurement.

Microanalyses were performed by Galbraith Laboratories, Knoxville, Tenn.

Melting points were taken with an Arthur H. Thomas Unimelt apparatus and are reported uncorrected.

Preparation of Ligands. $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ and $(C_6H_5)_2PCH_2P(C_6H_5)_2$.—These were prepared as previously described by Chatt and Hart.[9]

$[(C_6H_5)_2PCH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][Br^-]$.—This ligand was prepared by the method of Quagliano.[1]

$[(C_6H_5)_2PCH_2P^+(C_6H_5)_2CH_3][I^-]$.—To a solution of $(C_6H_5)_2PCH_2P(C_6H_5)_2$ (1.0 g, 0.0026 mol) in benzene (25 ml) was added CH_3I (0.37, 0.0026 mol). The product, a white solid, separated after several hours. The yield was essentially stoichiometric. The pmr spectrum consisted of, in addition to phenyl proton resonance, a doublet ($^2J_{P^+H}$ = 13.3 Hz) assigned to phosphonium–methyl proton coupling and a doublet ($^2J_{P^+H}$ = 15.0 Hz) assigned to phosphonium–methylene proton coupling centered at 2.73 and 4.10 ppm, respectively. The trivalent phosphorus–methylene proton coupling was too small to be resolved.

Preparation of Neutral Complexes. $(CO)_5WNH_2C_6H_5$.—This complex was prepared as previously described by Angelici and Malone.[10]

$(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2$ and $(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2W(CO)_5$.—To a solution of $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ (3.0 g, 0.0075 mol) in benzene (100 ml) was added $(CO)_5WNH_2C_6H_5$ (1.0 g, 0.0023 mol). The solution was allowed to stand for 12 hr at room temperature and was taken to a thick oil with a rotary evaporator. The oil was dissolved in dichloromethane and an equal volume of methanol was added. The solution was cooled to 5° and white crystals slowly formed. These crystals, $(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2$, were recrystallized from ethanol (yield 1.8 g). The pmr spectrum consisted of a complicated methylene region extending from 2.8 to 3.8 ppm and phenyl proton absorption. When $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ (6.8 g, 0.017 mol) in 100 ml of benzene was treated with $(CO)_5WNH_2C_6H_5$ (6.0 g, 0.014 mol), only $(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2W(CO)_5$ was recovered. The pmr spectrum consisted of a broad singlet at 2.30 ppm assigned to the methylene protons and the phenyl proton absorption.

$(CO)_5WP(C_6H_5)_2CH_2P(C_6H_5)_2$.—A solution of $(C_6H_5)_2PCH_2P(C_6H_5)_2$ (1.2 g, 0.003 mol) in acetone (25 ml) was mixed with a solution of $(CO)_5WNH_2C_6H_5$ (1.25 g) in benzene (50 ml). After 24 hr, the benzene–acetone solvent was removed with a rotary evaporator. The resulting oil was dissolved in equal volumes of dichloromethane and methanol. The solution was cooled to 5°, and white crystals slowly formed. Recrystallization from methanol gave the product (1.5 g). The pmr spectrum, in addition to the phenyl proton resonance, was found to be a doublet of doublets, centered at 3.29 ppm. The signal was first split by phosphorus coordinated to tungsten into a doublet ($^2J_{PH}$ = 8.60 Hz) and each member was further split into doublets ($^2J_{PH}$ = 2.40 Hz) by coupling with uncoordinated phosphorus.

Preparation of Positively Charged Complexes. $[(CO)_5WP(C_6H_5)_2CH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][Br^-]$.—To a solution of $(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2$ (1.0, 0.0014 mol) in benzene (50 ml) was added $C_6H_5CH_2Br$ (0.237 g, 0.0014 mol). The white solid which separated after the solution had been stirred for several hours was washed with benzene to give 1.2 g of product. Attempts at recrystallization were unsuccessful. The same complex was also prepared in much lower yields by the direct interaction of $(CO)_5WNH_2C_6H_5$ and $[(C_6H_5)_2PCH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][Br^-]$. The pmr spectrum of the complex consisted of phenyl proton resonance, a broad methylene region extending from 1.8 to 3.2 ppm, and a doublet ($^2J_{PH}$ = 15.4 Hz), assigned to phosphorus–benzyl proton coupling, at 5.42 ppm.

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TABLE I

MELTING POINTS AND ANALYTICAL DATA

Compound	Mp, °C	Analytical data, % — Calcd C	Calcd H	Calcd P	Calcd X[a]	Found C	Found H	Found P	Found X[a]
$(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2$	116–117	51.91	3.37	8.64		51.88	3.21	8.55	
$(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2W(CO)_5$	205–206 dec	41.33	2.31	5.92		41.22	2.27	5.80	
$[(CO)_5WP(C_6H_5)_2CH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][PF_6^-]$	175–185 dec	47.62	3.26	9.69	11.89	47.37	3.29	9.71	11.59
$(CO)_5WP(C_6H_5)_2CH_2P(C_6H_5)_2$	188–189 dec	50.60	3.17	8.87		50.87	3.13	8.74	
$[(CO)_5WP(C_6H_5)_2CH_2P^+(C_6H_5)_2CH_3][I^-]$	145–150 dec	44.05	2.98	7.33	15.01	44.32	3.12	7.11	15.30

[a] Halogen.

TABLE II

INFRARED CARBONYL STRETCHING FREQUENCIES AND FORCE CONSTANTS OF MONOSUBSTITUTED TUNGSTEN PENTACARBONYL COMPLEXES

Complex	Freq, cm^{-1} — B_1	$A_1^{(2)}$	$A_1^{(1)}$	E	Constants, mdyn cm^{-1} — k_i	k_1	k_2
$(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)$	1986	2074	. . .	1940	0.32	15.50	15.84[a]
$(CO)_5WP(C_6H_5)_2CH_2CH_2P(C_6H_5)_2W(CO)_5$	1983	2075	. . .	1942	0.32	15.53	15.87[a]
$[(CO)_5WP(C_6H_5)_2CH_2CH_2P^+(C_6H_5)_2(CH_2C_6H_5)][PF_6^-]$	1990	2076	. . .	1938	0.34	15.44	15.81[a]
$(CO)_5WP(C_6H_5)_2P(C_6H_5)_2$	1981	2072	. . .	1939	0.32	15.48	15.82[a]
$[(CO)_5WP(C_6H_5)_2CH_2P^+(C_6H_5)_2CH_3][I^-]$	1989	2078	1952	1938	0.34	15.63	15.84
$(CO)_5WP(C_6H_5)_3$	1981	2075	. . .	1942	0.32	15.53	15.87[a]

[a] These *k* values were calculated by estimating the $A_1^{(1)}$ mode to be 5 cm^{-1} larger than the E mode.

$[(CO)_5WP(C_6H_5)_2CH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][PF_6^-]$.—To a solution of NH_4PF_6 (0.3 g, 0.0018 mol) in water (50 ml) was added $[(CO)_5WP(C_6H_5)_2CH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][Br^-]$ (0.8 g, 0.0009 mol). The white product which formed immediately was washed with water and dried under vacuum to give the product (0.8 g). The pmr spectrum was obtained from an acetone solution and consisted of phenyl proton resonance, a doublet ($^2J_{PH}$ = 15.2 Hz), assigned to phosphorus–benzyl proton coupling at 4.70 ppm, and a broad methylene region.

$[(CO)_5WP(C_6H_5)_2CH_2P^+(C_6H_5)_2CH_3][I^-]$.—This complex was best prepared by dissolving $[(C_6H_5)_2PCH_2P^+(C_6H_5)_2CH_3][I^-]$ (1.37 g, 0.0026 mol) in chloroform (25 ml) and treating the solution with $(CO)_5WNH_2C_6H_5$ (1.08 g, 0.0026 mol). The solution was allowed to stand for 12 hr at room temperature and was then taken to a thick oil with a rotary evaporator. Extraction of the oil with benzene left the desired white solid. The pmr spectrum consisted of a doublet ($^2J_{PH}$ = 13.4 Hz) at 2.50 ppm assigned to phosphorus–methyl proton coupling and a doublet of doublets arising from the methylene protons centered at 4.82 ppm. The methylene signal was split by phosphorus coordinated to tungsten into a doublet ($^2J_{PH}$ = 18.6 Hz) and each member was further split into doublets ($^2J_{P^+H}$ = 7.60 Hz) by coupling with the positively charged phosphorus atom.

Results and Discussion

Good yields of complexes containing positively charged phosphorus ligands were obtained by displacing aniline from $(CO)_5WC_6H_5NH_2$. These reactions occur at room temperature and thus decomposition complications are greatly minimized. When a solution of $(CO)_5WC_6H_5NH_2$ is treated with $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ in 1:1 mole ratio, the dimetallic complex $(CO)_5W(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2W(CO)_5$ can be isolated in excellent yield. If the ratio of ligand to complex is 3:1, the predominant product is $(CO)_5W(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$. This complex is easily quaternized to give the desired $[(CO)_5W(C_6H_5)_2PCH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][Br^-]$. The bromide was converted to the hexafluorophosphate in order to achieve purification. The aniline complex will react directly with $[(C_6H_5)_2PCH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][Br^-]$ to give the positively charged complex, but low yields were realized by this method because of a side reaction which is now under investigation.

There is no tendency for the dimetallic complex $(CO)_5W(C_6H_5)_2PCH_2P(C_6H_5)_2W(CO)_5$ to form from the reaction of $(CO)_5WC_6H_5NH_2$ and $(C_6H_5)_2PCH_2P(C_6H_5)_2$ under the conditions employed in this study. The only product isolated from this reaction was $(CO)_5WP(C_6H_5)_2CH_2P(C_6H_5)_2$. Attempts to quaternize the dangling end with benzyl bromide were unsuccessful. Quaternization in low yields was accomplished with iodomethane to give $[(CO)_5WP(C_6H_5)_2CH_2P^+(C_6H_5)_2CH_3][I^-]$. This complex was better prepared from the direct reaction of $[(C_6H_5)_2PCH_2P^+(C_6H_5)_2CH_3][I^-]$ and $(CO)_5WC_6H_5NH_2$. Melting points and analytical data for the complexes are recorded in Table I.

The infrared spectra of the carbonyl region were characterized by the $A_1^{(2)}$, $A_1^{(1)}$, and E modes expected for complexes of C_{4v} symmetry. The forbidden B_1 mode was observed for all of the complexes reported here. The assignments shown in Table II are based upon previously reported work.[10–12] For most of the complexes it was not possible to resolve the $A_1^{(1)}$ mode and for those it was estimated that the band is found 5 cm^{-1} higher than the E mode. This procedure has been used previously for spectra for which resolution of the $A_1^{(1)}$ mode was not possible.[10,11] The method of Cotton and Kraihanzel was used to approximate the force constants k_i, k_1, and k_2.[12]

The complexes $(CO)_5W(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$, $(CO)_5W(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2W(CO)_5$, $[(CO)_5W(C_6H_5)_2PCH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][PF_6^-]$, and $(CO)_5W(C_6H_5)_2PCH_2P(C_6H_5)_2$ gave, within experimental error, identical carbonyl stretching frequency data. Apparently the positive charge is too far removed from the metal-bonded phosphorus atom to have any effect upon the CO stretching frequencies. Previously a comparison of infrared carbonyl data for $(CO)_5WP(OCH_2)_3P$ and $[(CO)_5WP(OCH_2)_3P^+CH_3][BF_4^-]$ also showed that the positive charge had no effect upon the stretching frequencies.[5]

When the positive charge is moved to the position β to the metal-bonded phosphorus in $[(CO)_5W(C_6H_5)_2PCH_2P^+(C_6H_5)_2CH_3][I^-]$, the $A_1^{(1)}$ mode appears as a distinct shoulder. These results may be interpreted to mean that $[(C_6H_5)_2PCH_2P^+(C_6H_5)_2CH_3][I^-]$ donates slightly less negative charge to tungsten or is a better

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π-bonding ligand than is $[(C_6H_5)_2PCH_2CH_2P^+(C_6H_5)_2CH_2C_6H_5][PF_6^-]$ or $(C_6H_5)_2PCH_2P(C_6H_5)_2$ but since the $A_1^{(1)}$ mode is not resolvable in complexes of the latter ligands, the interpretation is speculative. Attempts to put a positive charge in a position α to the bonding phosphorus atom by quaternizing such precursors as $(CO)_5WP(C_6H_5)_2N(C_6H_5)_2$ and $(CO)_5WP(C_6H_5)_2P(C_6H_5)_2$ were unsuccessful.

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Fluoroperoxytrifluoromethane, CF_3OOF. Preparation from Trifluoromethyl Hydroperoxide and Fluorine in the Presence of Cesium Fluoride

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Fluoroperoxytrifluoromethane, CF_3OOF, was first reported as a product in the fluorination of sodium trifluoroacetate.[1] It was later postulated to be an intermediate in the preparation of CF_3OOOCF_3 by the reaction of OF_2 and COF_2 in the presence of CsF[2] and has subsequently been prepared by this method.[3]

Recently we have obtained a number of new compounds containing the CF_3OO group by reactions involving CF_3OOH.[4] In this paper a convenient preparation of CF_3OOF from the reaction of CF_3OOH and F_2 in the presence of CsF is described and its physical properties are reported. The chemical properties of CF_3OOF are under investigation and will be the subject of a future paper.

Experimental Section

General Information.—All volatile compounds were handled in a stainless steel[5] or Pyrex glass vacuum system fitted with stainless steel and glass–Teflon valves. Connections to the vacuum system were made by means of Swagelok fittings or glass joints lubricated with Kel-F90 grease. Pressures were measured using a precision Heise Bourdon tube gauge in the metal system and Wallace and Tiernan differential pressure gauge in the glass line. Amounts of volatile materials were determined by *PVT* measurements assuming ideal gas behavior.

Infrared spectra were taken on a Beckman IR-10 using a 10-cm glass cell with silver chloride windows. Fluorine nmr spectra were recorded on a Varian A-56/60 at 15° using 80 mol % $CFCl_3$ as an internal reference. In order to observe the large negative chemical shifts of the OOF group, a Krohn–Hite oscillator operated at 72.5 kc was substituted for the frequency offset oscillator normally used for fluorine operation.

Molecular weights were determined by vapor density measurements and were accurate to 0.5% when taken on pure CF_3OF. The vapor pressure as a function of temperature was obtained in the usual way[6] and a least-squares fit of the data was used to obtain the vapor pressure equation. Liquid density was obtained using a calibrated dilatometer.

Reagents.—Fluorine was passed through a NaF scrubber before using. Cesium fluoride was pretreated with several atmospheres of F_2 at 250° and was pulverized in the usual way.[7] Trifluoromethyl hydroperoxide was prepared by a modification of the procedure reported by Talbott[4,8] and was checked for purity by means of its infrared spectrum and molecular weight.

Reaction of CF_3OOH with F_2.—The reaction of CF_3OOH with fluorine was carried out under a variety of conditions in attempting to optimize the yield of CF_3OOF. The procedure used involved condensing CF_3OOH into a 75-ml stainless steel reactor onto 10 g of CsF at −196° and adding the desired amount of F_2. The vessel was then warmed to the desired reaction temperature by placing it in a $CFCl_3$ or CF_2Cl_2 cold bath. After reaction had proceeded for the appropriate time, the vessel was cooled to −196° and the F_2 and O_2 were removed. The remaining material was then transferred to a glass sample tube and separated by pumping through traps at −111, −140, −160, and −196° as the sample tube warmed from −196° in an empty 1-pt dewar at 22°. The −111° trap retained CF_3OOCF_2OF and $(CF_3OO)_2CFOF$;[8] the CF_3OOOCF_3[2,9] and CF_3OOCF_3[10] were retained in the −140° trap; CF_3OOF collected at −160° and CF_3OF[6] at −196°. Small amounts of CF_3OOF passed through the −160° trap and a small amount of CF_3OOCF_3 was often observed in the −160° trap. This was removed by a second fractionation through the −140° trap. Known compounds were identified by ^{19}F nmr, infrared spectra, and molecular weight (molecular weights found were within 2% of the calculated values). The CsF was replaced every third run and was treated with several atmospheres of F_2 at 22° after each run. Essentially no volatile products were observed on treating with F_2. The data for the reactions are summarized in Table I and the physical

TABLE I

REACTIONS OF CF_3OOH WITH F_2

Amt of reactants,[a] mmol		Conditions		
CF_3OOH	F_2	Time, hr	Temp, °C	Products (mmol)[b]
1.8	1.8[c]	3	−111 to −100	CF_3OOH (1.8), F_2 (1.8), trace of COF_2–HF
0.43	0.43[d]	20	−78	CF_3OF (0.14), $CF_3O_3CF_3$ (0.14), O_2, F_2
6.0	6.0	6	−196 to −15	CF_3OF (1.0), CF_3OOF (1.0), $CF_3O_2CF_3$ (1.0), CF_3OOCF_2OF–$(CF_3OO)_2CFOF$ (0.7), O_2, F_2
6.0	6.0	1	−15	CF_3OF (1.0), CF_3OOF (0.7), $CF_3O_2CF_3$ (0.7), CF_3OOCF_2OF–$(CF_3OO)_2CFOF$ (1.4), O_2, F_2
4.6	4.6	18	−78	CF_3OF (1.8), CF_3OOF (0.6), $CF_3O_2CF_3$ (0.6), CF_3OOCF_2OF–$(CF_3OO)_2CFOF$ (0.3), O_2, F_2
6.0	6.0	14	−111 to −78	CF_3OF (0.5), CF_3OOF (1.8), COF_2 (0.6), $CF_3O_2CF_3$ (1.2), CF_3OOCF_2OF–$(CF_3OO)_2CFOF$ (0.1), O_2, F_2
6.0	18.0	2 5	−196 to −78 −78	CF_3OF (1.5), CF_3OOF (1.5), $CF_3O_2CF_3$ (0.6), CF_3OOCF_2OF–$(CF_3OO)_2CFOF$ (0.9), O_2, F_2
6.0	3.0	19	−78	CF_3OOF (2.0), COF_2 (0.17), CF_3OOH–$(CF_3OO)_2CO$ (1.8), O_2 (1.2)

[a] Reactor contained 10 g of CsF except where noted. [b] O_2 and F_2 were not measured quantitatively. Small amounts of CF_4 may also have been present. [c] No alkali metal fluoride. [d] Ten grams of dried NaF.

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