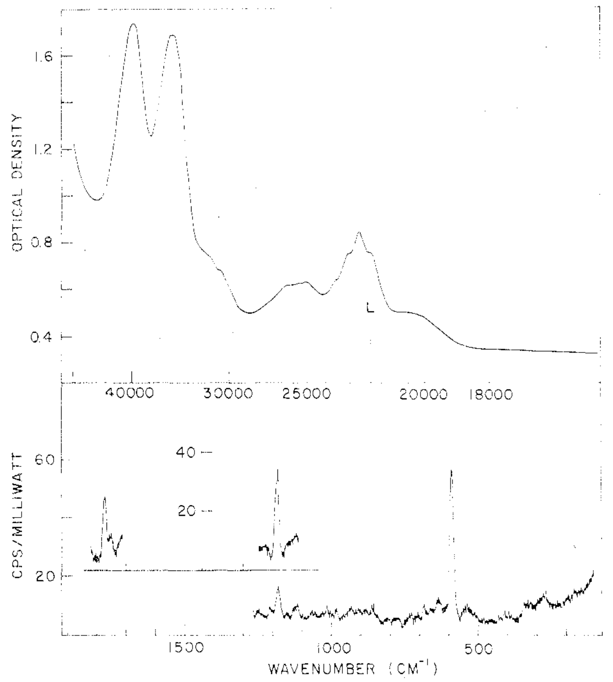

Figure 2. Top panel: absorption spectrum of argon matrix at 10 K deposited at rate of 0.01 mol/h of argon and containing an equivalent of about 10^{-6} mol of $(SN)_x$ of vapor species formed by heating $(SN)_x$ to 140 °C; $M/A \approx 3000$. Bottom panel: Raman spectrum at 10 K of argon matrix deposited at rate of 0.01 mol/h of argon and with $M/A \approx 1000$ and $A \approx 10^{-5}$ molar equiv of $(SN)_4$. Spectrum excited by 10-mW power in 22 000-cm^{-1} laser line (L in top panel) and recorded at scan rate of 100 cm^{-1}/min, 10 cm^{-1} spectral slit width, and 1-s rise time.

moreover that there is more than one unknown "X", i.e., more than one molecular entity or more than one kind of matrix trapping site. A possibility is that diffusion of molecules in the matrix at 40 K produces more X species at the expense of S_2N_2 and S_4N_4 molecules. Similar annealing of matrices containing only S_2N_2 and/or S_4N_4 molecules does not produce any of the "X" absorptions but only new absorptions which we attribute to weakly bound "aggregates" of these molecules, i.e., $(S_2N_2)_2$, $(S_2N_2)_3$, etc.

Qualitatively the same spectral changes resulting from annealing a dilute matrix can be achieved by depositing a more concentrated matrix. This is illustrated in Figure 1 by the spectrum of the dark orange matrix with an M/A of about 100. Finally, Figure 1 also contains the spectrum of a bright red solid film of the condensed vapors from $(SN)_x$. The dominant infrared absorptions of the film are in the frequency ranges of the X bands of the matrices.

In the uv–visible spectrum of a dilute matrix (Figure 2) the absorption maximizing at about 40 000 cm^{-1} is due to S_2N_2 and S_4N_4. The strong band at 35 000 cm^{-1} and the broad absorption from 28 000 to 19 000 cm^{-1} belong to the "X" substances. When the matrix is irradiated with a low-power level of the 22 000-cm^{-1} laser line (near the absorption maximum at about 22 500 cm^{-1}), a resonance Raman effect is observed with the appearance of a vibrational fundamental at 593 cm^{-1} and the first and second overtones at 1185 and 1773 cm^{-1} (Figure 2). However exposure of the matrix to higher power or prolonged irradiation by this blue line causes the resonance Raman effect to vanish and the matrix to turn grayish. (The red laser line at 15 454 cm^{-1} on the other hand does not degrade the matrix even at a power of 200 mW and yields a Raman line at 593 cm^{-1}, one due to S_2N_2 at 503 cm^{-1}, and a very broad feature centered at 950 cm^{-1}.) The 593- and 503-cm^{-1} lines have half-widths of less than 10 cm^{-1}, whereas the 950-cm^{-1} band is about 60 cm^{-1} wide.

Silver wool exposed to S_4N_4 vapor and presumably thus acquiring a coating of Ag_2S provides an effective surface for converting S_4N_4 to S_2N_2.[8] An experiment of passing the vapors from $(SN)_x$ through such silver wool plugs markedly increased the S_2N_2 absorptions, decreased the S_4N_4 bands, and completely eliminated the absorption by the unknown species X.

Our preliminary results show that under our conditions the volatilization of $(SN)_x$ at 140–160 °C produces some S_2N_2 molecules and possibly a trace of SN but mostly the well-known cyclic S_4N_4 molecule and more than one unidentified species. The unknowns are completely converted to S_2N_2 by the silver wool treatment. One or more of them are highly colored and give rise to broad bands in both the infrared and Raman spectra of the argon matrices. We might speculate that nonrigid, noncyclic biradical isomers of S_2N_2, S_4N_4, and possibly S_6N_6 could have these properties. When the blood red film of condensed vapor is warmed from 10 K, the infrared spectrum shows some slight continuous changes in band intensities, but quite suddenly at about 200 K the film becomes blue to transmission and its infrared spectrum is then characteristic of polymeric films. This all suggests that one or more of the unknown materials play a key role in the polymerization process. Further efforts in identifying the unknowns will be pursued.

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Registry No. S_2N_2, 25474-92-4; S_4N_4, 28950-34-7; SN, 12033-56-6; $(SN)_x$, 55892-53-0.

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Direct Synthesis of Mono- and Disubstituted Phosphorus Ligand Derivatives of Dodecacarbonyltetrairidium

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Substitution of carbonyl ligands in $Ir_4(CO)_{12}$ by phosphine and phosphite ligands requires elevated temperatures (ca. 110 °C).[1-6] The only complexes observed are the final substitution products $Ir_4(CO)_8L_4$ or $Ir_4(CO)_9L_3$, depending on the bulk of the entering ligand. Drakesmith and Whyman[3] observed

Table I. Physical Data

L	Mp, °C	ν_{CO}(in cyclohexane), cm^{-1}
		$Ir_4(CO)_{11}L$
PPh_3	239–245 dec	2087 m, 2069 w, 2054 vs, 2033 m, 2023 s, 2015 m, 1888 vw, 1854 m, 1831 m
PMe_2Ph	153–154	2087 m, 2069 m, 2055 s, 2030 m, 2022 s, 2014 m, 1853 m, 1828 m
P(*p*-tol)$_3$	205–208	2086 m, 2054 vs, 2033 m, 2021 s, 2014 m, 1888 vw, 1853 m, 1830 m
PEt_3	125–126	2088 m, 2069 w, 2050 vs, 2032 m, 2021 s, 2013 m, 1885 vw, 1851 m, 1825 m
$P(OMe)_3$	147–148	2095 m, 2068 sh, 2059 vs, 2037 s, 2026 m, 2016 m, 1852 m, 1833 m
$P(OPh)_3$^{a}	162–164	2096 m, 2059 vs, 2039 s, 2018 m, 1839 m, br
		$Ir_4(CO)_{10}L_2$
PPh_3	251–253 dec	2070 s, 2043 s, 2010 s, br, 1835 m, 1795 m
PMe_2Ph	124–125	2066 s, 2040 s, 2002 s, br, 1979 sh, 1830 m, 1795 m
P(*p*-tol)$_3$	190–195 dec	2064 s, 2038 s, 2006 s, br, 1835 m, 1796 m
PEt_3	120–121	2064 s, 2036 s, 2012 m, 2003 s, br, 1984 sh, 1823 m, 1786 m
$P(OMe)_3$	140–143	2074 s, 2049 s, 2016 s, 2012 s, 1834 m, 1811 m
$P(OPh)_3$	143–144	2076 s, 2051 s, 2026 s, 2012 sh, 1881 vw, 1844 m, 1826 m
$^1/_2$ diphos	228–235 dec	2071 s, 2042 s, 2014 s, br, 1988 m, 1874 w, 1838 m, 1789 m

[a] Spectrum obtained in chloroform due to low solubility in cyclohexane.

the formation of $Ir_4(CO)_{11}P(i\text{-}Pr)_3$ as a minor product from the high-temperature, high-pressure (150 °C, 80–100 atm of CO) carbonylation of $Ir_4(CO)_9(P(i\text{-}Pr)_3)_3$. Karel and Norton[6] utilized this method to prepare $Ir_4(CO)_{11}(PPh_3)$ and $Ir_4(CO)_{10}(PPh_3)_2$ from $Ir_4(CO)_9(PPh_3)_3$. However, the complexes $Ir_4(CO)_8L_4$ (L = PEt_3, P(*n*-Pr)$_3$, and P(*n*-Bu)$_3$) are much more resistant to carbonylation, with $Ir_4(CO)_{10}L_2$ being formed only under very severe conditions (200 °C, 400–450 atm of CO).[3] $Ir_4(CO)_{10}(PPh_3)_2$ has also been isolated from the addition of triphenylphosphine to a solution of $HIr_4(CO)_{11}^-$ ($Ir_4(CO)_{12}$, K_2CO_3, and CO in ethanol)[7,8] as well as from the reaction of $Ir_2(CO)_6(PPh_3)_2$ with perchloric acid.[9]

We recently reported a preparation of $Ir_4(CO)_{12}$ from the carbonylation of $Ir(CO)_2$(*p*-toluidine)Cl in the presence of zinc.[10] We have now found that addition of a phosphine or phosphite ligand to the reaction mixture allows direct synthesis of $Ir_4(CO)_{11}L$ and $Ir_4(CO)_{10}L_2$ in moderate yields. Since $Ir(CO)_2$(*p*-toluidine)Cl can be prepared in nearly quantitative yield from the commercially available iridium trichloride hydrate,[11] overall yield of substituted tetranuclear iridium products is good. Furthermore, only a simple apparatus suitable for pressures up to about 5 atm[12] is required, eliminating the need for high-pressure equipment.

Experimental Section

$Ir(CO)_2$(*p*-toluidine)Cl[11] and dimethylphenylphosphine[13] were prepared according to literature methods. The other ligands were obtained commercially. Trimethyl and triphenyl phosphite were distilled prior to use. Separations were carried out by preparative-scale TLC on silica gel (Merck) eluting with either petroleum ether (bp 60–68 °C) or petroleum ether–dichloromethane mixtures. Infrared spectra were recorded on a Beckman IR-12 spectrophotometer and calibrated with cyclohexane (2138 cm^{-1}) and polystyrene. Microanalyses were performed by the University of Illinois analytical laboratory. Mass spectra were obtained on a Varian-MAT CH-5 mass spectrometer using an ionizing voltage of 70 eV. Melting points were taken in evacuated, sealed capillary tubes on a Büchi melting point apparatus.

$Ir_4(CO)_{11}(PMe_2Ph)$ and $Ir_4(CO)_{10}(PMe_2Ph)_2$. $Ir(CO)_2$(*p*-toluidine)Cl (155 mg, 0.397 mmol) and dimethylphenylphosphine (13.7 mg, 0.099 mmol) were added to a 250-ml heavy-walled glass pressure bottle containing acid-washed mossy zinc (3 g), a magnetic stirring bar, and a solution of 2-methoxyethanol (30 ml) and water (1 ml) which had been saturated with carbon monoxide prior to use. After thorough flushing with carbon monoxide, the pressure bottle was charged to 4 atm and heated in an oil bath at 90 °C with stirring for 30 min. The cooled yellow solution was then filtered and the solvent removed at reduced pressure. The yellow residue was separated by preparative-scale TLC (petroleum ether–dichloromethane, 5:1), affording $Ir_4(CO)_{11}(PMe_2Ph)$ and $Ir_4(CO)_{10}(PMe_2Ph)_2$ as orange solids, which were recrystallized from pentane. The yields were 20% and 35%, respectively. Anal. Calcd for $C_{19}H_{11}O_{11}Ir_4P$: C, 18.78; H, 0.90; P, 2.55. Found: C, 19.00; H, 0.97; P, 2.78. Calcd for $C_{26}H_{22}O_{10}Ir_4P_2$: C, 23.56; H, 1.66; P, 4.68. Found: C, 23.49; H, 1.56; P, 4.76. A small amount of $Ir_4(CO)_{12}$ was formed as a yellow precipitate in this reaction. It was extracted from the zinc residues with boiling chlorobenzene (50 ml).

The preparations of $Ir_4(CO)_{11}L$ and $Ir_4(CO)_{10}L_2$ (L = PPh_3, P(*p*-tol)$_3$, PEt_3), and of $Ir_4(CO)_{10}$(diphos) proceeded analogously. For L = $P(OMe)_3$ and $P(OPh)_3$, tetrahydrofuran was substituted for 2-methoxyethanol to avoid exchange reactions between the ligand and solvent. Satisfactory carbon and hydrogen analyses were obtained for all of these compounds.

Results and Discussion

Reduction of $Ir(CO)_2$(*p*-toluidine)Cl by zinc in the presence of carbon monoxide and 0.25 equiv of a phosphine or phosphite ligand provides a mixture of $Ir_4(CO)_{11}L$ and $Ir_4(CO)_{10}L_2$. This mixture is separated readily by TLC and the compounds are isolated as air- and moisture-stable yellow or orange solids (for melting points, see Table I). The trimethyl phosphite, triethylphosphine, and dimethylphenylphosphine derivatives provide clean mass spectra with molecular ions, stepwise loss of carbonyls, and isotope patterns consistent with the presence of four iridium atoms. In several cases loss of hydrogen molecules accompanies loss of the last few carbonyl ligands.[14] Although molecular ions are seen for $Ir_4(CO)_{11}(PPh_3)$, $Ir_4(CO)_{11}$(P(*p*-tol)$_3$), and $Ir_4(CO)_{10}(P(OPh)_3)_2$, in general the temperatures required to observe spectra for derivatives of the heavier ligands cause extensive decomposition. Complex fragments and ions resulting from $Ir_4(CO)_{12}$ dominate the spectra.

Table I contains infrared data for the compounds prepared. There is reasonable agreement with previously reported values for $Ir_4(CO)_{11}(PPh_3)$,[6] $Ir_4(CO)_{10}(PPh_3)_2$,[8] and $Ir_4(CO)_{10}(PEt_3)_2$[3]. Bands for bridging carbonyls are observed for all of the compounds, indicating structural rearrangement upon substitution into the unbridged structure of $Ir_4(CO)_{12}$.[15] A constant structural type for the $Ir_4(CO)_{11}L$ compounds is suggested by their closely similar spectra.[16] Though broadly similar, the $Ir_4(CO)_{10}L_2$ spectra exhibit larger variations, which may be due to a ligand-dependent distribution of structural isomers. Preliminary ^1H NMR results for $Ir_4(CO)_{10}(P(OMe)_3)_2$ and $Ir_4(CO)_{10}(PMe_2Ph)_2$ indicate the presence of stereochemically nonrigid isomers in solution; this dynamic behavior is being studied further and will be reported separately.[20]

Isolated yields of the $Ir_4(CO)_{11}L$ and $Ir_4(CO)_{10}L_2$ clusters are consistently 15–20% and 25–40%, respectively, based on iridium, which represents a high percentage of the added ligand. With 0.5 equiv of added ligand $Ir_4(CO)_{10}L_2$ is obtained in ca. 50% yield but only traces of $Ir_4(CO)_{11}L$ are observed. When the reduction of $Ir(CO)_2$(*p*-toluidine)Cl is conducted in the presence of excess triphenylphosphine (>1:1),

$Ir_2(CO)_6(PPh_3)_2$ is isolated in high yield. Malatesta, Angoletta, and Caglio[9] observed that $Ir_2(CO)_6(PPh_3)_2$ is converted to $Ir_4(CO)_9(PPh_3)_3$ in refluxing benzene, and we have confirmed that the reaction occurs readily in chlorobenzene at 100 °C. Furthermore, Hanlan and Ozin[21] have recently reported that matrix-isolated $Ir_2(CO)_8$, when warmed above −58 °C, is transformed to a bridged form of $Ir_4(CO)_{12}$. These results suggest that reduction of $Ir(CO)_2(p\text{-toluidine})Cl$ under the conditions described proceeds first to iridium dimers, which then react further to give the final tetranuclear products.

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Registry No. $Ir_4(CO)_{11}PPh_3$, 53565-22-3; $Ir_4(CO)_{11}PMe_2Ph$, 59532-78-4; $Ir_4(CO)_{11}P(p\text{-tol})_3$, 59532-79-5; $Ir_4(CO)_{11}PEt_3$, 59532-80-8; $Ir_4(CO)_{11}P(OMe)_3$, 59532-81-9; $Ir_4(CO)_{11}P(OPh)_3$, 59532-82-0; $Ir_4(CO)_{10}(PPh_3)_2$, 59532-83-1; $Ir_4(CO)_{10}(PMe_2Ph)_2$, 59532-55-7; $Ir_4(CO)_{10}(P(p\text{-tol})_3)_2$, 59532-58-0; $Ir_4(CO)_{10}(PEt_3)_2$, 29132-21-6; $Ir_4(CO)_{10}(P(OMe)_3)_2$, 59532-54-6; $Ir_4(CO)_{10}(P(OPh)_3)_2$, 59532-57-9; $Ir_4(CO)_{10}(diphos)$, 59532-56-8; $Ir(CO)_2(p\text{-toluidine})Cl$, 14243-22-2; PPh_3, 603-35-0; PEt_3, 554-70-1; $P(OMe)_3$, 121-45-9; $P(OPh)_3$, 101-02-0.

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Preparation and Properties of a Stable Semiquinone Complex

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In a recent article, we reported the synthesis and characterization of a series of cobalt(III) catecholato complexes.[1] Cyclic voltametric studies of those systems indicated that they tend to undergo two one-electron oxidations, the first of which is quasireversible. Therefore, it appeared to us as if a relatively stable one-electron oxidation product might be found. Such a product would very likely contain a coordinated *o*-semiquinone. Of the several systems we have studied we find a rather stable product formed upon oxidation of (3,5-di-*tert*-butylcatecholato)triethylenetetraminecobalt(III) chloride, [Co(trien)(DBcat)]Cl.[2] The oxidation was found to be a *one*-electron oxidation and, as will be shown below, the compound formed is a stable *o*-semiquinone complex, (3,5-di-*tert*-butyl-*o*-benzosemiquinonato)triethylenetetraminecobalt(III) chloride, $[Co(trien)(DBsq)]Cl_2$. The complex has been isolated as a stable solid and fully characterized.

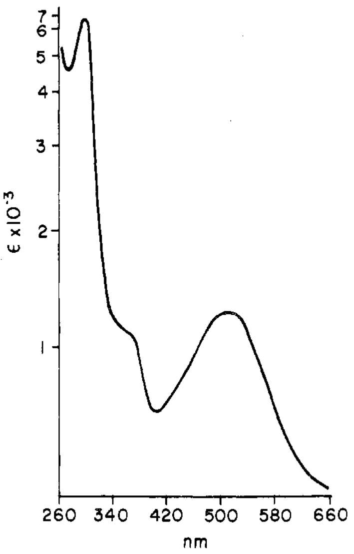

Figure 1. Electronic spectrum of $[Co(trien)(DBsq)]Cl_2$ obtained on an 8.5×10^{-4} M solution of the complex in methanol.

Results

The original cyclic voltametric work on [Co(trien)(DBcat)]Cl indicated that a one-electron oxidized species was generated at approximately +0.2 V vs. SCE.[1] Initial attempts to generate this product by chemical oxidation gave a bright red species in solution. The uv–visible spectrum of this species is given in Figure 1. Chemical oxidants which can produce this red complex include Fe^{3+}, Cu^{2+}, Ce^{4+}, and $Pb(OAc)_4$. Spectral titration indicated that when 1 equiv of oxidant had been added, the spectrum shown in Figure 1 was fully formed and additional oxidant caused no appreciable further spectral changes. Thus the intense red species appeared indeed to be the one-electron oxidation product observed in the cyclic voltamograms. Controlled-potential electrolysis at +0.4 V vs. SCE also generated the same species. The observation that the species is a *one*-electron oxidation product was reconfirmed by coulometry.

The fact that this one-electron oxidation product contained a coordinated semiquinone is demonstrated by the ESR spectrum given in Figure 2. The obvious eight-line pattern arises from hyperfine coupling to the cobalt-59 nucleus. This spectrum can be obtained by oxidizing [Co(trien)(DBcat)]Cl with a variety of oxidants, $Pb(OAc)_4$ being the most convenient.

After having generated and characterized this species in solution and observing its stability, we made attempts to isolate it as a pure solid. The complex $[Co(trien)(DBsq)]Cl_2$ has been successfully prepared and isolated by oxidizing [Co(trien)(DBcat)]Cl with hydrogen peroxide in acid solution. The semiquinone complex is isolated as an amorphous red-purple solid. It is soluble in water, methanol, and ethanol and slightly soluble in acetone. Characterization has been accomplished by elemental analysis, conductivity, magnetism, and spectral measurements. The ir spectrum of the complex shows a very