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XXXIII *. SOME NEW REACTIONS OF CYCLOPENTADIENYLTETRACARBONYLNIOBIUM **

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Summary

Photolysis of $C_5H_5Nb(CO)_4$ with excess cycloheptatriene gives the dark brown tetrahapto complex $C_5H_5Nb(CO)_2C_7H_8$ but no $C_5H_5NbC_7H_7$ analogous to the corresponding reaction of $C_5H_5V(CO)_4$ with cycloheptatriene. Photolysis of $C_5H_5Nb(CO)_4$ with cyclooctatetraene gives the dark green tetrahapto complex $C_5H_5Nb(CO)_2C_8H_8$, the C_8H_8 ring in this complex remains fluxional below −86°C. Reaction of $C_5H_5Nb(CO)_4$ with I_2 gives red-brown $C_5H_5Nb(CO)_3I_2$ in which the carbonyl groups are relatively labile. Thus reaction of $C_5H_5Nb(CO)_3I_2$ with $(CH_3)_2PCH_2CH_2P(CH_3)_2$ under ambient conditions results in the rapid replacement of two CO groups to give $C_5H_5Nb(CO)[(CH_3)_2PCH_2CH_2P(CH_3)_2]I_2$. Treatment of $C_5H_5V(CO)_4$ with I_2 at room temperature gives the carbonyl-free complex $C_5H_5VI_2$ with no evidence for any cyclopentadienylvanadium carbonyl iodide intermediates.

Introduction

A key intermediate for the preparation of cyclopentadienylniobium derivatives is $C_5H_5Nb(CO)_4$. However, until recently a reliable and efficient preparation of this important organometallic compound has not been available. Thus the original method [2] for the preparation of $C_5H_5Nb(CO)_4$ gave very low yields (~2%) and was unreliable. Subsequent work [3] led to the development

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* For part XXXII of this series see ref. 1.

** This paper is dedicated to Professor Henry Gilman in appreciation of all his contributions to organometallic chemistry.

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of a method for preparing $C_5H_5Nb(CO)_4$ in improved yields (40%) but this method is difficult and has not been widely used. Nevertheless the improved availability of $C_5H_5Nb(CO)_4$ allowed a limited study of its chemistry [4] including its reactions with triphenylphosphine [5], diphenylacetylene [6], and cyclopentadiene [7].

The recent development of methods for the preparation of $C_5H_5NbCl_4$ in large quantities [8,9] has spawned a renaissance in the chemistry of $C_5H_5Nb(CO)_4$ since reductive carbonylation of $C_5H_5NbCl_4$ with active metals gives good to excellent yields of $C_5H_5Nb(CO)_4$. Thus we found that carbonylation of $C_5H_5NbCl_4$ with zinc in tetrahydrofuran for two days at 25°C and 200 atmospheres CO readily gives a 27% yield of $C_5H_5Nb(CO)_4$. After we carried out most of the reactions of $C_5H_5Nb(CO)_4$ outlined in this paper, a report appeared by Hermann and Biersack [10] describing a better method for preparing $C_5H_5Nb(CO)_4$ by the reductive carbonylation of $C_5H_5NbCl_4$ with sodium sand in the presence of a copper-aluminum alloy as a halogen acceptor. These workers achieved yields of 89—94% by carrying out such a reductive carbonylation at 135°C for 135 h under 330 atmospheres CO.

This paper describes our studies over the last several years of the reactions of $C_5H_5Nb(CO)_4$ with cycloheptatriene, cyclooctatetraene, and iodine. These reactions are of interest since they yield products different from those obtained in corresponding reactions of $C_5H_5V(CO)_4$.

Experimental

All manipulations were carried out under an atmosphere of dry nitrogen using standard Schlenk tube techniques. Solvents were distilled from an appropriate drying agent before use and degassed by evacuating and filling with nitrogen a Schlenk tube containing the solvent. Cycloheptatriene and cyclooctatetraene were purchased from Aldrich Chemical Company, Milwaukee, Wisconsin, and distilled before use. The reported [8] reaction of $(n\text{-}C_4H_9)_3SnC_5H_5$ with $NbCl_5$ was used to prepare $C_5H_5NbCl_4$. Analyses were performed by Schwarzkopf Microanalytical Laboratories, Woodside, New York.

Preparation of cyclopentadienyltetracarbonylniobium

The bulk of the preparative work described in this paper was done before the report by Hermann and Biersack [10] of the high yield preparation of $C_5H_5Nb(CO)_4$ by the reductive carbonylation of $C_5H_5NbCl_4$ using the Na/Al/Cu trimetallic system. We therefore obtained $C_5H_5Nb(CO)_4$ by the reductive carbonylation of $C_5H_5NbCl_4$ with zinc as described below.

An autoclave of 250 ml internal capacity was loaded with 15 g (50 mmol) of $C_5H_5NbCl_4$ and 8 g (122 mg-atom) of zinc metal. Freshly distilled tetrahydrofuran (100 ml) was added and the autoclave was closed. After flushing with 35 atmospheres CO, the autoclave was filled with 200 atmospheres CO and rocked for two days at room temperature. After venting, the contents of the autoclave were transferred to a 1 liter Schlenk tube and a mixture of 500 ml of dichloromethane and 200 ml of hexane was added. The red solution was filtered through a Florisil column and then the volatile products were removed in vacuum. The resulting red solid was sublimed at 90°C/0.1 mmHg to give 3.6 g

(27% yield) of $C_5H_5Nb(CO)_4$, identified by its infrared and proton NMR spectra.

Reactions of $C_5H_5Nb(CO)_4$ with cycloheptatriene

A solution of 3.0 g (11 mmol) of $C_5H_5Nb(CO)_4$ and 13.0 g (141 mmol) of cycloheptatriene in approximately 300 ml of heptane was exposed to sunlight. A steady evolution of gas through the exit bubbler was observed. Monitoring the reaction by its infrared $\nu(CO)$ frequencies showed steady conversion of $C_5H_5Nb(CO)_4$ to $C_5H_5Nb(CO)_2C_7H_8$. After the reaction was complete, the volatile components were removed in vacuum. The solid residue was dissolved in approximately 200 ml of hexane and filtered into another Schlenk tube. The clear red-brown solution was stored at −10°C for 2 days. During this time brown crystals of the product appeared. The crystals were collected, washed with hexane and dried in vacuum to give 2.6 g (77% yield) of air-sensitive $C_5H_5Nb(CO)_2C_7H_8$; infrared $\nu(CO)$ frequencies (heptane): 1978s and 1910s cm^{-1}; proton NMR spectrum ($C_6D_5CD_3$): δ (ppm) 6.29 (double doublet, J_1 8 Hz, J_2 10 Hz, 1 H) 5.25 (multiplet, 1 H), 4.96 (multiplet, 2 H), 4.84 (singlet, 5 H), 2.87 (multiplet, 2 H), and 2.59 (multiplet, 2 H): mass spectrum (80°C, 12 eV, relative intensities in parentheses): $C_5H_5Nb(CO)_2C_7H_8^+$ (1), $C_5H_5Nb(CO)C_7H_8^+$ (15), $C_5H_5NbC_7H_8^+$ (81), $C_5H_5NbC_7H_7^+$ (92), $C_5H_5NbC_5H_6^+$ (4), $(C_5H_5)_2Nb^+$ (2), $C_5H_5Nb(CO)_2^+$ (5), $C_5H_5Nb(CO)^+$ (5), $C_6H_7Nb^+$ (2), $C_6H_6Nb^+$ (1), $C_5H_5Nb^+$ (11), $Nb(CO)_2^+$ (6), Nb^+ (10), $C_7H_8^+$ (100), $C_7H_7^+$ (65), $C_5H_5Nb^{2+}$ (6), $C_6H_6^+$ (2), $C_5H_6^+$ (3), and $C_5H_5^+$ (1). Anal. Found: C, 54.3; H, 4.3; Nb, 29.2. $C_{14}H_{13}NbO_2$ calcd.: C, 54.9; H, 4.2; Nb, 30.4%.

Slow sublimation at 70°C/0.01 mmHg could also be used to purify $C_5H_5Nb(CO)_2C_7H_8$; during this sublimation the compound melted at ~50°C to give a purple liquid from which bubbles of gas evolved slowly. However, 60% of the original material was recovered.

Boiling solutions of $C_5H_5Nb(CO)_4$ and cycloheptatriene in heptane under reflux also yielded $C_5H_5Nb(CO)_2C_7H_8$ as indicated by infrared spectroscopy. Boiling a solution of $C_5H_5Nb(CO)_4$ in cycloheptatriene under reflux similar to the reported [11] preparation of $C_5H_5VC_7H_7$ did not give significant quantities of either $C_5H_5Nb(CO)_2C_7H_8$ or $C_5H_5NbC_7H_7$ even though the latter has been synthesized by a different method [12].

Reaction of $C_5H_5Nb(CO)_4$ with cyclooctatetraene

In a procedure analogous to that described above for the reaction of $C_5H_5Nb(CO)_4$ with cycloheptatriene, 2.2 g (8.1 mmol) of $C_5H_5Nb(CO)_4$ was photolyzed with 20.0 g (192 mmol) of cyclooctatetraene to give 1.60 g (62% yield) of dark green, very air-sensitive crystalline $C_5H_5Nb(CO)_2C_8H_8$; infrared $\nu(CO)$ frequencies (heptane): 1985s and 1920s cm^{-1}; proton NMR spectrum ($C_6D_5CD_3$): δ (ppm) 5.57 (singlet, 8 H) and 4.38 (singlet, 5 H); mass spectrum (52°C, 70 eV, relative intensities in parentheses): $C_5H_5Nb(CO)_2C_8H_8^+$ (5), $C_5H_5Nb(CO)C_8H_8^+$ (0.6), $C_5H_5NbC_8H_8^+$ (100), $C_5H_5NbC_6H_6^+$ (44), $(C_5H_5)_2Nb^+$ (8), $C_5H_5NbC_4H_2^+$ (19), $C_8H_8Nb^+$ (1), $C_5H_5NbC_2H_2^+$ (3), $C_5H_5Nb^+$ (51), $Nb(CO)_2^+$ (2), $C_8H_8^+$ (30), $C_6H_6^+$ (16), and $C_5H_5^+$ (3). Anal. Found: C, 54.2; H, 4.3; Nb, 30.0. $C_{15}H_{13}NbO_2$ calcd.: C, 56.6; H, 4.1; Nb, 29.2%.

This compound is readily oxidized losing C_8H_8: a second analysis on the same sample which had been stored in a glove box showed 40% carbon indicating substantial loss of the C_8H_8 ligand. Rigorous exclusion of air was necessary to obtain the other spectroscopic data.

Several attempts were made to convert $C_5H_5Nb(CO)_2C_8H_8$ into the carbonyl-free complex $C_5H_5NbC_8H_8$. Continued photolysis of $C_5H_5Nb(CO)_4$ in the presence of excess cyclooctatetraene gave solutions exhibiting a single infrared $\nu(CO)$ frequency at 1910 cm^{-1} and finally a dark solution exhibiting no $\nu(CO)$ frequencies. Attempts to obtain pure products from these very sensitive solutions failed. Heating $C_5H_5Nb(CO)_2C_8H_8$ in a sealed evacuated tube at 100°C for prolonged periods gave $C_5H_5Nb(CO)_4$ and a black solid which could not be recrystallized.

Reaction of $C_5H_5V(CO)_4$ with iodine

A filtered red-orange solution of 1.0 g (4.4 mmol) of $C_5H_5V(CO)_4$ in 150 ml of dichloromethane was stirred vigorously at room temperature for 1 h with 1.0 g (4.0 mmol) of I_2. During this time rapid gas evolution occurred with concurrent precipitation of a brown-black solid. This solid was filtered, washed with dichloromethane followed by pentane, and dried in vacuum to give 1.4 g (95% yield) of $C_5H_5VI_2$; infrared spectrum: no $\nu(CO)$ frequencies; mass spectrum (223°C, 70 eV, relative intensities in parentheses): $C_5H_5VI_2^+$ (5), $(C_5H_5)_2VI^+$ (22), VI_2^+ (2), I_2^+ (28), $C_5H_5VI^+$ (9), $(C_5H_5)_2V^+$ (100), VI^+ (18), I^+ (27), $C_5H_5V^+$ (90), $C_3H_3V^+$ (19), $C_5H_5^+$ (3), and V^+ (8). Anal. Found: C, 16.5; H, 1.6; I, 68.2; V, 13.6. $C_5H_5I_2V$ calcd.: C, 16.2, H, 1.4; I, 68.6; V, 13.8%.

Continuous monitoring of the infrared spectrum in the $\nu(CO)$ region of the solution obtained by treatment of $C_5H_5V(CO)_4$ with a deficiency of iodine failed to reveal any vanadium carbonyl derivatives other than unreacted $C_5H_5V(CO)_4$.

Reaction of $C_5H_5Nb(CO)_4$ with iodine

A heptane solution of 1.2 g (4.4 mmol) of $C_5H_5Nb(CO)_4$ was stirred vigorously with 1.0 g (4.0 mmol) of solid I_2 for 1 h after which time all of the iodine had dissolved and a red-brown solid precipitated out of solution. The precipitate was collected, washed with heptane, and dried in vacuum to give 1.8 g (91% yield) of $C_5H_5Nb(CO)_3I_2$; infrared $\nu(CO)$ frequencies (KBr pellet): 2020m and 1960s cm^{-1}. Anal. Found: C, 17.5; H, 1.4; I, 51.5; Nb, 19.1. $C_8H_5I_2NbO_3$ calcd.; C, 19.4; H, 1.0; I, 51.2; Nb, 18.7%.

This product readily loses CO upon evacuation, which may account for the low carbon analysis. Prolonged evaluation of $C_5H_5Nb(CO)_3I_2$ gives a darker color solid whose dichloromethane solution shows no infrared $\nu(CO)$ frequencies. However, upon bubbling CO through this solution, new infrared frequencies appear at 1995 and 1965 cm^{-1}. This process was found to be reversible through several cycles of evacuation and refilling with CO.

The stoichiometry of the reaction of $C_5H_5Nb(CO)_4$ with I_2 was also monitored by titrating dichloromethane solutions of the reactants using infrared spectra in the $\nu(CO)$ region to analyze the niobium carbonyl derivatives present in the reaction mixture. A quantitative yield of $C_5H_5Nb(CO)_3I_2$ was formed at a Nb/I ratio of 1/2.

The mass spectrum (70 eV) of $C_5H_5Nb(CO)_3I_2$ could only be obtained at relatively high temperatures (214°C) under which conditions there were no major ions showing retention of coordinated CO. The following ions were observed in such a spectrum (relative intensities in parentheses): $(C_5H_5)_2Nb_2I_3^+$ (9), $C_5H_5NbI_3^+$ (16), $(C_5H_5)_2NbI_2^+$ (36), $C_5H_5NbI_2^+$ (85), $(C_5H_5)_2NbI^+$ (68), NbI_2^+ (10), $C_5H_5NbOI^+$ (17), $C_5H_5NbI^+$ (100), $C_3H_3NbI^+$ (33), $(C_5H_5)_2Nb^+$ (71), $C_5H_5NbO^+$ (17), $C_5H_5Nb^+$ (52), NbO^+ (3), NbC^+ (3), Nb^+ (4) and $C_5H_5^+$ (3).

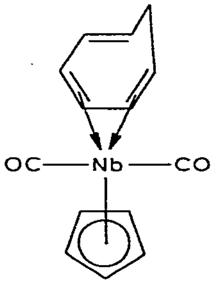
Reaction of $C_5H_5Nb(CO)_3I_2$ with $(CH_3)_2PCH_2CH_2P(CH_3)_2$

A red-brown solution of 0.28 g (0.56 mmol) of $C_5H_5Nb(CO)_3I_2$ in 10 ml of tetrahydrofuran was treated with a solution of 0.25 g (1.67 mmol) of $(CH_3)_2PCH_2CH_2P(CH_3)_2$ in 10 ml of tetrahydrofuran. The reaction mixture immediately turned green with gas evolution. After stirring for 30 minutes, 100 ml of heptane was added to the dark green solution. Filtration of the resulting microcrystalline green precipitate followed by washing with an additional 20 ml of heptane and drying in vacuum gave a nearly quantitative yield of $C_5H_5Nb(CO)I_2[(CH_3)_2PCH_2CH_2P(CH_3)_2]$, identified by comparison of its infrared ν(CO) frequencies and proton NMR spectrum with those reported in the literature [13] for authentic material obtained from $C_5H_5Nb(CO)_2[(CH_3)_2PCH_2CH_2P(CH_3)_2]$ and methyl iodide.

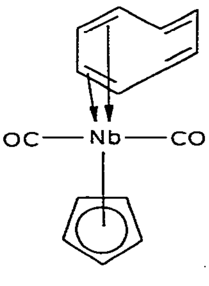
Results and discussion

A. Reactions of $C_5H_5M(CO)_4$ (M = V and Nb) with cycloheptatriene and cyclooctatetraene

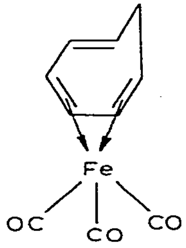
The photochemical reactions of $C_5H_5Nb(CO)_4$ with cycloheptatriene and cyclooctatetraene give the tetrahapto complexes $C_5H_5Nb(CO)_2C_7H_8$ (I) and $C_5H_5Nb(CO)_2C_8H_8$ (II), respectively, as air-sensitive solids soluble in organic solvents and exhibiting the expected two infrared ν(CO) frequencies. The proton NMR spectra of the tetrahapto ligands in I and II resemble closely those of the corresponding tetrahapto ligands in their well-known $Fe(CO)_3$ complexes $C_7H_8Fe(CO)_3$ (III) [14,15] and $C_8H_8Fe(CO)_3$ (IV) [16,17,18], respectively. Thus the proton NMR spectrum of the C_7H_8 ligand in $C_5H_5Nb(CO)_2C_7H_8$ (I) reveals a 1/1/2/2/2 pattern of multiplets similar to that found [14,15] for $C_7H_8Fe(CO)_3$ (III). The proton NMR spectrum of the C_8H_8 ligand in $C_5H_5Nb(CO)_2C_8H_8$ (II) shows a single sharp resonance at ambient temperature indicating a fluxional [19] C_8H_8 ring similar to that found in $C_8H_8Fe(CO)_3$. Cooling a tolu-



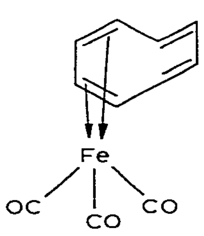
(I)



(II)



(III)



(IV)

ene-d_8 solution of $C_5H_5Nb(CO)_2C_8H_8$ (II) to −86°C resulted in only slight broadening of the C_8H_8 singlet relative to the C_5H_5 singlet indicating that the C_8H_8 ring in $C_5H_5Nb(CO)_2C_8H_8$ remains fluxional at relatively low temperatures like the C_8H_8 ring in $C_8H_8Fe(CO)_3$ [20,21,22].

Some attempts were made to substitute more than two CO groups in $C_5H_5Nb(CO)_4$ with cycloheptatriene and cyclooctatetraene thereby providing complexes with more than four carbons of the cyclic polyolefin bonded to a single niobium atom. However, all such attempts were unsuccessful. Thus reaction of $C_5H_5Nb(CO)_4$ with cycloheptatriene under forcing conditions failed to provide any evidence for the formation of significant quantities of $C_5H_5NbC_7H_7$ even though this niobium complex has been prepared by a different method [12] and corresponding reactions of $C_5H_5V(CO)_4$ with cycloheptatriene give relatively good yields of the vanadium analogue $C_5H_5VC_7H_7$ [11]. Possibly the C_5H_5Nb system is not as effective as the C_5H_5V system in abstracting the hydrogen from cycloheptatriene to form C_7H_7 derivatives. Photolysis of $C_5H_5Nb(CO)_4$ with excess cyclooctatetraene appeared to result eventually in substitution of more than two CO groups; at one point a sensitive solution was obtained exhibiting a single ν(CO) frequency at 1910 cm^{-1} suggesting the hexahapto complex $C_5H_5Nb(CO)C_8H_8$. However, the extreme sensitivity of this solution precluded the isolation of any pure compounds.

B. Reactions of $C_5H_5M(CO)_4$ (M = V and Nb) with iodine

Many mononuclear carbonyl derivatives react with iodine to replace one CO group with two iodine atoms. Thus iodine reacts with $Fe(CO)_5$ and $C_5H_5Co(CO)_2$ to give $Fe(CO)_4I_2$ [23] and $C_5H_5Co(CO)I_2$ [24], respectively. The reaction of $C_5H_5Nb(CO)_4$ with iodine appears to proceed analogously to give red-brown $C_5H_5Nb(CO)_3I_2$. However, the carbonyl groups in this complex are relatively labile and can be removed by prolonged evacuation. Carbonyl-free material obtained in this manner reversibly absorbs CO to give a cyclopentadienylniobium carbonyl derivative with a ν(CO) infrared spectrum different from that of the original $C_5H_5Nb(CO)_3I_2$; this product could be a bridging dimer such as $[C_5H_5Nb(CO)_2I_2]_2$ since it exhibits two relatively closely spaced ν(CO) frequencies. The lability of the CO groups in $C_5H_5Nb(CO)_3I_2$ is also indicated by the failure to observe any niobium carbonyl ions in its mass spectrum: observed ions possibly indicating a complex decomposition mode of $C_5H_5Nb(CO)_3I_2$ include $(C_5H_5)_2Nb_2I_3^+$, $C_5H_5NbI_3^+$, $(C_5H_5)_2NbI_2^+$, and $C_5H_5NbI_2^+$. In addition the chelating ditertiary phosphine $(CH_3)_2PCH_2CH_2P(CH_3)_2$ rapidly displaces two CO groups from $C_5H_5Nb(CO)_3I_2$ at ambient conditions to give a nearly quantitative yield of the known [13] complex $C_5H_5Nb(CO)I_2[(CH_3)_2PCH_2P(CH_3)_2]$. The complex $C_5H_5Nb(CO)_3I_2$ is an analogue of the reported [25] $C_5H_5Nb(CO)_3Cl_2$ which has been prepared by the reductive carbonylation of $C_5H_5NbCl_4$ at room temperature and atmospheric pressure using amalgamated aluminum.

The corresponding reaction of $C_5H_5V(CO)_4$ with iodine proceeds differently from that of $C_5H_5Nb(CO)_4$ since no cyclopentadienylvanadium carbonyl iodide can be found in the reaction mixture, at least under ambient conditions. Instead the reaction of $C_5H_5V(CO)_4$ with iodine leads directly to the carbonyl-free vanadium(III) derivatives $C_5H_5VI_2$, which apparently has not been reported before. Corresponding reactions of $C_5H_5V(CO)_4$ with the lighter halogens Cl_2 and Br_2 lead to the vanadium(IV) derivatives [26] $C_5H_5VX_3$ (X = Cl and Br) providing another example of the well-known tendency of iodine to stabilize lower metal oxidation states than its lighter congeners [27]. Also the failure for vanadium to form $C_5H_5V(CO)_3I_2$ analogous to $C_5H_5Nb(CO)_3I_2$ may relate to the difficulty of a 3*d* transition metal such as vanadium assuming a coordination number of 8; in such systems the C_5H_5 ring can be regarded as a tridentate ligand occupying 3 coordination positions [28].

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