posed instantaneously upon contact with carbon tetrachloride. The proposed structure is supported by the magnetic properties as well as by its infrared spectrum. Compound (I) showed paramagnetism, corresponding to 1.78 B.M., which is equivalent to one unpaired electron. The presence of only one strong absorption in its infrared spectrum, taken in a KBr disk, at 1845 cm\(^{-1}\) suggests a trans position for the phosphine ligands in the crystalline solid. In \(\text{CH}_3\text{Cl}_2\) the infrared spectrum also exhibits an additional absorption at 1969 cm\(^{-1}\) the intensity of which increases with standing and which is, probably, due to formation of \(\text{V} (\text{CO})_6\) in this solvent. The analogous triphenylarsine and triphenylstibine derivatives were also prepared, but were distinctly less stable, especially in their solutions. For this reason, they could not easily be purified. The greenish-yellow triphenylstibine compound was pyrophoric, reacted with most solvents and showed a strong infrared absorption at 1855 cm\(^{-1}\) (KBr wafer);

\[
\text{C}_{6}\text{H}_{4}\text{O}_{3}\text{SbV}:
\]

Calculated. C 55.27 H 3.48 V 5.76.

Found C 54.2 H 4.75 V 5.3.

It has been shown that the central metal in the mixed triarylpalladium carbonyls of cobalt and manganese \(^3\) can achieve rare gas configuration either by dimerization of the complex \(^2\) or by reduction to an anion \(^6\), \(^7\), thus maintaining the coordination possessed in the neutral complex. For the same sterical reasons which can be advanced to explain the monomeric state of \(\text{V}(\text{CO})_6\), the possible existence of a stable dimeric form of \([(\text{C}_6\text{H}_5\text{J})_3\text{P}]_2\text{V}(\text{CO})_4\) seemed quite improbable. Accordingly, the reduction of the complex was studied.

A suspension of (I) (3 g., 4.36 mmole) in 80 ml. of ethanol was shaken for 15 to 30 minutes with an excess of freshly prepared liquid sodium amalgam of about 1% Na content until the originally colorless solvent had assumed a redbrown color. Addition of excess aqueous tetraethylammonium iodide solution to the clear filtered precipitate which was washed exhaustively with water, ethanol and ether and dried. Recrystallization from acetone/ether afforded 1.25 g. (49%) of crystalline yellow solid, stable in air, m.p. 180—182\(^\circ\), to which structure (II), tetraethylammonium-[pentacarbonyl-triphenylphosphine-vanadium-(1)] has been assigned. Analysis:

\[
\text{C}_{6}\text{H}_{6}\text{NPO}_{3}\text{V}:
\]

Calculated. C 63.8 H 6.04 N 2.4 P 5.31 V 8.73 CO 24.0.

Found C 63.8 H 6.19 N 2.41 P 5.4 V 8.7 CO 23.6.

Compound (II) was found to be diamagnetic; the proposed structure seems compatible with its infrared spectrum which exhibits absorptions in the metal carbonyl region at 1767 (s.), 1805 (v.s.), 1842 (sh.) and 1953 (m.) cm\(^{-1}\).

Other salts of the novel pentacarbonyl-triphenylphosphine-vanadate-(1) anion were obtained in a like fashion and showed very similar infrared absorptions in the carbonyl region. Thus, the tetramethylammonium salt, \([(\text{CH}_3\text{H})_3\text{N}] [(\text{CH}_3\text{H})_3\text{P}]_2\text{V}(\text{CO})_5\) :  

Calculated. C 61.48 H 3.61 N 5.58 Ni 3.9 V 6.76.

Found C 64.6 H 4.2 N 5.0 Ni 4.0 V 6.2, was obtained as a yellow crystalline solid, stable in air for several hours. The tri(o-phenanthroline)-nickel(II) salt, \([(\text{CH}_3\text{H})_3\text{N}] [(\text{CH}_3\text{H})_3\text{P}]_2\text{V}(\text{CO})_5\) :  

Calculated. C 64.8 H 3.61 N 5.58 Ni 3.9 V 6.76.

Found C 64.6 H 4.2 N 5.0 Ni 4.0 V 6.2, was a purple-brown crystalline solid, m. p. 134—136\(^\circ\) (dec.).

This reduction is noteworthy for the surprising redistribution of the ligands whereby one of the bulky triphenylphosphine groups is replaced by carbon monoxide, thus giving rise to the hexacoordinated and diamagnetic air pentacarbonyl-triphenylphosphine-vanadium(-1) anion which could not be obtained directly by reacting a hexacarbonylvanaadate(-1) with triphenylphosphine. It can be assumed that this loss of triphenylphosphine is primarily due to sterical reasons; the comparable reduction of \((\text{C}_6\text{H}_5\text{H})_3\text{PMn}(\text{CO})_4\) to \([(\text{C}_6\text{H}_5\text{H})_3\text{PMn}(\text{CO})_4]^{-}\) follows, accordingly, a more regular pattern.

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**Tetracarbonyl-nitrosyl-triphenylphosphine-vanadium**

*(Chemistry of Vanadiumhexacarbonyl III)*

By Robert P. M. Werner

Research Laboratories of the Ethyl Corporation,
Ferndale 20, Detroit, Michigan, U.S.A.

(Z. Naturforschg. 16b, 476—479 [1961]; eingegangen am 8. Mai 1961)

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5 I am indebted to Professor S. Kirschner at Wayne State University, Detroit, for carrying out the magnetic susceptibility measurements.


7 W. Hierer and E. Linderer, Z. Naturforschg. 16b, 137 [1961].

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Einwirkung von Stickoxyd auf eine Suspension von paramagnetischem Tetracarbonyl-bis-triphenylphosphin-vanadium in einem inerten Kohlenwasserstoff führte zur Bildung von diamagnetischem und petroleumätherlöslichem Tetracarbonyl-nitrosyl-triphenylphosphine-vanadium:

\[
\text{Phi}_{3}\text{P} (\text{CO})_4 \text{V} (\text{NO}) ---\longrightarrow \Phi_{3}\text{PV} (\text{NO}) (\text{CO})_4,
\]

Dieses stellt die erste Nitrosyl-carbonyl-Verbindung des Vanadiums dar.

In previous communications we have reported the novel compounds obtained by the reaction of vanadium-hexacarbonyl with triphenylphosphine \(^1\) and cyclo-
heptatriene, respectively. We now wish to describe the first stable nitrosyl compound derived from V(CO)₆.

Nitric oxide was bubbled for several minutes into the agitated suspension of tetracarbonyl-bis-tripheny1phosphine-vanadium in n-hexane at 25 °C, punctiliously maintaining a protective nitrogen blanket at all times. The clear yellow solution obtained after quickly filtering from the solid mixture of unreacted starting material and dark decomposition products was slowly concentrated and afforded an orange crystalline solid which was subsequently recrystallized from n-hexane. The structure of this compound, tetracarbonyl-nitrosyl-tripheny1phosphine-vanadium, is supported by its analysis C₂₂H₁₅NO₅PV:

Calcd. C 58.40 H 3.29 N 3.05 P 6.74 V 11.09,
Found C 58.8 H 3.47 N 3.14 P 6.2 V 11.2,
diamagnetism and infrared spectrum. From the simplicity of its infrared spectrum (in CCl₄), which shows only one carbonyl stretching mode at 1935 cm⁻¹, as well as a sharp NO-stretching band at 1630 cm⁻¹, it is concluded that the nitrosyl and triphenylphosphine groups are in a trans position, whereas the four carbonyl groups are located in one plane. Here, as well as in the course of the previously reported reduction of tetracarbonyl-bis-tripheny1phosphine-vanadium to the [(C₆H₅)₃PV(CO)₅] anion, the achievement of rare gas configuration is accompanied by loss of one of the tripheny1phosphine ligands.

3 I am indebted to Professor S. Kirchner at Wayne State University, Detroit, for carrying out the magnetic susceptibility measurement.

Die Bildung von Plaques durch das Virus der Maul- und Klauenseuche unter methylcellulose-haltigen Medien

Von Wilhelm Schwöbel

Bundesforschungsanstalt für Viruskrankheiten der Tiere, Tübingen


Derartige Plaques erhält man auch, wenn man an Stelle des Agars ein Medium über die Zellen gibt, dessen Viskosität durch geeignete Zusätze erhöht wurde. Wir benutzen zu diesem Zweck Methylcellulose (Tylose MH 4000 der Firma Kalle — Wiesbaden). Als abgewogene, trocken sterilisierte Substanz wird sie einer bestimmten Menge Kulturmedium zugegeben. Geeignet ist dazu jedes der zur Viruszüchtung üblichen Me-