species, transport by the perhaps more plausible ions SbI_2^+ or SbI_4^- as well as by I^- affects only the coefficient of the solvent term $\ln N_0$ in eq. 3 and, as has been seen, this is of no consequence. However, it is obvious that conduction by ions derived from the solute would directly obviate the previous conclusions.[10] Although the presence of such ions is not reflected by an appreciable change in the conductivity of SbI_3 on reaction with metal, substantiation of the earlier conclusions regarding the nature of the solute and the fact that the solvent furnishes the conducting ions comes from concentration cell experiments in which 2 to 5 mole % KI had been added to the SbI_3. In this case, concentration changes in the catholyte readily can be produced by electrolysis, since even with 2% KI the conductivity of the melt is 100 times greater than that of pure solvent. Furthermore, from what is known concerning conduction processes in fused salt mixtures, it is highly probable that in this solution the potassium ion carries most if not all of the current (with perhaps a small contribution by iodide), so that the interpretation of the cell data is free of any ambiguity associated with conduction by antimony-containing ions. The data obtained still indicate that *nm* is between 1.9 and 2.5. The results are less accurate than those shown in Fig. 2 as they are affected by a change in N_M^0 with KI concentration,[11] for which satisfactory corrections can be applied, and by the continuous diffusion through the diaphragm during the 1 to 4 hr. necessary for equilibrium after each electrolysis. Correction of the data with the diffusion equations derived for a constant-composition anolyte gives more consistent results for *nm*, $2.0_5 \pm 0.1$ (average deviation) for three runs with 2% KI and 1.9 to 2.2 for one with 5% KI.

Although it seems certain that each molecule of the product is formed by a two-electron reduction of SbI_3, as noted in the earlier discussion, the method is rather insensitive to changes in the amount of solvent. Hence, it cannot be ascertained with any accuracy *how many* SbI_3 molecules are involved in the two-electron reduction, that is, how the product is solvated. Although $\cdot\underset{..}{\dot{Sb}}–I$ is not allowed magnetically, spin pairing as a result of strong coördination by the solvent might be possible, giving, for example, $I_2\underset{..}{Sb}–I \rightarrow \underset{..}{\ddot{Sb}}–I$. This is considered unreasonable since the relative acidities expected for the two oxidation states involved would not favor a strong interaction. On the other hand, the isomeric $I_2\underset{..}{Sb}–\underset{..}{Sb}I_2$ does have some precedence in the structure known for P_2I_4[12]; the molecular As_2I_4[13] also may be catenated similarly. It is striking that the chemistry of antimony apparently does not resemble that of bismuth in this respect. Reduction of at least bismuth(III) chloride or bromide gives the bismuth(I) halide,[14] for which the structure $X—\underset{..}{Bi}{=}\underset{..}{Bi}—X$ has been suggested.[15]

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[10] For sample, *nm* would equal 4 with $Sb_mI_m{}^{+(3-n)-1}$.

[11] At 326°, the temperature of these experiments, $N_M{}^0 = 4.26^2$, 3.6 ± 0.1, and 2.6 ± 0.15 for 0, 2.0 and 5.0 mole % KI in SbI_3, respectively. A compound is formed between KI and SbI_3 with a molar ratio greater than unity.

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AMES, IOWA

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, STATE UNIVERSITY OF IOWA]

Metal–Olefin Compounds. I. The Preparation and Molecular Structure of Some Metal–Olefin Compounds Containing Norbornadiene (Bicyclo[2.2.1.]hepta-2,5-diene)

BY R. A. ALEXANDER, N. C. BAENZIGER, C. CARPENTER AND J. R. DOYLE[1]

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The preparation of a number of metal–olefin compounds containing norbornadiene and either platinum or palladium is described. The physical and chemical properties of these compounds indicate that both olefinic bonds of the hydrocarbon are coördinated to a single metal atom in the *cis* positions of the coördination sphere. In addition, two new compounds formed by the 1,5-homoconjugative addition of mercury salts to norbornadiene are described.

Introduction

Several cyclic polyolefins have been reported to form compounds with platinum, palladium and other metallic halides.[2–8] Physical measurements on a number of these compounds, *i.e.*, 1,3,5,7-cyclooctatetraene–dibromoplatinum(II), dicyclopentadiene–dichloroplatinum(II) and 1,5-cycloöctadiene–dichloroplatinum(II) indicate that the hydrocarbon is chelated to the metal atoms. The resulting compounds, in solution, are monomeric species in contrast with 1,3-bis-(ethylene)-2,4-dichloro-μ-dichlorodiplatinum or 1,3-bis-(ethylene)-3,4-dichloro-μ-dichlorodipalladium.[9–12]

Recent work has shown that norbornadiene will undergo a number of addition reactions, and it was of interest to attempt to prepare and determine the molecular configuration of the products formed by the reactions of norbornadiene and platinum or palladium salts. In addition the relative simplicity of norbornadiene would facilitate X-ray crystal-

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[1] To whom communications concerning this paper should be addressed.

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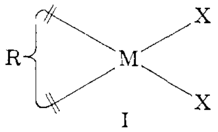
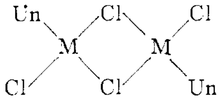


I

R = 1,3,5,7-cycloöctatetraene, dicyclopentadiene or 1,5-cycloöctadiene
X = Chloride, bromide or iodide
M = Platinum or palladium



II

M = Platinum or palladium
Un = Ethylene, styrene or other monoölefins

lographic studies on the diolefin compounds of platinum and palladium.[13]

Experimental

Infrared Spectra.[14]—The infrared absorption spectra were obtained on a Perkin–Elmer Model 21 Spectrophotometer, equipped with sodium chloride optics and a linear wave length scale. The spectra of all solids were observed using potassium bromide pressed discs and checked for spurious bands by obtaining comparison spectra in mineral oil or fluorinated hydrocarbon mulls.

TABLE I

INFRARED ABSORPTION SPECTRA[a] (IN CM.$^{-1}$) OF METAL–OLEFIN COMPLEXES CONTAINING NORBORNADIENE, $C_7H_8MX_2$, KBr discs

Norbornadiene[b]	C_7H_8-$PtCl_2$	C_7H_8-$PtBr_2$	C_7H_8-PtI_2	C_7H_8-$PdCl_2$	C_7H_8-$PdBr_2$
3070S	3400M[c]	3420M[c]	3420M[c]	3420M[c]	3430M[c]
2940VS	3050M	3060M	3030W	3060M	3040M
2870S	2950Shld	2960Shld	2960W	2930W	2970Shld
2120W	2920VW	2920W	2910W	2860Shld	2920W
2040W	1628M[c]	1622M[c]	1624M[c]	1625M[c]	1625M[c]
1650M	1440M	1439M	1437M	1449M	1449M
1608M	1411M	1411W	1410Shld	1428M	1428M
1550S	1392M	1392M	1391M	1410S	1410M
1452S	1312S	1309S	1309S	1388W	1388W
1390M	1252W	1251W	1222W	1311S	1312S
1335S	1243W	1241W	1175M	1227M	1228M
1310VS	1228M	1226M	1157W	1178M	1175S
1269M	1180M	1178M	1072W	1160M	1160VW
1228VS	1103W	1113M	1031W	1082W	1082W
1204VS	1032W	1031W	997W	1042W	1038W
1106M	982M	992W	950M	1000W	997W
1062M	949M	972M	930W	958M	957M
1013M	933W	949M	895W	939M	948M
935S	903W	932W	770M	895M	941Shld
912S	877W	903W		877M	893M
889S	772M	881W		807M	876W
870S		773M		782S	768S
795–790W				769S	
770Shld					
728VS					

[a] VW = very weak intensity; W = weak intensity; M = medium intensity; S = strong intensity; VS = very strong intensity. [b] The referee of this paper has determined the infrared spectra of a gas chromatographically pure sample of norbornadiene and finds a strong band at 797 cm.$^{-1}$ and a weak to medium band at 773 cm.$^{-1}$. The sample used in the present determination was purified by vacuum distillation through a packed column of about thirty theoretical plates. [c] These bands are absent in the spectra obtained by use of a mull and presumably are due to water in the potassium bromide.

Molecular Weight Determinations.—The molecular weights were obtained ebullioscopically, using a modified Menzies–Wright apparatus, according to the procedure described by Smith and Shriner.[15] Measurements were made on solutions containing the solute dissolved in 1,2-dichloroethane and temperature changes were measured by the use of a water vapor thermometer. The concentrations used in these determinations ranged from 0.208 to 0.508 weight %. The solvent was calibrated by using triphenylmethane as a standard and naphthalene as an unknown.

Anal. Calcd. for $C_{10}H_8$: mol. wt., 128.2. Found: mol. wt., 123.6, 140.4.

Norbornadiene-dichloroplatinum(II).[16]—A solution of 0.793 g. (13.52 mmoles) of sodium chloride in 5 ml. of water was added to a solution of 3.5 g. (6.75 mmoles) of chloroplatinic acid hexahydrate in 5 ml. of water. The mixture was evaporated on a steam-bath yielding orange hydrated sodium hexachloroplatinate(IV). The product was pulverized and dried at 105° yielding anhydrous sodium hexachloroplatinate(IV) which was placed in a 125-ml. round bottom flask and dissolved in 25 ml. of absolute ethanol. A reflux condenser was attached to the flask and the solution was maintained at 50° for a three day period. At the end of this period the solution was golden yellow in color and solid sodium chloride had precipitated from the mixture. The mixture was filtered and to the filtrate containing Zeise's acid,[17] $H(C_2H_4PtCl_3)$, was added 5 ml. of norbornadiene and a few drops of concentrated hydrochloric acid. The mixture was heated on a steam-bath for ten minutes during which time a fine white crystalline product separated from the solution. The reaction mixture was cooled and stored at room temperature for 12 hr. The product was removed by filtration and washed with 10 ml. of ethanol and then 10 ml. of diethyl ether. The crude product was redissolved in 1.0 liter of methylene chloride, and the solution was decolorized with charcoal and filtered. The product was recovered by evaporation of the filtrate on a steam-bath to the point of crystallization and on subsequent cooling to room temperature it separated as white needle-like crystals. Evaporation of the mother liquor yielded a further quantity of product resulting in a total yield of 1.50 g. (theory 2.42 g.) of norbornadiene-dichloroplatinum(II).

Anal. Calcd. for $C_7H_8PtCl_2$: C, 23.46; H, 2.25; mol. wt., 358.3. Found: C, 23.45; H, 2.24; mol. wt., 418.4, 390.0, 378.6.

Norbornadiene-dibromoplatinum(II).—A 0.400 g. (1.12 mmoles) sample of norbornadiene–dichloroplatinum(II) was suspended in 150 ml. of acetone. To this mixture was added 0.600 g. (5.73 mmoles) of lithium bromide monohydrate, and the suspension then was warmed on a steam-bath until solution was complete. The hot solution was filtered and the filtrate evaporated to the point of crystallization. The product was removed by filtering and along with a second crop of crystals was recrystallized from methylene chloride. The yield was 0.400 g. (theory 0.501 g.) of pale yellow needle-like crystals.

Anal. Calcd. for $C_7H_8PtBr_2$: C, 18.79; H, 1.80. Found: C, 18.44; H, 1.77.

Norbornadiene-diiodoplatinum(II).—Twenty grams (41.13 mmoles) of potassium hexachloroplatinate(IV) was added to a solution of 41 g. of potassium iodide in 400 ml. of water. An immediate chocolate brown precipitate occurred and to this mixture was added 15 g. (162.7 mmoles) of norbornadiene. The container was stoppered and shaken for a few minutes at which time a deep red solution formed and a light yellow precipitate settled to the bottom of the flask. The mixture was kept at room temperature for one week and then filtered to remove the dark brown residue. The residue was washed with 25 ml. of absolute ethanol and then 25 ml. of diethyl ether. The product was purified by dissolving in 2 l. of boiling methylene chloride, and the resulting solution was decolorized with charcoal and filtered. Upon evaporation of the filtrate to the point of crystallization with subsequent cooling the product separated in the form of orange needle-like crystals. A second crop of crystals was recovered in a similar manner giving a combined yield of 18.5 g. (theory 22.26 g.).

Anal. Calcd. for $C_7H_8PtI_2$: C, 15.53; H, 1.48; Pt, 36.07. Found: C, 15.15, 15.70, 16.17; H, 1.48, 1.41, 1.53; Pt, 36.05.

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(13) X-Ray diffraction studies on single crystals of a number of the metal–olefin compounds containing dienes and such metals as platinum, palladium, silver and copper are now in progress and will be reported in the near future.

(14) The determination of the infrared absorption spectra was aided by a grant from the National Science Foundation.

(15) W. T. Smith and R. L. Shriner, "The Examination of New Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 1956, p. 63.

(16) The norbornadiene and 1,3,5-cycloheptatriene were kindly furnished by the Shell Chemical Corporation.

(17) W. C. Zeise, *Chem. Zentr.*, **2**, 677 (1831).

Norbornadiene–dichloropalladium(II).—A 3.00 g. (7.82 mmoles) sample of bis-benzonitrile-dichloropalladium(II), prepared by the method described by Kharasch,[18] was dissolved in 300 ml. of boiling benzene and the resulting solution was filtered. The product was formed by adding 8 ml. of norbornadiene to the filtrate from which the product separated as a light yellow solid. The product was separated by filtration and washed with 20 ml. of methanol followed by 10 ml. of diethyl ether. A single recrystallization from acetone gave 1.86 g. (theory 2.11 g.) of yellow needle-shaped crystals.

Norbornadiene–dichloropalladium(II) also was prepared by dissolving 3.87 g. (13.13 mmoles) of sodium tetrachloropalladate(II) in 150 ml. of absolute methanol. The solution, initially dark brown, was filtered and upon addition of 6 ml. of norbornadiene it turned light yellow and the product separated as a yellow crystalline material. A single recrystallization from acetone yielded 2.40 g. (theory 3.54 g.) of yellow needle-shaped crystals.

Anal. Calcd. for $C_7H_8PdCl_2$: C, 31.17; H, 2.99. Found: C, 31.60, 31.93; H, 3.35, 3.16.

Norbornadiene–dibromopalladium(II).—To a solution of 1.00 g. (2.61 mmoles) of bis-benzonitrile-dichloropalladium(II) in 100 ml. of thiophene-free benzene was added a solution of 0.600 g. of lithium bromide monohydrate dissolved in 50 ml. of acetone. After the addition the solution turned from red to dark brown at which time 4 ml. of norbornadiene was added and the mixture then was heated on a steam-bath until it commenced to boil. Upon cooling to room temperature orange needle-shaped crystals separated and after standing at room temperature for 3 hr. they were removed by filtration.

The product was washed with absolute ethanol and diethyl ether and recrystallized from methylene chloride. The yield was 0.487 g. (theory 0.935 g.) of an orange crystalline material.

Anal. Calcd. for $C_7H_8PdBr_2$: C, 23.46; H, 2.25. Found: C, 23.42; H, 2.12.

1,3,5-Cycloheptatriene–dichloroplatinum(II).[19]—A solution of 2.000 g. (3.4 mmoles) of 1,3-bis-(ethylene)-2,4-dichloro-μ-dichlorodiplatinum, ethylene platinous chloride, in 25 ml. of absolute ethanol was mixed with 2.0 g. of 1,3,5-cycloheptatriene.[15] The mixture was stored at room temperature for 24 hr. during which time the product separated in the form of yellow crystals. The product was separated, washed with absolute ethanol and recrystallized from methylene chloride. The yield was 0.959 g. (theory 2.450 g.) of light yellow crystals.

Anal. Calcd. for $C_7H_8PtCl_2$: C, 23.46; H, 2.25; mol. wt., 358.3. Found: C, 23.54; H, 2.09; mol. wt., 372.4.

1-(Chloromercuri)-5-methoxynortricyclene.—To a solution of 35 g. of mercuric chloride in 300 ml. of acetone-free methanol was added 12 g. of norbornadiene. The container was stoppered and kept at room temperature for three days during which time a large quantity of solid separated from the mixture. The solid was removed by filtering, washed with cold methanol and dried in a desiccator over phosphorus pentoxide. The yield of crude product was 14 g. and on recrystallization from methanol 5.5 g. of white, needle-shaped crystals was recovered, m.p. 121–121.5°.

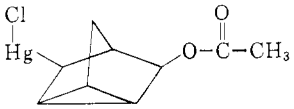
Anal. Calcd. for $C_8H_{11}HgOCl$: C, 26.75; H, 3.09. Found: C, 27.03; H, 3.39.

1-(Chloromercuri)-5-ethoxynortricyclene.—A solution of 35 g. of mercuric chloride in 200 ml. of absolute ethanol was mixed with 15 g. of norbornadiene. The container was stoppered and kept at room temperature for three days then heated to the boiling point of the mixture, decolorized with charcoal and filtered. The filtrate was reheated and water added until a permanent turbidity occurred and on cooling overnight a yellow oil and white flocculent precipitate separated from the mixture. The white crystalline product was decanted off and recrystallized from an ethanol–water mixture. The yield of recrystallized product was 4.1 g., m.p. 101–101.5°.

Anal. Calcd. for $C_9H_{13}HgOCl$: C, 28.96; H, 3.51. Found: C, 28.67; H, 3.44.

Discussion

Among the addition reactions of norbornadiene and ionic reagents several have been reported which involve the use of metallic salts. Winstein and Shatavsky[20] found that mercuric chloride dissolved in glacial acetic acid, added to norbornadiene *via* a 1,5-homoconjugative addition and yielded a nortricyclene derivative as shown in structure III



III

A number of nortricyclene derivatives have been characterized by the absence of the usual olefinic double bond absorption band in the 1550–1650 cm.$^{-1}$ region and the presence of an absorption band in the 790–815 cm.$^{-1}$ region of the infrared spectrum.[21] Recently Cristol and LaLonde[22] have reported that tetrasubstituted nortricyclene derivatives and nortricyclene derivatives that are doubly substituted at C_3 do not show the absorption band in the 790–815 cm.$^{-1}$ region of the infrared spectra.

To examine the infrared spectra of the products of a 1,5-homoconjugative addition to norbornadiene two new addition products containing mercury were synthesized by the reaction of mercuric chloride and norbornadiene dissolved in methanol or ethanol. These reactions result in a variety of products; however, the major constituents have been tentatively identified as 1-(chloromercuri)-5-methoxynortricyclene and 1-(chloromercuri)-5-ethoxynortricyclene depending on whether methanol or ethanol is used as the solvent medium. Neither of these compounds had absorption bands in the 1650–1550 cm.$^{-1}$ region, indicating absence of an olefinic double bond. Both compounds gave absorption bands of medium intensity at 795 cm.$^{-1}$ indicative of a nortricyclene ring system and in addition both derivatives displayed a strong absorption band at 1105 cm.$^{-1}$ which is attributed to the carbon–oxygen–carbon stretching vibration of ethers.[23]

In recent studies on silver nitrate–olefin complexes, Traynham and Olechowski[24] have reported the equilibrium constant for the formation of a 1 to 1 norbornadiene–silver nitrate complex in solution. By the treatment of a 1 *M* solution of silver nitrate with norbornadiene they isolated a white solid and analysis indicated the formation of a 1 to 1 complex. On the basis of similarities of olefin–halogen complexes to the olefin–silver nitrate complexes they proposed that the silver ion coördinated to one of the olefinic bonds of norbornadiene as shown in IV.

A third possible mode of addition is exemplified

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[18] M. S. Kharasch, R. C. Seyler and F. R. Mayo, THIS JOURNAL, **60**, 882 (1938).

[19] Evidence for the formulation of this compound as a metal–olefin compound will be published in the near future. See also: E. O. Fisher and H. P. Fritz, *Z. physik. Chem., Neue Folge*, **17**, [132], 135 (1958).

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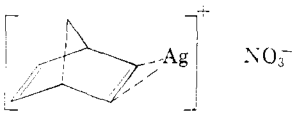
[22] S. J. Cristol and R. L. LaLonde, *ibid.*, **80**, 4355 (1958).

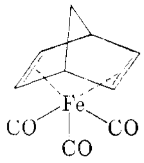
[23] W. West, "Techniques of Organic Chemistry," Vol. IX, Interscience Publishers, Inc., New York, N. Y., 1956, p. 574.

[24] J. G. Traynham and J. R. Olechowski, THIS JOURNAL, **81**, 5711 (1959).



IV

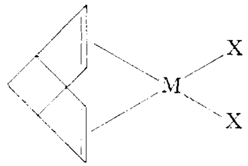
by the reaction of iron pentacarbonyl and norbornadiene as reported by Pettit.[25] Heating a mixture of iron pentacarbonyl and excess norbornadiene produced norbornadiene-tricarbonyl-iron(0) with the proposed structure V



V

In the present study it was found that the analytical data for the metal–olefin compounds containing norbornadiene and platinum or palladium agree with the general formula of $C_7H_8MX_2$, where X represents a halogen atom. The molecular weight of norbornadiene–dichloroplatinum (II) indicates a monomeric species in solution and the rapid reaction of the dichloro derivatives containing platinum or palladium with bromide or iodide ion to form the corresponding dibromo or diiodo derivatives indicates a strong *trans* labilizing ligand. The infrared spectra of all of the norbornadiene–platinum or palladium halides do not display an absorption band in the 1650–1550 cm.$^{-1}$ region indicating that both olefinic bonds of the hydrocarbon moiety are altered on formation of the complexes. This would eliminate structures similar to II and IV which can be considered as norbornene derivatives and as such would be expected to display an absorption band in this region.[22] In addition the absence of an absorption band in the 790–815 cm.$^{-1}$ region indicates that the platinum and palladium halides did not add *via* a 1,5-homoconjugative addition to yield nortricyclene derivatives thus a structure similar to III can be eliminated.

The structure of these compounds can best be explained by an arrangement in which the norbornadiene is chelated to the platinum or palladium in *cis* positions of the coördination sphere of the metal; such a structure is shown in VI. This arrangement is similar to the structure of norbornadiene–tricarbonyliron(0) and norbornadiene–tetracarbonylmolybdenum(0), and both of these compounds are reported to have medium intensity absorption bands at 1430, 1291 and 1177 cm.$^{-1}$. It is interesting to note that the norbornadiene platinum and palladium derivatives also display medium to strong intensity bands close to those cited for the iron and molybdenum carbonyl compounds (see Table I). The 1430–1440 cm.$^{-1}$ absorption bands in the spectra of the platinum and palladium complexes are in the range reported by Chatt and Duncanson[26] for a vibration arising from an olefinic double bond coördinated to platinum.



VI

M = platinum or palladium
X = chloride, bromide or iodide ion

The possible isomerization of norbornadiene to 1,3,5-cycloheptatriene followed by coördination to platinum or palladium was examined by comparison of the infrared spectra 1,3,5-cycloheptatriene–dichloroplatinum(II) and norbornadiene-dichloroplatinum(II). The 1,3,5-cycloheptatriene–dichloroplatinum(II) was prepared by the displacement of ethylene from 1,3-bis-(ethylene)-2,4-dichloro-μ-dichlorodiplatinum and the infrared spectrum of the light yellow crystalline product was distinctly different from norbornadiene–dichloroplatinum(II).

A further confirmation of the proposed structure VI for the norbornadiene platinum and palladium compounds has been obtained from X-ray diffraction data on single crystals of norbornadiene–dichloropalladium(II). Patterson and Fourier projections utilizing *hk*0 data indicate the chlorine atoms occupy *cis* positions in the coördination sphere of the palladium.

Acknowledgments.—This research was supported in part by the Office of Ordnance Research, U. S. Army. J. R. D. wishes to acknowledge the receipt of a du Pont Summer Research Fellowship.

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(26) J. Chatt and L. A. Duncanson, *J. Chem. Soc.*, 2939 (1953).