Table VII. The Ultraviolet Spectra of R'-[Co(d- or l-pn)(trien)]Cl_3

Complexes	Absorption maxima ν_1, 10^3 cm^{-} (log ϵ)	 ν_2, 10^3 cm^{-1} (log ϵ)	 ν_3, 10^3 cm^{-1} (log ϵ)
R'-β-[Co(d-pn)(trien)]Cl_3	21.23 (2.09)	29.50 (2.08)	42.05 (3.97)
R'-β-[Co(l-pn)(trien)]Cl_3	21.23 (2.09)	29.50 (2.08)	42.05 (3.97)

The samples were purified by reprecipitation, as above, and were dried at 100° for 2 hr before analysis.

Reaction of Optically Active α-[$CoCl_2$(trien)]Cl with Optically Active Propylenediamine in Aqueous Solution (the reaction described in Table IV). Elemental analysis gave the following results. Found for L*-β-[Co(d-pn)(trien)]Cl_3: C, 27.75; N, 20.41; H, 7.91. Found for L*-β-[Cl(l-pn)(trien)]Cl_3: C, 27.90; N, 20.75; H, 7.50. Found for D*-β-[Co(d-pn)(trien)]Cl_3: C, 27.91; N, 20.85; H, 7.56. Found for D*-β-[Co(l-pn)(trien)]Cl_3: C, 27.74; N, 20.55; H, 7.39.

The ultraviolet spectra in aqueous solution and the infrared spectra are collected in Tables V and VI.

The experiment using *rac*-[$CoCl_2$(trien)]Cl and optically active propylenediamine was carried out and the products were treated in a similar way. The yield, in each case, was 1.4 g (73%).

Anal. Found for R'-[Co(d-pn)(trien)]Cl_3: C, 27.93; N, 20.21; H, 7.65. Found for R'-[Co(l-pn)(trien)]Cl_3: C, 27.62; N, 20.06; H, 7.77.

The ultraviolet spectra in aqueous solution are shown in Table VII. The infrared spectra of both were identical.

Optical rotatory dispersion curves were measured on 5.0×10^{-3} M solutions.

Spectrophotometric measurements were done on 1.0×10^{-2} M aqueous solutions.

The apparatus was described in an earlier paper.

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The Mass Spectrum and Preparation of Pure Manganese Pentacarbonyl Hydride[1]

Walter F. Edgell and William M. Risen, Jr.[2]

Contribution from the Department of Chemistry, Purdue University, Lafayette, Indiana. Received June 29, 1966

Abstract: The mass spectrum of $HMn(CO)_5$, prepared by the standard literature procedure, shows it to contain impurities. Modifications in the preparative scheme have resulted in a method for the preparation of pure $HMn(CO)_5$. The mass spectrum of the pure $HMn(CO)_5$ is reported. The fragment $Mn\text{–}H^+$ has been observed in the mass spectrum, providing direct evidence for the manganese–hydrogen bond. In addition, the loss of CO and H from $HMn(CO)_5$ are found to be competitive fragmentation processes.

Recently there have appeared several mass spectroscopic studies of organometallic compounds.[3–7] These studies have revealed a number of interesting aspects of the nature of such compounds. Winters and Kiser have investigated in detail the mass spectra of the monometallic transition metal carbonyls $Ni(CO)_4$ and $Fe(CO)_5$,[3] and the hexacarbonyls of chromium, molybdenum, and tungsten.[4] On the basis of their experimental data, they have proposed that the fragmentation of these transition metal carbonyl ions, produced in the spectrometer, occurs by successive removal of neutral CO groups. This proposed successive removal of CO groups has been substantiated for both the $M(CO)_n{}^+$ and $M(CO)_n{}^{2+}$ ions in a detailed metastable transition study of $Fe(CO)_5$.[8] Winters and Kiser[6] and King[7] have studied the mass spectra of polymetallic transition metal carbonyl compounds. Winters and Kiser reported that the spectrum of $Mn_2(CO)_{10}$ contained a peak for the $Mn_2{}^+$ ion, in accordance with the structure containing a manganese–manganese bond. King's[7] spectra of the polynuclear carbonyls $Co_4(CO)_{12}$ and $Ru_3(CO)_{12}$ exhibit mass peaks assigned to the bare metal cluster ions $Co_4{}^+$ and $Ru_3{}^+$, respectively. These ion clusters are in keeping with the established structures of the parent molecules. Thus, the mass spectra of organometallics clearly contain valuable information about the nature of these compounds.

In connection with studies of the infrared spectrum of $HMn(CO)_5$, we were led to examine the mass spectrum of the substance as prepared by the usual method.[9,10] It was found to be impure, but a modification of the method led to a pure product. We report here the mass spectrum of the resulting pure $HMn(CO)_5$.

Experimental Section

Reagents. The $Mn_2(CO)_{10}$ used was supplied by Alfa Inorganics, Inc., and was freshly sublimed, before use, to yield a bright yellow crystalline solid. Reagent grade tetrahydrofuran (Fisher Certified

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(1) Abstracted from the Ph.D. Thesis of W. M. Risen, Jr., Purdue University, 1966.

(2) Monsanto Predoctoral Fellow, 1965–1966.

(3) R. E. Winters and R. W. Kiser, *Inorg. Chem.*, **3**, 699 (1964).

(4) R. E. Winters and R. W. Kiser, *ibid.*, **4**, 157 (1965).

(5) R. E. Winters and R. W. Kiser, *J. Phys. Chem.*, **69**, 1618 (1965).

(6) R. E. Winters and R. W. Kiser, *J. Organometal. Chem.* (Amsterdam), **4**, 190 (1965).

(7) R. B. King, *J. Am. Chem. Soc.*, **88**, 2075 (1966).

(8) R. E. Winters and J. H. Collins, *J. Phys. Chem.*, **70**, 2057 (1966).

(9) W. Hieber and G. Wagner, *Z. Naturforsch.*, **12b**, 47 (1957); **13b**, 339 (1958).

(10) R. B. King, "Transition Metal Compounds," Vol. I, "Organometallic Synthesis," Academic Press Inc., New York, N. Y., 1965, p 158.

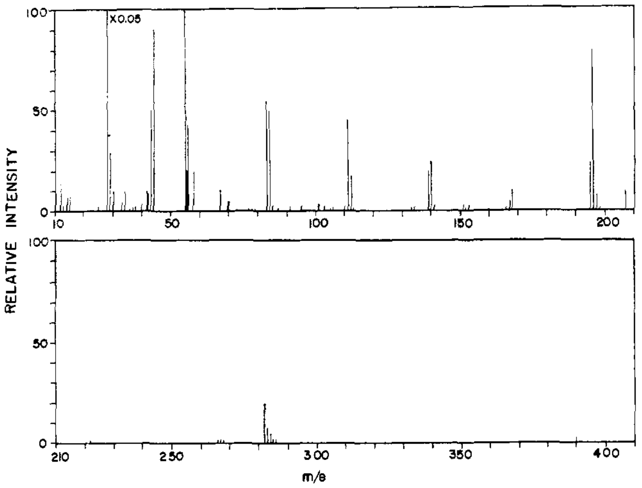

Figure 1. Mass spectrum of $HMn(CO)_5$ prepared by the H_3PO_4 method. The peaks of this spectrum are taken relative to the *m/e* 55 peak set at 100. The peak at *m/e* 28 has intensity 2000.

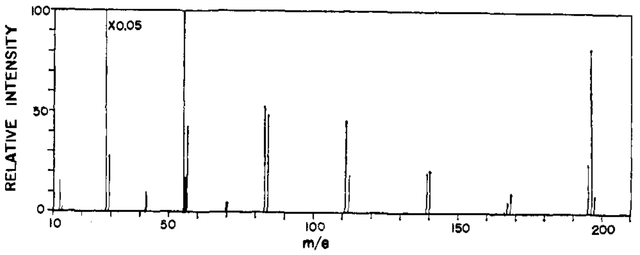

Figure 2. The mass spectrum of $HMn(CO)_5$. The peaks in the mass spectrum of $HMn(CO)_5$ are taken relative to the *m/e* 55 peak set at 100. The peak at *m/e* 28 has intensity 2000.

Reagent) was distilled under nitrogen. It was then refluxed for several days over freshly pressed sodium ribbon. The refluxed THF was then redistilled under N_2 from freshly pressed Na ribbon. This THF was vacuum deaerated. The sodium used was reagent grade. Whenever sodium was used, it was freshly pressed, as a shiny ribbon, directly into the reaction flask, all operations being carried out in a nitrogen atmosphere. The H_2SO_4 used was reagent grade, assay 96.7% (J. T. Baker Reagent Grade). Before use, it was partially deoxygenated by bubbling nitrogen through it for a period of about 15 min. It was then vacuum deaerated. HP grade nitrogen was used throughout, without further purification. Linde 4A molecular sieves were dried by placing them in a specially designed vessel and evacuating it. The flask was flamed with a cool flame, *in vacuo*, for about 1 hr to assure that the sieves were dehydrated and free of all volatiles. The phosphoric acid used was prepared by passing the vapors of deaerated H_2O over reagent grade P_2O_5 in a stream of nitrogen.

Preparation of $NaMn(CO)_5$. $Mn_2(CO)_{10}$ (5 g) was placed in a round-bottom flask fitted with two necks, each with vacuum-tight stopcocks. The flask was connected by one neck to a vacuum line, the other neck being fitted with a serum stopple. The flask was evacuated, being pumped on for about 2 min, to free the $Mn_2(CO)_{10}$ of any volatiles and trapped oxygen. Tetrahydrofuran, purified as described above, was then vacuum distilled onto the $Mn_2(CO)_{10}$ until, upon warming to room temperature, the volume of solution was 50 ml. Nitrogen was then admitted to the flask. The solution was transferred from this flask to a three-necked flask, fitted with serum stopples, containing 4 g of freshly pressed sodium ribbon under a nitrogen atmosphere. This transfer was effected by forcing the solution through a double-ended needle, one end through the stopple on one of the flask's three necks, and one through the stopple of the solution flask, by a positive pressure of nitrogen. The mixture of sodium and THF solution of $Mn_2(CO)_{10}$ was stirred for 4 to 8 hr. During this time the color changed from the golden color of the $Mn_2(CO)_{10}$ solution to the dark green color of the $NaMn(CO)_5$ solution. The reaction product was transferred to a dry, nitrogen-filled centrifuge tube, which was fitted with a serum stopple. It was then centrifuged for 1-2 hr. The centrifuged solution was then transferred *via* the double-ended needle, under nitrogen, through a glass frit into the $HMn(CO)_5$ preparation vessel.

Preparation of $HMn(CO)_5$. The reaction vessel for the preparation of $HMn(CO)_5$ consists of a round-bottom flask with three necks. One neck contains a vacuum stopcock and leads to a fine glass frit at the base of a 50-ml tube, the top of which is fitted with a serum stopple, through which the $NaMn(CO)_5$ solution is added to the vessel. A second neck leads to a vacuum line through a vacuum stopcock. The third neck is a ground-glass joint to which is joined the H_2SO_4-containing flask in which the acid was deaerated.

The sodium pentacarbonyl manganate(−I) solution was admitted, through the fine frit, into the reaction vessel, and the stopcock to the frit was closed. The THF solvent was vacuum distilled from the $NaMn(CO)_5$ into a receiver at −196°, without heating the solution. The salt was gently warmed by the air heat of a hair dryer, and evacuated to a high vacuum in order to remove complexed tetrahydrofuran molecules. The resulting solid $NaMn(CO)_5$ was nearly white.

Deaerated sulfuric acid (1.0 *M*) was added dropwise over a period of 1 hr. The evolved gases, $HMn(CO)_5$, H_2O, and very small amounts of THF were collected in an empty, evacuated trap which served as a bridge between the line in which the preparation took place and a second vacuum line used for the purification steps.

The gases collected were then distilled to a condensing finger which is built onto a 2-l. bulb, in which the treated 4A molecular sieves were contained. In a closed system within this bulb the gases were allowed to equilibrate with the sieves to remove the H_2O. Vapors above the sieves were collected quickly in a bulb at −76°. After distillation to a bulb at −20°, the $HMn(CO)_5$ was collected at −76° in a trap fitted with a manometer and flask for removal for mass spectral analysis.

$HMn(CO)_5$ was also prepared by the phosphoric acid method of Hieber and Wagner.[9,10] The sodium pentacarbonyl manganate(−I) was prepared, however, in the manner given above to avoid any spurious reaction with $Hg[Mn(CO)_5]_2$. Drying was effected using P_2O_5, and further purification by bulb-to-bulb distillation.

Mass Spectra. The mass spectra reported here were measured with an Hitatchi-Perkin-Elmer RMU-6A magnetic deflection mass spectrometer, at an ionizing potential of 75 ev, and with a Bendix Time of Flight mass spectrometer, at 70 ev. The spectrometers were completely cooled to room temperature, except for the electron source filaments, which raised the ionization chamber temperature to 40°. Samples were admitted to the instruments through gas leak systems. Spectra were calibrated with known mass standards. Peaks due to background and traces of THF were subtracted from the spectra of Figures 1 and 2.

Results and Discussion

The preparation of $HMn(CO)_5$ by the standard phosphoric acid method of Hieber and Wagner[9] was carried out as described by King,[10] with two noteworthy modifications. First, the preparation of the sodium salt of manganese pentacarbonyl, $NaMn(CO)_5$, did not involve mercury at any stage. King[11] has shown that $Hg[Mn(CO)_5]_2$ is isolated in 11.5% yield from a THF solution of $NaMn(CO)_5$ prepared by the reaction of sodium amalgam with $Mn_2(CO)_{10}$ in THF. Therefore, in the present preparation, the $NaMn(CO)_5$ used was prepared from Na ribbon according to the procedure described in the Experimental Section. Second, all operations commencing with the acidification of $NaMn(CO)_5$ were carried out *in vacuo*. The mass spectrum of the $HMn(CO)_9$ prepared by this literature method is given in Figure 1. This spectrum shows a number of peaks which do not belong to $HMn(CO)_5$. The intense peaks in the region *m/e* 282–286 and the less intense peaks at *m/e* 222 and 207 occur at *m/e* values higher than that of the parent ion $HMn(CO)_5^+$ which occurs at *m/e* 196. In addition, there are peaks at *m/e* 133, 67, 58, and 34, not readily assignable to singly or multiply charged fragments of $HMn(CO)_5$.

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[11] R. B. King, *J. Inorg. Nucl. Chem.*, **25**, 1296 (1963).

A number of possible sources of systematic error or introduction of impurities, other than by the preparation itself, have been carefully checked. They have been eliminated as the explanation for these extra peaks. Less intense peaks in the regions about *m/e* 222, 207, and 133 coupled with those in the region *m/e* 282–286 suggested the possibility of interference from stopcock grease. However, the impurity peaks simply do not correspond to the known spectrum of this grease.[12] The peak in the impurity spectrum at *m/e* 67 is of nearly the same intensity as that at *m/e* 282, whereas the grease spectrum contains no peak at *m/e* 67. The grease spectrum, however, does contain a peak at *m/e* 73 of intensity comparable to that of its *m/e* 281 peak. In the impurity spectrum, the *m/e* 73 peak is only about one-twentieth as intense as the *m/e* 282 peak. There is a moderate *m/e* 58 impurity peak, but none in the grease spectrum. Thus, it is clear that the impurity is not this stopcock grease. The presence of peaks at *m/e* 58, 43, and 15 suggests acetone as one possible impurity. However, acetone was not used at any stage of the cleaning of equipment or preparative procedure. Similarly, the presence of the *m/e* 44 peak suggests interference from CO_2. The vacuum techniques employed in the preparation and purification of $HMn(CO)_5$ should not have allowed admission of CO_2, and the rigorous exclusion of oxygen appears to rule out reaction of it with CO to form CO_2. Therefore, we cannot assign this peak to CO_2.

A careful review of the experimental and instrumental techniques has eliminated a number of possible interferences as the explanation for the extraneous peaks. A number of species can be postulated to explain the *m/e* 282–286 peaks. One such postulate would involve THF complexed in a substituted manganese carbonyl, and might be supported by the *m/e* 58 and 44 peaks. However, much more data are necessary to solve this problem; consequently, the nature of the species giving rise to the extraneous peaks is obscure.

The mass spectrum of $HMn(CO)_5$ prepared by the method of this paper, described in the Experimental Section, is given in Figure 2. It is immediately apparent that there are no peaks at *m/e* greater than that of the parent ion $HMn(CO)_5^+$. In addition, the peaks in the spectrum are all readily identifiable as peaks of $HMn(CO)_5^+$ and its fragments. Thus, the method of preparation is shown to yield pure $HMn(CO)_5$.

The mass spectrum of the $HMn(CO)_5$, prepared by the method of this paper, contains a series of pairs of peaks, those of highest *m/e* at 196 and 195, and the lowest at *m/e* 55 and 56. The individual peaks of each pair are 1 amu/e apart. The pairs are spaced at intervals of 28 amu/e, which is, of course, the mass of carbon monoxide. Therefore, we must consider the possibility that the higher peak of each pair is due to isotopic C^{13} in one or more of the CO ligands on the fragments. The natural abundance of C^{13} is 1.112%. It is clear from the relative intensities that the higher *m/e* peak of each pair is not due primarily to C^{13} in the molecule. Manganese is monoisotopic, Mn^{55} having a natural abundance of 100%. Therefore, the higher *m/e* peak of each pair cannot be an isotopic manganese peak. Clearly, then, the higher *m/e* peak of each pair is due to the hydrogen atom remaining on the $HMn(CO)_5$ and its fragments. These fragments are $HMn(CO)_n$, where $n = 0$–5. The lower peak of each pair is due to the analogous fragment without the hydrogen. Thus, the *m/e* 196 peak is that of the molecular ion $HMn(CO)_5^+$, while the other peak of the pair, *m/e* = 195, corresponds to $Mn(CO)_5^+$. Similarly, the peaks at *m/e* 168 and *m/e* 167 are for $HMn(CO)_4^+$ and $Mn(CO)_4^+$, respectively.

Several features of the observed spectrum remain to be discussed. The peaks at *m/e* 69.5 and 70 correspond to the doubly charged ions $Mn(CO)_3^{2+}$ and $HMn(CO)_3^{2+}$, respectively. In addition, the doubly charged ions $Mn(CO)^{2+}$ and $HMn(CO)^{2+}$ are observed at *m/e* 41.5 and 42.[13] The peak at *m/e* 55.5 is assigned to the doubly charged ion $Mn(CO)_2^{2+}$. Now, the existence of a peak at *m/e* 56 could be understood in terms of the species $HMn(CO)_2^{2+}$. However, the intensity of this *m/e* 56 peak is not what would be expected if the peak were due to $HMn(CO)_2^{2+}$ alone. Winters and Kiser[14] have recently shown that a direct relationship between the intensities of singly and doubly charged ions of metal carbonyls does exist. In the case of tungsten hexacarbonyl, the doubly charged species are approximately one-fourth to one-fifth as intense as the corresponding singly charged species. The intensities of the $HMn(CO)_3^{2+}$, $Mn(CO)_3^{2+}$, $Mn(CO)_2^{2+}$, $HMn(CO)^{2+}$, and $Mn(CO)^{2+}$ with respect to those of the analogous singly charged ions are in full accord with these findings. The *m/e* 55.5 peak for $Mn(CO)_2^{2+}$ is less than half the intensity of the *m/e* 111 peak. Now the *m/e* 111 peak is also about twice as great as the *m/e* 112 peak due to $HMn(CO)_2^+$. The peak due to $HMn(CO)_2^{2+}$ is expected to be much less intense than that due to $HMn(CO)_2^+$, but in fact it is not less but more intense than that peak. In addition, it is expected to be considerably less intense than the *m/e* 55.5 peak, but it is more than twice as intense as that peak. Therefore, we conclude that the major portion of the intensity of the *m/e* 56 peak cannot be assigned to $HMn(CO)_2^{2+}$.

We assign the major portion of the *m/e* 56 peak to the $Mn{-}H^+$ fragment. This assignment is supported by the appearance of the series of pairs of peaks corresponding to $HMn(CO)_n^+$ and $Mn(CO)_n^+$ with $n = 1$–5 and represents the case for $n = 0$. This observation of the MnH^+ fragment is direct evidence for a Mn–H bond in $HMn(CO)_5$.

The complete mass spectral assignment for $HMn(CO)_5$ is given in Table I.

It is clear that the fragmentation of $HMn(CO)_5$, upon ionization in the mass spectrometer, can be thought of as a stepwise process. However, the spectra taken at 70- and 75-ev ionizing potential are not sufficient to elucidate the mechanism of this process. The mass spectra of similar metal carbonyls have been analyzed by Winters and Kiser[3–6] and generally show such a stepwise fragmentation for metal carbonyls. It is concluded, therefore, that in $HMn(CO)_5$, also, the series of peaks observed results from loss of neutral CO molecules in a stepwise manner. Now the stepwise fragmentation of $HMn(CO)_5$, with both the $HMn(CO)_n^+$ and $Mn(CO)_n^+$ peaks evident, indicates that

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[12] K. Biemann, "Mass Spectrometry, Organic Chemical Applications," McGraw-Hill Book Co., Inc., New York, N. Y., 1962, p 172.

[13] The peak for $HMn(CO)_4^{2+}$, if present, is obscured by the intense $HMn(CO)^+$ peak at *m/e* 84.

[14] R. E. Winters and R. W. Kiser, *J. Phys. Chem.*, 70, 1680 (1966).

Table I. Identity of the Principal Peaks in the Mass Spectrum of $HMn(CO)_5$

m/e	Identity
197	$HMn(CO)_4(^{13}CO)^+$
196	$HMn(CO)_5^+$
195	$Mn(CO)_5^+$
168	$HMn(CO)_4^+$
167	$Mn(CO)_4^+$
140	$HMn(CO)_3^+$
139	$Mn(CO)_3^+$
112	$HMn(CO)_2^+$
111	$Mn(CO)_2^+$
84	$HMn(CO)^+$
83	$Mn(CO)^+$
70	$HMn(CO)_3^{2+}$
69.5	$Mn(CO)_3^{2+}$
56	HMn^+, $HMn(CO)_2^{2+}$
55.5	$Mn(CO)_2^{2+}$
55	Mn^+
42	$HMn(CO)^{2+}$
41.5	$Mn(CO)^{2+}$
29	$^{13}CO^+$
28	CO^+
13	$^{13}C^+$
12	$^{12}C^+$

the loss of hydrogen and of neutral CO fragments are competitive processes.

The infrared spectra of $HMn(CO)_5$ and $DMn(CO)_5$ have been discussed by several investigators.[15–18] While no complete vibrational assignment has been made, Edgell, *et al.*,[15] and Cotton, *et al.*,[16] have identified the bands at 1783 cm^{-1} in the hydride and 1287 cm^{-1} in the deuteride as being due to a vibration primarily MnH stretching in character. From this evidence for a MnH bond, Edgell, *et al.*,[15] have estimated that the Mn–H stretching force constant is about 1.87 mdynes/A. Force field calculations on a number of related metal carbonyls show that M–C stretching force constants fall in the range 2.0–2.7 mdynes/A. Thus, the presence of a Mn–H bond and a competitive loss of H and CO fragments from $HMn(CO)_5$ are in keeping with conclusions from infrared investigations.

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(15) W. F. Edgell, G. Asato, W. Wilson, and C. Angell, *J. Am. Chem. Soc.*, **81**, 2022 (1959).

(16) F. A. Cotton, J. L. Down, and G. Wilkinson, *J. Chem. Soc.*, 833 (1959).

(17) W. E. Wilson, *Z. Naturforsch.*, **13b**, 349 (1958).

(18) D. K. Huggins and H. D. Kaesz, *J. Am. Chem. Soc.*, **86**, 2784 (1964).

Infrared Spectra and Normal Coordinate Analysis of Metal–Olefin Complexes. I. Zeise's Salt Potassium Trichloro(ethylene)platinate(II) Monohydrate[1]

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Abstract: To date the majority of infrared studies on platinum–olefin complexes have been limited to the NaCl region and particularly to the C═C stretching vibration. The present study was undertaken primarily to assign the platinum–olefin stretching vibration and to estimate the relative strength of the platinum–olefin bond in terms of force constants. For this purpose we have measured the infrared spectra of Zeise's salt, $K[Cl_3Pt(C_2H_4)]\cdot H_2O$, and its deuterio analog, $K[Cl_3Pt(C_2D_4)]\cdot H_2O$, from 4000 to 33 cm^{-1}, and have carried out approximate normal coordinate analysis for coordinated ethylene of V_h symmetry and for the remaining square-planar complex ion, in which the ethylene molecule is treated as a single atom having the mass of C_2H_4 or C_2D_4. The results indicate that the platinum–olefin stretching band is at 407 cm^{-1} (nondeuterated compound) and the corresponding force constant is 2.23 mdynes/A including repulsive force constants between nonbonded atoms. It has also been found that the C═C stretching vibration is coupled with the in-plane CH_2 scissoring vibration. This result suggests that the lowering of the C═C stretching frequency upon coordination can be used only as a qualitative measure of the strength of the platinum–olefin bond.

The infrared spectrum of Zeise's salt, $K[Cl_3Pt(C_2H_4)]\cdot H_2O$,[2a] has been observed by many investigators.[3,4] Chatt and Duncanson[3a] observed the infrared spectra of Zeise's salt and the dimer, $[Cl_2Pt(C_2H_4)]_2$,[2b] from 4000 to 650 cm^{-1}. From a comparison of the C–H stretching frequencies in saturated and

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[2] (a) Potassium trichloro(ethylene)platinate(II) monohydrate; (b) *sym-trans*-di-*μ*-chloro-dichlorobis(ethylene)diplatinum(II); (c) *sym-trans*-di-*μ*-chloro-dichlorobis(ethylene)dipalladium(II); (d) *trans*-dichloro(ammine)(ethylene)platinum(II).

[3] (a) J. Chatt and L. A. Duncanson, *J. Chem. Soc.*, 2939 (1953); (b) H. B. Jonassen and J. E. Field, *J. Am. Chem. Soc.*, **79**, 1275 (1957); (c) D. B. Powell and N. Sheppard, *Spectrochim. Acta*, **13**, 69 (1958).

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