This method has the advantages of relatively simple manipulations and no apparent risk of overreaction to form $SeCl_4$ in contrast to the reported[10] reaction of SeO_2 with $SOCl_2$.

B. $SeCl_4$ Reactions. Reactions of $SeCl_4$ with secondary amines are complicated by difficulties in separating the selenium products from the dialkylammonium chloride byproducts. Accordingly, the substitution of chlorine atoms in $SeCl_4$ with dimethylamido groups was investigated by using Me_2NSiMe_3. Thus reaction of $SeCl_4$ with excess Me_2NSiMe_3 was found to give a white solid, which is sparingly soluble in tetrahydrofuran, is much more resistant toward hydrolysis than the other (dialkylamido)selenium derivatives and analyses for $(Me_2N)_3SeCl$. This product is formulated as the tris(dimethylamido)selenonium salt $[(Me_2N)_3Se]^+Cl^-$ containing a cation analogous to that in $[(Me_2N)_3S]^+[BF_4]^-$ obtained from the reaction of SF_4 with $(Me_2N)_3B$.[15] Reaction of $SeCl_4$ with a smaller amount of Me_2NSiMe_3 gave a more soluble and much more hydrolysis-sensitive yellow solid analyzing for $(Me_2N)_2SeCl_2$, which could either be a tetracoordinate nonionic selenium(IV) derivative or an ionic selenonium salt $[(Me_2N)_2SeCl]^+Cl^-$. Unfortunately, neither of these (dimethylamido)selenium(IV) chlorides formed crystals that were suitable for resolving these structural uncertainties by X-ray crystallography. Furthermore, these compounds were insufficiently soluble in nonreactive ionizing solvents for meaningful conductivity measurements.

C. SeO_2 Reactions. The ultimate hydrolysis products of (dialkylamido)selenium(IV) derivatives are the dialkylammonium salts $[R_2NH_2]_2[Se_2O_5]$. A number of authentic samples of such salts (Table I) were prepared by reactions of SeO_2 with excess secondary amine in order to provide information on their NMR spectra and other properties for comparison with hydrolysis products. The products, although ionic, did not exhibit high conductivities in nitromethane since they are salts of the weakly basic dialkylamines with the weakly acidic H_2SeO_3 ($K_1 \sim 3.5 \times 10^{-3}$)[16] and thus in solution dissociate into the un-ionized free amine and free selenious acid. Ionic formulations are supported by the Nujol mull infrared spectrum of the diisopropylammonium derivative, which exhibited relatively strong bands at 865, 808, and 620 cm^{-1} close to bands found[17] in authentic $HSeO_3^-$ salts (e.g., strong bands at 875, 845, 825, 790, 615, and 605 cm^{-1} in solid $NaHSeO_3$). The selenium-77 NMR spectra of the products listed in Table I all exhibit a single resonance at δ 1369 ± 5, indicating the same selenium species in each of the products. The proton NMR spectra of the products in Table I exhibit N–H resonances in the range δ 9.3–9.8 in addition to the expected alkyl resonances. These $[R_2NH_2]_2[Se_2O_5]$ salts are reasonably soluble in chlorinated organic solvents such as CH_2Cl_2 and $CHCl_3$ undoubtedly owing to the effects of the two organic cations as well as the weak dissociation of these salts of weak acids with weak bases.

Acknowledgment. We are indebted to the National Science Foundation for partial support of this work under Grant CHE-82-12166.

Registry No. $SeOCl_2$, 7791-23-3; SeO_2, 7446-08-4; Me_3SiCl, 75-77-4; Me_2NH, 124-40-3; $(Me_2N)_2SeO$, 2424-09-1; $(Me_2N)_2S$, 2424-06-8; $(Ph_2P)_2Se$, 109243-13-2; $(Bu_2P)_2Se$, 109243-14-3; $((Me_2N)_2P)_2Se$, 109243-15-4; *i*-$Pr_2Se(O)Cl$, 109243-16-5; $(i\text{-}Pr)_2NH$, 108-18-9; *i*-Pr_2NSiMe_3, 17425-88-6; *meso*-$[i\text{-}Pr_2NSe(O)]_2O$, 109243-17-6; (±)-$[i\text{-}Pr_2NSe(O)]_2O$, 109243-18-7; $(i\text{-}PrN)_2Se$, 109243-19-8; $(OC_4H_8N)_2Se$, 109243-20-1; $SeCl_4$, 10026-03-6; $(Me_2N)_3SeCl$, 109243-23-4; $(Me_2N)_2SeCl_2$, 109243-24-5; $[Me_2NH_2][Se_2O_5]$, 2881-47-2; $[Et_2NH_2]_2[Se_2O_5]$, 44594-94-7; $[n\text{-}Pr_2NH_2]_2[Se_2O_5]$, 109243-21-2; $[i\text{-}Pr_2NH_2]_2[Se_2O_5]$, 109243-22-3; Et_2NH, 109-89-7; Pr_2NH, 142-84-7; morpholinotrimethylsilane, 13368-42-8; (dimethylamino)trimethylsilane, 2083-91-2.

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Gas-Phase Structure of $AsCl_2F_3$ and a Vibrational Analysis of $AsCl_nF_{5-n}$ (n = 1–5)

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After preparing the very unstable $AsCl_5$ by photolytic low-temperature chlorination of $AsCl_3$,[1] we recently succeeded in synthesizing the entire series of mixed chlorofluoroarsoranes from $AsCl_4^+AsF_6^-$.[2–6] These arsoranes are significantly less stable than the homologous phosphoranes, whose gas-phase structures and force fields have been reported recently in this journal.[7] An independent electron diffraction study has also recently been reported for PCl_2F_3.[8] $AsCl_4F$ forms upon the pyrolysis of $AsCl_4^+AsF_6^-$ and can be solidified on cooled surfaces below −196 °C, after a short distance of flight. Because of its thermal instability the compound is not condensable.[5] The byproduct AsF_5 is separated in a reaction with activated KF.[9]

The primary pyrolysis products undergo halogen-exchange reactions during longer times of flight to form $AsCl_2F_3$ and $AsClF_4$.[2,3] There are no indications that the postulated intermediary species $AsCl_3F_2$ forms. This arsorane is accessible, however, by a gas-phase/solid reaction of $AsCl_2F_3$ and $CaCl_2$ and may be characterized by vibrational spectroscopy below −196 °C.[4] $AsCl_2F_3$ and $AsClF_4$ decompose in their liquid phases at temperatures above −75 and −90 °C, respectively, to give mainly $AsCl_4^+AsF_6^-$,[2] but $AsCl_2F_3$ is recondensable at its melting point, thus allowing an investigation of its gas-phase structure by electron diffraction. Analysis of the radial distribution curve (Figure 1) confirms the conformation predicted by the VSEPR theory[10] or the electronegativity rule,[11] i.e., C_{2v} symmetry with equatorial positions of the two chlorine atoms. The geometry of $AsCl_2F_3$ is characterized by five geometric parameters that are well-determined by the six "characteristic features" (peaks or shoulders) of the radial distribution curve. A diagonal-weight matrix has been applied to the intensities in the least-squares analysis, and the scattering amplitudes and phases of Haase[12] are used. The ratio of axial and equatorial As–F vibrational amplitudes is constrained to the spectroscopic value. The five independent geometric parameters and seven vibrational amplitudes have been refined simultaneously. Four correlation coefficients have values larger than $|0.6|$: AsF_{eq}/AsF_{ax}, 0.89; $AsF_{eq}/F_{ax}AsF_{eq}$, −0.66; $AsF_{ax}/l(AsF)$, −0.80; $AsF_{eq}/l(AsF)$, 0.90. The results of the least-squares analysis are summarized in Table I. The r_a distances have been converted to r_g values by using the experimental vibrational amplitudes.

The angular distortions from the ideal trigonal bipyramid are small. The angles $F_{ax}AsF_{eq}$ and $Cl_{eq}AsCl_{eq}$ deviate by less than 1° from the ideal angles of 90 and 120°, respectively. The equatorial As–F bond in $AsCl_2F_3$ (As–F_{eq} = 166.3 (7) pm) appears to be slightly longer than in AsF_5 (As–F_{eq} = 165.6 (4) pm),[13] but the difference (Δ(As–F_{eq}) = 0.7 (8) pm) is within the combined experimental uncertainties. The axial As–F bonds lengthen by 1.2 (7) pm upon substitution of two equatorial fluorines by chlorines (As–F_{ax} = 171.1 (5) and 172.3 (4) pm in AsF_5[13] and $AsCl_2F_3$, respectively). These trends in the As–F bond lengths are equal to those observed in the analogous phosphoranes: Δ(P–F_{eq}) = 0.6 (7) pm and Δ(P–F_{ax}) = 1.3 (5) pm.[7] No gas-phase values for As(V)–Cl bond lengths are known in the literature for comparison.

Valence force fields for the chlorofluoroarsoranes $AsCl_nF_{5-n}$ with n = 1–5 were determined with the program NORCOR[14] from

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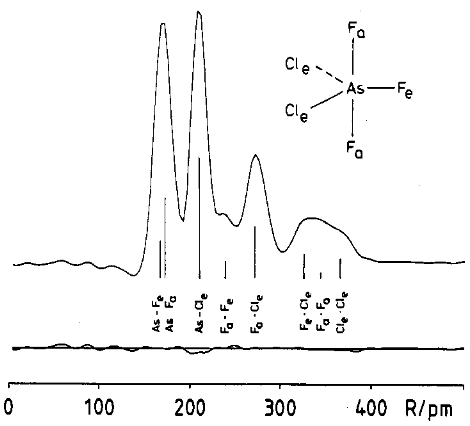

Figure 1. Experimental radial distribution function and difference curve.

Table I. Geometric Parameters (r_g Values in pm and deg)[a] and Vibrational Amplitudes from Electron Diffraction (ed)[b] and Spectroscopic Data (sp) for $AsCl_2F_3$

	r_g	l(ed)	l(sp)
$As-F_{eq}$	166.3 (7)	5.0 (6)[c]	4.1
$As-F_{ax}$	172.3 (4)	5.2 (6)[c]	4.3
$As-Cl_{eq}$	209.7 (3)	5.6 (2)	5.1
$F_{ax}\cdots F_{eq}$	237.8 (8)[d]	5.4 (8)	7.8
$F_{ax}\cdots Cl_{eq}$	272.3 (6)[d]	9.4 (4)	8.6
$F_{eq}\cdots Cl_{eq}$	325.9 (9)[d]	10.9 (12)	12.9
$F_{ax}\cdots F_{ax}$	344.6 (6)[d]	5.9 (31)	5.8
$Cl_{eq}\cdots Cl_{eq}$	364.3 (11)[d]	11.9 (16)	14.2
$F_{ax}AsF_{eq}$	89.2 (2)		
$Cl_{eq}AsCl_{eq}$	120.6 (5)		

[a] Error limits are 2σ values and include a possible scale error of 0.1% for bond lengths. [b] Error limits are 3σ values. [c] Ratio constrained to spectroscopic value. [d] Dependent geometry parameter.

Table II. Diagonal Force Constants (10^2 N/m) of the Arsoranes $AsCl_nF_{5-n}$ (n = 0–5)[a]

	AsF_5[b]	$AsClF_4$	$AsCl_2F_3$	$AsCl_3F_2$	$AsCl_4F$	$AsCl_5$
$f(AsF_{ax})$	4.51	4.21	3.91	3.61	3.48	
$f(AsF_{eq})$	5.47	5.13	4.81			
$f(AsCl_{ax})$					1.92	1.81
$f(AsCl_{eq})$		2.83	2.60	2.33	2.29	2.30
$f(F_{ax}AsF_{eq})$	1.96	1.79	1.77			
$f(F_{ax}AsCl_{eq})$		1.84	1.80	1.77	1.73	
$f(Cl_{ax}AsCl_{eq})$					1.46	1.51
$f(F_{eq}AsF_{eq})$	0.22	0.21				
$f(F_{eq}AsCl_{eq})$		0.22	0.22			
$f(Cl_{eq}AsCl_{eq})$			0.19	0.18	0.17	0.17

[a] Values for the off-diagonal constants can be obtained on request from R.M. [b] From ref 15.

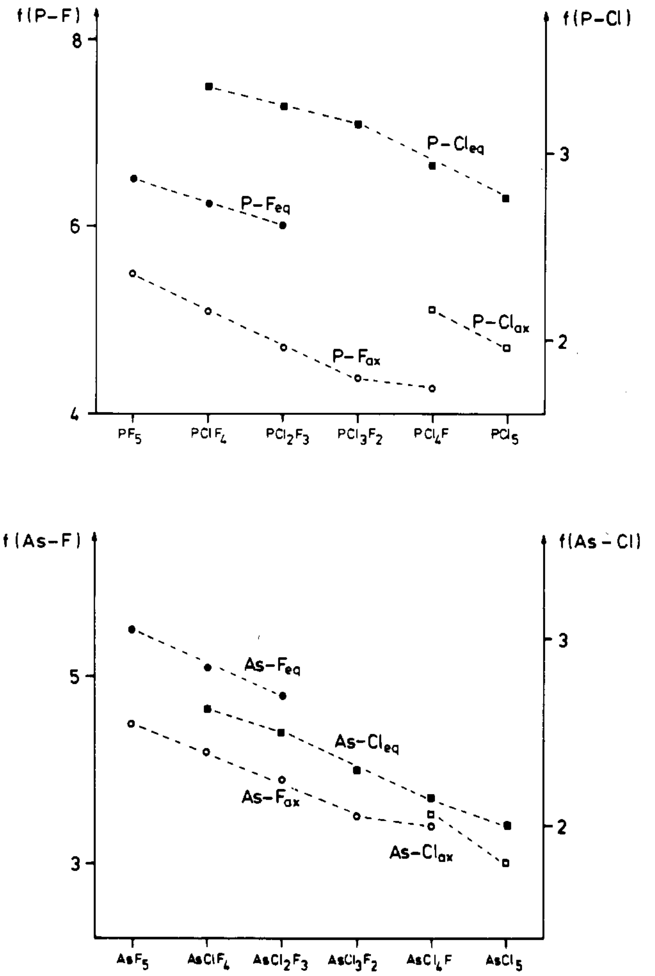

Figure 2. Stretching force constants in PCl_nF_{5-n}[7] (top) and $AsCl_nF_{5-n}$ (bottom) (n = 0–5).

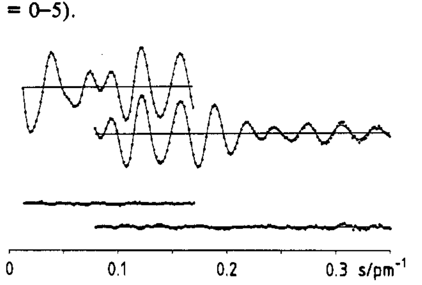

Figure 3. Experimental (···) and calculated (—) molecular intensities, $sM(s)$, and differences.

IR-matrix or Raman frequencies reported in the literature.[2–5] In all cases the mean deviation between experimental and calculated frequencies is smaller then 1 cm^{-1}. The diagonal force constants are listed in Table II. The variations of the As–F and As–Cl stretching force constants (Figure 2) are very similar to those for the homologous phosphoranes. This observation indicates trends of equatorial and axial bond lengths similar to those determined for the analogous phosphorane series.[7]

Experimental Section

$AsCl_2F_3$ has been isolated at −80 °C after pyrolysis of $AsCl_4^+AsF_6^-$.[3] In the ^{19}F NMR spectrum (solvent SO_2ClF, external standard $CFCl_3$) at −90 °C only one signal is observed at −44.4 ppm (half-width 98 Hz), which is slightly shifted from that of AsF_5 at −49.8 ppm (half-width 44 Hz in SO_2ClF, −90 °C).[16] The ^{75}As NMR spectrum shows a signal at +205 ppm with a half-width of 5000 Hz (solvent $CFCl_3$, external standard $KAsF_6$, T = −90 °C). Vapor pressures were recorded in the temperature range of −70 to −30 °C: $\log p = 7.035 - 1385/T$ (p in torr).

The electron diffraction intensities were recorded with a Balzers KD-G 2 Gas Diffractograph[17] at two camera distances (25 and 50 cm) with an

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accelerating voltage of about 60 kV. The sample was kept at -40 °C and the inlet system and nozzle were at room temperature. The camera pressure never exceeded 5×10^{-6} Torr during the experiment. Two plates for each camera distance were analyzed by the usual procedures.[18] The averaged molecular intensities in the ranges 0.014-0.17 and 0.08-0.35 pm^{-1} are presented in Figure 3.

Acknowledgment. Financial support by the Fonds der Chemischen Industrie is gratefully acknowledged.

Registry No. AsF_5, 7784-36-3; $AsClF_4$, 94649-43-1; $AsCl_2F_3$, 109278-32-2; $AsCl_3F_2$, 95036-82-1; $AsCl_4F$, 87198-15-0; $AsCl_5$, 22441-45-8.

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Cationic Iridium(I) Complexes of Unsaturated Aldehydes and Their Catalytic Activities

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The iridium complexes of unsaturated nitriles $[Ir^{I}L(CO)(PPh_3)_2]ClO_4$ and $[Ir^{III}(H)_2L(CO)(PPh_3)_2]ClO_4$ (L = unsaturated nitriles) have been prepared and investigated with respect to their catalytic activities for the hydrogenation and isomerization of L.[1] The rhodium(I) analogues $[RhL(CO)(PPh_3)_2]ClO_4$ have also been prepared and studied for L being unsaturated nitriles[2] and unsaturated aldehydes.[3]

In this paper, we wish to report the synthesis of iridium(I) complexes of unsaturated aldehydes, [Ir(*trans*-$CH_3CH{=}CHCHO)(CO)(PPh_3)_2]ClO_4$ (**1**) and [Ir(*trans*-$C_6H_5CH{=}CHCHO)(CO)(PPh_3)_2]ClO_4$ (**2**), and their catalytic activities for the hydrogenation of *trans*-$CH_3CH{=}CHCHO$ and *trans*-$C_6H_5CH{=}CHCHO$, respectively. Catalytic hydrogenation of $CH_2{=}CHCHO$ with $Ir(ClO_4)(CO)(PPh_3)_2$ (**3**) is also included in this report.

Complexes **1** and **2** have been prepared according to eq 1. Attempts have been made to prepare a $CH_2{=}CHCHO$ complex of iridium without success, although the rhodium complex, $[Rh(CH_2{=}CHCHO)(CO)(PPh_3)_2]ClO_4$, was previously prepared.[2]

$$\underset{\mathbf{3}}{Ir(ClO_4)(CO)(PPh_3)_2} + L \rightarrow [IrL(CO)(PPh_3)_2]ClO_4 \qquad (1)$$

1, L = *trans*-$CH_3CH{=}CHCHO$
2, L = *trans*-$C_6H_5CH{=}CHCHO$

Experimental Section

Caution. Perchlorate salts of transition-metal complexes containing organic ligands are potentially explosive.[4]

Materials. $Ir(ClO_4)(CO)(PPh_3)_2$ was prepared by the literature method.[5] $CH_2{=}CHCHO$ and *trans*-$CH_3CH{=}CHCHO$ (Aldrich) were distilled before use. *trans*-$C_6H_5CH{=}CHCHO$ (Aldrich) was used as purchased.

Physical Measurements. Electronic and infrared adsorption spectra were obtained with Shimadzu UV-240 and Shimadzu IR-440 instruments. ^1H NMR spectra were recorded on a Varian 60-MHz instrument (Model EM-360A). Conductance measurements were performed in the corresponding aldehydes with a Wiss-Tech LBR conductivity meter at 25 °C under nitrogen. Elemental analyses were carried out by Spang Microanalytical Laboratory, Eagle Habor, MI.

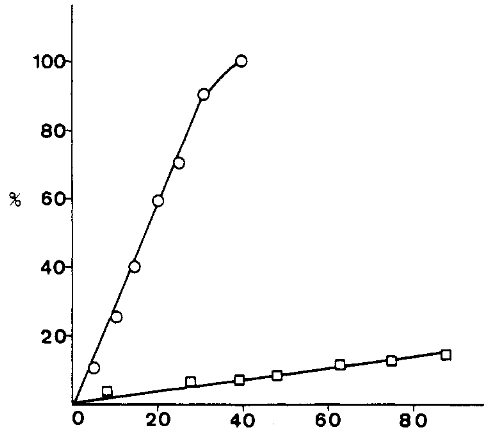

Figure 1. Hydrogenation of $CH_2{=}CHCHO$ (37 mmol) to CH_3CH_2CHO with $Ir(ClO_4)(CO)(PPh_3)_2$ (**3**) (0.1 mmol) (○) and of *trans*-$CH_3CH{=}CHCHO$ (37 mmol) to $CH_3CH_2CH_2CHO$ with [Ir(*trans*-$CH_3CH{=}CHCHO)(CO)(PPh_3)_2]ClO_4$ (**1**) (0.1 mmol) (or **3**, 0.1 mmol) (□) at 25 °C under hydrogen (P_{H_2} + vapor pressure of the solution = 1 atm).

Preparation. Standard vacuum-line and Schlenk-type glassware were used in handling metal complexes. Complexes **1** and **2** are stable in the solid state in air and in solution under nitrogen at 25 °C.

[Ir(*trans*-$CH_3CH{=}CHCHO)(CO)(PPh_3)_2]ClO_4$ (1). Addition of *trans*-$CH_3CH{=}CHCHO$ (1 mL, ca. 12 mmol) to a benzene solution (35 mL) of **3** (0.42 g, 0.50 mmol) under nitrogen at 25 °C immediately resulted in yellow crystals, which were collected by filtration, washed with benzene (20 mL), and dried under vacuum; yield 0.36 g (79% based on **1**). Anal. Calcd for $IrC_{41}H_{36}O_6ClP_2$: C, 53.86; H, 3.97; Cl, 3.88; P, 6.78. Found: C, 53.71; H, 3.89; Cl, 3.99; P, 6.92. Molar conductance = 105 ohm^{-1} cm^2 mol^{-1} ([Ir] = 5.0×10^{-5} M in *trans*-$CH_3CH{=}CHCHO$).

[Ir(*trans*-$C_6H_5CH{=}CHCHO)(CO)(PPh_3)_2]ClO_4$ (2). This compound was prepared in the same manner described for **1**. A 1-mL quantity (ca. 8.0 mmol) of *trans*-$C_6H_5CH{=}CHCHO$ and 0.42 g (0.50 mmol) of **3** were used; yield 0.40 g (82% based on **2**). Anal. Calcd for $IrC_{46}H_{38}O_6ClP_2$: C, 56.59; H, 3.92; Cl, 3.63; P, 6.34. Found: C, 57.01; H, 3.89; Cl, 3.71; P, 6.27. Molar conductance = 100 ohm^{-1} cm^2 mol^{-1} ([Ir] = 5.0×10^{-5} M in *trans*-$C_6H_5CH{=}CHCHO$).

Hydrogenation of $CH_2{=}CHCHO$ with $Ir(ClO_4)(CO)(PPh_3)_2$ (3). A 37-mmol sample of $CH_2{=}CHCHO$ and 0.1 mmol of **3** in a 25-mL round-bottomed flask were stirred at 25 °C under hydrogen (P_{H_2} + vapor pressure of the solution = 1 atm). A part (ca. 0.5 mL) of the reaction mixture was taken out and analyzed by ^1H NMR spectroscopy at intervals. Disappearance of $CH_2{=}CH$ signals of $CH_2{=}CHCHO$ at 5.5-6.1 ppm (or appearance of CH_3 signals of CH_3CH_2CHO at 0.7 ppm) was follwed to obtain the data given in Figure 1.

Hydrogenation of *trans*-$CH_3CH{=}CHCHO$ with [Ir(*trans*-$CH_3CH{=}CHCHO)(CO)(PPh_3)_2]ClO_4$ (1) (or $Ir(ClO_4)(CO)(PPh_3)_2$ (3)). This reaction was carried out in the same manner described above, using 37 mmol of *trans*-$CH_3CH{=}CHCHO$ and 0.1 mmol of **1** (or **3**). After a certain period of time, the volatile materials were separated from the nonvolatile materials by using a dry ice/acetone bath trap. *trans*-$CH_3CH{=}CHCHO$ and $CH_3CH_2CH_2CHO$ in the volatile materials were analyzed by ^1H NMR spectroscopy. The nonvolatile materials (oily oligomers of *trans*-$CH_3CH{=}CHCHO$ and iridium complex) were dissolved in $CDCl_3$ and analyzed by ^1H NMR spectroscopy.

Bonding between Iridium and Unsaturated Aldehydes

Spectral data suggest that *trans*-$CH_3CH{=}CHCHO$ in **1** and *trans*-$C_6H_5CH{=}CHCHO$ in **2** are coordinated through the oxygen atom to iridium. It is well-known that the related four-coordinated iridium(I) complexes, $IrA(CO)(PPh_3)_2$ (A = monodentate ligand coordinated through oxygen and nitrogen atom), show the three bands very similar to those for **1** and **2** (Table I) in the visible region.[1,6] With the exception of $[Ir(C_2H_4)_2$-

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