kanenitrile oxide bound to transition metals.[13] It appears that in **2a** the nitrogen is attached in a σ fashion to Co1, as is C16 to Co2. The bonding to Co3 is best described as a π-type interaction with the CN double bond. The O–N–C_{16}–C_{17} atoms define an approximate plane, Co1 and -2 located closer (0.32 and 0.53 Å, respectively) to it than Co3 (1.57 Å and on the opposite side). The carbyne half of the remaining cluster has been left relatively unchanged. The structure **2a** is reminiscent of that of related (CpM)$_3$alkyne(CO) complexes[14] and $Fe_3(CO)_9(CH_3CN)(H)$.[15] As will be noted from the examples **1b–d** → **2b–d**, the insertion is *completely* regiospecific, apparently seeking out the more electron-rich metal–carbon bond.

A highly unusual transformation ensued when attempts were made to deprotonate either **2a** or **2b** ($CH_3CH_2CH_2CH_2Li$ or $NaOCH_3$). An isomer was formed in either case, **3a** (37%) and **3b** (41%) on the basis of MS (*m/e* for M^+ and M^+ – alkyne) and elemental analysis. A strong IR peak at 1420 cm^{-1} suggests the presence of a bridging NO ligand,[11] the ^{1}H NMR spectrum of **3a** reveals two *equivalent* butyl groups and that of **3b** an upfield shift of the original carbyne hydrogen (δ 23.13) to 11.73 pm. The Cp's give sharp signals even at -80 °C. The $^{13}C\{^1H\}$ NMR spectra exhibit peaks typical[16] for those of σ,π-coordinated alkynes (**3a** δ 172.9, **3b** δ 176.7, 139.7) and the IR stretch at 1385 cm^{-1} (seen only for **3b**) is that expected for a cationic (CpCo)$_3$alkyne.[17] On the basis of these data we assign the structure **3** shown for these molecules, the third example[8a,b] of carbyne–carbyne coupling in **1**, occurring under exceedingly mild conditions and involving simultaneous NO deinsertion.

It is interesting to note that both the C–N (1.362 Å) as well as the N–O bond length in **2a** (1.280 Å) are *identical* with the analogous bonds in pyridine *N*-oxide.[18] Since the latter is readily deoxygenated,[19] it became of interest to determine the potential of **2** in oxidation reactions. Indeed, when exposed to CO (10 atm) both **2a** and **2b** converted to two new compounds **4a** (38%) and **4b** (50%), respectively,[10] to which, on the basis of spectral data, we assign the structures of their nitrile analogues μ_3-η^1-CR^2-$(CpCo)_3(R^1CN)^+BF_4^-$. Significantly, the IR spectra reveal the absence of NO and CO absorptions (but strong signals for BF_4^-, 1000–1150 cm^{-1})[20] and the ^{1}H and ^{13}C NMR spectra show that reaction must have occurred at the side of the nitrile oxide ligand. For example, the carbyne hydrogen in **4b** resonates at 22.70 ppm, the corresponding carbon at 373.9 ppm. A ^{13}C peak at 204.7 is assigned to the nitrile carbon. Because suitable crystals for X-ray could not be grown, we are not sure about the exact mode of coordination of the R^1CN group, but it is very likely similar to the iron system studied by Kaesz et al.[15] Finally, preliminary results show that both **2a** and **3a** react with H_2 (or D_2, 10 atm) in the presence of catalytic Pd (10% on C) to give new difficult to characterize salts, with spectral data suggesting hydrogenolysis of the N–O bond [from **2a**: IR ν_{N-H} 3279 cm^{-1}, ν_{N-D} 2438 cm^{-1}, ν_{BF_4} 1000–1150 (vs, br) cm–^{1}]. A precise structural analysis will have to await the outcome of experiments employing more readily crystallizable systems.

There is considerable current effort to understand the heterogeneously catayzed reduction of NO by H_2 and/or CO,[21] which, under some condition, furnishes C–H bonded products. This observation, in conjunction with the implication of surface carbynes in Fischer–Tropsch catalysis,[22] suggests potential relevance of the chemistry reported here to heterogeneously catalyzed processes.

Acknowledgment. This work was supported by the NSF-CHE 8504987. The crystal structure analyses were carried out by Dr. F. J. Hollander, staff crystallographer on the U.C. Berkeley, Department of Chemistry, X-ray facility (CHEXRAY). K.P.C.V. is a Miller Professor in residence, M.W. was the recipient of a Deutscher Akademischer Austauschdienst Postdoctoral Fellowship (1984–1985).

Supplementary Material Available: Complete spectral data on compounds **2c–e**, **3a**, and **4a**, elemental analyses on the new compounds **1b–d**, **2a–e**, and **3a,b**, a listing of positional and thermal parameters and tables of bond lengths and angles of **2a,b**, and an ORTEP drawing of **2b** (17 pages). Ordering information is given on any current masthead page.

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Rhodium–Rhodium Stretching Frequencies in ^{16}O, ^{18}O, and CD_3 Derivatives of $Rh_2(O_2CCH_3)_4(PPh_3)_2$

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The assignment of rhodium–rhodium stretching frequencies, ν(RhRh), in dirhodium tetracarboxylate complexes has long been controversial. The initial indications of San Filippo and Sniadoch,[2] subsequently supported by Kharitonov et al.[3] and Kireeva et al.,[4] were in favor of the range 150–170 cm^{-1} for ν(RhRh) in a variety of complexes of the type $Rh_2(O_2CR)_4L_2$ (L = axial ligand). On the other hand Ketteringham and Oldham,[5] who also studied a range of complexes of this sort, favored the range 288–351 cm^{-1} for ν(RhRh). Recent Raman and extensive electronic spectral analyses by Miskowski et al.[6] on $Rh_2(O_2CCH_3)_4(H_2O)_2$ and $Li_2Rh_2(O_2CCH_3)_4Cl_2{\cdot}8H_2O$ were based on the lower value for ν(RhRh). Since in our view there was no firm evidence one way or the other regarding the correct wavenumber region of ν(RhRh) in dirhodium tetracarboxylate complexes, we undertook detailed electronic, infrared, Raman, and resonance Raman studies of $Rh_2(O_2CCH_3)_4(PPh_3)_2$, as its ^{16}O, ^{18}O, and CD_3 variants, in order to resolve this problem definitively in one case at least. The choice of triphenylphosphine as axial ligand is advantageous for resonance

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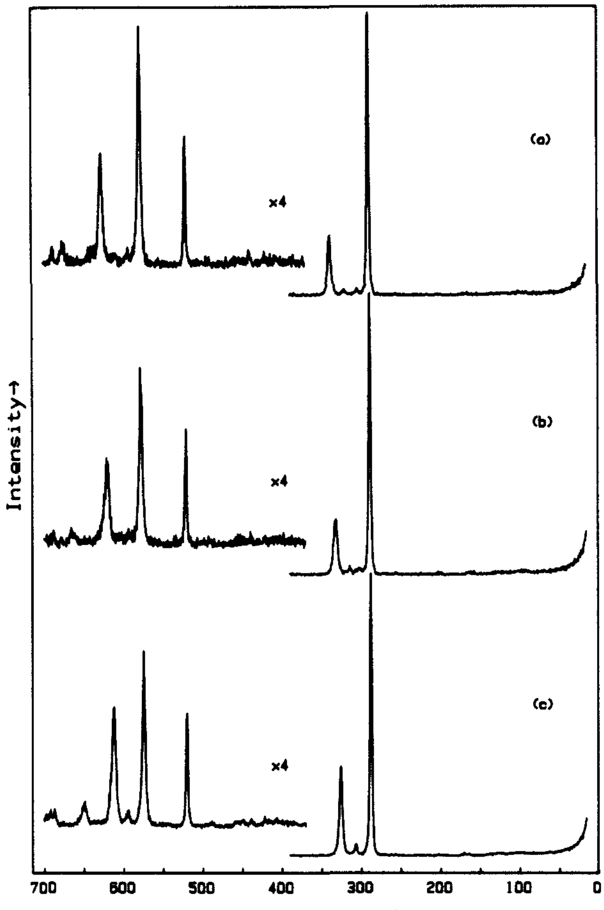

Figure 1. Resonance Raman spectrum of (a) $Rh_2(^{16}O_2CCH_3)_4(PPh_3)_2$, (b) $Rh_2(^{18}O_2CCH_3)_4(PPh_3)_2$, and (c) $Rh_2(^{16}O_2CCD_3)_4(PPh_3)_2$ as KCl disks at ca. 80 K (λ_0 = 363.8 nm, slit width ~ 2.5 cm^{-1}).

Table I. Resonance Raman Data on $Rh_2(^{16}O_2CCH_3)_4(PPh_3)_2$, $Rh_2(^{18}O_2CCH_3)_4(PPh_3)_2$ and $Rh_2(^{16}O_2CCD_3)_4(PPh_3)_2$ in the Range 700–250 cm^{-1}

	Band Wavenumber/cm^{-1}		
assignment	^{16}O	^{18}O	CD_3
ν(RhRh), ν_1	289.3 vs	289.1 vs	287.5 vs
ν(RhO), ν_2	338.4 m	332.0 m	325.8 m
ν(RhO)	321 w	314 w	307 w
ν(RhO)?	305 w	302 w	
y X-sensitive PPh$_3$[a]	520.4 m	520.7 m	520.7 m
$2\nu_1$	577.5 m	577.1 m	574.7 m
$\nu_1 + \nu_2$	626.5 m	619.5 m	612.4 m
$2\nu_2$	675 w	663 w	650 w

[a] Nomenclature as per: Whiffen, D. H. *J. Chem. Soc.* **1956**, 1350.

Raman studies since the electric-dipole-allowed metal-based $\sigma \rightarrow \sigma^*$ transition[7,8] occurs in the near-ultraviolet region and is accessible with the Ar^{2+} 363.8-nm excitation line. Raman spectra so excited are much simpler than off-resonance spectra; under these (A-term)[9] conditions, it is only bands attributable to totally symmetric modes which are enhanced at resonance with an electric-dipole-allowed transition, together with bands attributable to overtones and combination tones involving the enhanced bands.

The key resonance Raman spectra[10] are shown in Figure 1 and selected band wavenumbers are given in Table I. The Raman spectrum of $Rh_2(O_2CCH_3)_4(PPh_3)_2$ taken at resonance with the $\sigma \rightarrow \sigma^*$ transition displays no bands in the 150–170-cm^{-1} region but does give rise to a strong band at 289.3 cm^{-1} (labeled ν_1), to the first overtone $2\nu_1$, and to a combination band $\nu_1 + \nu_2$, where ν_2 is a band of moderate intensity at 338.4 cm^{-1}. Any structural change attendant upon $\sigma \rightarrow \sigma^*$ excitation would be expected to occur primarily along the RhRh bond rather than along the nearly orthogonal RhO bonds. Hence it is ν(RhRh) and its overtones that are expected to suffer the greatest intensification at resonance with the $\sigma \rightarrow \sigma^*$ transition. This indicates that ν_1 should be identified with ν(RhRh) and suggests that ν_2 is attributable to the totally symmetric ν(RhO) mode. Although ν(RhP) might also be expected to be enhanced at resonance with the $\sigma \rightarrow \sigma^*$ transition, it has not been positively identified. It cannot be identified with ν_1 since this mode moves from 289.3 cm^{-1} to *higher* wavenumbers for the analogous AsPh$_3$ and SbPh$_3$ derivatives (297 and 307 cm^{-1}, respectively), i.e., in the opposite direction to that expected on both mass and Rh–M bond-length grounds (for the complexes $Rh_2(O_2CCH_3)_4\cdot(MPh_3)_2$, r(Rh–M) increases in the order RhP, 2.477 Å, < RhAs, 2.576 Å, < RhSb, 2.732 Å).[11,12] The axial nature of the resonant transition is confirmed by the fact that the depolarization ratio of the ν_1 band is $^1/_3$ at resonance.

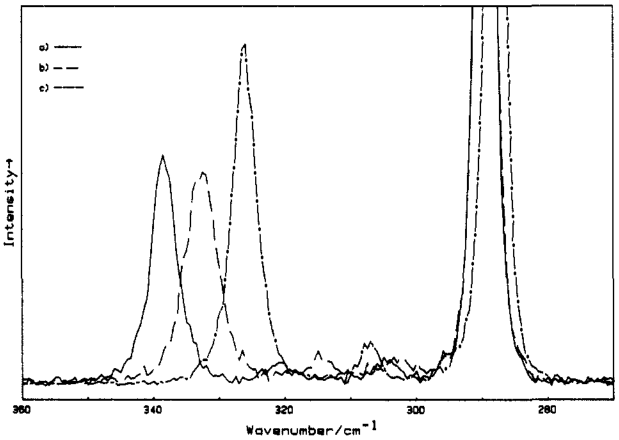

Figure 2. Expanded version of Figure 1, with the ν_1 bands truncated, showing the shifts of ν_1 and ν_2 on ^{18}O substitution and on deuteration. (a), (b), (c) as for Figure 1.

The resonance Raman spectra of the ^{18}O and CD$_3$ analogues[13-15] of $Rh_2(O_2CCH_3)_4(PPh_3)_2$ confirm this assignment convincingly. The most resonance-enhanced band, ν_1, occurs at 289.3, 289.1, and 287.5 cm^{-1} for the ^{16}O, ^{18}O, and CD$_3$ derivatives, respectively, indicating the virtual independence of ν_1 of the equatorial modes, which is as expected for ν(RhRh). On the other hand the two bands at 338.4 and 321 cm^{-1} in the resonance Raman spectrum of the ^{16}O complex are shifted by 6.4 and 7 cm^{-1}, respectively, to lower wavenumbers by ^{18}O substitution, consistent with the behavior expected for modes in which the RhO stretching coordinate dominates (Figure 2). On deuteration, these modes move by rather more (12–14 cm^{-1}), a matter that will be discussed elsewhere.

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(14) Acetic acid 100.0 atom % D (Aldrich) was used to synthesise the deuterated derivative.

(15) [^{18}O]Acetic acid (250 mg) was added dropwise over a period of several minutes to a suspension of $Na_4[Rh_2(CO_3)_4]\cdot 2.5H_2O$ (233 mg) in ca. 3 cm^3 of H$_2$O. The mixture was stirred for a further 5 min while being kept at a constant 80 °C. The mixture was then cooled and the resulting green solid, the ^{18}O-enriched $Rh_2(O_2CCH_3)_4(H_2O)_2$, filtered off. The solid was then dissolved in methanol, to which was added a saturated methanol solution of triphenylphosphine to yield the enriched bis(triphenylphosphine) adduct.

The combination of resonance Raman and isotopic work thus demonstrates conclusively that, for $Rh_2(O_2CCH_3)_4(PPh_3)_2$, ν(RhRh) is at 289.3 cm^{-1}. Since the RhRh bond length in this complex is 2.4505 (2) Å,[11] longer than in the analogous $AsPh_3$ (2.427 (1) Å),[12] $SbPh_3$ (2.421 (4) Å),[12] and H_2O (2.3855 (5) Å)[16] complexes, the implication is that ν(RhRh) in these complexes must all lie above 289.3 cm^{-1}, as indeed is found to be the case for the $AsPh_3$ and $SbPh_3$ derivatives (vide supra). The high value for ν(RhRh) is thought to arise from the significant contribution to the RhRh restoring force brought about by the four chelating acetate groups (primarily via the four OCO bending coordinates, which are coupled only in second order to the RhRh stretching coordinate). Full details of these and related studies will be published elsewhere.

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Reactions of Cobalt Carbene Complexes with Alkynes—η^4-Vinylketene Complex Intermediates and a Novel Synthesis of Bovolide[†]

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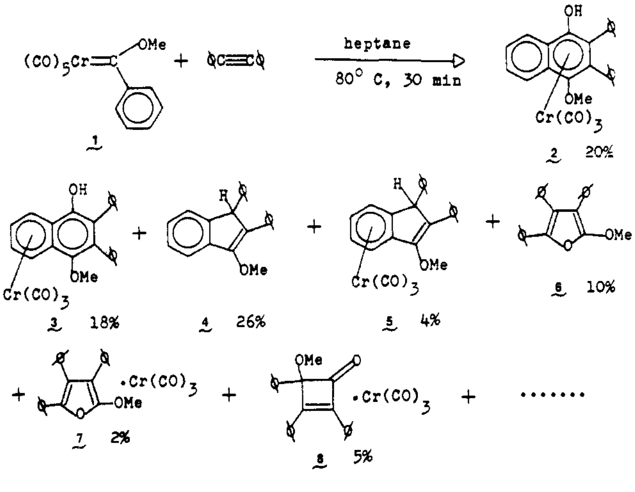
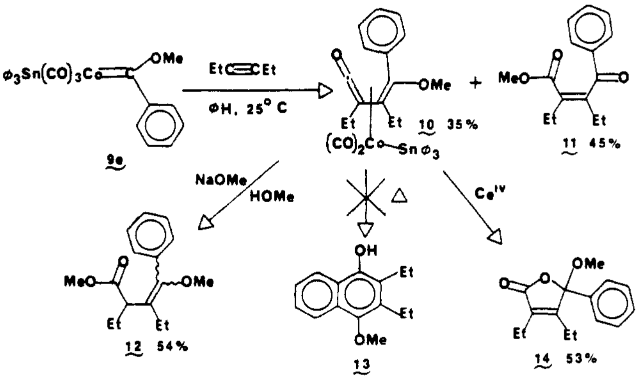
The report 8 years ago of the reaction of the chromium carbene complex **1** with diphenylacetylene in heptane (Scheme I) was the first to indicate the complexity of reactions of this type with regard to structural variations in the products and the sensitivity to reaction conditions.[3a] The reaction had previously been reported to give only the naphthol complex **2** in *n*-butyl ether.[3b] It has since been well established[4,5] that the benzannulated product (naphthol **2**) is the normal product of the reaction of chromium carbene complexes and that indenes,[4b,6] furans,[5b,7,8] and cyclobutenones[4b,5e,g,9] have only been observed on rare occasions and in low yields or as minor products. We report here that the reactions of (methoxyalkylidene)(triphenylstannyl)tricarbonylcobalt(0) complexes of the type **9** with alkynes occur with exclusive formation of 2-alkoxyfurans.

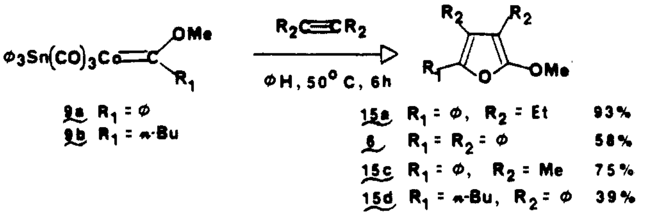
Scheme I



Scheme II



Tricarbonylcobalt carbene complexes of the type **9** have been previously prepared;[10] however, their reactivity with either olefins or acetylenes has not been previously reported. Complex **9a** was allowed to react with 1.2 equiv of diethylacetylene at room temperature in a deoxygenated benzene solution. After 48 h the η^4-vinylketene complex **10** and the γ-keto unsaturated ester **11** could be isolated after silica gel chromatography in air with a 1:1 mixture of benzene and hexanes as eluent. It has been suggested that η^4-vinylketene complexes are key intermediates in the reactions of chromium[4,8] and iron[5k] carbene complexes with acetylenes; however, until now such complexes have never been isolated from the reaction of a transition-metal carbene complex



[†] This work was presented at the 190th National Meeting of the American Chemical Society, Chicago, IL, Sept 8-13, 1985.

(1) The University of Chicago.

(2) Merck Institute for Therapeutic Research.

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