especially in the "red" region of each spectrum, where the photomultiplier response is low.

The spectra reported here were from ions collisionally excited in C2; therefore only relatively long lived excited states (0.1 μs) are involved. At present we cannot *quantitatively* correct for other identified sources of radiation resulting from a collision experiment performed in C3, e.g. target gas emissions and emissions from metal surfaces caused by collisions with scattered ions.

The ions, H_3^+ and $H_2^{\bullet+}$, were studied as model systems, using He target gas, to limit the number of emitting species. Before presenting our results, previous studies will be reviewed. Ford et al.[9] have also studied H_3^+ and $H_2^{\bullet+}$ using ion beams of 75–400 keV and He target gas. For H_3^+, the lifetime of $H^\bullet(n=3)$ produced was assessed by detecting Balmer-α photons. Emissions between 550 and 580 nm, recorded ca. 3×10^{-8} s after neutralization of keV beams of H_3^+ using alkali metal vapors as the electron transfer target, have been reported by Figger et al.[10,11] They proposed that the emission arose from $H_3^\bullet$ transitions originating in the n = 3 and 2 electronic levels. Measurements of the kinetic energy released in the formation of product ions and neutrals by keV collisions of H_3^+ with He target gas showed[12] $H^\bullet$ (2p) and H_2 (B $^1\Sigma_u^+$) as the only excited state products. Yenen and Jaecks[13] detected Lα photons in the reaction $H_3^+ + He \rightarrow H_2^{\bullet+} + H^\bullet(n=2) + He$. Emissions from H_3^+, $H_3^\bullet$, $H_2^{\bullet+}$,[14-16] and H_2[16] may lie outside either our wavelength range or time scale. Indeed, our only observed emission, 650–680 nm, coincided with the Balmer-α transition for $H^\bullet$ (n = 3 to 2, 656 nm), indicating that some $H^\bullet$ resulting from the dissociation of H_3^+ is in the n = 3 electronic state, consistent with the work by Ford et al.[9]

The $H_2^{\bullet+}$ spectrum in the present work was more complex. A strong signal in the 650–680-nm region implies that excited-state $H^\bullet$ was formed, consistent with earlier observations.[9,17] Spectral structure observed between 375 and 500 nm may be due to neutralization of $H_2^{\bullet+}$ to form excited H_2.[17] Huber and Herzberg[18] list numerous singlet–singlet transitions within this region, terminating in the B $^1\Sigma_u^+$ electronic state of neutral H_2. A gap between 500 and 600 nm is consistent with the above singlet–singlet transitions. UV emission between 180 and 340 nm was also observed by Gellene et al.[19] in the emission spectrum of $H_2^{\bullet+}$ using K as the target gas. It was assigned to the triplet–triplet transition, $a^3\Sigma_g^+ \rightarrow b^3\Sigma_u^+$. Once wavelength resolution is improved by the use of a monochromator, transitions will be more accurately identified.

In contrast, the emission spectra for polyatomic organic ions were more complex. The isomeric ions $CH_3CH_2Cl^{\bullet+}$ and $CH_3ClCH_2^{\bullet+}$ are distinguishable by their O_2 CID mass spectra,[20] with the former displaying a much more intense m/z 28 peak than the latter, for which loss of $CH_3^\bullet$ is more significant. Figure 2 shows their collision-induced emission spectra which have significant differences in the 300–500-nm spectral range. Spectral differences are probably due to both parent ion emissions and different product ions and neutrals (and hence different emitting species) being formed.

The He CID mass spectra of the isomers $CH_3CHO^{\bullet+}$ and $CH_2{=}CHOH^{\bullet+}$ are distinguishable only in that the latter ion produces a significant peak at m/z 30.[21,22] Their He CID emission spectra (Figure 2), however, are markedly different, with $CH_2{=}CHOH^{\bullet+}$ exhibiting more signal between 500 and 680 nm. Since predominantly the same product ions and neutrals are formed, this result suggests that either different electronic states are involved or only the parent ions emit radiation. These preliminary results show that collision-induced emission spectroscopy may evolve into a sensitive probe of ion structure.

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Coordination and Interconversion of a Ketonic Grouping between One and Two Transition-Metal Atoms

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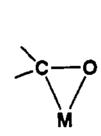
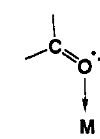
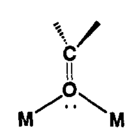
Electrophilic activation of carbonyl groups with Lewis acids is a well-established method for enhancing their reactivity toward nucleophiles.[1] Coordination of ketones to chiral metal centers has been found to promote nucleophilic additions with significant asymmetric induction.[2] The two principal modes of coordination of ketones to metals are the η^2-π-bonded and the η^1-σ-bonded modes **A** and **B**.[3] Double electrophilic activation of ketones, types **C**,[4] **D**,[5] and **E**,[6] should produce a much greater reactivity toward nucleophilic addition. Examples of these bridging types of co-



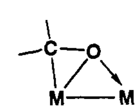
A



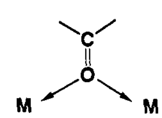
B



C



D



E

ordination are rare, and the μ-di-σ mode **E** has been especially elusive.[6] We have now obtained the first structural characterization of a ketonic grouping exhibiting type **E** coordination to two transition-metal atoms and have also demonstrated its reversible conversion to the η^1-σ mode **B**.

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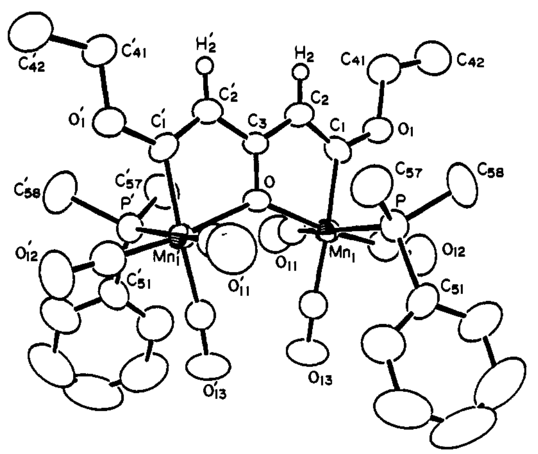


Figure 1. An ORTEP diagram of $Mn_2(CO)_6(PMe_2Ph)_2\{\mu\text{-}O{=}C[C(H){=}C(OEt)]_2\}$ (3). Selected interatomic distances (Å) are Mn(1)···Mn(1′) = 3.988 (2). Mn(1)–O = 2.136 (2), Mn(1)–C(1) = 1.992 (6), Mn(1)–P = 2.326 (2), C(3)–O = 1.312 (9), C(1)–O(1) = 1.345 (6), C(1)–C(2) = 1.376 (8), C(2)–C(3) = 1.413 (7).

From the reaction of 50 mg (0.124 mmol) of $Mn_2(CO)_9NCMe$[7] (I) with 60.5 μL (0.310 mmol) of HC≡COEt in hexane at 35 °C for 3 h, we have isolated by TLC the new compound $Mn_2(CO)_8\{\mu\text{-}O{=}C[C(H){=}C(OEt)]_2\}$ (2) in 15% yield.[8] The empirical formula of 2 was established by mass spectrometry.[8] Its structure was deduced by an X-ray crystallographic analysis of its $(PMe_2Ph)_2$ derivative 3 and its CO adduct 4; see below. An ORTEP drawing of the molecular structure of $Mn_2(CO)_6(PMe_2Ph)_2\{\mu\text{-}O{=}C[C(H){=}C(OEt)]_2\}$ (3) is shown in Figure 1.[9-11] The molecule contains two octahedrally coordinated manganese atoms bridged by a $(EtOC{=}CH)_2C{=}O$ ligand. There is a crystallographically imposed C_2 axis that lies along the C(3)–O bond. The long Mn(1)···Mn(1′) distance of 3.988 (2) Å shows that the metal atoms are not mutually bonded. They are instead separated by the bridging oxygen atom O, Mn(1)–O = 2.136 (2) Å. The ketonic group C(3)–O has substantial double-bond character, 1.312 (9) Å, and there is delocalized unsaturation throughout the entire planar C(1)–C(2)–C(3)–C(2′)–C(1′) chain, C(1)–C(2) = 1.376 (8) Å and C(2)–C(3) = 1.413 (7) Å. The $Mn_2O{=}C$ grouping is planar, and the coordination of the ketonic group C(3)–O is clearly of the μ-di-σ type **E**. The $(EtOC{=}CH)_2C{=}O$ ligand was evidently formed by the addition of two HC≡COEt molecules at their unsubstituted end to the carbon atom of a carbonyl ligand.[12] This is supported by a ^{13}CO-labeling study of 2, which showed a shift of the IR absorption that is attributed to the ketonic double bond from 1498 to 1483 cm^{-1}.[14] Although the frequency reduction is augmented by conjugation from the adjacent olefinic groups, we feel that the low frequency of this absorption is indicative of a higher degree of C=O bond reduction than occurs with the type **B** mode of coordination; see below.[2,15] The ligand is also σ-coordinated to the metal atoms through the carbons C(1) and C(1′). The hydrogen atom on C(2) shows the expected low-field resonance, δ = 5.90.

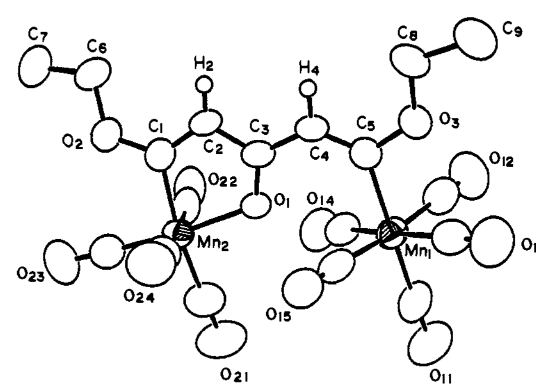
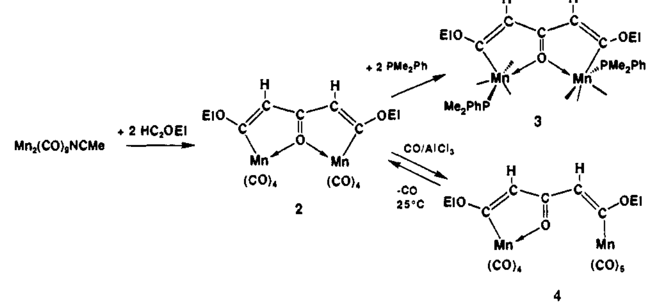


Figure 2. An ORTEP diagram of $Mn_2(CO)_9\{\mu\text{-}O{=}C[C(H){=}C(OEt)]_2\}$ (4). Selected interatomic distances (Å) are Mn(2)–O(1) = 2.027 (3), Mn(1)···O(1) = 3.278 (3), Mn(1)–C(1) = 1.999 (5), Mn(1)–C(5) = 2.079 (5), C(3)–O(1) = 1.282 (9), C(1)–O(2) = 1.338 (6), C(1)–C(2) = 1.368 (7), C(2)–C(3) = 1.410 (7), C(3)–C(4) = 1.446 (7), C(5)–O(3) = 1.381 (6), C(4)–C(5) = 1.353 (7).

Scheme I



When compound 2 was treated with CO (1 atm) at 25 °C for 3 h in the presence of $AlCl_3$, the CO addition product $Mn_2(CO)_9\{\mu\text{-}O{=}C[C(H){=}C(OEt)]_2\}$ (4) was obtained in 90% yield.[16] Compound 4 was characterized by a combination of IR, ^1H NMR, and single-crystal X-ray diffraction analyses. An ORTEP drawing of the molecular structure of 4 is shown in Figure 2.[11,17] Compound 4 contains a bridging $(EtOC{=}CH)_2C{=}O$ ligand similar to that found in 3, but the ketonic oxygen atom O(1) is coordinated to only one manganese atom, Mn(2)–O(1) = 2.027 (3) Å. A

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(8) HC≡COEt (60.5 μL, 0.310 mmol) in hexane (purchased from Aldrich) was allowed to react with 50.0 mg (0.124 mmol) of $Mn_2(CO)_9(MeCN)$ in 50 mL of hexane at 35 °C for 3 h. The principal product, yellow $Mn_2(CO)_8\{\mu\text{-}O{=}C[C(H){=}C(OEt)]_2\}$ (2), 9.8 mg (15%), was separated from unreacted $Mn_2(CO)_9(MeCN)$, 4.8 mg, by TLC with a hexane/CH_2Cl_2 (4/1) solvent mixture. For 2: IR (ν_{CO} in hexane) 2096 (m), 2083 (w), 2012 (s), 1993 (m), 1969 (w), 1955 (s), 1498 (m, br); ^1H NMR (δ in $CDCl_3$) 5.90 (1 H, s, CH), 4.11 (2 H, q, $^3J_{H-H}$ = 7.1 Hz, CH_2), 1.45 (3 H, t, $^3J_{H-H}$ = 7.0 Hz, CH_3). The mass spectrum of 2 showed the parent ion m/e = 502 and ions corresponding to the loss of each of two through eight carbonyl ligands.

(9) $Mn_2(CO)_6(PMe_2Ph)_2\{\mu\text{-}O{=}C[C(H){=}C(OEt)]_2\}$ (3) was obtained in 95% yield by reaction of 2 (30.0 mg) with PMe_2Ph (20.0 μL) in 80 mL of hexane at 68 °C for 1 h. For 3: IR (ν_{CO} in hexanes) 2020 (s), 2007 (s), 1992 (w), 1940 (s), 1934 (m, sh), 1906 (s); ^1H NMR (δ in $CDCl_3$) 7.39 (5 H, m, Ph), 5.77 (1 H, d, $^4J_{P-H}$ = 2.3 Hz, CH), 3.84 (1 H, dq, C*H*H, $^1J_{H-H}$ = 9.4 Hz, $^3J_{H-H}$ = 7.0 Hz), 3.58 (1 H, dq, CH*H*, $^1J_{H-H}$ = 9.4 Hz, $^3J_{H-H}$ = 7.4 Hz), 1.63 (3 H, d, $^2J_{P-H}$ = 8.19 Hz, PMe), 1.45 (3 H, d, $^2J_{P-H}$ = 8.6 Hz, PMe), 1.23 (3 H, t, $^3J_{H-H}$ = 6.9 Hz, Me).

(10) Crystal data for 3: space group = $C2/c$, a = 26.271 (7) Å, b = 9.130 (2) Å, c = 15.616 (5) Å, β = 113.90 (2)°, Z = 4, 1226 reflections, R = 0.038.

(11) Diffraction measurements were made at 20 °C on a Rigaku AFC6S four-circle diffractometer using Mo Kα radiation. Structure solutions and refinements were made on a VAXstation 3520 computer by using the TEXSAN structure solving program library (v5.0) of the Molecular Structure Corp., The Woodlands, TX.

(12) A similar coupling of terminal alkynes to CO was observed in products obtained from the reactions of alkynes with $Os_3(CO)_{10}(\mu\text{-}H)_2$ except that in these products the ketonic oxygen atom was not coordinated.[13]

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(14) A sample of $Mn_2(CO)_9(MeCN)$ enriched with ^{13}CO to approximately 45% was converted to 2* by treatment with HC≡COEt. The IR spectrum of 2* in CCl_4 showed an absorption at 1498 cm^{-1} due to the unlabeled C=O grouping and a second absorption of approximately equal intensity at 1483 cm^{-1} that is attributed to the labeled C=O grouping.

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(16) $AlCl_3$ (50 mg, 0.374 mmol) was added to 10 mg (0.020 mmol) of 2 in 50 mL of CH_2Cl_2 at 25 °C. After 20 min, this solution was purged with CO for 3 h and then filtered over a silica gel column to remove $AlCl_3$. The solvent was evaporated and the residue chromatographed by TLC with hexane to give 9.5 mg of yellow $Mn_2(CO)_9\{\mu\text{-}O{=}C[C(H){=}C(OEt)]_2\}$ (4), 90%. For 4: IR (ν_{CO} in hexanes) 2122 (w), 2084 (w), 2065 (w), 2032 (s), 2005 (s), 1995 (s), 1954 (s), 1553 (w, br); ^1H NMR (δ in C_6D_6) 6.31 (1 H, s, CH), 5.89 (1 H, s, CH), 3.75 (2 H, q, $^3J_{H-H}$ = 7.0 Hz, CH*H*), 3.43 (2 H, q, $^3J_{H-H}$ = 7.0 Hz, CH*H*), 1.12 (3 H, t, $^3J_{H-H}$ = 7.0 Hz, Me), 1.00 (3 H, t, $^3J_{H-H}$ = 6.9 Hz, Me).

(17) Crystal data for 4: space group = $P2_1/n$, a = 7.565 (2) Å, b = 22.788 (4) Å, c = 13.015 (2) Å, β = 92.71 (2)°, Z = 4, 1515 reflections, R = 0.032.

carbonyl ligand was added to Mn(1), and the Mn(1)···O(1) distance was lengthened to a nonbonding value, 3.278 (3) Å. The ketonic C(3)–O(1) distance is slightly shorter than that in **3**, 1.282 (6) Å, and the C=O absorption frequency is increased to 1553 cm^{-1}.[16] The CO addition to **2** is fully reversible, and when solutions of **4** were purged with nitrogen for 24 h at 25 °C, compound **2** was regenerated in essentially a quantitative yield.[18] The results of this study are summarized in Scheme I. Studies of the reactivity of the ketonic grouping are in progress.

Acknowledgment. These studies were supported by the U.S. Department of Energy under Grant No. DEFG84ER13296.

Supplementary Material Available: Tables of crystal data, positional parameters, bond distances and angles, and anisotropic thermal parameters for **3** and **4** (18 pages); tables of structure factors for **3** and **4** (20 pages). Ordering information is given on any current masthead page.

(18) A solution of 10.0 mg of **4** in 50 mL of hexane was purged with nitrogen at 25 °C for 24 h. **2**, 9.0 mg, 96% yield, was isolated after workup by TLC.

A Multiply-Substituted Buckminsterfullerene (C_{60}) with an Octahedral Array of Platinum Atoms

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Fundamental questions concerning the chemistry of the recently isolated carbon clusters (C_{60}, C_{70}, C_{84}, etc.)[1] include how many substituents can be attached and what, if any, geometrical preferences or electronic directing effects guide the substitution chemistry of these molecules. Several structurally-characterized derivatives of C_{60} have been reported, including (t-$BuC_5H_4N)_2OsO_4C_{60}$[2] and $[(C_6H_5)_3P]_2Pt(\eta^2\text{-}C_{60})$.[3] In these cases only two of the C_{60} carbon atoms are derivatized. There is evidence that multiply-substituted compounds exist, but these usually occur as mixtures and have not been structurally defined.[2,4] Here we describe the characterization of the hexa-substituted platinum derivative $\{[(C_2H_5)_3P]_2Pt\}_6C_{60}$. This is the first example of a reaction that selectively forms a *single isomer* of a highly-substituted C_{60} derivative in high yield. The phosphorus, platinum, and C_{60} atoms have nearly ideal and rarely observed T_h point group symmetry.

Addition of 0.724 g (1.08 mmol) of $[(C_2H_5)_3P]_4Pt$[5] to a solution of 75 mg (0.10 mmol) of C_{60} in 5 mL of benzene produced a dark orange-brown solution. After 10 min, solvent and released triethylphosphine were removed in vacuo. Benzene was added until the compound all dissolved, and the solution was filtered. After removal of solvent, hexane (ca. 6–10 mL) was added to the flask; the air-sensitive, orange, crystalline solid was collected by filtration, washed three times with 1–2-mL portions of hexane, and dried in vacuo to obtain an 88% yield of $\{[(C_2H_5)_3P]_2Pt\}_6C_{60}$.[6]

The simplicity of the ^{31}P NMR spectrum (one resonance) and the ^{13}C NMR spectrum (three resonances for C_{60} in a 2:2:1 ratio) as well as the observed couplings among the spin $^1/_2$ nuclei allowed unambiguous assignment of the structure.[6] The molecule has a C_{60} core bearing six octahedrally-disposed $[(C_2H_5)_3P]_2Pt$ groups

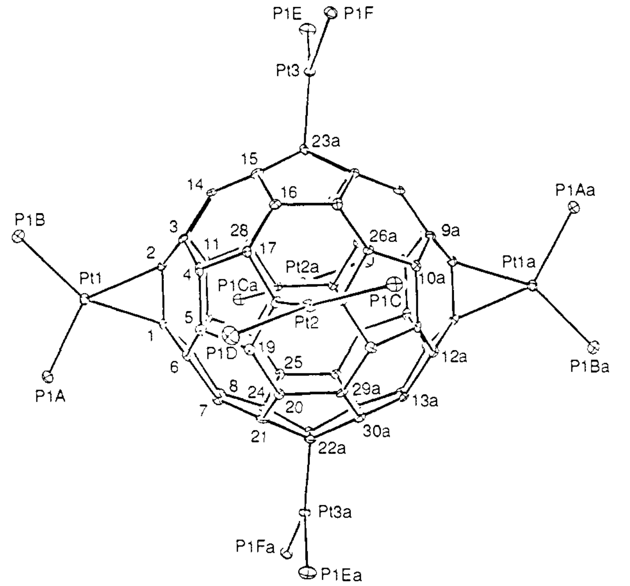

Figure 1. Diagram of one of the two independent molecules of $\{[(C_2H_5)_3P]_2Pt\}_6C_{60}$. For clarity, phosphine ethyl groups are not shown. As in $[(C_6H_5)_3P]_2Pt(\eta^2\text{-}C_{60})$,[3] carbons attached to Pt are pulled out from the original C_{60} frame with their average distance to the C_{60} centroid being 3.687 (±0.009) Å (range 3.663–3.697 Å). Under idealized T_h symmetry, there are two other unique symmetry related sets of carbon atoms in the C_{60} frame, i.e. those two bonds from Pt and those three bonds from Pt (average distance to centroid = 3.538 (±0.013) and 3.526 (±0.010) Å, respectively). The twists of the P–Pt–P planes relative to the C–Pt–C planes are 13.2° (Pt1), 15.8° (Pt2), and 9.6° (Pt3). (Values of 12.5°, 2.6°, and 17.0° are found for the other molecule.) The Pt atoms bend away to different extents from the normal to the C_{60} surface. For example, the Pt3–midpoint (C22,C23a)–centroid (C_{60}) angle is 175.1° whereas for Pt1 and Pt2 these angles are 177.8° and 179.4°, respectively (values of 173.8, 178.5, and 178.8° are observed for the other molecule). Bond distances and angles about the Pt atoms are as follows: average Pt–C (bonds G, Figure 2) = 2.115 (±0.017) Å, range 2.084 (8)–2.137 (9) Å; average Pt–P (bonds F, Figure 2) = 2.261 (±0.007) Å, range 2.251 (3)–2.272 (3) Å; average P–Pt–P = 111.8 (±1.2)°, range 110.5 (1)–114.0 (1)°; average C–Pt–C = 41.4 (±0.2)°, range 41.3 (3)°–41.8 (3)°; average P–Pt–C (smaller angle) = 103.7 (±3.6)°, range 98.0 (3)°–110.9 (2)°.

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