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The Preparation and Crystallographic Determination of Stereochemistry for the Monoclinic and Triclinic Crystal Modifications of 4,6,8-Trimethylazulenetetraruthenium Enneacarbonyl, $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$

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Received March 24, 1969

The reaction of 4,6,8-trimethylazulene with $Ru_3(CO)_{12}$ leads to the formation of 4,6,8-trimethylazulenetetraruthenium enneacarbonyl, $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$, which crystallizes in two forms—a monoclinic and a triclinic modification. The crystal structure of each polymorph has been determined using molybdenum Kα radiation and counter techniques. Monoclinic crystals are obtained as small red parallelepipeds, crystallizing in space group $P2_1/n$ (C_{2h}^5; no. 14) with $a = 8.99 \pm 0.02$ Å, $b = 18.56 \pm 0.03$ Å, $c = 14.44 \pm 0.02$ Å, $\beta = 96.5 \pm 0.1°$, $\rho_{obsd} = 2.29 \pm 0.02$ g cm^{-3}, and $\rho_{calcd} = 2.293$ g cm^{-3} for $Z = 4$. Triclinic crystals are obtained as thin red platelets crystallizing in space group $A\bar{1}$ (C_i^1; no. 2) with $a = 15.267 \pm 0.010$ Å, $b = 10.610 \pm 0.007$ Å, $c = 14.862 \pm 0.010$ Å, $\alpha = 92.03 \pm 0.05°$, $\beta = 101.18 \pm 0.05°$, $\gamma = 90.08 \pm 0.05°$, $\rho_{obsd} = 2.35 \pm 0.02$ g cm^{-3}, and $\rho_{calcd} = 2.326$ g cm^{-3} for $Z = 4$. Both structures were refined by the method of least squares, all nonhydrogen atoms having been located in each case. Final discrepancy indices are $R_F = 9.49\%$ (1844 reflections) for the *monoclinic* and $R_F = 6.34\%$ (3158 reflections) for the *triclinic* modification. The two crystal forms contain experimentally indistinguishable molecular units of 4,6,8-trimethylazulenetetraruthenium enneacarbonyl, separated by normal van der Waals distances. The molecule possesses approximate C_s symmetry, with the four ruthenium atoms defining a distorted tetrahedron in which interatomic distances are found to range from 2.702 to 2.902 Å (±0.005 Å) in the monoclinic determination and from 2.698 to 2.908 Å (±0.003 Å) in the triclinic study. The 4,6,8-trimethylazulene ligand is arched across the largest face of the tetrahedral ruthenium cluster (*i.e.*, is directly bonded to *three* ruthenium atoms) and is bent across C(9)–C(10), C(8)···C(4), and C(7)···C(5) such that there is a dihedral angle of 126° between the planar five-membered ring and the plane defined by C(5), C(6), and C(7). The three ruthenium atoms that are associated with the azulene ligand are each bonded to two terminal carbonyl ligands while the apical ruthenium is bonded to three terminal carbonyl groups. The bonding between the 4,6,8-trimethylazulene ligand and the adjacent three ruthenium atoms is best considered in terms of delocalized "ligand-to-cluster" bonding.

Introduction

The present paper is the seventh in a series of structural studies on transition metal complexes of azulene or substituted azulenes, and follows detailed crystallographic reports on $C_{10}H_8Fe_2(CO)_5$,[2,3] $[C_{10}H_8Mo(CO)_3CH_3]_2$,[4] $[(i\text{-}C_3H_7)(CH_3)_2C_{10}H_5]Mo_2(CO)_6$,[5] $C_{10}H_8Mn_2(CO)_6$,[6] $(C_{10}H_8)_2Fe$,[7] and $(C_{10}H_8)_2Fe_4(CO)_{10}$.[8] Studies on $C_{10}H_8Mo_2(CO)_6$ have been reported by ourselves[5b] and two other groups of research workers.[9,10] In each of the above-mentioned complexes, the azulene π-electrons are factored between the constituent metal atoms in such a manner that a certain group of carbon atoms is obviously associated with one metal atom, while a second group of carbon atoms is linked to another metal atom. In no case is a carbon atom of the azulene system shared between two or more metal atoms.

It is interesting, however, that of complexes with a 1:2 azulene-to-metal ratio, only $C_{10}H_8Fe_2(CO)_5$ has any noncoordinated π density, and it is not easy to provide an unambiguous explanation as to why this is the case. It was in attempting to clarify this anomaly that the reaction of 4,6,8-trimethylazulene with triruthenium dodecacarbonyl was originally investigated.[11,12] However, the product isolated from this reaction—$[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$—had a stoichiometry completely different from that expected.

A brief account of the preparation of this new complex and of a crystallographic study of the *monoclinic* crystal modification has appeared previously.[13] A crystallographic investigation of the *triclinic* crystal form was undertaken subsequently in order to ascertain whether or not the configuration of the $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule in this crystal habit was the same as in the monoclinic modification.

Experimental Section

Triruthenium dodecacarbonyl (0.48 g) and 4,6,8-trimethylazulene (0.58 g) were heated under reflux in ligroin (bp 90–120°; 25 ml) in an atmosphere of nitrogen. After removal of the solvent under vacuum, residual $Ru_3(CO)_{12}$ and 4,6,8-trimethylazulene were removed from the reaction mixture by sublimation. The resulting dry residue was dissolved in the minimum necessary quantity of 60:40 1,2-dichloroethane–cyclohexane; chromatography of this solution on Florisil yielded two bands. A mass spectral examination of the products showed them to be $Ru_6(CO)_{17}C$[14] and $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$—in order of elution. The latter compound showed a parent-ion peak at m/e 826, corre-

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[1] Research Fellow of the Alfred P. Sloan Foundation, 1968–1970.

[2] M. R. Churchill, *Inorg. Chem.*, **6**, 190 (1967).

[3] M. R. Churchill, *Chem. Commun.*, 450 (1966).

[4] (a) P. H. Bird and M. R. Churchill, *Inorg. Chem.*, **7**, 349 (1968); (b) P. H. Bird and M. R. Churchill, *Chem. Commun.*, 705 (1967).

[5] (a) M. R. Churchill and P. H. Bird, *Inorg. Chem.*, **7**, 1545 (1968); (b) M. R. Churchill and P. H. Bird, *Chem. Commun.*, 746 (1967).

[6] (a) M. R. Churchill and P. H. Bird, *Inorg. Chem.*, **7**, 1793 (1968); (b) P. H. Bird and M. R. Churchill, *Chem. Commun.*, 145 (1968).

[7] (a) M. R. Churchill and J. Wormald, *Inorg. Chem.*, **8**, 716 (1969); (b) M. R. Churchill and J. Wormald, *Chem. Commun.*, 1033 (1968).

[8] (a) M. R. Churchill and P. H. Bird, *Inorg. Chem.*, **8**, 1941 (1969); (b) M. R. Churchill and P. H. Bird, *J. Am. Chem. Soc.*, **90**, 3241 (1968).

[9] J. S. McKechnie and I. C. Paul, *Chem. Commun.*, 747 (1967).

[10] A. W. Schleuter and R. A. Jacobsen, *Inorg. Chim. Acta*, **2**, 241 (1968).

[11] This substituted azulene was used in order to avoid disorder problems such as those found in $C_{10}H_8Mo_2(CO)_6$[5b,9,10] and azulene itself.[12]

[12] J. M. Robertson, H. M. M. Shearer, G. A. Sim, and D. G. Watson, *Acta Cryst.*, **15**, 1 (1962).

[13] M. R. Churchill and P. H. Bird, *J. Am. Chem. Soc.*, **90**, 800 (1968).

[14] B. F. G. Johnson, R. D. Johnston, and J. Lewis, *Chem. Commun.*, 1057 (1967); B. F. G. Johnson, R. D. Johnston, and J. Lewis, *J. Chem. Soc.*, *A*, 2865 (1968).

sponding to $(CH_3)_3C_{10}H_5{}^{101}Ru_4(CO)_9{}^+$, along with species consistent with stepwise loss of CO—*i.e.*, $(CH_3)_3C_{10}H_5Ru_4(CO)_{9-n}{}^+$ (n = 1–9); an extremely strong feature of the spectrum at lower mass numbers was the presence of ions of the type $(CH_3)_3C_{10}H_5Ru_2(CO)_{4-n}{}^+$ (n = 0–4).

An infrared spectrum of $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ (taken in 1,2-dichloroethane) showed bands typical of terminal carbonyl stretches at 2059 (s), 2000 (s), 1962 (w), and 1927 (w) cm^{-1}.

The crystals of $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ obtained by crystallization of the original eluent were extemely small red parallelepipeds which were found (*vide infra*) to belong to the monoclinic system. Subsequent attempts to grow larger crystals by recrystallization of the complex from solution in 60:40 1,2-dichloroethane–cyclohexane yielded only bright red platelets belonging to the triclinic crystal system. Both crystal forms of the complex are air stable in the solid state; solutions appear to be stable under nitrogen but decompose slowly on exposure to air.

Unit Cells

Monoclinic Crystal.—Optical examination and X-ray diffraction photographs for the zones *Nkl, hNl, hkN* (N = 0–2) proved the initially obtained small red parallelepipeds to belong to the monoclinic system. The systematic absences $h0l$ for $h + l = 2n + 1$ and $0k0$ for $k = 2n + 1$ are compatible with space group $P2_1/n$, a nonstandard setting of space group $P2_1/c$ (C_{2h}^5; no. 14) having the equipoints: $\pm(x, y, z)$; $\pm(^1/_2 + x, {}^1/_2 - y, {}^1/_2 + z)$. Unit cell parameters, from calibrated (NaCl; a = 5.640 Å) precession photographs taken with Mo Kα radiation ($\bar{\lambda}$ 0.7107 Å) at 24 ± 2°, are a = 8.99 ± 0.02 Å, b = 18.56 ± 0.03 Å, c = 14.44 ± 0.02 Å, β = 96.5 ± 0.1°. The unit cell volume is 2394 Å^3. The observed density (by flotation in aqueous zinc iodide) was 2.29 ± 0.02 g cm^{-3}; that calculated for Z = 4 and M = 826.63 is 2.293 g cm^{-3}.

Triclinic Crystal.—A survey of $h0l$, $hk0$, $hk1$ Weissenberg photographs (Cu Kα, $\bar{\lambda}$ 1.5418 Å) and $0kl$, $1kl$, $2kl$, $h0l$ precession photographs (Mo Kα, $\bar{\lambda}$ 0.7107 Å) showed that the thin red platelets were members of the triclinic system. The systematic extinctions hkl for $k + l = 2n + 1$ are consistent with either of the nonstandard space groups A1 (C_1^1; no. 1) or A$\bar{1}$ (C_i^1; no. 2). Unit cell dimensions, obtained by a least-squares analysis of NaCl-calibrated precession photographs (Mo Kα; 23 ± 3°) are a = 15.267 ± 0.010 Å, b = 10.610 ± 0.007 Å, c = 14.862 ± 0.010 Å, α = 92.03 ± 0.05°, β = 101.18 ± 0.05°, γ = 90.08 ± 0.05°, and V = 2360 Å^3. Observed and calculated densities are 2.35 ± 0.02 and 2.326 g cm^{-3} (for Z = 4, M = 826.63). The asymmetric unit is therefore either (i) one molecule in space group A$\bar{1}$ or (ii) two molecules in space group A1. Although no piezoelectric test was performed, it is statistically more likely that the true space group is the centrosymmetric A$\bar{1}$.[15,16] The analysis was initiated anticipating A$\bar{1}$ to be the true space group; the accurate solution to the structure (*vide infra*) proves this assumption to be valid.

Although the A-centered cell is used throughout this paper, it may be noted that the associated primitive "reduced cell," obtained by a Delaunay reduction,[17] is defined by a' = 15.267 ± 0.010 Å, b' = 9.282 ± 0.008 Å, c' = 8.921 ± 0.007 Å, α' = 109.15 ± 0.05°, β' = 99.37 ± 0.05°, and γ' = 90.07 ± 0.05°.

Collection and Reduction of the X-Ray Diffraction Data

For both the monoclinic and triclinic crystal modifications of $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$, intensity data were collected with a 0.01°-incrementing Supper-Pace Buerger automated diffractometer operating in conjunction with a fully stabilized Philips X-ray generator run at 45kV/18mA, a Philips transistorizeds cintillation counter, and a Philips electronics panel, which includes a pulse-height analyzer (PHA). The PHA was adjusted to receive the central 95% of pulses produced by Mo Kα radiation ($\bar{\lambda}$ 0.7107 Å), the Kβ component being virtually eliminated by the use of a 3.0-mil zirconium filter at the X-ray source.

Using drive tapes prepared *via* the IBM 7094 program PREPAR (by G. N. Reeke) the diffractometer was programmed to collect all data within a given zone using a "stationary-background, ω-scan, stationary-background" counting sequence and equiinclination Weissenberg geometry. Within each zone, the stability of the entire assembly was monitored by remeasuring a carefully preselected check reflection after each batch of 20 reflections had been collected. No significant variations from the mean values were detected; this indicates stability of the electronics, the continued alignment of the crystal, and the stability (toward X-rays) of the crystal within ~1–2%.

A chart recorder was employed to check on peak profile, centering of the reflection within the ω scan, and possible overlap of adjacent reflections.

The intensity of each reflection was recorded by means of the "stationary-background, ω-scan, stationary-background" sequence that has been described in detail previously.[18] For a reflection hkl initial and final backgrounds, $B_1(hkl)$ and $B_2(hkl)$, were counted for one-fourth the time of the ω scan, the count associated with this scan being $C(hkl)$.

$I(hkl)$, the integrated intensity for reflection hkl, was calculated as: $I(hkl) = C(hkl) - 2[B_1(hkl) + B_2(hkl)]$.

Data for the monoclinic modification of $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ were collected from two very small parallelepipeds *ca.* 0.1 × 0.1 × 0.1 mm in size and mounted along their a and b axes, respectively. The scan angle was chosen as $\omega = [2.0 + (1.0/L)]°$ (where $1/L$ is the Lorentz factor)[19] after a study of the mosaicity of the crystals as determined from Weissenberg photographs. Intensities for the data sets Hkl and $Hk\bar{l}$ (H = 0–9) were collected from the a-mounted crystal. This represents data to $\sin\theta$ = 0.38 complete save for the few reflections with $\theta \leqslant 4°$ which are shielded from the counter by a lead backstop; the limit

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(15) J. D. H. Donnay, G. Donnay, E. G. Cox, O. Kennard, and M. V. King, "Crystal Data, Determinative Tables," Monograph No. 5, 2nd ed, American Crystallographic Association, 1963, pp 17–67.

(16) W. Nowacki, T. Matsumotu, and A. Edenharter, *Acta Cryst.*, **22**, 935 (1967).

(17) "International Tables for X-Ray Crystallography," Vol. I, The Kynoch Press, Birmingham, England, 1965, pp 530–535.

(18) M. R. Churchill and J. P. Fennessey, *Inorg. Chem.*, **7**, 1123 (1968).

(19) The term involving the Lorentz factor is included to allow for divergence of the X-ray beam which results in extended low-order reflections on upper level data when Υ is positive: D. C. Phillips, *Acta Cryst.*, **7**, 746 (1954).

$\sin\theta = 0.38$ leads to the inclusion of all reflections visible on long-exposure Weissenberg films taken with Mo $K\alpha$ radiation. Data for the quadrants hKl and $hK\bar{l}$ ($K = 0$–4; $\sin\theta_{\max} = 0.38$) were collected from the b-mounted crystal in order to provide a basis for correlation of the a-axis data.

Two platelike crystals were used during the collection of data from the triclinic form of $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$. Crystal A ($0.05 \times 0.19 \times 0.34$ mm) was mounted along its extended c direction and crystal B ($0.04 \times 0.20 \times 0.35$ mm) was mounted on its b axis. [Dimensions refer sequentially to $\vec{a}$, $\vec{b}$, $\vec{c}$.] A careful study of Weissenberg photographs led to the choice of scan angle as $\omega = [1.5 + (1.0/L)]°$, the limit of data collection being $\sin\theta_{\max} = 0.42$. A total of 3731 reflections in the quadrants hkL, $\bar{h}kL$, $h\bar{k}L$, $\bar{h}\bar{k}L$ ($L = 0$–11) were collected from crystal A, and 2423 reflections in the quadrants hKl, $hK\bar{l}$, $\bar{h}Kl$, $\bar{h}K\bar{l}$ ($K = 0$–4) were collected from crystal B for correlation purposes.

Data for the two species were reduced in approximately the same way. Standard deviations were assigned according to the scheme: $I(hkl) > 1225$, $\sigma\{I(hkl)\} = 0.1[I(hkl)]$; $I(hkl) \leqslant 1225$, $\sigma\{I(hkl)\} = 3.5[(I(hkl)]^{1/2}$. Reflections were rejected from the subsequent analysis on two criteria: (1) reflection not significantly above background, *i.e.*, $I(hkl) \leqslant 3[C(hkl) + B_1(hkl) + B_2(hkl)]^{1/2}$; (2) backgrounds grossly asymmetric (indicating a possible overlap problem), *i.e.*, $[B_1(hkl)/B_2(hkl)]$ or $[B_2(hkl)/B_1(hkl)] \geqslant 3$.

All data were adjusted for Lorentz and polarization effects $[(Lp)^{-1} = 2\cos^2\bar{\mu}\sin\Upsilon/(1 + \cos^2 2\theta)]$.[20,21] Absorption corrections were not applied to data from the small monoclinic crystals but were applied[22,23] to data from the platelike crystals of the triclinic modification. With $\mu = 24.74\ \text{cm}^{-1}$, transmission coefficients were found to range from 0.627 to 0.907 for data from crystal A (volume 0.0031 mm^3) and from 0.547 to 0.906 for data from crystal B (volume 0.0038 mm^3). We estimate that neglect of absorption corrections for the approximately cube-shaped monoclinic crystals will involve a maximum error of some 5% in F^2. For each of the two species, the corrected data were merged to a common scale.[24–27] The R factors for scaling (based on F^2 in each case) were *ca.* 7.5% for the monoclinic crystal and *ca.* 6.5% for the triclinic form. The structural analyses were performed using 1844 independent nonzero reflections from the monoclinic crystal, and 3158 independent nonzero reflections from the triclinic polymorph.

The absolute scale of each data set was determined by means of a Wilson plot;[28] this showed the data for the monoclinic crystal form to be about one-third the strength of the triclinic data. Over-all isotropic thermal parameters obtained from the Wilson plot were $\bar{B}$(monoclinic) $= 1.52$ Å^2 and $\bar{B}$(triclinic) $= 1.64$ Å^2.

Elucidation and Refinement of the Crystal Structures

All crystallographic routines were (unless otherwise stated) performed under the CRYM system[29] using the Harvard University IBM 7094 computers. The function minimized during least-squares refinement procedures was $\Sigma w(|F_o|^2 - |F_c|^2)^2$ with $w = [\sigma\{F^2(hkl)\}]^{-2}$ in each case. Discrepancy indices referred to are $R_F = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$ and $R_{wF^2} = \Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w|F_o|^4$. Scattering factors for neutral oxygen and carbon were taken from the compilation of Ibers.[30a] The Thomas–Fermi–Dirac values for neutral ruthenium[30b] were corrected to allow for dispersion ($\Delta f'(\text{Ru}) = 1.2$ e$^-$ and $\Delta f''(\text{Ru}) = +1.1$ e$^-$ for Mo $K\alpha$ radiation).[31] The anisotropic thermal parameter (T) used in the latter stages of refinement is defined by: $T = \exp[-b_{11}h^2 - b_{22}k^2 - b_{33}l^2 - b_{12}hk - b_{13}hl - b_{23}kl]$.

For both monoclinic and triclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$, the positions of all four ruthenium atoms were obtained by inspection of sharpened three-dimensional Patterson functions. The subsequent solution and refinement of the individual crystal structures are outlined below.

Monoclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$.—A difference-Fourier synthesis, phased by the four ruthenium atoms ($R_F = 25.64\%$), led to the unambiguous location of all 31 carbon and oxygen atoms. Seven cycles of full-matrix least-squares refinement of individual positional and isotropic thermal parameters led to convergence at $R_F = 11.13\%$, $R_{wF^2} = 7.31\%$. A second difference-Fourier synthesis now revealed asymmetric features about each ruthenium atom. Four cycles of refinement using anisotropic thermal parameters for ruthenium atoms and isotropic thermal parameters for carbon and oxygen atoms led to convergence at the reduced discrepancy indices—$R_F = 9.88\%$, $R_{wF^2} = 5.95\%$. A third difference-Fourier synthesis now suggested the use of anisotropic thermal parameters for oxygen atoms also. Three cycles of refinement led to convergence ($\Delta/\sigma < 0.05$) at $R_F = 9.49\%$ and $R_{wF^2} = 5.61\%$. A final difference-Fourier synthesis showed no significant features, thus validating the results of the least-squares refinement process. The final "goodness-of-fit"[32] was 2.11. Observed and calculated structure factors for

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(20) Where $\bar{\mu}$ is the equiinclination angle, Υ is the vertical Weissenberg coordinate,[21] and θ is the Bragg angle.

(21) C. T. Prewitt, *Z. Krist.*, **13**, 355 (1960).

(22) Using a locally modified version of GNABS—a general absorption correction program for the IBM 7094, by C. W. Burnham.[23]

(23) C. W. Burnham, *Am. Mineralogist*, **51**, 159 (1966).

(24) Owing to the systematic absence $k + l = 2n + 1$, diffraction data for the *triclinic* crystal fall into two sets—reflections of types h, $2n$, $2n'$ and h, $2n + 1$, $2n' + 1$ (n, n' = integer). Since data have been collected from b and c mountings of the crystal, there is no correlation between the two sets. It was assumed initially that the two sets of data were on the same scale. At a later stage in the analysis a separate scale factor was applied to each set and allowed to refine (see text).

(25) Data were merged using a least-squares program[26] which minimizes a set of residuals linear in the logarithms of the individual scale factors.[27]

(26) DIFCOR, by G. N. Reeke.

(27) A. D. Rae, *Acta Cryst.*, **19**, 683 (1965).

(28) A. J. C. Wilson, *Nature*, **150**, 152 (1942).

(29) CRYM is an integrated sequence of crystallographic routines for the IBM 7094, compiled under the direction of Professor R. E. Marsh at the California Institute of Technology.

(30) "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962: (a) p 202; (b) p 211.

(31) Reference 30, p 216.

(32) The "goodness-of-fit" or "standard error in an observation of unit weight" is defined as $[\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - m)]^{1/2}$ where n is the number of observed reflections and m is the number of variables.

TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS (IN ELECTRONS × 10.00) FOR MONOCLINIC $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$[a]

H	K	FO	FC
L = 0			
2	0	1158	1196
4	0	1072	1077
6	0	2690	2683
8	0	1058	843
2	1	601	650
3	1	1833	1681
4	1	1018	922
5	1	675	701
6	1	926	906
7	1	525	50
9	1	784	794
2	2	1087	900
3	2	355	253
4	2	946	1089
5	2	961	1027
6	2	732	628
7	2	1664	1650
8	2	446	356
2	3	551	429
3	3	1101	1026
4	3	841	874
5	3	611	654
6	3	734	739
7	3	1523	1552
8	3	383	212
9	3	522	472
0	4	970	1233
1	4	1273	1250
2	4	1466	1294
3	4	2290	2204
4	4	460	343
5	4	381	131
6	4	766	698
7	4	918	899
8	4	386	158
9	4	380	193
1	5	2230	2153
2	5	1167	1121
3	5	919	799
4	5	381	355
5	5	424	337
6	5	1310	1298
7	5	333	174
8	5	799	853
9	5	483	516
0	6	4066	4257
1	6	1382	1258
2	6	1120	977
3	6	1056	998
6	6	1563	1558
7	6	841	681
8	6	625	648
9	6	536	480
1	7	1194	1120
2	7	1316	1330
4	7	517	554
5	7	978	963
6	7	404	471
7	7	784	717
8	7	499	400
0	8	2149	1981
1	8	2941	2987
2	8	1487	1524
5	8	1579	1606
6	8	612	489
7	8	823	856
8	8	1074	1010
2	9	1230	1249
3	9	906	931
4	9	1230	1197
5	9	627	661
6	9	723	702
7	9	491	402
0	10	1886	1930
1	10	1173	1204
2	10	853	698
4	10	1194	1170
6	10	935	921
7	10	895	922
1	11	1038	981
2	11	612	690
6	11	632	680
5	11	1081	1107
7	11	570	455
0	12	1099	1078
3	12	475	29
5	12	615	584
6	12	485	434
1	13	1268	1190
2	13	662	562
3	13	498	519
4	13	565	494
5	13	571	524
6	13	797	806
7	13	609	613
0	14	1235	1303
2	14	960	1047
5	14	519	284
6	14	429	241
2	15	1230	1261
0	16	426	441
1	16	896	1120
2	16	445	101
3	16	902	904
4	16	492	513
1	17	812	743
4	17	523	580
0	18	849	782
1	18	558	393
2	18	680	586
4	18	709	695
1	19	588	512
L = 1			
-9	0	498	515
-7	0	1688	1672
-5	0	1829	1827
-3	0	3234	2714
3	0	605	497
5	0	1793	1690
7	0	1815	1818
9	0	600	311
-9	1	897	926
-8	1	484	540
-7	1	726	703
-6	1	406	430
-5	1	534	620
-4	1	2296	2314
-3	1	1940	1825
3	1	567	525
4	1	1160	1144
5	1	625	718
7	1	1061	1022
8	1	367	294
9	1	526	450
-8	2	369	400
-7	2	697	228
-6	2	1477	1488
-5	2	642	638
-4	2	369	372
-3	2	289	306
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4	0	883	826
-2	1	1322	1402
-1	1	613	271
1	1	674	673
2	1	468	353
4	1	736	710
-4	2	803	670
-2	2	677	443
-1	2	494	74
5	2	608	711
-5	3	520	344
-3	3	1325	1250
-2	3	563	414
-1	3	1443	1485
0	3	622	474
3	3	696	694
-6	4	544	529
-5	4	718	811
-3	4	696	671
-2	4	437	294
0	4	498	554
2	4	441	396
3	4	573	567
-3	5	619	585
-2	5	1584	1431
0	5	503	628
4	5	1409	1293
-2	7	881	825
2	7	674	604
3	7	621	633
4	7	528	358
-3	8	530	635
-2	8	510	384
0	8	832	804
-3	9	614	723
-2	9	484	334
0	9	448	483
-4	10	796	770
-3	10	463	529
-2	10	574	488
-3	11	680	661
-2	11	667	661
-1	11	875	892
1	11	650	460
-2	12	552	362
-1	12	567	581
L = 13			
-5	0	511	457
-3	0	565	8
1	0	458	354
3	0	483	504
-5	1	419	358
-3	1	554	519
-2	1	526	429
-1	1	659	515
1	1	647	680
2	1	583	608
-5	2	807	827
0	2	525	409
2	2	417	107
3	2	495	502
4	2	497	592
-5	3	530	232
-3	3	451	273
-2	3	1472	1515
4	3	1273	1274
-5	4	466	440
-4	4	1002	986
-3	4	467	122
-2	4	535	448
0	4	611	607
1	4	618	517
2	4	498	617
4	4	523	617
-3	5	774	767
-2	5	690	661
-1	5	839	913
3	5	814	818
-5	6	909	806
-3	6	477	262
0	6	675	838
-7	7	423	477
1	7	856	880
-1	8	407	453
1	8	489	15
2	8	428	136
-2	9	492	537
0	9	407	225
L = 14			
-2	0	498	400
0	0	393	425
2	0	490	384
-3	1	469	5
-2	1	729	669
0	1	675	747
1	1	860	784
2	1	562	580
-4	2	596	647
0	2	542	591
1	2	547	520
2	2	479	445
-3	3	757	704
-2	3	601	83
1	3	560	508
-4	4	408	202
-3	4	546	388
0	4	727	680
1	4	657	642
-2	5	850	837
1	5	430	448
-1	6	632	521
0	6	444	493
-1	7	593	478
0	7	652	615
L = 15			
-3	0	546	570
-1	0	445	85
1	0	614	584
-1	1	507	404
0	1	684	637
1	1	959	974
0	2	594	514
1	2	540	513
-1	3	525	76
0	3	569	609
0	4	591	589

[a] This table shows h, k, $10|F_o|$, $10|F_c|$ in blocks of constant l.

monoclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ are shown in Table I. Atomic parameters are collected in Table II.

Triclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$.—A three-dimensional difference-Fourier synthesis, phased by the four ruthenium atoms ($R_F = 34.01\%$), yielded the positions for all remaining nonhydrogen atoms. Three cycles of full-matrix least-squares refinement of positional and individual isotropic thermal parameters resulted in convergence at $R_F = 11.21\%$ and $R_{wF^2} = 9.60\%$. A second difference-Fourier synthesis now showed evidence of anisotropic motion for many atoms, but no attempt was made to locate hydrogen atoms. Three cycles of refinement of individual positional parameters for all atoms, anisotropic thermal parameters for ruthenium atoms, and isotropic thermal parameters for other atoms resulted in convergence at $R_F = 7.71\%$ and $R_{wF^2} = 5.03\%$. At this point the two sets of data (l even, l odd)[24] were separated, and the scale factor for each set was refined. The two sets were found to have scales which differed by *ca.* 4%. The two sets were placed on a common scale and a single scale factor was used during subsequent refinement processes. Refinement was continued using anisotropic thermal parameters for all atoms. The limited size of the computer storage (32*K*) necessitated the blocking of variables into three submatrices. Matrix I contained the scale factor, plus positional and anisotropic thermal parameters for the ruthenium atoms and C(1) through C-

Table II

Final Parameters for Monoclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$

(A) Atomic Positions and Isotropic Thermal Parameters, with Esd's[a]

Atom	x	y	z	B, Å^2
Ru(1)	0.11695 (24)	0.18200 (15)	0.16656 (18)	...
Ru(2)	0.22572 (27)	0.04454 (15)	0.23734 (19)	...
Ru(3)	0.25189 (26)	0.16887 (16)	0.35808 (19)	...
Ru(4)	0.42366 (24)	0.15130 (16)	0.21653 (19)	...
O(1)	0.2969 (32)	0.3245 (19)	0.3884 (22)	...
O(2)	0.5268 (28)	0.1526 (14)	0.4902 (22)	...
O(3)	0.4678 (27)	−0.0609 (13)	0.2783 (21)	...
O(4)	0.1976 (39)	−0.0144 (19)	0.0394 (21)	...
O(5)	0.1856 (28)	0.1538 (17)	−0.0261 (17)	...
O(6)	0.2671 (26)	0.3260 (15)	0.1526 (18)	...
O(7)	0.5956 (28)	0.2906 (14)	0.2268 (26)	...
O(8)	0.6819 (29)	0.0748 (20)	0.3289 (19)	...
O(9)	0.5303 (29)	0.0953 (16)	0.0357 (20)	...
C(1)	−0.1034 (30)	0.1271 (18)	0.1434 (21)	2.2 (6)
C(2)	−0.1219 (37)	0.2047 (22)	0.1375 (25)	3.6 (7)
C(3)	−0.0748 (32)	0.2260 (18)	0.2341 (22)	2.5 (6)
C(4)	0.0179 (31)	0.1662 (18)	0.3997 (22)	2.4 (6)
C(5)	0.0982 (30)	0.1145 (17)	0.4417 (20)	2.1 (6)
C(6)	0.1610 (36)	0.0536 (20)	0.3990 (24)	3.4 (7)
C(7)	0.0696 (36)	0.0052 (20)	0.3290 (24)	3.0 (6)
C(8)	−0.0187 (35)	0.0218 (20)	0.2534 (24)	2.9 (7)
C(9)	−0.0417 (26)	0.0975 (15)	0.2284 (18)	1.0 (5)
C(10)	−0.0287 (36)	0.1660 (20)	0.2888 (25)	3.2 (7)
C(11)	−0.0590 (47)	0.2356 (26)	0.4444 (31)	5.4 (9)
C(12)	0.2564 (40)	0.0061 (24)	0.4749 (27)	4.3 (8)
C(13)	−0.1062 (42)	−0.0331 (24)	0.1999 (27)	4.4 (8)
C(14)	0.2836 (43)	0.2625 (24)	0.3664 (29)	4.2 (9)
C(15)	0.4239 (37)	0.1594 (19)	0.4351 (24)	3.0 (7)
C(16)	0.3827 (44)	−0.0263 (24)	0.2666 (28)	4.0 (8)
C(17)	0.2168 (43)	0.0081 (25)	0.1161 (30)	4.4 (9)
C(18)	0.1659 (37)	0.1625 (21)	0.0489 (26)	3.2 (7)
C(19)	0.2223 (40)	0.2718 (24)	0.1618 (27)	3.5 (8)
C(20)	0.5286 (41)	0.2443 (23)	0.2215 (27)	3.8 (8)
C(21)	0.5782 (41)	0.1046 (23)	0.2836 (27)	3.8 (8)
C(22)	0.4861 (38)	0.1174 (22)	0.0988 (27)	3.7 (7)

(B) Anisotropic Thermal Parameters (with Esd's[a]) for Ruthenium and Oxygen Atoms

Atom	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
Ru(1)	52.3 (2.7)	18.1 (0.9)	30.7 (1.4)	5.4 (2.7)	0.4 (3.2)	−0.3 (1.9)
Ru(2)	86.3 (3.3)	17.2 (0.9)	37.9 (1.6)	9.0 (3.0)	18.4 (3.7)	2.7 (2.0)
Ru(3)	75.5 (3.2)	21.7 (1.0)	32.5 (1.6)	4.6 (3.0)	−10.6 (3.6)	−7.4 (2.0)
Ru(4)	49.6 (2.9)	23.1 (1.0)	39.4 (1.6)	6.8 (2.8)	12.2 (3.4)	3.2 (2.1)
O(1)	203 (41)	51 (15)	113 (24)	−96 (44)	−149 (54)	−7 (31)
O(2)	148 (39)	26 (10)	101 (22)	37 (33)	−135 (51)	−32 (24)
O(3)	114 (33)	20 (9)	107 (23)	46 (31)	−5 (45)	20 (23)
O(4)	304 (61)	46 (15)	63 (20)	32 (50)	−64 (59)	−49 (27)
O(5)	181 (38)	58 (14)	33 (16)	−59 (40)	75 (41)	20 (24)
O(6)	162 (37)	22 (10)	54 (17)	35 (33)	24 (41)	−7 (21)
O(7)	132 (37)	10 (8)	195 (32)	1 (31)	147 (58)	5 (27)
O(8)	147 (40)	86 (19)	52 (17)	−37 (46)	53 (45)	64 (29)
O(9)	174 (41)	34 (11)	67 (19)	19 (37)	69 (48)	−28 (25)

(C) Thermal Vibration Ellipsoids for Ruthenium and Oxygen Atoms[b,c]

Atom	B_{maj}, Å^2	B_{med}, Å^2	B_{min}, Å^2	Atom	B_{maj}, Å^2	B_{med}, Å^2	B_{min}, Å^2
Ru(1)	2.65	2.49	1.60	O(4)	12.05	6.78	2.91
Ru(2)	3.22	2.79	2.21	O(5)	9.15	5.58	1.38
Ru(3)	3.53	2.66	2.01	O(6)	5.73	4.55	2.45
Ru(4)	3.39	3.07	1.54	O(7)	16.38	3.31	0.144
O(1)	13.91	8.02	1.95	O(8)	13.28	5.21	2.08
O(2)	12.19	2.99	2.36	O(9)	6.38	5.84	2.82
O(3)	9.19	4.76	1.44				

[a] Esd's (estimated standard deviations) are shown in parentheses in sections A and B of this table; they are right-adjusted to the least significant digit of the preceding number. [b] The major, median, and minor axes of the thermal vibration ellipsoids are defined in terms of the isotropic thermal parameter B, which is related to the root-mean-square displacement, $(\overline{U^2})^{1/2}$, by: $(\overline{U^2})^{1/2} = [B/8\pi^2]^{1/2}$. [c] The vibration ellipsoids for monoclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ are depicted in Figure 1.

(13); matrix II contained all parameters for C(14) through C(17) and O(1) through O(4); matrix III contained appropriate parameters for C(18) through C(22) and O(5) through O(9). Four cycles of refinement led to convergence ($\Delta/\sigma < 0.05$ for each parameter) at $R_F = 6.35\%$ and $R_{wF^2} = 3.84\%$. A final difference-Fourier synthesis was devoid of significant features, thus confirming the correctness of the structure. The final "goodness-of-fit"[32] was 1.99. Observed and calculated structure factors for triclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ are collected in Table III; all atomic parameters are compiled in Table IV.

The Molecular Structure

The monoclinic and triclinic crystal modifications of $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ contain molecules whose configurations are identical within experimental error. Interatomic distances for the two species are compared in Table V. Bond angles are similarly collected in Table VI. Figures 1 and 2 show the monoclinic and triclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecules, respectively, projected onto the least-squares plane through C(4), C(8), C(9), and C(10) and also show the 68% probability envelopes for the thermal vibration ellipsoids.[33] The $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule possesses approximate (although not exact) C_s symmetry about the plane which passes through Ru(1), Ru(4), and the midpoint of the Ru(2)–Ru(3) bond.

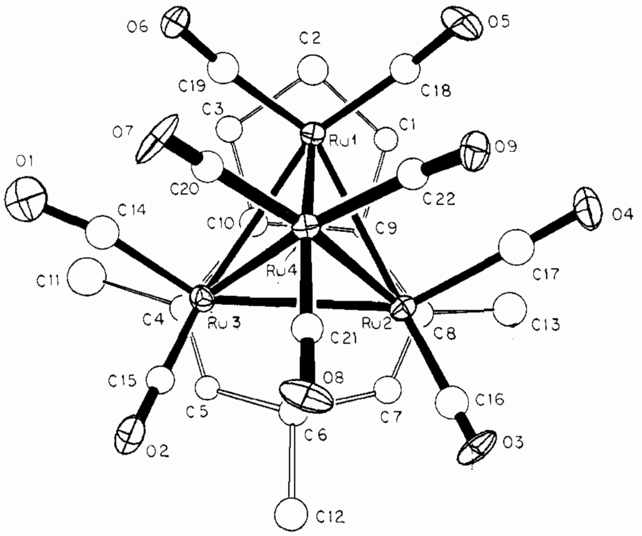

Figure 1.—The monoclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule, projected onto the plane defined by C(4), C(8), C(9), C(10). The 68% probability envelopes of the thermal vibration ellipsoids are shown (OTLIPS[33] diagram).

Since the crystallographic analysis of the triclinic crystal form is more accurate (*i.e.*, lower esd's on bond lengths, bond angles, etc.) than that of the monoclinic modification,[34] all data in the discussion will (unless otherwise stated) pertain to the triclinic crystal form.

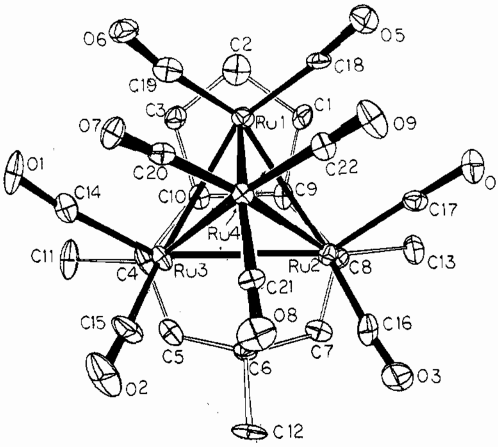

Figure 2.—The triclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule, projected onto the plane defined by C(4), C(8), C(9), C(10) (OTLIPS[33] diagram).

In each case, however, the corresponding data for the monoclinic form are available in the appropriate table.

The $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule consists of a 4,6,8-trimethylazulene ligand arched across the largest face of an irregular tetrahedron of ruthenium atoms in which individual ruthenium–ruthenium distances vary from 2.698 (3) to 2.908 (3) Å [2.702 (5) to 2.90 (5) Å in the monoclinic form]. The three ruthenium atoms which are associated with the azulene ligand [*i.e.*, Ru(1), Ru(2), Ru(3)] are each associated with two terminal carbonyl ligands; the apical Ru(4) is associated with three terminal carbonyl groups and is not directly linked to the azulene ligand—rather, it is linked to the other three ruthenium atoms *via* σ bonds.

The present $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule may be regarded as derived from the as yet unknown $Ru_4(CO)_{14}$ molecule. Although neither $Ru_4(CO)_{14}$ nor the isoelectronic $Fe_4(CO)_{14}$ is known, derivatives of each of these species have been reported. Thus, salts of the $Fe_4(CO)_{13}^{2-}$ dianion are known[35–43] and the crystal structure of $[Fe(C_5H_5N)_6^{2+}][Fe_4(CO)_{13}^{2-}]$ has been described.[44] Similarly, the known ruthenium carbonyl hydrides $H_2Ru_4(CO)_{13}$[45,46] and $H_4Ru_4(CO)_{12}$[45–48] may be regarded as derived from the unknown $Ru_4(CO)_{14}$.

The over-all stereochemistry of the $[(CH_3)_3C_{10}H_5]$-$Ru_4(CO)_9$ molecule is best seen in Figure 3, which shows the molecule projected onto the plane defined by Ru(1), Ru(2), and Ru(4). The 4,6,8-trimethylazulene ligand

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[33] Diagrams of thermal vibration ellipsoids were constructed using OTLIPS—an IBM 1620/CALCOMP program written by Dr. P. H. Bird.

[34] The lower accuracy of the crystallographic analysis of the monoclinic modification may primarily be attributed to the use of very small crystals for collection of diffraction data (see text).

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TABLE III

OBSERVED AND CALCULATED STRUCTURE FACTORS (IN ELECTRONS × 10.00) FOR TRICLINIC $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$[a]

H	K	FO	FC
L = -17			
0	1	259	80
1	1	295	353
2	1	735	654
3	1	244	277
4	1	529	496
6	1	258	223
7	1	420	353
2	3	314	270
3	3	460	461
5	3	614	676
L = -16			
1	2	1001	994
2	2	400	407
3	2	799	894
4	2	522	579
6	2	394	330
7	2	599	541
8	2	347	308
9	2	249	299
10	2	355	369
0	4	503	493
1	4	295	272
3	4	307	317
4	4	402	396
6	4	672	680
7	4	378	382
9	4	545	581
L = -15			
0	1	926	937
2	1	1261	1295
3	1	616	672
4	1	748	735
5	1	247	261
7	1	564	581
8	1	490	424
9	1	1170	1165
11	1	735	756
12	1	460	401
0	3	880	938
2	3	1229	1290
3	3	283	309
4	3	581	621
5	3	302	364
9	3	301	92
10	3	273	200
11	3	565	580
L = -14			
0	2	434	380
1	2	1863	1952
3	2	1563	1635
5	2	842	896
6	2	484	514
7	2	354	326
8	2	747	765
11	2	278	322
13	2	802	753
0	4	395	331
1	4	621	569
7	4	234	182
9	4	886	870
10	4	852	813
11	4	716	647
L = -13			
0	1	1209	1282
1	1	228	258
2	1	1436	1483
3	1	340	340
4	1	603	653
5	1	386	385
6	1	330	258
7	1	979	1004
8	1	435	372
9	1	495	640
12	1	1349	1257
13	1	340	233
14	1	1352	1219
0	3	1115	1138
1	3	273	291
2	3	1346	1413
4	3	843	913
5	3	870	881
6	3	576	637
7	3	1226	1239
8	3	591	609
9	3	342	354
10	3	474	498
11	3	835	820
12	3	329	237
13	3	536	497
L = -12			
0	2	536	526
1	2	1374	1389
2	2	406	453
3	2	918	922
4	2	985	996
5	2	518	565
6	2	1751	1712
7	2	924	877
8	2	1370	1328
9	2	683	671
10	2	273	328
11	2	530	476
12	2	667	625
13	2	1275	1191
15	2	803	725
0	4	601	608
1	4	713	757
2	4	424	439
4	4	771	778
5	4	208	170
6	4	1313	1345
8	4	878	854
9	4	732	648
10	4	272	157
11	4	246	289
12	4	409	348
13	4	283	233
L = -11			
1	1	572	545
3	1	399	400
4	1	228	245
5	1	965	954
6	1	702	713
7	1	684	737
8	1	860	903
9	1	252	245
11	1	838	826
12	1	1110	1129
13	1	349	274
14	1	1083	1147
0	3	1218	1290
1	3	395	378
2	3	276	204
3	3	932	954
4	3	220	220
5	3	2382	2376
6	3	510	495
7	3	1893	1907
8	3	279	261
9	3	740	797
10	3	231	218
12	3	661	643
13	3	220	164
1	5	1159	1023
4	5	932	844
5	5	621	563
6	5	402	365
14	5	759	807
1	7	678	639
2	7	477	332
3	7	767	737
4	7	285	385
5	7	701	723
7	7	1263	1152
8	7	461	442
9	7	1443	1349
11	7	497	563
2	9	876	816
4	9	425	467
7	9	427	346
8	9	698	569
L = -10			
0	2	448	457
1	2	425	436
3	2	1045	978
4	2	1334	1333
5	2	248	258
6	2	2295	2292
7	2	676	701
8	2	1236	1272
11	2	601	647
13	2	642	666
14	2	365	274
15	2	337	346
16	2	808	859
0	4	1151	1193
1	4	1140	1186
2	4	367	339
3	4	262	245
4	4	1658	1674
6	4	1784	1806
8	4	1003	1065
9	4	897	854
10	4	428	430
11	4	1081	1085
12	4	516	582
14	4	590	566
15	4	674	649
1	6	1179	1176
2	6	385	370
6	6	329	346
8	6	884	841
10	6	897	838
11	6	302	338
13	6	381	376
14	6	510	567
1	8	872	816
3	8	810	772
6	8	651	673
7	8	1053	907
8	8	1361	1310
9	8	764	697
10	8	1145	1094
1	10	1012	1000
3	10	684	688
L = -9			
0	1	1555	1584
1	1	1554	1473
2	1	2505	2467
3	1	370	371
4	1	1985	1991
5	1	791	799
6	1	1139	1184
7	1	329	345
8	1	1210	1256
9	1	238	224
12	1	459	478
14	1	231	142
15	1	625	642
16	1	371	153
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8	-9	241	125

adopts a distinctly nonplanar conformation [see Figure 4] and bends about C(9)–C(10), C(8)···C(4), and C(7)···C(5) such that there is a dihedral angle of 125° 44′ between the planar five-membered ring and the plane defined by C(5), C(6), and C(7) [see Table VII]. The three methyl groups (for which the central carbon atoms are C(11), C(12), and C(13)) are each bent away from the $Ru_4(CO)_9$ moiety. Each of these carbon atoms is sufficiently distant (>3.3Å; see Table V) from the azulene-bonded ruthenium atoms as to exclude the possibility of hydride abstraction from a methyl group with concomitant metal···methylene interaction (as has been observed in 1,3,5-trimethyl-7-methylenecycloocta-1,3,5-trienediiron pentacarbonyl[49]).

The metal-to-azulene bonding in $[(CH_3)_3C_{10}H_5]Ru_4$-

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(49) F. A. Cotton and J. Takats, *J. Am. Chem. Soc.*, **90**, 2031 (1968).

TABLE III (*Continued*)

H	K	FO	FC
***************			
L = 5			
10	-9	375	455
0	-7	666	682
1	-7	439	419
2	-7	896	892
3	-7	800	838
4	-7	1689	1743
6	-7	1144	1157
9	-7	366	312
11	-7	378	376
12	-7	337	408
0	-5	1100	1080
1	-5	678	133
2	-5	762	756
3	-5	733	852
4	-5	369	354
5	-5	223	128
6	-5	697	577
7	-5	255	389
8	-5	475	610
9	-5	320	137
10	-5	585	580
12	-5	884	943
13	-5	360	363
14	-5	786	772
0	-3	1706	1722
1	-3	929	901
2	-3	2134	2078
3	-3	636	587
4	-3	943	993
5	-3	2112	2170
6	-3	1972	2066
7	-3	1603	1669
8	-3	2003	2119
9	-3	563	586
10	-3	795	795
12	-3	598	657
14	-3	485	499
0	-1	218	156
1	-1	1552	1508
2	-1	321	286
3	-1	1415	1421
4	-1	1596	1634
5	-1	1190	1262
6	-1	446	436
7	-1	1031	1023
8	-1	590	625
9	-1	432	425
10	-1	428	466
11	-1	634	661
14	-1	584	640
-16	1	352	382
-15	1	418	319
-14	1	1187	1351
-13	1	690	865
-12	1	1515	1656
-11	1	1418	1441
-10	1	341	319
-9	1	197	252
-8	1	385	388
-7	1	491	478
-6	1	245	237
-5	1	633	621
-4	1	324	322
-3	1	1422	1510
-2	1	1623	1603
-1	1	596	552
0	1	1594	1505
1	1	1975	1895
2	1	1911	1944
3	1	1916	1927
4	1	3718	3934
5	1	554	490
6	1	1876	1949
7	1	667	725
8	1	891	892
9	1	1530	1584
10	1	323	310
11	1	828	784
12	1	339	265
13	1	277	268
14	1	504	511
15	1	443	420
-17	3	258	309
-16	3	273	105
-15	3	673	738
-13	3	471	498
-10	3	869	883
-9	3	412	366
-8	3	651	657
-7	3	204	197
-6	3	892	854
-5	3	2996	3111
-4	3	918	924
-3	3	4232	4362
-2	3	684	700
-1	3	3292	3318
1	3	913	961
2	3	1250	1272
3	3	208	154
4	3	1764	1729
5	3	386	393
7	3	771	797
8	3	673	706
9	3	1323	1408
10	3	619	591
11	3	457	489
12	3	990	1027
13	3	508	563
14	3	531	561
15	3	678	692
-15	5	373	452
-14	5	273	356
-12	5	1106	1247
-11	5	269	325
-10	5	1807	1972
-9	5	956	1093
-8	5	608	751
-7	5	1069	1123
-6	5	441	536
-5	5	1395	1152
-4	5	350	354
-3	5	1515	1557
-2	5	312	321
-1	5	204	215
0	5	253	146
1	5	551	443
2	5	529	567
3	5	1182	1206
4	5	1384	1411
5	5	1276	1306
6	5	2291	2387
7	5	1095	1192
8	5	1490	1631
9	5	622	510
11	5	390	431
12	5	729	788
13	5	519	566
14	5	360	412
-13	7	557	570
-12	7	350	364
-11	7	440	457
-10	7	570	652
-8	7	249	223
-7	7	1011	1061
-6	7	360	316
-5	7	811	888
-3	7	1082	1183
-2	7	354	436
-1	7	2234	2348
0	7	375	359
1	7	1836	1958
3	7	434	385
5	7	1282	1305
6	7	901	926
7	7	931	994
8	7	949	989
9	7	629	658
12	7	245	151
-12	9	544	527
-11	9	588	688
-10	9	526	503
-8	9	1014	1117
-7	9	289	224
-6	9	727	747
-5	9	250	217
-4	9	378	419
-3	9	338	208
-2	9	1150	1180
-1	9	926	983
0	9	668	677
1	9	1295	1351
2	9	329	363
3	9	342	383
4	9	330	356
7	9	534	555
8	9	319	336
9	9	687	706
-6	11	381	408
-5	11	716	682
-3	11	1097	1095
-2	11	453	489
-1	11	560	589
1	11	349	265
2	11	552	561
4	11	592	582
***************			
L = 6			
0	-10	510	519
1	-10	444	369
2	-10	529	456
6	-10	611	498
7	-10	321	344
8	-10	356	328
0	-8	261	164
2	-8	834	806
3	-8	1231	1221
5	-8	1379	1362
6	-8	577	604
7	-8	502	449
8	-8	519	493
10	-8	714	672
0	-6	1092	1040
1	-6	493	487
2	-6	582	598
3	-6	1195	1158
4	-6	500	564
5	-6	771	749
6	-6	408	416
8	-6	852	903
9	-6	281	236
10	-6	727	698
11	-6	632	652
12	-6	262	155
13	-6	692	645
1	-4	549	589
2	-4	283	226
3	-4	605	637
4	-4	802	777
5	-4	480	517
6	-4	1780	1879
7	-4	1536	1597
8	-4	785	837
9	-4	913	890
11	-4	478	514
13	-4	782	772
14	-4	310	226
0	-2	2814	2687
1	-2	1366	1355
2	-2	1584	1529
4	-2	980	1063
5	-2	512	456
6	-2	2607	2697
7	-2	1474	1490
8	-2	1219	1229
9	-2	874	896
10	-2	386	380
14	-2	361	386
15	-2	412	469
-15	0	991	1004
-14	0	752	822
-13	0	1640	1551
-12	0	1115	1046
-11	0	1649	1540
-10	0	651	630
-9	0	1426	1308
-8	0	929	868
-7	0	1439	1339
-6	0	1510	1403
-5	0	699	646
-3	0	2095	1967
-2	0	632	713
-1	0	2457	2429
0	0	3451	3157
1	0	843	804
2	0	2024	1881
3	0	1648	1542
4	0	372	225
5	0	1347	1372
8	0	759	765
9	0	361	320
10	0	840	780
11	0	392	396
13	0	431	408
14	0	433	481
15	0	1006	954
-17	2	384	374
-15	2	357	411
-14	2	342	277
-13	2	732	742
-12	2	438	386
-11	2	583	516
-9	2	691	684
-8	2	538	540
-7	2	1170	1221
-6	2	1449	1392
-5	2	1308	1249
-4	2	3327	3294
-3	2	749	797
-2	2	2821	2721
-1	2	881	853
0	2	1183	1250
1	2	809	806
2	2	234	195
3	2	2617	2527
4	2	573	509
5	2	591	657
6	2	1575	1661
8	2	2472	2527
10	2	1435	1482
11	2	600	587
13	2	244	235
14	2	393	384
15	2	338	317
-15	4	484	108
-13	4	682	682
-11	4	1962	1962
-10	4	782	852
-9	4	1389	1403
-8	4	996	985
-6	4	399	445
-5	4	713	699
-4	4	2772	2765
-2	4	2295	2246
-1	4	738	743
0	4	791	772
1	4	1844	1771
2	4	983	997
3	4	1270	1280
4	4	1096	1101
5	4	745	735
6	4	377	396
7	4	1169	1209
8	4	541	579
9	4	272	307
10	4	700	743
11	4	381	382
12	4	281	208
13	4	400	427
14	4	310	273
-15	6	329	119
-13	6	473	516
-12	6	435	261
-11	6	1567	1628
-9	6	1466	1545
-8	6	313	311
-7	6	400	416
-6	6	527	122
-3	6	620	354
-2	6	1188	1191
-1	6	258	266
0	6	1847	1827
1	6	595	618
3	6	546	524
4	6	1626	1609
5	6	773	704
6	6	1297	1219
7	6	1440	1350
8	6	959	936
10	6	989	986
11	6	297	288
12	6	888	887
-13	8	402	427
-12	8	524	578
-9	8	615	627
-7	8	913	902
-6	8	412	441
-4	8	548	512
-3	8	1197	1162
-2	8	1334	1315
-1	8	1727	1681
0	8	2147	2131
1	8	790	729
2	8	1048	1040
3	8	378	320
4	8	324	346
5	8	617	592
7	8	522	491
9	8	523	473
10	8	282	286
-9	10	963	958
-7	10	1232	1235
-4	10	304	228
-3	10	731	695
-1	10	600	533
6	10	834	790
***************			
L = 7			
0	-11	667	633
1	-11	452	393
2	-11	350	313
0	-9	989	993
1	-9	787	710
3	-9	755	685
4	-9	642	671
9	-9	359	257
0	-7	589	530
1	-7	693	677
2	-7	847	825
3	-7	790	804
4	-7	1069	1061
5	-7	920	942
6	-7	481	341
7	-7	1080	1020
8	-7	413	408
9	-7	1265	1220
11	-7	817	749
0	-5	1241	1242
1	-5	412	463
2	-5	1494	1457
3	-5	508	540
4	-5	886	890
5	-5	821	797
6	-5	377	313
8	-5	718	771
9	-5	605	598
11	-5	307	271
12	-5	714	631
0	-3	600	582
1	-3	1120	1092
2	-3	486	480
3	-3	695	708
4	-3	204	215
5	-3	2496	2614
6	-3	811	798
7	-3	2042	2117
8	-3	1036	1079
9	-3	903	949
11	-3	545	623
12	-3	483	518
13	-3	541	539
0	-1	1567	1528
1	-1	2239	2218
2	-1	568	617
4	-1	275	285
5	-1	1525	1476
6	-1	768	735
7	-1	320	326
8	-1	259	269
10	-1	694	708
11	-1	363	407
13	-1	734	766
14	-1	441	506
-15	1	372	324
-14	1	944	999
-13	1	534	545
-12	1	1315	1361
-10	1	1602	1636
-9	1	1264	1269
-8	1	2360	2456
-7	1	1202	1196
-6	1	2001	2079
-4	1	811	760
-3	1	1089	1117
-2	1	2267	2221
-1	1	465	438
0	1	1850	1770
1	1	1045	1020
2	1	614	606
3	1	481	476
4	1	369	289
5	1	866	834
6	1	557	567
7	1	1877	1882
9	1	1571	1614
10	1	388	401
11	1	540	559
13	1	262	169
14	1	539	605
-17	3	271	237
-15	3	472	462
-13	3	434	476
-12	3	532	558
-11	3	402	393
-10	3	789	781
-9	3	1556	1620
-8	3	417	436
-7	3	402	422
-6	3	861	876
-5	3	2140	2108
-3	3	2526	2458
-2	3	571	568
-1	3	1323	1251
0	3	1935	1922
1	3	760	749
2	3	2449	2445
3	3	1738	1710
4	3	1298	1244
5	3	568	576
6	3	988	974
7	3	1531	1537
8	3	596	628
9	3	1838	1940
11	3	791	802
14	3	261	215
-14	5	319	229
-12	5	1440	1557
-11	5	458	186
-10	5	2135	2310
-8	5	1088	1158
-7	5	383	407
-6	5	577	548
-5	5	1246	1061
-4	5	470	404
-3	5	343	283
-2	5	239	236
-1	5	411	430
0	5	1529	1485
1	5	523	509
2	5	1925	1934
3	5	982	1022
4	5	513	510
5	5	1058	1065
6	5	550	556
7	5	317	288
8	5	557	532
11	5	882	856
13	5	619	646
-13	7	358	93
-12	7	251	374
-11	7	410	114
-10	7	459	438
-9	7	276	326
-7	7	622	543
-5	7	579	621
-4	7	918	1103
-3	7	741	715
-2	7	1254	1251
-1	7	1875	1837
0	7	862	696
1	7	1463	1464
3	7	360	364
4	7	454	437
5	7	489	734
6	7	703	738
7	7	706	510
8	7	524	269
9	7	707	728
10	7	860	794
11	7	1104	1119
-11	9	324	272
-10	9	808	800
-8	9	1403	1379
-6	9	827	821
-4	9	328	318
-3	9	616	596
-2	9	994	956
-1	9	877	837
0	9	767	712
1	9	396	381
2	9	976	968
3	9	371	374
4	9	1233	1175
6	9	476	470
7	9	770	732
8	9	329	377
-1	11	257	268
0	11	531	585
1	11	791	724
2	11	403	441
***************			
L = 8			
0	-10	835	796
1	-10	986	969
2	-10	679	613
3	-10	261	108
6	-10	586	495
0	-8	759	668
1	-8	374	341
2	-8	545	531
4	-8	357	290
6	-8	502	474
8	-8	953	837
0	-6	625	528
1	-6	1620	1531
3	-6	1455	1351
5	-6	300	246
6	-6	536	564
7	-6	670	629
8	-6	1154	1076
10	-6	1002	945
0	-4	686	625
1	-4	1756	1748
2	-4	313	318
3	-4	1558	1534
4	-4	1230	1205
5	-4	562	588
6	-4	1120	1143
7	-4	717	692
10	-4	303	277
11	-4	486	469
12	-4	350	309
13	-4	226	262
0	-2	2579	2479
1	-2	671	626
2	-2	876	830
4	-2	1549	1557
5	-2	391	393
6	-2	1455	1434
7	-2	258	259
8	-2	420	465
9	-2	489	519
10	-2	866	922
11	-2	684	707
12	-2	1115	1131
13	-2	265	146
-17	0	299	214
-16	0	441	373
-15	0	567	501
-13	0	603	532
-12	0	250	35
-11	0	788	775
-10	0	631	623
-9	0	1959	1791
-8	0	1278	1176
-7	0	2907	2846
-6	0	653	648
-5	0	628	609
-4	0	503	69
-3	0	3015	2907
-2	0	890	903
-1	0	3065	2990
0	0	1534	1283
1	0	1652	1537
2	0	246	74
3	0	1307	1373
4	0	718	611
5	0	1825	1843
7	0	696	609
8	0	952	965
9	0	671	680
10	0	341	313
11	0	597	612
12	0	417	392
14	0	667	490
-16	2	407	431
-15	2	318	325
-14	2	715	745
-13	2	402	399
-11	2	247	240
-10	2	1614	1635
-9	2	834	848
-8	2	2017	2055
-7	2	1606	1623
-5	2	801	740
-4	2	466	444
-3	2	600	577
-2	2	200	159
-1	2	169	161
1	2	1239	1206
2	2	854	810
3	2	1218	1141
4	2	1087	1033
5	2	1018	997
6	2	1105	1076
7	2	966	937
8	2	2112	2132
9	2	253	139
10	2	1488	1458
12	2	506	486
13	2	643	660
-16	4	391	430
-14	4	674	744
-13	4	526	509
-12	4	436	449
-11	4	1420	1429
-10	4	283	266
-9	4	809	861
-8	4	232	177
-7	4	414	411
-6	4	799	760
-5	4	451	465
-4	4	735	717
-3	4	357	260
-2	4	272	246
-1	4	2699	2594
0	4	271	263
1	4	3171	3139
2	4	719	689
3	4	2375	2427
4	4	478	496
5	4	335	344
6	4	401	366
7	4	303	182
8	4	987	984
12	4	362	291
-14	6	306	137
-13	6	313	350
-12	6	559	185
-11	6	892	988
-10	6	433	443
-9	6	682	732
-8	6	994	1006
-7	6	429	416
-6	6	1656	1609
-5	6	855	793
-4	6	927	961
-3	6	849	846
-2	6	490	371
0	6	1383	1234
1	6	800	773
3	6	369	359
4	6	363	299
5	6	234	207
7	6	272	56
8	6	480	449
9	6	622	666
10	6	1147	1076
11	6	648	627
-12	8	337	324
-10	8	268	268
-9	8	608	658
-7	8	745	783
-6	8	491	483
-3	8	1030	952
-2	8	846	870
-1	8	818	981
0	8	789	743
1	8	1120	1080
2	8	265	276
3	8	1826	1761
4	8	451	485
5	8	1347	1230
8	8	342	88
-8	10	628	603
-7	10	1042	1015
-6	10	429	435
-5	10	332	364
-4	10	598	598
-2	10	465	419
2	10	760	743
3	10	704	678
4	10	463	520
***************			
L = 9			
0	-9	1329	1317
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2	-9	652	689
4	-9	351	328
5	-9	306	216
6	-9	384	387
0	-7	398	395
1	-7	506	304
2	-7	539	574
6	-7	443	479
7	-7	804	736
8	-7	653	629
9	-7	1116	998
0	-5	2001	2021
1	-5	334	306
2	-5	2431	2362
3	-5	577	584
4	-5	1075	1099
5	-5	607	636
6	-5	267	264
7	-5	382	272
8	-5	454	410
9	-5	554	567
0	-3	603	231
1	-3	975	956
2	-3	1073	1110
3	-3	531	518
4	-3	441	425
5	-3	702	728
6	-3	246	102
8	-3	296	317
9	-3	500	390
10	-3	962	1018
11	-3	1034	1039
12	-3	411	447
0	-1	1612	1584
1	-1	381	384
2	-1	1493	1492
3	-1	1035	1017
4	-1	1554	1560
5	-1	1267	1247
6	-1	939	985
8	-1	515	559
10	-1	1073	1125
11	-1	675	713
12	-1	617	643
13	-1	554	702
-15	1	489	497
-13	1	308	128
-12	1	332	267
-11	1	841	976
-10	1	844	805
-9	1	1987	2081
-8	1	1698	1859
-7	1	1382	1476
-6	1	1727	1825
-5	1	981	1013
-3	1	1155	1116
-2	1	991	965
-1	1	777	814
0	1	296	255
1	1	1073	1057
2	1	1029	1021
3	1	1400	1365
4	1	1850	1837
5	1	538	573
6	1	1255	1327
7	1	1163	1189
9	1	1132	1173
11	1	361	301
12	1	324	327
-16	3	590	692
-15	3	932	1086
-14	3	372	264
-13	3	592	656
-12	3	267	193
-11	3	354	360
-10	3	318	356
-9	3	1154	1221
-7	3	548	534
-5	3	299	290
-4	3	577	556
-3	3	929	756
-2	3	604	601
-1	3	605	627
0	3	1932	1928
1	3	243	188
2	3	2256	2366
3	3	497	447
4	3	780	867
5	3	250	272
6	3	312	302
7	3	1166	1151
9	3	846	833
10	3	275	350
12	3	1010	996
-15	5	262	240
-12	5	403	453
-11	5	318	333
-10	5	469	537
-9	5	631	675
-8	5	420	426
-7	5	1346	1435
-6	5	642	677
-5	5	1395	1414
-4	5	658	706
-3	5	316	303
-2	5	577	546
-1	5	581	591
0	5	2222	2209
1	5	595	565
2	5	1811	1865
3	5	343	341
4	5	648	594
5	5	745	740
6	5	322	330
7	5	512	487
8	5	632	628
9	5	309	330
10	5	509	542
11	5	823	771
-13	7	379	346
-12	7	465	507
-11	7	263	351
-8	7	381	443
-7	7	1464	1403
-6	7	292	175
-5	7	1726	1762
-4	7	637	659
-3	7	533	533
-2	7	631	618
-1	7	227	243
0	7	432	330
1	7	275	203
2	7	632	660
3	7	520	565
4	7	1152	1149
5	7	363	289
6	7	398	301
8	7	847	807
9	7	591	571
-9	9	348	372
-8	9	819	828
-6	9	451	437
-5	9	420	417
-3	9	686	696
0	9	314	131
1	9	593	491
2	9	1058	1057
3	9	929	888
4	9	1410	1403
***************			
L = 10			
0	-10	423	225
1	-10	585	593
1	-8	549	443
2	-8	543	527
3	-8	387	376
4	-8	792	695
5	-8	746	728
7	-8	777	714
0	-6	623	601
1	-6	1836	1730
3	-6	808	782
4	-6	360	327
6	-6	440	412
7	-6	468	469
8	-6	850	769
9	-6	392	325
10	-6	463	455
0	-4	1286	1193
1	-4	2237	2254
2	-4	560	588
3	-4	1405	1368
5	-4	279	259
6	-4	474	468
7	-4	375	79
8	-4	572	561
9	-4	695	693
10	-4	383	252
11	-4	346	272
0	-2	497	457
2	-2	292	240
3	-2	1070	1073
4	-2	234	220
5	-2	868	815
7	-2	329	214
9	-2	1272	1296
10	-2	663	654
11	-2	1024	1031
12	-2	689	714
-15	0	353	290
-13	0	764	720
-12	0	536	538
-11	0	480	304
-10	0	1238	1176
-9	0	846	499
-8	0	1525	1463
-7	0	1582	1586
-6	0	470	468
-5	0	384	357
-4	0	834	337
-3	0	1149	1158
-2	0	904	947
-1	0	1430	1257
0	0	538	378
1	0	1505	1465
2	0	1677	1610
3	0	2073	1999
4	0	1695	1653
5	0	1753	1683
6	0	726	591
7	0	512	524
8	0	299	249
9	0	456	468
10	0	295	164
11	0	425	354
12	0	312	209
-16	2	780	836
-15	2	388	335
-14	2	862	948
-12	2	273	326
-11	2	236	209
-10	2	1408	1470
-9	2	509	503
-8	2	1360	1364
-7	2	432	401
-6	2	1193	1213
-5	2	274	302
-4	2	1804	1785
-2	2	1718	1739
-1	2	375	353
0	2	507	477
1	2	981	953
2	2	1201	1199
4	2	833	844
5	2	600	586
6	2	589	547
8	2	696	656
9	2	249	226
11	2	774	737
-15	4	742	792
-14	4	704	721
-13	4	368	355
-12	4	277	184
-11	4	267	292
-9	4	380	310
-8	4	515	491
-7	4	389	378
-6	4	980	941
-5	4	984	968
-4	4	203	144
-3	4	404	408
-2	4	838	863
-1	4	1582	1507
0	4	753	736
1	4	2130	2114
2	4	570	544
3	4	1211	1203
4	4	597	611
5	4	298	301
6	4	1026	1021
7	4	905	929
8	4	561	556
9	4	503	455
10	4	337	345
11	4	593	546
-12	6	381	352
-11	6	280	277
-9	6	296	304
-8	6	1411	1454
-6	6	2452	2344
-5	6	316	298
-4	6	1544	1472
-2	6	318	341
-1	6	816	743
0	6	307	131
1	6	659	653
4	6	903	862
5	6	313	304
6	6	1215	1132
7	6	503	498
9	6	833	760
-9	8	275	319
-8	8	565	322
-7	8	405	378
-6	8	766	595
0	8	575	565
1	8	435	403
2	8	803	838
3	8	1433	1323
5	8	1143	1127
-2	10	702	722
***************			
L = 11			
0	-9	631	560
1	-9	554	526
2	-9	383	400
3	-9	1024	912
4	-9	702	655
0	-7	431	385
1	-7	334	266
3	-7	777	707
4	-7	416	269
6	-7	609	535
0	-5	1474	1308
1	-5	1002	959
2	-5	1419	1270
3	-5	360	342
5	-5	810	716
7	-5	937	808
0	-3	1395	1290
1	-3	568	566
2	-3	579	546
3	-3	598	589
4	-3	377	327
5	-3	819	857
7	-3	926	901
8	-3	767	830
10	-3	902	959
11	-3	386	469
1	-1	1167	1126
2	-1	830	791
3	-1	1205	1191
4	-1	1470	1414
5	-1	849	847
6	-1	523	565
7	-1	300	225
8	-1	602	663
9	-1	299	265
10	-1	771	815
-15	1	753	860
-14	1	511	621
-13	1	380	424
-12	1	541	535
-11	1	788	732
-10	1	504	312
-9	1	943	1037
-8	1	369	380
-7	1	670	780
-6	1	234	152
-5	1	1371	1408
-4	1	965	948
-3	1	1966	1984
-2	1	601	615
-1	1	844	894
0	1	442	436
1	1	1449	1399
2	1	320	209
3	1	1753	1671
4	1	1123	1112
5	1	711	831
6	1	918	874
9	1	564	536
11	1	380	328
-15	3	768	839
-14	3	547	555
-13	3	293	296
-12	3	713	742
-11	3	491	484
-10	3	889	905
-9	3	730	727
-8	3	200	172
-7	3	239	242
-6	3	1309	1246
-5	3	942	924
-4	3	1295	1286
-3	3	1612	1585
-2	3	249	232
-1	3	1260	1335
0	3	513	507
1	3	245	218
2	3	579	556
5	3	789	757
6	3	569	567
7	3	622	641
8	3	645	651
9	3	324	333
10	3	510	435
-12	5	380	361
-11	5	333	349
-10	5	748	717
-9	5	461	413
-7	5	1847	1776
-5	5	1661	1553
-4	5	503	534
-3	5	262	399
-2	5	504	598
0	5	1245	1098
2	5	514	512
3	5	765	710
5	5	1592	1476
7	5	1303	1204
8	5	530	526
-11	7	368	361
-7	7	1351	1196
-5	7	1176	1064
-4	7	485	441
-3	7	558	524
-2	7	1037	1071
-1	7	868	789
0	7	645	614
1	7	885	854
2	7	485	387
4	7	1095	986
5	7	612	526
6	7	290	351
-5	9	565	560
-3	9	759	740
-2	9	445	344
0	9	306	165
1	9	514	469
2	9	489	464
***************			
L = 12			
1	-4	780	864
2	-4	583	588
3	-4	248	143
4	-4	1036	1093
6	-4	1329	1419
7	-4	240	227
8	-4	659	740
9	-4	365	353
1	-2	701	704
2	-2	625	615
3	-2	636	650
5	-2	443	443
6	-2	580	611
7	-2	321	246
8	-2	532	533
9	-2	611	695
-15	0	869	806
-14	0	535	494
-13	0	1478	1347
-11	0	517	610
-7	0	237	124
-6	0	230	203
-5	0	620	593
-4	0	1112	1163
-2	0	563	588
-1	0	643	564
0	0	999	954
1	0	449	307
2	0	1322	1408
3	0	953	905
4	0	1082	1280
5	0	989	880
6	0	915	818
8	0	777	782
9	0	499	492
10	0	691	681
-14	2	695	669
-11	2	671	660
-10	2	572	539
-9	2	371	408
-8	2	380	386
-7	2	683	647
-6	2	656	678
-5	2	1795	1815
-4	2	1698	1709
-3	2	1487	1545
-2	2	1678	1710
-1	2	409	437
2	2	907	892
3	2	356	348
4	2	239	250
7	2	640	559
8	2	720	718
9	2	246	279
10	2	730	751
-13	4	771	730
-12	4	430	435
-11	4	1285	1230
-10	4	424	394
-9	4	1112	1077
-6	4	593	579
-5	4	1205	1190
-4	4	205	204
-3	4	759	751
-2	4	298	299
0	4	387	251
2	4	306	273
4	4	1123	1146
6	4	1396	1456
7	4	499	504
8	4	494	506
***************			
L = 13			
1	-3	340	413
3	-3	288	284
4	-3	277	291
5	-3	1096	1149
7	-3	978	1050
1	-1	995	1018
3	-1	819	942
4	-1	339	534
5	-1	368	327
8	-1	384	456
-14	1	622	576
-13	1	516	442
-11	1	516	393
-9	1	494	553
-8	1	357	414
-7	1	381	385
-6	1	1175	1142
-5	1	771	730
-4	1	1231	1475
-3	1	1685	1721
-2	1	329	405
-1	1	582	520
1	1	948	902
2	1	408	461
3	1	810	838
5	1	710	672
6	1	600	570
7	1	859	831
8	1	826	823
9	1	982	1031
-13	3	435	362
-12	3	1064	1026
-11	3	353	398
-10	3	1204	1192
-8	3	255	240
-7	3	201	174
-6	3	1140	1155
-5	3	746	755
-4	3	1434	1474
-3	3	813	877
-2	3	991	1047
0	3	725	776
1	3	682	749
2	3	796	859
3	3	276	308
4	3	444	459
5	3	760	778
6	3	459	464
7	3	241	253
8	3	491	503
***************			
L = 14			
3	-4	560	652
4	-4	952	960
5	-4	445	527
6	-4	1030	1084
1	-2	969	998
2	-2	332	338
4	-2	374	371
6	-2	430	503
-13	0	860	719
-12	0	624	585
-11	0	325	286
-10	0	1180	1089
-9	0	342	304
-8	0	1257	1231
-7	0	575	534
-6	0	496	473
-5	0	508	464
-4	0	1040	994
-3	0	513	462
-2	0	912	916
-1	0	724	772
0	0	404	313
2	0	664	639
3	0	309	360
4	0	238	288
5	0	517	451
6	0	428	366
7	0	953	870
-11	2	688	673
-10	2	371	379
-8	2	459	409
-7	2	811	761
-5	2	1221	1192
-4	2	558	586
-3	2	991	1008
-1	2	1070	1105
0	2	806	806
1	2	1261	1303
2	2	404	434
3	2	649	629
5	2	593	586
7	2	966	941
-11	4	1082	1042
-10	4	631	630
-9	4	760	717
-8	4	458	444
-6	4	608	589
-5	4	469	431
-4	4	405	303
-2	4	552	563
-1	4	337	425
0	4	776	834
1	4	1021	985
3	4	954	969
4	4	587	603
5	4	273	322
***************			
L = 15			
1	-3	241	229
3	-3	348	396
4	-3	481	532
5	-3	592	702
1	-1	270	91
3	-1	340	461
6	-1	432	509
-11	1	673	637
-9	1	1323	1305
-7	1	988	956
-6	1	535	525
-3	1	329	299
-1	1	381	382
0	1	754	725
1	1	352	262
2	1	512	545
3	1	264	324
4	1	405	472
5	1	225	92
-10	3	774	755
-7	3	299	301
-6	3	482	463
-5	3	276	323
-4	3	245	150
-3	3	206	195
-2	3	515	485
-1	3	803	827
0	3	1204	1202
1	3	759	775
2	3	1236	1303
3	3	222	199
***************			
L = 16			
1	-4	369	332
1	-2	265	255
2	-2	582	648
-10	0	1347	1233
-9	0	376	180
-8	0	1407	1303
-6	0	420	334
-4	0	262	284
-3	0	654	590
-1	0	343	313
2	0	905	883
4	0	840	818
-6	2	270	115
-2	2	688	757
-1	2	674	689
0	2	902	936
1	2	1053	1058
3	2	392	378
-7	4	425	392
-6	4	913	907
-4	4	273	336
-2	4	509	470
-1	4	264	307
0	4	863	845
***************			
L = 17			
1	-1	882	946
-7	1	536	500
-6	1	646	644
-5	1	291	247
-4	1	810	794
-3	1	410	455

[a] This table shows h, k, $10|F_o|$, $10|F_o|$ in blocks of constant l.

$(CO)_9$ is considerably more complex than that found in previously studied transition metal complexes of azulene.[2-10] Ru(1) lies asymmetrically below the five-membered ring of the azulene system, with ruthenium-carbon bond lengths (in order of increasing distance) being: Ru(1)–C(1) = 2.218 (14) Å, Ru(1)–C(2) = 2.242 (14) Å, Ru(1)–C(3) = 2.244 (13) Å, Ru(1)–C(9) = 2.336 (13) Å, Ru(1)–C(10) = 2.348 (13) Å. The other azulene-bonded ruthenium atoms (*i.e.*, Ru(2) and Ru(3)] are in approximately equivalent environments interrelated by a noncrystallographic mirror plane. Important distances for these atoms are: Ru(2)–C(8) = 2.260 (13) Å, Ru(3)–C(4) = 2.260 (15) Å; Ru(2)–C(7) = 2.213 (14) Å, Ru(3)–C(5) = 2.182 (14) Å; Ru(2)–C(6) = 2.489 (13) Å, Ru(3)–C(6) = 2.441 (13) Å; Ru(2)–C(9) = 2.613(13) Å, Ru(3)–C(10) = 2.559 (13) Å.

It is immediately apparent that the azulene-to-metal bonding in this complex cannot be described in any simple manner. In fact, it is difficult, on any *a priori* basis, to decide which ruthenium-to-carbon distances are intrinsically bonding and which are nonbonding. However, the conformation of the azulene ligand does provide some information on this, strongly suggesting that C(6) is bonded both to Ru(2) and to Ru(3). Thus, with Ru(2)–C(6) and Ru(3)–C(6) distances of 2.489 (13) and 2.441 (13) Å, respectively, considered as bonding interactions, it is apparent that the Ru(2)–C(9) and Ru(3)–C(10) distances of 2.613 (13) and 2.559 (13) Å must also be indicative of a bonding situation.

Although it is probably more appropriate to consider the molecule in terms of a sophisticated molecular orbital treatment, it is, nevertheless, possible to show that each ruthenium atom attains the noble gas configuration appropriate to the diamagnetism of the complex. The key to this approach is the recognition that the p_z orbitals on atoms C(6), C(9), and C(10) point toward the midpoints of the metal–metal vectors Ru(2)–Ru(3), Ru(1)–Ru(2), and Ru(1)–Ru(3), respectively. This suggests two-electron three-center bonds over

TABLE IV

FINAL PARAMETERS FOR TRICLINIC $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$

(A) Atomic positions with Esd's[a]

Atom	x	y	z
Ru(1)	−0.13316 (7)	0.01653 (11)	−0.18355 (7)
Ru(2)	−0.26947 (7)	0.10780 (11)	−0.32618 (7)
Ru(3)	−0.30752 (7)	−0.10415 (11)	−0.22052 (8)
Ru(4)	−0.20227 (7)	−0.12394 (11)	−0.34827 (7)
O(1)	−0.2340 (11)	−0.3276 (12)	−0.1120 (9)
O(2)	−0.4361 (11)	−0.2828 (15)	−0.3382 (10)
O(3)	−0.3844 (8)	0.1069 (14)	−0.5136 (8)
O(4)	−0.1372 (9)	0.2593 (12)	−0.4022 (8)
O(5)	0.0120 (8)	0.0845 (12)	−0.2869 (8)
O(6)	−0.0458 (7)	−0.2383 (11)	−0.1529 (8)
O(7)	−0.1452 (8)	−0.3960 (11)	−0.3262 (8)
O(8)	−0.3487 (9)	−0.1932 (13)	−0.5074 (8)
O(9)	−0.0809 (9)	−0.0548 (14)	−0.4766 (9)
C(1)	−0.1353 (9)	0.2086 (12)	−0.1199 (10)
C(2)	−0.0952 (11)	0.1266 (16)	−0.0500 (9)
C(3)	−0.1612 (9)	0.0431 (14)	−0.0413 (8)
C(4)	−0.3305 (10)	0.0082 (14)	−0.0952 (9)
C(5)	−0.4036 (9)	0.0177 (13)	−0.1695 (10)
C(6)	−0.4049 (8)	0.0763 (13)	−0.2592 (9)
C(7)	−0.3725 (9)	0.2052 (15)	−0.2639 (9)
C(8)	−0.2889 (8)	0.2541 (13)	−0.2176 (9)
C(9)	−0.2273 (9)	0.1794 (13)	−0.1530 (8)
C(10)	−0.2454 (9)	0.0696 (13)	−0.1013 (8)
C(11)	−0.3472 (13)	−0.0494 (16)	−0.0100 (10)
C(12)	−0.4917 (9)	0.0504 (18)	−0.3305 (12)
C(13)	−0.2698 (11)	0.3925 (17)	−0.2236 (11)
C(14)	−0.2650 (11)	−0.2495 (18)	−0.1547 (11)
C(15)	−0.3859 (11)	−0.2126 (20)	−0.2936 (13)
C(16)	−0.3414 (11)	0.1036 (15)	−0.4398 (9)
C(17)	−0.1892 (9)	0.1968 (15)	−0.3740 (9)
C(18)	−0.0458 (8)	0.0560 (16)	−0.2524 (9)
C(19)	−0.0853 (10)	−0.1493 (17)	−0.1717 (11)
C(20)	−0.1648 (9)	−0.2948 (16)	−0.3339 (9)
C(21)	−0.2923 (9)	−0.1635 (15)	−0.4436 (9)
C(22)	−0.1250 (10)	−0.0785 (15)	−0.4254 (10)

(B) Anisotropic Thermal Parameters with Esd's[a]

Atom	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
Ru(1)	27.8 (0.5)	65.7 (1.3)	25.3 (0.6)	−13.5 (1.2)	3.7 (0.8)	−8.6 (1.3)
Ru(2)	34.0 (0.6)	53.3 (1.2)	24.6 (0.6)	−0.5 (1.3)	7.5 (0.8)	8.3 (1.3)
Ru(3)	34.0 (0.6)	51.6 (1.2)	37.3 (0.6)	−29.7 (1.3)	28.7 (0.9)	−15.1 (1.4)
Ru(4)	31.5 (0.6)	55.7 (1.2)	27.6 (0.6)	−6.4 (1.3)	16.7 (0.8)	−16.0 (1.3)
O(1)	120 (13)	79 (14)	71 (8)	11 (21)	42 (15)	74 (18)
O(2)	103 (12)	194 (23)	87 (9)	−205 (28)	41 (16)	−102 (23)
O(3)	79 (7)	210 (23)	43 (7)	−92 (20)	−34 (11)	31 (18)
O(4)	74 (8)	115 (15)	66 (7)	−15 (17)	53 (12)	38 (17)
O(5)	41 (6)	137 (16)	77 (7)	−7 (16)	43 (11)	27 (17)
O(6)	49 (5)	88 (14)	65 (7)	36 (14)	4 (10)	6 (16)
O(7)	82 (8)	60 (12)	68 (7)	34 (16)	27 (11)	27 (15)
O(8)	76 (9)	156 (20)	56 (7)	−3 (20)	−7 (13)	−95 (18)
O(9)	92 (9)	150 (19)	81 (8)	−92 (20)	100 (14)	−51 (19)
C(1)	49 (7)	29 (12)	45 (8)	15 (15)	7 (12)	4 (16)
C(2)	58 (10)	96 (19)	29 (7)	−18 (22)	12 (13)	−41 (19)
C(3)	48 (7)	78 (17)	23 (6)	7 (17)	12 (10)	9 (16)
C(4)	40 (8)	75 (16)	41 (7)	−63 (19)	32 (13)	−36 (17)
C(5)	48 (7)	38 (14)	62 (9)	−9 (16)	63 (13)	−36 (18)
C(6)	36 (6)	56 (14)	39 (7)	−2 (15)	11 (10)	−14 (17)
C(7)	34 (6)	95 (18)	34 (7)	−9 (16)	19 (10)	−27 (18)
C(8)	32 (6)	49 (13)	40 (7)	7 (14)	21 (10)	7 (16)
C(9)	38 (7)	50 (13)	28 (6)	−7 (16)	21 (11)	−7 (15)
C(10)	39 (7)	54 (14)	21 (6)	−11 (15)	17 (10)	−21 (14)
C(11)	89 (14)	80 (18)	33 (8)	−10 (25)	44 (16)	22 (19)
C(12)	26 (6)	139 (25)	67 (10)	−7 (18)	−16 (12)	−50 (25)
C(13)	80 (9)	79 (20)	51 (9)	−42 (22)	25 (14)	42 (23)
C(14)	68 (9)	108 (21)	51 (10)	−16 (22)	50 (15)	29 (24)
C(15)	60 (9)	148 (26)	69 (11)	−98 (25)	22 (15)	−5 (27)

TABLE IV (*Continued*)

Atom	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
C(16)	57 (9)	96 (18)	21 (6)	−27 (20)	15 (21)	−5 (17)
C(17)	36 (6)	88 (18)	35 (17)	−3 (17)	5 (11)	−5 (18)
C(18)	28 (5)	124 (20)	41 (8)	28 (16)	7 (10)	37 (20)
C(19)	44 (8)	109 (20)	50 (9)	6 (21)	28 (13)	−29 (22)
C(20)	41 (7)	84 (19)	36 (7)	15 (18)	23 (11)	−27 (20)
C(21)	44 (7)	87 (17)	32 (7)	27 (17)	15 (11)	−16 (17)
C(22)	43 (8)	87 (18)	41 (8)	−6 (19)	35 (13)	−11 (19)

(C) Thermal Vibration Ellipsoids for Atoms[a,b]

Atom	B_{maj}, Å^2	B_{med}, Å^2	B_{min}, Å^2
Ru(1)	3.24	2.66	1.83
Ru(2)	3.14	2.45	2.00
Ru(3)	4.22	2.45	1.65
Ru(4)	3.20	2.59	1.83
O(1)	10.81	7.09	2.25
O(2)	15.98	7.51	2.04
O(3)	12.41	6.05	2.69
O(4)	7.19	6.12	3.55
O(5)	6.99	5.87	3.13
O(6)	6.33	4.87	3.00
O(7)	7.60	5.87	2.38
O(8)	9.91	7.01	2.53
O(9)	12.13	5.07	3.62
C(1)	4.87	3.61	1.24
C(2)	5.50	4.85	1.76
C(3)	4.38	3.50	1.94
C(4)	6.02	2.77	1.45
C(5)	6.65	2.73	1.25
C(6)	3.64	3.21	2.25
C(7)	4.85	2.94	2.37
C(8)	3.52	2.67	2.16
C(9)	3.57	2.43	2.00
C(10)	3.67	2.69	1.31
C(11)	8.02	3.87	2.08
C(12)	8.17	4.90	1.89
C(13)	7.78	4.95	2.22
C(14)	6.52	5.33	2.89
C(15)	9.29	5.89	2.82
C(16)	5.71	3.78	1.78
C(17)	4.03	3.58	2.69
C(18)	5.99	3.43	2.17
C(19)	5.73	4.19	3.08
C(20)	4.59	3.82	2.09
C(21)	4.96	3.34	2.31
C(22)	4.67	3.69	2.60

[a] See footnotes *a* and *b* of Table II. [b] The vibration ellipsoids for triclinic [$(CH_3)_3C_{10}H_5$]$Ru_4(CO)_9$ are exhibited pictorially in Figures 2 and 3.

Ru(2)–C(6)–Ru(3), Ru(1)–C(9)–Ru(2), and Ru(1)–C(10)–Ru(3). It should be understood that the first of these three-center bonds is symmetric, while the other two are asymmetric—as may be seen by consideration of the appropriate metal–carbon distances (see Table V).

Utilizing the above-defined three-center bonds, the ruthenium atoms may each be shown to obey the "noble gas rule" in the following way. The atom Ru(1) is assigned a formal charge of +1 (d^7 configuration) and obtains four electrons from its two terminal carbonyl ligands, three electrons from its three Ru–Ru bonds, three electrons from the C(1)–C(2)–C(3) π-allyl system, and a total of one electron from the asymmetric three-center bonds over Ru(1)–C(9)–Ru(2) and Ru(1)–C(10)–Ru(3). Atoms Ru(2) and Ru(3) are electronically equivalent (although they have different stereochemical environments, interrelated by a mirror plane) and it is necessary to consider only one of the two atoms, Ru(2). This has a formal oxidation state of zero (d^8) and obtains four electrons from its two terminal carbonyl ligands, three electrons from three Ru–Ru bonds, two electrons from the C(7)–C(8) double bond, and half of an electron from each of the three-center bonds over Ru(2)–C(9)–Ru(1) and Ru(2)–C(6)–Ru(3). Finally, Ru(4) is assigned a formal oxidation state of −1 (d^9) and obtains six electrons from its three carbonyl groups and three electrons from Ru–Ru bonds.

It should be emphasized that the consideration of electronic requirements at each metal center in a cluster is of questionable value. It is probably more meaningful to consider valence electrons as a property of the Ru_4 cluster as a whole. The total number of valence electrons required to achieve an over-all stable, dia-

TABLE V

INTERATOMIC DISTANCES, WITH ESTIMATED STANDARD DEVIATIONS, FOR MONOCLINIC AND TRICLINIC $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$

	Distances, Å	
Atoms	Monoclinic	Triclinic
(a) Ruthenium–Ruthenium Distances		
Ru(1)---Ru(2)	2.877 (5)	2.864 (3)
Ru(1)---Ru(3)	2.902 (5)	2.898 (3)
Ru(1)---Ru(4)	2.827 (5)	2.840 (3)
Ru(2)---Ru(3)	2.886 (5)	2.908 (3)
Ru(2)---Ru(4)	2.702 (5)	2.698 (3)
Ru(3)---Ru(4)	2.717 (5)	2.717 (3)
(b) Ruthenium–Azulene Distances		
Ru(1)---C(1)	2.219 (30)	2.218 (14)
Ru(1)---C(2)	2.182 (36)	2.242 (14)
Ru(1)---C(3)	2.229 (31)	2.244 (13)
Ru(1)---C(10)	2.332 (35)	2.348 (13)
Ru(1)---C(9)	2.362 (25)	2.336 (13)
Ru(1)···C(4)	3.591 (31)	3.512 (14)
Ru(1)···C(8)	3.498 (34)	3.435 (13)
Ru(2)---C(9)	2.586 (25)	2.613 (13)
Ru(2)---C(8)	2.274 (34)	2.260 (13)
Ru(2)---C(7)	2.161 (34)	2.213 (14)
Ru(2)---C(6)	2.475 (35)	2.489 (13)
Ru(3)---C(6)	2.388 (35)	2.441 (13)
Ru(3)---C(5)	2.182 (29)	2.182 (14)
Ru(3)---C(4)	2.252 (31)	2.260 (15)
Ru(3)---C(10)	2.606 (35)	2.559 (13)
Ru(2)···C(13)	3.303 (40)	3.335 (15)
Ru(2)···C(12)	3.484 (39)	3.452 (15)
Ru(3)···C(12)	3.457 (39)	3.425 (15)
Ru(3)···C(11)	3.417 (45)	3.332 (15)
(c) Carbon–Carbon Distances in Azulene Ligand		
C(1)---C(2)	1.452 (47)	1.424 (21)
C(2)---C(3)	1.466 (48)	1.368 (21)
C(3)---C(10)	1.399 (47)	1.449 (18)
C(10)---C(9)	1.539 (47)	1.472 (18)
C(9)---C(1)	1.401 (39)	1.425 (19)
C(10)---C(4)	1.610 (47)	1.472 (19)
C(4)---C(5)	1.307 (43)	1.416 (20)
C(5)---C(6)	1.434 (45)	1.486 (19)
C(6)---C(7)	1.522 (49)	1.462 (19)
C(7)---C(8)	1.311 (48)	1.415 (19)
C(8)---C(9)	1.458 (42)	1.462 (18)
C(4)---C(11)	1.629 (54)	1.491 (23)
C(6)---C(12)	1.580 (52)	1.546 (21)
C(8)---C(13)	1.456 (52)	1.506 (21)
(d) Ruthenium–Carbonyl Distances		
Ru(1)---C(18)	1.838 (36)	1.885 (14)
Ru(1)---C(19)	1.922 (39)	1.907 (16)
Ru(2)---C(16)	1.938 (41)	1.826 (15)
Ru(2)---C(17)	1.870 (43)	1.810 (14)
Ru(3)---C(14)	1.763 (42)	1.902 (17)
Ru(3)---C(15)	1.808 (34)	1.827 (19)
Ru(4)---C(20)	1.965 (39)	1.908 (14)
(d) Ruthenium–Carbonyl Distances (*Continued*)		
Ru(4)---C(21)	1.820 (39)	1.809 (14)
Ru(4)---C(22)	1.953 (38)	1.871 (13)
Ru(1)···O(5)	2.964 (27)	3.018 (12)
Ru(1)···O(6)	3.011 (25)	3.011 (11)
Ru(2)···O(3)	2.935 (26)	2.991 (12)
Ru(2)···O(4)	3.045 (33)	2.968 (12)
Ru(3)···O(1)	2.942 (32)	2.963 (14)
Ru(3)···O(2)	2.961 (28)	3.027 (15)
Ru(4)···O(7)	3.007 (30)	3.014 (12)
Ru(4)···O(8)	3.030 (30)	3.012 (13)
Ru(4)···O(9)	3.064 (28)	2.995 (13)
(e) Carbon–Oxygen Distances		
C(14)---O(1)	1.196 (52)	1.110 (22)
C(15)---O(2)	1.156 (44)	1.161 (25)
C(16)---O(3)	1.000 (49)	1.165 (20)
C(17)---O(4)	1.179 (54)	1.180 (19)
C(18)---O(5)	1.129 (45)	1.149 (18)
C(19)---O(6)	1.096 (46)	1.134 (20)
C(20)---O(7)	1.047 (49)	1.118 (19)
C(21)---O(8)	1.209 (49)	1.183 (19)
C(22)---O(9)	1.114 (47)	1.142 (19)
(f) Distances between Carbonyl Ligands		
C(14)···C(15)	2.440 (54)	2.519 (26)
C(15)···C(16)	4.210 (53)	4.123 (24)
C(16)···C(17)	2.572 (59)	2.538 (21)
C(17)···C(18)	3.042 (56)	2.963 (20)
C(18)···C(19)	2.616 (53)	2.608 (22)
C(19)···C(14)	2.949 (57)	2.997 (24)
C(20)···C(21)	2.764 (56)	2.676 (20)
C(21)···C(22)	2.714 (54)	2.671 (20)
C(22)···C(20)	2.946 (55)	2.792 (21)
C(14)···C(20)	3.222 (57)	3.352 (23)
C(15)···C(21)	2.903 (52)	2.918 (24)
C(16)···C(21)	2.991 (57)	2.934 (20)
C(17)···C(22)	3.190 (57)	3.219 (21)
C(18)···C(22)	3.005 (52)	2.979 (21)
C(19)···C(20)	2.837 (55)	2.920 (22)
O(1)···O(2)	3.989 (42)	4.127 (21)
O(2)···O(3)	4.999 (38)	5.030 (20)
O(3)···O(4)	4.081 (42)	4.142 (18)
O(4)···O(5)	3.259 (43)	3.172 (17)
O(5)···O(6)	4.121 (37)	4.141 (16)
O(6)···O(1)	3.387 (41)	3.192 (18)
O(7)···O(8)	4.307 (42)	4.278 (18)
O(8)···O(9)	4.317 (41)	4.281 (19)
O(9)···O(7)	4.555 (41)	4.460 (18)
O(1)···O(7)	3.803 (44)	3.765 (18)
O(2)···O(8)	3.192 (41)	3.210 (20)
O(3)···O(8)	3.204 (39)	3.228 (18)
O(4)···O(9)	3.624 (43)	3.665 (18)
O(5)···O(9)	3.309 (39)	3.253 (18)
O(6)···O(7)	3.095 (39)	3.196 (16)

magnetic electronic configuration in a tetrahedral cluster is 60 (*i.e.*, 18 outer electrons for each of the four metal atoms, less the 12 electrons which are shared in the six metal–metal bonds). In the present $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule, the required 60 electrons are obtained from the following sources: 32 from the four d^8 Ru(0) atoms, 18 electrons from the nine carbonyl groups, and 10 electrons by the total utilization of the π electrons of the substituted azulene ligand. We feel that the latter interpretation of electronic fulfillment is the more satisfactory, for it requires no charge separation between ruthenium atoms. Thus, we regard the $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule as an example of the "ligand-to-cluster" bonding which has been observed in a very few organometallic molecules and of which the most obvious previous example occurs in $(\pi\text{-}C_5H_5)_4$-

TABLE VI

INTERATOMIC ANGLES, WITH ESTIMATED STANDARD DEVIATIONS, FOR MONOCLINIC AND TRICLINIC $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$

	Angles, deg	
Atoms	Monoclinic	Triclinic
(a) Angles within Ruthenium Cluster		
Ru(2)---Ru(1)---Ru(3)	59.91 (09)	60.61 (04)
Ru(2)---Ru(1)---Ru(4)	56.54 (09)	56.47 (04)
Ru(3)---Ru(1)---Ru(4)	56.59 (09)	56.51 (04)
Ru(1)---Ru(2)---Ru(3)	60.48 (09)	60.28 (04)
Ru(1)---Ru(2)---Ru(4)	60.80 (09)	61.32 (04)
Ru(3)---Ru(2)---Ru(4)	58.07 (09)	57.83 (04)
Ru(1)---Ru(3)---Ru(2)	59.61 (09)	59.10 (04)
Ru(1)---Ru(3)---Ru(4)	60.31 (09)	60.66 (04)
Ru(2)---Ru(3)---Ru(4)	57.58 (09)	57.22 (04)
Ru(1)---Ru(4)---Ru(2)	62.66 (09)	62.21 (04)
Ru(1)---Ru(4)---Ru(3)	63.10 (09)	62.83 (04)
Ru(2)---Ru(4)---Ru(3)	64.36 (10)	64.95 (04)
(b) Other Angles within the $Ru_4(CO)_9$ Cluster		
Ru(2)---Ru(1)---C(18)	92.7 (11)	90.1 (4)
Ru(2)---Ru(1)---C(19)	129.2 (12)	128.1 (5)
Ru(3)---Ru(1)---C(18)	138.3 (11)	136.1 (5)
Ru(3)---Ru(1)---C(19)	87.3 (12)	86.4 (4)
Ru(4)---Ru(1)---C(18)	82.4 (11)	80.4 (4)
Ru(4)---Ru(1)---C(19)	73.4 (12)	72.2 (5)
C(18)---Ru(1)---C(19)	88.1 (16)	88.4 (7)
O(5)···Ru(1)···O(6)	87.2 (7)	86.8 (3)
Ru(1)---Ru(2)---C(16)	152.6 (12)	155.1 (5)
Ru(1)---Ru(2)---C(17)	90.6 (13)	92.0 (5)
Ru(3)---Ru(2)---C(16)	113.7 (12)	111.0 (5)
Ru(3)---Ru(2)---C(17)	147.9 (13)	148.9 (5)
Ru(4)---Ru(2)---C(16)	92.8 (12)	92.3 (5)
Ru(4)---Ru(2)---C(17)	96.9 (13)	97.8 (5)
C(16)---Ru(2)---C(17)	84.9 (18)	87.9 (7)
O(3)···Ru(2)···O(4)	86.0 (18)	88.1 (3)
Ru(1)---Ru(3)---C(14)	91.7 (14)	93.0 (5)
Ru(1)---Ru(3)---C(15)	146.2 (11)	148.0 (6)
Ru(2)---Ru(3)---C(14)	146.2 (14)	148.2 (5)
Ru(2)---Ru(3)---C(15)	107.4 (11)	109.7 (6)
Ru(4)---Ru(3)---C(14)	93.9 (14)	97.2 (5)
Ru(4)---Ru(3)---C(15)	86.1 (11)	87.7 (6)
C(14)---Ru(3)---C(15)	86.2 (18)	85.4 (8)
O(1)···Ru(3)···O(2)	85.0 (8)	87.1 (4)
Ru(1)---Ru(4)---C(20)	106.4 (12)	108.8 (4)
Ru(1)---Ru(4)---C(21)	153.5 (13)	150.9 (5)
Ru(1)---Ru(4)---C(22)	102.1 (11)	102.5 (4)
Ru(2)---Ru(4)---C(20)	163.7 (12)	164.8 (4)
Ru(2)---Ru(4)---C(21)	93.7 (12)	91.8 (5)
Ru(2)---Ru(4)---C(22)	96.8 (11)	97.0 (4)
Ru(3)---Ru(4)---C(20)	100.3 (12)	100.3 (4)
Ru(3)---Ru(4)---C(21)	96.9 (12)	95.5 (5)
Ru(3)---Ru(4)---C(22)	159.5 (11)	160.2 (4)
C(20)---Ru(4)---C(21)	93.7 (17)	93.4 (6)
C(20)---Ru(4)---C(22)	97.5 (16)	97.1 (6)
C(21)---Ru(4)---C(22)	91.9 (17)	92.9 (6)
O(7)···Ru(4)···O(8)	91.0 (8)	90.4 (3)
O(7)···Ru(4)···O(9)	97.2 (8)	95.8 (4)
O(8)···Ru(4)···O(9)	90.2 (8)	90.9 (4)

	Angles, deg	
Atoms	Monoclinic	Triclinic
(c) Angles between $Ru_4(CO)_9$ Cluster and Azulene Ligand		
C(1)---Ru(1)---C(2)	38.5 (13)	37.3 (1)
C(2)---Ru(1)---C(3)	38.8 (13)	35.5 (1)
C(3)---Ru(1)---C(10)	35.6 (12)	36.7 (1)
C(10)---Ru(1)---C(9)	38.3 (11)	36.6 (1)
C(9)---Ru(1)---C(1)	35.4 (10)	36.3 (1)
C(9)---Ru(2)---C(8)	34.2 (10)	33.1 (4)
C(8)---Ru(2)---C(7)	34.3 (13)	36.6 (5)
C(7)---Ru(2)---C(6)	37.5 (12)	35.6 (5)
C(6)---Ru(3)---C(5)	36.2 (11)	36.6 (5)
C(5)---Ru(3)---C(4)	34.2 (11)	36.6 (5)
C(4)---Ru(3)---C(10)	37.8 (11)	34.2 (5)
Ru(1)---C(9)---Ru(2)	70.9 (7)	69.1 (4)
Ru(1)---C(10)---Ru(3)	71.7 (10)	71.5 (4)
Ru(2)---C(6)---Ru(3)	72.8 (10)	71.4 (4)
(d) Angles within the 4,6,8-Trimethylazulene Ligand		
C(9)---C(1)---C(2)	118.1 (28)	112.1 (13)
C(1)---C(2)---C(3)	101.1 (28)	105.2 (13)
C(2)---C(3)---C(10)	111.0 (29)	112.5 (13)
C(3)---C(10)---C(9)	109.8 (28)	105.2 (11)
C(10)---C(9)---C(1)	99.8 (24)	104.9 (11)
C(10)---C(4)---C(5)	121.9 (28)	118.7 (13)
C(4)---C(5)---C(6)	127.0 (30)	127.9 (13)
C(5)---C(6)---C(7)	122.8 (30)	121.2 (12)
C(6)---C(7)---C(8)	130.2 (32)	125.7 (13)
C(7)---C(8)---C(9)	119.2 (30)	121.8 (12)
C(8)---C(9)---C(10)	131.0 (26)	129.6 (12)
C(9)---C(10)---C(4)	124.1 (27)	130.4 (12)
C(1)---C(9)---C(8)	128.7 (26)	125.2 (12)
C(3)---C(10)---C(4)	126.0 (29)	123.7 (12)
C(10)---C(4)---C(11)	108.7 (27)	123.0 (13)
C(5)---C(4)---C(11)	129.3 (30)	117.8 (13)
C(5)---C(6)---C(12)	110.5 (29)	113.9 (12)
C(7)---C(6)---C(12)	110.0 (29)	111.3 (12)
C(7)---C(8)---C(13)	121.2 (33)	118.5 (12)
C(9)---C(8)---C(13)	119.3 (30)	119.0 (12)
(e) Angles within Carbonyl Ligands		
Ru(1)---C(18)---O(5)	174.1 (33)	173.5 (13)
Ru(1)---C(19)---O(6)	171.3 (35)	168.0 (15)
Ru(2)---C(16)---O(3)	175.8 (39)	175.9 (14)
Ru(2)---C(17)---O(4)	174.0 (38)	176.8 (13)
Ru(3)---C(14)---O(1)	167.6 (37)	173.6 (16)
Ru(3)---C(15)---O(2)	174.4 (31)	178.4 (18)
Ru(4)---C(20)---O(7)	173.5 (37)	178.4 (18)
Ru(4)---C(21)---O(8)	178.7 (34)	177.0 (13)
Ru(4)---C(22)---O(9)	174.6 (34)	175.9 (13)

Rh_3H where a π-cyclopentadienyl ligand lies above a triangle of rhodium atoms.[50,51]

Bond Lengths within the Tetrahedral Ruthenium Cluster.—Within the tetrahedral cluster, ruthenium–ruthenium distances vary from 2.698 (3) to 2.908 (3)—a range of 0.21 Å—*i.e.*, 70σ. This large discrepancy must result from the difference in coordination environment of the various ruthenium atoms. Ruthenium–ruthenium distances in other molecules are 2.848 ± 0.006 Å for $Ru_3(CO)_{12}$[52] and 2.853–2.956 Å (σ = 0.007 Å) for $Ru_6C(CO)_{14}[C_6H_3(CH_3)_3]$.[53]

The mean Ru–C(carbonyl) bond length in $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ is 1.861 (15) Å, as compared to 1.91 ± 0.02 Å for $Ru_3(CO)_{12}$[52] and 1.92 ± 0.04 Å for the *terminal* carbonyl ligands of $Ru_6C(CO)_{14}[C_6H_3(CH_3)_3]$.[53] The mean carbon–oxygen bond length of 1.149 (20) Å

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Table VII

Important Planes within the Monoclinic and Triclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ Molecules

(A) Equations of Planes (Monoclinic Molecule)[a,b]

Plane	Atoms	c_1	c_2	c_3	d
I	C(1), C(2), C(3), C(9), C(10)	−0.9643	−0.1216	+0.2349	+1.2932
II	C(4), C(8), C(9), C(10)	−0.9860	−0.0817	+0.1452	+1.0681
III	C(4), C(5), C(7), C(8)	−0.8471	−0.3110	+0.4308	+1.9321
IV	C(5), C(6), C(7)	−0.3204	−0.5836	+0.7460	+3.4380
V	Ru(1), Ru(2), Ru(3)	−0.9360	−0.2013	+0.2885	−0.7193

(B) Equations of Planes (Triclinic Molecule)[a,b]

Plane	Atoms	c_1	c_2	c_3	d
I	C(1), C(2), C(3), C(9), C(10)	−0.3988	+0.5875	+0.7040	+0.8036
II	C(4), C(8), C(9), C(10)	−0.2611	+0.5976	+0.7580	+0.2745
III	C(4), C(5), C(7), C(8)	−0.5822	+0.5896	+0.5597	+2.0880
IV	C(5), C(6), C(7)	−0.9372	+0.3485	+0.0071	+5.3994
V	Ru(1), Ru(2), Ru(3)	−0.4390	+0.6097	+0.6598	−0.9365

(C) Dihedral Angles between Planes

Planes	Angles	
	Monoclinic	Triclinic
I–II	5° 51′	8° 35′
I–III	17° 05′	13° 27′
I–IV	56° 17′	54° 16′
II–III	22° 36′	21° 46′
II–IV	61° 51′	62° 44′
III–IV	39° 15′	41° 00′

(D) Deviations from Planes

Atom	Dev, Å	
	Monoclinic	Triclinic
	(i) From Plane I	
C(1)	+0.026 (31)	−0.009 (14)
C(2)	−0.018 (37)	+0.011 (16)
C(3)	+0.004 (32)	−0.010 (14)
C(9)	−0.021 (25)	+0.003 (13)
C(10)	+0.009 (35)	+0.004 (13)
C(4)	+0.155 (31)	+0.208 (15)
C(5)	−0.217 (30)	−0.111 (14)
C(6)	−0.835 (35)	−0.733 (13)
C(7)	−0.281 (35)	−0.180 (14)
C(8)	+0.073 (34)	+0.128 (13)
C(11)	+0.885 (45)	+0.896 (18)
C(12)	−1.180 (40)	−1.156 (17)
C(13)	+0.692 (41)	+0.808 (17)
Ru(1)	−1.894 (03)	−1.925 (03)
Ru(2)	−2.176 (03)	−2.107 (03)
Ru(3)	−2.086 (03)	−2.023 (03)
Ru(4)	−4.236 (03)	−4.207 (03)
	(ii) From Plane II	
C(4)	−0.001 (31)	+0.006 (15)
C(8)	+0.001 (34)	−0.006 (13)
C(9)	−0.002 (25)	+0.015 (13)
C(10)	+0.002 (35)	−0.015 (13)
C(1)	+0.186 (31)	+0.212 (14)
C(2)	+0.210 (37)	+0.334 (16)
C(3)	+0.118 (32)	+0.169 (14)
C(5)	−0.479 (30)	−0.494 (14)
C(6)	−1.101 (35)	−1.147 (13)
C(7)	−0.479 (35)	−0.514 (14)
C(11)	+0.740 (45)	+0.687 (18)
C(12)	−1.595 (40)	−1.782 (17)
C(13)	+0.663 (41)	+0.726 (17)
Ru(1)	−1.765 (03)	−1.745 (03)
Ru(2)	−2.259 (03)	−2.258 (03)
Ru(3)	−2.233 (03)	−2.237 (03)
Ru(4)	−4.252 (03)	−4.250 (03)
	(iii) From Plane III	
C(4)	−0.002 (31)	−0.001 (15)
C(5)	+0.002 (30)	+0.001 (14)
C(7)	−0.002 (35)	−0.001 (14)
C(8)	+0.002 (34)	+0.001 (13)
C(1)	−0.793 (31)	−0.721 (14)
C(2)	−1.145 (37)	−0.925 (16)
C(3)	−0.895 (32)	−0.777 (14)
C(6)	−0.447 (35)	−0.474 (13)
C(9)	−0.448 (25)	−0.400 (13)
C(10)	−0.487 (35)	−0.432 (13)
C(11)	+0.520 (45)	+0.598 (18)
C(12)	−0.326 (40)	−0.542 (17)
C(13)	+0.581 (41)	+0.640 (17)
Ru(1)	−2.613 (03)	−2.547 (03)
Ru(2)	−2.112 (03)	−2.121 (03)
Ru(3)	−2.115 (03)	−2.102 (03)
Ru(4)	−4.393 (03)	−4.379 (03)
	(iv) From Plane IV	
C(4)	−0.800 (31)	−0.887 (15)
C(8)	−0.775 (34)	−0.895 (13)
C(12)	+1.091 (40)	+0.961 (17)
	(v) From Plane V	
Ru(4)	−2.182 (03)	−2.184 (03)
C(1)	+1.928 (31)	+1.928 (14)
C(2)	+1.760 (37)	+1.867 (16)
C(3)	+1.833 (32)	+1.862 (14)
C(4)	+2.215 (31)	+2.204 (15)
C(5)	+1.971 (30)	+1.972 (14)
C(6)	+1.428 (35)	+1.414 (13)
C(7)	+1.980 (35)	+1.980 (14)
C(8)	+2.232 (34)	+2.223 (13)
C(9)	+2.002 (25)	+2.008 (13)
C(10)	+1.978 (35)	+1.966 (13)
C(11)	+2.856 (45)	+2.843 (18)
C(12)	+1.232 (40)	+1.077 (17)
C(13)	+2.871 (41)	+2.927 (17)

[a] Planes are defined as $c_1X + c_2Y + c_3Z = d$, where X, Y, Z are Cartesian coordinates which are related to the monoclinic or triclinic cell coordinates (x, y, z) by: $X = xa \sin\gamma + zc(\cos\beta - \cos\alpha\cos\gamma)/\sin\gamma$, $Y = yb + xa\cos\gamma + zc\cos\alpha$, and $Z = zc[(1 - \cos^2\alpha - \cos^2\beta - \cos^2\gamma + 2\cos\alpha\cos\beta\cos\gamma)/\sin^2\gamma]^{1/2}$. [b] Planes are derived using unit weights for atoms specified in sections A and B of this table.

in $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ agrees well with the value of 1.14 ± 0.02 Å for $Ru_3(CO)_{12}$.[52]

The 4,6,8-Trimethylazulene Ligand.—In $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$, the 4,6,8-trimethylazulene is severely distorted from planarity (see Figure 4). Relative to the plane (rms deviation only 0.008 Å) of the five-membered ring, the C(8)–C(9)–C(10)–C(4) plane bends away from the ruthenium atoms by 8° 35′; the ligand further bends across C(4)···C(8) such that there is a dihedral angle of 21° 46′ between the planes defined by C(8)–C(9)–C(10)–C(4) and C(5)–C(4)–C(7)–C(8); finally, the ligand bends across C(5)···C(7) by 41° 00′ relative to the plane defined by C(5)–C(4)–C(7)–C(8), such that C(6) lies 0.733 (13) Å below the plane of the five-membered ring. Methyl substituents on the azulene ligand are each significantly bent away from the $Ru_4(CO)_9$ system. Thus, while C(4) and C(8) lie 0.208 (15) and 0.128 (13) Å, respectively, above the plane of the five-membered ring, the methyl substituents at these positions are 0.896 (18) [C(11)] and 0.808 (17) Å [C(13)] above this plane. As may conveniently be seen in Figure 4, C(12)—which is attached to C(6)—is similarly displaced upward [by 0.961 (17) Å] from the C(5)–C(6)–C(17) plane.

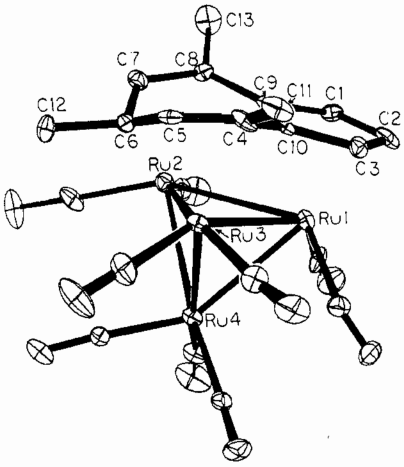

Figure 3.—The triclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule, projected onto the plane defined by Ru(1), Ru(3), Ru(4) (OTLIPS[38] diagram).

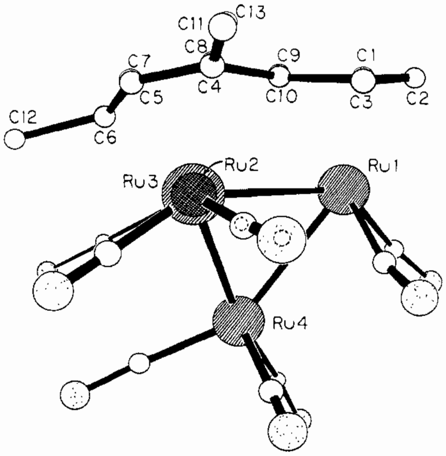

Figure 4.—The triclinic $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$ molecule, projected onto the plane defined by Ru(1), Ru(4), and the midpoint of Ru(2)–Ru(3).

Individual carbon–carbon distances within the five-membered ring range from 1.368 (21) to 1.472 (18) Å, while internal bond angles vary from 104.9 (1.1) to 112.5 (1.3)°. In neither case does any individual value vary significantly from the mean of 1.428 Å and 108.0°, respectively. The five-membered ring therefore has D_{5h} symmetry within the limits of experimental error.

Bond distances within the seven-membered ring range from 1.415 (19) to 1.486 (19) Å, averaging 1.455 Å. The similarity of bond lengths is indicative of delocalization around the system and is in keeping with our proposal that the azulene binds to the $Ru_4(CO)_9$ cluster *as a whole* (*vide supra*), rather than being partitioned between individual ruthenium atoms.

Angles within the seven-membered ring range from 118.7 (1.3) to 130.4 (1.2)°, reflecting the distortion of the ring from planarity. [Angles within the planar seven-membered rings in $C_{10}H_8C_6H_3(NO_2)_3$[54] and 1,3-$(HO_2CC_2H_4)_2C_{10}H_6$[55] range from 127.15 to 129.47° and from 125.9 to 130.2°, respectively.]

Intermolecular Contacts.—There are no abnormally short intermolecular contacts either in the monoclinic or in the triclinic crystal, thus confirming that the crystals contain discrete molecular units of 4,6,8-trimethylazulenetetraruthenium enneacarbonyl.

Shortest contacts (of each type) in the triclinic crystal are O···O = 2.94 Å, C···O = 3.12 Å, C···C = 3.49 Å. In the monoclinic crystal, the minimum contacts are O···O = 3.03 Å, C···O = 3.04 Å, and C···C > 3.5 Å.

Acknowledgments.—This work has been generously supported by the National Science Foundation (Grant GP-8077) and the Advanced Research Projects Agency (Contract SD-88). K. G. acknowledges, with gratitude, the receipt of a PHS predoctoral research fellowship.

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