formula $Os_6(CO)_{16}[C(H)NMe_2](\mu_3\text{-}S)(\mu_4\text{-}S)(\mu\text{-}H)_2$ (**2**). These products can be separated by TLC on silica gel. In order of elution they are as follows: yellow **2a**, 2.5 mg (11%), red **2b**, 8.8 mg (38%), and orange **2c**, 6.0 mg (26%).[11] Compounds **2a** and **2b** have been characterized by single-crystal X-ray diffraction analyses.[12,13]

An ORTEP diagram of the molecular structure of **2a** is shown in Figure 1.[14] The molecule consists of two triangular triosmium groupings Os(1)–Os(2)–Os(3) and Os(3)–Os(4)–Os(5) that share the atom Os(3). Each triangle is bridged by a sulfido ligand. A sixth osmium atom Os(6) bridges the two triangular groups through a metal–metal bond to Os(5) and a coordinate bond to S(1). Os(6) contains a terminally coordinated secondary (dimethylamino)carbene ligand, C(17), N, C(18), C(19). The Os(6)–C(17) distance of 2.05 (2) Å is similar to the osmium–carbon (carbene) distances of 2.026 (15) Å [2.04 (2) Å] observed in $Os_3(CO)_8[C(H)NMe_2](\mu_3\text{-}S)(\mu\text{-}H)_2$ (**3**).[3] Compound **2a** contains two bridging hydride ligands (^1H NMR, $\delta = -18.90$ and -27.00). These were not located crystallographically. There are 16 linear terminal carbonyl ligands distributed as shown in Figure 1.

An ORTEP diagram of the molecular structure of **2b** is shown in Figure 2.[15] The molecule contains a cluster of six osmium atoms. The group Os(1)–Os(2)–Os(3)–Os(4) is arranged in the form of a butterfly tetrahedron. Atoms Os(5) and Os(6) extend from the wing-tip atom Os(4). Sulfido ligand S(1) bridges the Os(1)–Os(2)–Os(4) open triangular group of the butterfly tetrahedron and is linked to Os(6). Sulfido ligand S(2) bridges the open triangular group Os(4)–Os(5)–Os(6). A secondary (dimethylamino)carbene ligand is terminally coordinated to Os(6). The Os(6)–C(17) distance of 2.04 (3) Å is equivalent to those in **3** and in **2a**. Compound **2b** contains two bridging hydride ligands (^1H NMR, $\delta = -13.67$ and -15.25). These were not located crystallographically, but they are beleived to bridge the two elongated osmium–osmium bonds Os(1)–Os(3) = 2.991 (1) Å and Os(5)–Os(6) = 2.981 (1) Å. There are 16 terminal carbonyl ligands distributed shown in Figure 2. Compound **2b** is structurally very similar to its carbonyl homologue $Os_6(CO)_{17}(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}H)_2$.[16]

When refluxed in octane solvent under an atmosphere of carbon monoxide, compound **2a** is converted into **2b** (16% yield) and $Os_5(CO)_{14}(\mu_3\text{-}S)_2(\mu\text{-}H)_2$ (13% yield).[17] The latter is formed by the removal of the carbene-containing osmium atom Os(6). The transformation of **2a** into **2b** is dependent on the CO atmosphere, and little or no **2b** is formed in its absence. A transformation pathway of **2a** into **2b** is shown in Scheme I. This is clearly a multistep process that cannot be delineated at this time, but it is believed that the shift of a CO ligand from Os(6) to Os(3) in **2a** is of pivotal importance. Although this could be accomplished by a sequence of bridge/terminal rearrangements, the importance of the CO atmosphere suggests a dissociation/readdition process as shown in Scheme I.

Compounds **2b** and **2c** equilibrate slowly in solution at 25 °C. The equilibrium mixture **2b**/**2c** is 1.00/0.89 in $CDCl_3$ solvent. Structural studies of **2c** are currently in progress.

The transformation of **1** into the **2a–c** mixture of isomers represents another example of the arene elimination/cluster condensation sequence that was observed for $Os_3(CO)_{10}(\mu\text{-}SPh)(\mu\text{-}H)$;[18] however, the most interesting features demonstrated by this reaction are the transformation of a coordinated trimethylamine ligand into a secondary (dimethylamino)carbene ligand in a high-nuclearity cluster and its stability in the presence of major framework rearrangements in the cluster. It is believed that tertiary amines containing methyl groups may provide a general route to secondary aminocarbenes in polynuclear metal complexes.

Acknowledgment. These studies were supported by the Office of Basic Energy Science of the U.S. Department of Energy. We wish to thank Johnson-Matthey for a loan of osmium tetraoxide.

Supplementary Material Available: Tables of crystal data, positional and thermal parameters, and selected interatomic distances and angles for **2a** and **2b** (14 pages); tables of observed and calculated structure factor amplitudes for both structural analyses (36 pages). Ordering information is given on any current masthead page.

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(11) IR (ν(CO), in hexane, cm^{-1}): for **2a**, 2097 w, 2079 s, 2072 s, 2053 s, 2020 s, 2012 m, 2008 s, 1969 w, 1941 w; for **2b**, 2099 w, 2080 s, 2069 s, 2046 m, 2037 w, 2029 s, 2020 w, 2003 m, 1991 w, 1987 w, 1973 w, br; for **2c** (in CH_2Cl_2 solvent), 2098 w, 2080 s, 2066 s, 2045 s, 2029 s, 1997 m, br, 1986 m, br. ^1H NMR (δ, in CD_2Cl_2: for **2a**, 10.00 (s, 1 H), 3.80 (s, 3 H), 3.73 (s, 3 H), −18.90 (s, 1 H), −27.00 (s, 1 H); for **2b**, 11.56 (s, 1 H), 3.75 (s, 3 H), 3.69 (s, 3 H), −13.67 (s, 1 H), −15.25 (s, 1 H); for **2c**, 9.98 (s, 1 H), 3.53 (s, 3 H), 3.45 (s, 3 H), −13.49 (s, 1 H), −15.28 (s, 1 H). Satisfactory elemental analyses have been obtained: Anal. Calcd (found) for **2a**: C, 13.32 (13.41); H, 0.53 (0.58); N, 0.82 (0.86). Calcd for **2b**: C, 13.32 (13.68); H, 0.53 (0.61); N, 0.82 (0.84). The latter was crystallized in the absence of benzene solvent.

(12) Crystals of **2a** and **2b** were grown by slow evaporation of solvent from CH_2Cl_2/hexane/benzene solutions at −20 °C. Compound **2a** crystallizes in the triclinic crystal system, space group $P\bar{1}$, with $a = 15.652$ (3) Å, $b = 9.587$ (2) Å, $c = 10.768$ (4) Å, $\alpha = 99.70$ (2)°, $\beta = 95.92$ (2)°, $\gamma = 92.80$ (2)°, $Z = 2$, and $\rho_{calcd} = 3.58$ g/cm^3. The structure was solved by a combination of Patterson and difference Fourier techniques and was refined (3412 reflections) to the final values of the residuals $R = 0.040$ and $R_w = 0.044$. The carbonyl ligand C(8)–O(8) was found to be partially disordered. Two sites were satisfactorily refined. Compound **2b** crystallizes in the monoclinic crystal system with $^1/_2$ mol of C_6H_6/mol of complex in space group $P2_1/c$, with $a = 16.070$ (4) Å, $b = 12.054$ (5) Å, $c = 18.387$ (13) Å, $\beta = 107.08$ (4)°, $Z = 4$, and $\rho_{calcd} = 3.41$ g/cm^3. The structure of **2b** was solved by direct methods (MULTAN) and was refined (2820 reflections) to the final values of the residuals $R = 0.044$ and $R_w = 0.045$.

(13) Diffraction measurements for **2a** were made on a Rigaku AFC6 automatic diffractometer by using Mo Kα radiation. Diffraction measurements for **2b** were made on an Enraf-Nonius CAD-4 automatic diffractometer. The structure solution and refinements were performed on a Digital Equipment Corp. VAX 11/782 computer by using the Enraf-Nonius SDP program library. Diffraction data were corrected for the effects of absorption.

(14) Selected interatomic distances (Å) and angles (deg) for **2a** are as follows: Os(1)–Os(2) = 2.808 (1), Os(1)–Os(3) = 2.819 (1), Os(2)–Os(3) = 2.838 (1), Os(3)–Os(4) = 2.808 (1), Os(3)–Os(5) = 2.884 (1), Os(4)–Os(5) = 2.877 (2), Os(5)–Os(6) = 2.864 (1), Os(1)–S(1) = 2.338 (5), Os(2)–S(1) = 2.338 (5), Os(3)–S(1) = 2.381 (5), Os(3)–S(2) = 2.399 (5), Os(4)–S(2) = 2.370 (5), Os(5)–S(2) = 2.384 (6), Os(6)–S(1) = 2.459 (5), Os(6)–C(17) = 2.05 (2), C(17)–N = 1.30 (2); Os(6)–C(17)–N = 135 (1).

(15) Selected interatomic distances (Å) and angles (deg) for **2b** are as follows: Os(1)–Os(2) = 2.815 (1), Os(1)–Os(3) = 2.991 (1), Os(2)–Os(3) = 2.844 (1), Os(2)–Os(4) = 2.868 (1), Os(3)–Os(4) = 2.823 (1), Os(4)–Os(5) = 2.802 (1), Os(5)–Os(6) = 2.981 (1), Os(1)–S(1) = 2.365 (5), Os(2)–S(1) = 2.397 (5), Os(4)–S(1) = 2.334 (5), Os(6)–S(1) = 2.434 (5), Os(4)–S(2) = 2.443 (5), Os(5)–S(2) = 2.371 (6), Os(6)–S(2) = 2.442 (5), Os(6)–C(17) = 2.04 (3), C(17)–N = 1.31 (3); Os(6)–C(17)–N = 134 (2).

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Synthesis of a New Family of Water-Soluble Clusters: $Ru_3(CO)_{12-x}L_x$ (x = 1-3), $Os_3(CO)_{10}L_2$, and $Ir_4(CO)_9L_3$ (L = Trisodium Salt (Hydrated) of Tris(m-sulfonatophenyl)phosphine)

Sir:

The synthesis of water-soluble clusters is of interest for their possible use as homogeneous catalysts in this solvent. There are several advantages for carrying out catalytic reactions in water: easy recovery of the costly transition metal in a biphasic system (e.g. in hydroformylation of olefins with rhodium),[1,2] use of water

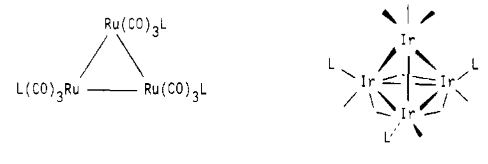
Table I. Spectroscopic Data for the Water-Soluble Clusters[17]

compd[a]	ν(CO),[b] cm^{-1}	$\delta(^{31}P)$[c]	ref
1	2053 (m), 1992 (m), 1973 (s)	38.7 (s)	*e*
PPh_3	2044 (m), 1978 (m), 1967 (s)		8
2	2080 (sm), 2030 (ms), 2005 (sh), 1990 (vs)	37.9 (s)	*e*
PPh_3	2072 (sm), 2047 (sm), 2019 (s), 1996 (vs)		8
3	2100 (sm), 2050 (ms), 2025 (s), 2010 (sh), 2000 (sh), 1982 (m, br)	35.4 (s)	*e*
PPh_3	2098 (m), 2046 (s), 2038 (sh), 2024 (sh), 2015 (vs), 1985 (sm)		8
4	2075 (sm), 2020 (s), 1992 (vs), 1965 (s), 1940 (sh)	−2.3 (s)	*e*
PPh_3	2088 (sm), 2035 (s), 2002 (vs), 1963 (s), 1946 (m), 1935 (sh)		15
5	2049 (m), 2012 (s), 1985 (vs), 1790 (vs), 1784 (sh)	+21.3 (s), P_{rad};[d] −15 (s), P_{ax}	*e*
PPh_3	2042 (ms), 2015 (sh), 1982 (vs), 1772 (vs)	+20 (s), P_{rad};[d] −17 (s), P_{ax}	16

[a] PPh_3: Values for analogous PPh_3 complex. [b] IR taken in KBr. [c] In D_2O; s = singlet. [d] P_{rad} = phosphorous in radial configuration, and P_{ax} = phosphorus in axial position with an intensity of 2:1. [e] This work.

as a reagent (as in the Wacker process[3]), and/or obtention of a better regioselectivity with amphiphilic compounds. A better understanding of the chemical behavior and stability of transition metals, especially low-valent group VIII (groups 8–10[21]) complexes, in water is important for the development of homogeneous catalysis in this solvent.[4] We describe here the synthesis and characterization of a new family of water-soluble metal clusters based on the hydrosoluble ligand L = $P[C_6H_4\text{-}m\text{-}SO_3^-Na^+(H_2O)]_3$ first described by Kuntz.[1,5]

The red-violet trisubstituted cluster $Ru_3(CO)_9[P(C_6H_4\text{-}m\text{-}SO_3^-Na^+(H_2O))_3]_3$[6] (**1**) has been obtained by three different routes: (a) thermal substitution of $Ru_3(CO)_{12}$ (1 mmol) with the ligand L (3.2 mmol in boiling methanol for 12 h); this reaction was followed by precipitation of the cluster in a methanol–diethyl ether mixture (80% yield); (b) activation of $Ru_3(CO)_{12}$ (1 mmol) by Me_3NO (anhydrous, 3 mmol) in the presence of L (3 mmol) in dichloromethane–methoxyethanol (2:1) for 6 h;[7] (c) activation of $Ru_3(CO)_{12}$ (1 mmol) by [PPN]X salts (X = Cl^-, CH_3COO^-)[8] (0.3 mmol) in the presence of L (3.2 mmol) at room temperature for 4 h in a solvent mixture of tetrahydrofuran–methoxyethanol–dichloromethane (2:1:0.1) (95% yield).[9] The red-violet cluster can be recrystallized in methanol–diethyl ether at −5 °C.[10]



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5

Figure 1. Ligand positions in substituted complexes.

The mono- and disubstituted clusters $Ru_3(CO)_{11}L$ (**2**) and $Ru_3(CO)_{10}L_2$[6] (**3**) have been obtained in almost pure form (ca. 90% yield) by the stoichiometric reactions at 0 °C of respectively $Ru_3(CO)_{11}(CH_3CN)$ (1 mmol) or $Ru_3(CO)_{10}(CH_3CN)_2$ (1 mmol)[11] with 1 or 2 equiv of L in a mixture of acetonitrile–methoxyethanol (2:1).[9,12] The compounds can be recrystallized from methanol–diethyl ether at −5 °C. Compounds **2** and **3** give the red-violet compound **1** on refluxing in methanol with sufficient ligand L.

The disubstituted yellow osmium derivative $Os_3(CO)_{10}L_2$ (**4**) has been obtained via a similar route by thermal substitution at 40 °C of $Os_3(CO)_{10}(CH_3CN)_2$[11] (1 mmol) with 2 equiv of L in a mixture of acetonitrile–methoxyethanol (2:1) for 4 h. The cluster can be purified by recrystallization from methanol–diethyl ether at −5 °C (75% yield).

The trisubstituted yellow iridium derivative $Ir_4(CO)_9L_3$ (**5**) has been obtained by thermal treatment of $Ir_4(CO)_{12}$ (1 mmol) in boiling methanol in the presence of 3 equiv of L. The cluster slowly precipitates in almost pure form from the reaction medium during the reaction (70% yield).

All these clusters are easily soluble in water[13] in methanol and methoxyethanol, giving colored, somewhat air-stable solutions. The stability to air as well as the solubility increases with the degree of substitution (e.g. compound **1** can be kept in aqueous solution in air for more than 15 days without apparent decomposition). They are practically insoluble in the usual nonpolar or less polar organic solvents, which makes them potentially useful for carrying out catalytic reactions in water or at the liquid–liquid interface between water and an organic solvent.

The compound $Ru_3(CO)_9L_3$ (**1**) has been used as an useful intermediate for the preparation of water-soluble tetrameric ruthenium hydrides.[14]

The infrared ν(CO) frequencies and ^{31}P NMR resonances are presented in Table I.[17] Upon comparison of these frequencies for the expected water soluble clusters with those for the analogous known tris(triphenylphosphine) complexes[8,11,16] and other substituted clusters,[15] we notice a close similarity in the pattern, intensities, and position of the IR bands in the ν(CO) region, for both sets of clusters, suggesting possible isostructural conformations. The other regions of the spectra clearly show the presence

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(6) Compound **1** (red-violet) shows absorption bands (in MeOH) with λ_{max} at 382 and 520 nm. The analogous deep violet $Ru_3(CO)_9(PPh_3)_3$ has λ_{max} at 387 and 507 nm. Compound **2** (red-orange) shows λ_{max} at 383 and 488 nm (in MeOH), compared with the analogous deep red $Ru_3(CO)_{10}(PPh_3)_2$, which has bands at 376 and 494 nm.[20] Compound **3** (orange) has λ_{max} at 376 and 488 nm (in MeOH).

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(8) (a) Lavigne, G.; Kaesz, H. D. *J. Am. Chem. Soc.* **1984**, *106*, 4647. (b) $PPN^+ = N(P(C_6H_5)_3)_2^+$.

(9) Due to the low solubility of the ligand L in organic solvents (except methanol and methoxyethanol), the success of the synthesis depends greatly on the solvent mixture used. The ligand L, dissolved in methoxyethanol or methanol, is added slowly to the $Ru_3(CO)_{12}$ dissolved in tetrahydrofuran with strong stirring. The Me_3NO is added, dissolved in CH_2Cl_2. After initial cloudiness, the components dissolve completely in the solvent mixture. A similar procedure is used for the acetonitrile complexes as starting material. The compounds produced precipitate slowly from the solution upon cooling. Differences in solubility in the solvent mixture allow final purification. The complexes are strongly absorbed on silica or alumina columns thus not permitting chromatographic separation.

(10) The compounds give satisfactory elemental analysis, e.g. $Ru_3(CO)_9[P(C_6H_4\text{-}m\text{-}SO_3^-Na^+(H_2O))_3]_3$ Anal. Calcd: C, 31.2; H, 2.2; S, 11.9; P, 3.8; Na 8.5; Ru, 12.5. Found: C, 29.7; H, 2.6; S, 10.9; P, 3.6, Na, 8.1, Ru, 10.8.

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(12) Similar results are obtained if the intermediate acetonitrile (in dichloromethane–acetonitrile) or trimethylamine complexes (in dichloromethane or chloroform)[11] are used directly in solution with the appropriate amounts of ligand L dissolved in methoxyethanol or methanol, with strong Ar bubbling.

(13) Solubility (in g/L) in water: compound **1**, 800; compound **2**, 300; compound **3**, ca. 250; compound **4**, ca 200; compound **5**, 500.

(14) The reactivity of these tetrameric hydrides is presently under investigation in our laboratories.

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(17) ν(SO) frequencies: compound **1**, 1224 (sh), 1188 (vs, br), 1041 (s), 623 (s); compound **2**, 1239 (sh), 1199 (vs, br), 1040 (s), 623 (s); compound **3**, 1238 (sh), 1199 (vs, br), 1040 (s), 624 (s); compound **4**, 1238 (sh), 1199 (vs, br), 1040 (s), 622 (s); compound **5**, 1211 (sh), 1196 (s), 1035 (s), 620 (s); free ligand, 1211–1196 (vs, br), 1040 (s), 1039 (s), 620 (s).

of the ligands L[17] in the complexes.

All the ruthenium and osmium clusters give singlets in the ^{31}P NMR (Table I, in D_2O) spectra at room temperature, suggesting that all the P atoms are equivalent (unfortunately the NMR data for the analogous PPh_3 complexes of Ru and Os, as well as crystallographic data, are not easily available[18]). Concerning the Ir cluster (**5**) (the X-ray structure is available for the PPh_3 analogue[19]), two ligands L adopt a radial configuration and one adopts an axial configuration like the analogous PPh_3 cluster (Figure 1).[16]

The number and position of the UV-Vis bands for the Ru clusters[6] suggest a triangular metal-metal-bonded framework as discussed by Gray et al.[20] for the analogous PPh_3-substituted clusters.

The catalytic properties in water for these clusters and derived related compounds are presently being investigated in our laboratories and will be described later.

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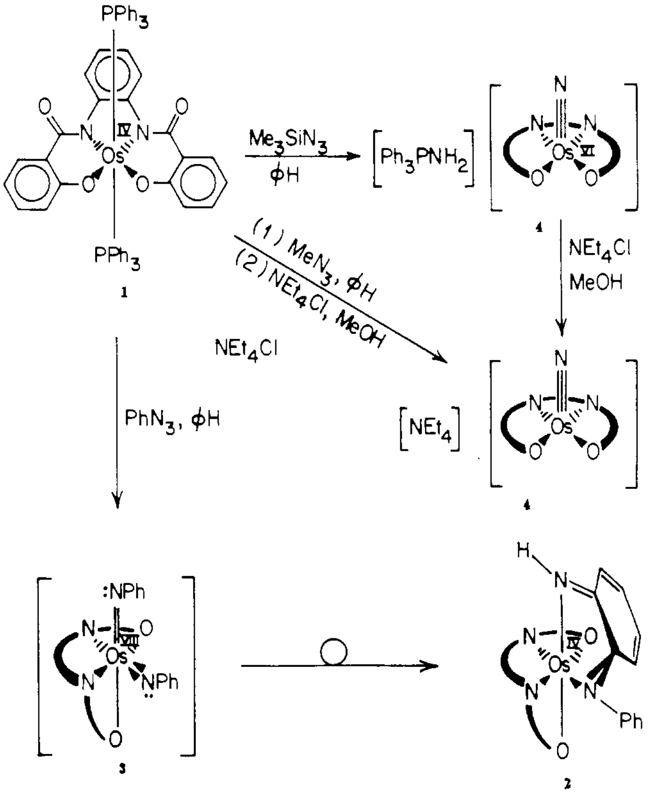
(21) The periodic group notation in parentheses is in accord with recent actions by IUPAC and ACS nomenclature committees. A and B notation is eliminated because of wide confusion. Groups IA and IIA become groups 1 and 2. The d-transition elements comprise groups 3 through 12, and the p-block elements comprise groups 13 through 18. (Note that the former Roman number designation is preserved in the last digit of the new numbering: e.g., III → 3 and 13.)

Reactions of Osmium(IV) Complexes of PAC Ligands with Azide Species

Sir:

The search for new oxidizing agents is an interdisciplinary theme.[1] We have been developing new highly oxidized and highly oxidizing inorganic complexes by utilizing oxidation-resistant polyanionic chelating (PAC) ligands.[2-6] As part of this work, we sought to incorporate multiply bonded nitrogen ligands[7] in the same coordination sphere as PAC ligands and discovered that organic azides react in several ways with the osmium(IV) complex *trans*-Os(η^4-HBA-B)(PPh_3)$_2$ (**1**) (Scheme I), depending upon the nature of the azide. Here we report a thermal C-H bond activation via a putative osmium(VIII) bis(imido) complex and the synthesis of a nitrido complex derived from methyl or trimethylsilyl azide.

Scheme I



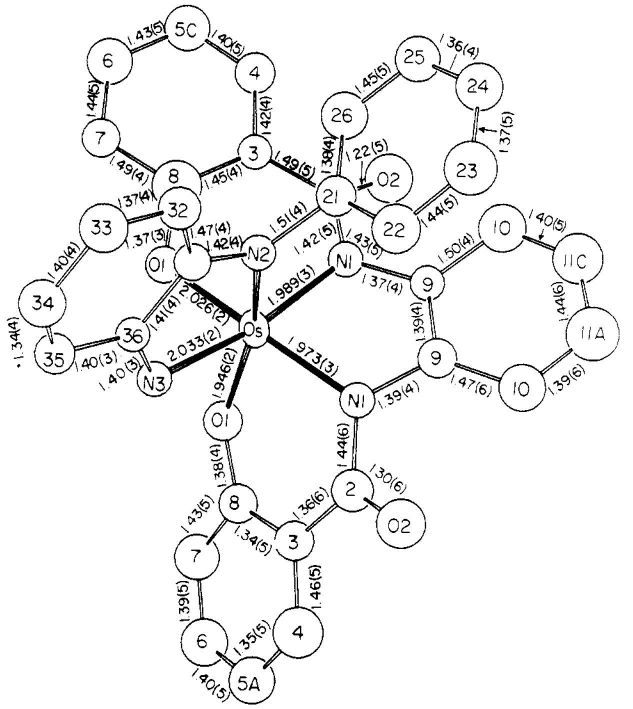


Figure 1. Molecular structure of *cis*-β-Os(η^2-NPhC_6H_4NH)(η^4-HBA-B) (**2**).

The reaction of **1** with a twofold excess of phenyl azide (*caution*!) in benzene proceeds at room temperature[8] to yield the

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(8) Reaction proceeds identically in presence or absence of laboratory lighting.