Reactions of triosmium clusters $Os_3(CO)_{11}(MeCN)$ and $(\mu\text{-}H)Os_3(CO)_{10}(\mu\text{-}OH)$ with *L*-α-serine ethyl ester and ethanolamine

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A series of novel chiral complexes with μ,η^1 and μ,η^2 coordination of organic ligands were prepared by reactions of $Os_3(CO)_{11}(MeCN)$ and $(\mu\text{-}H)Os_3(CO)_{10}(\mu\text{-}OH)$ with *L*-α-serine ethyl ester and ethanolamine. The diastereomeric cluster complexes with serine ligands were separated by crystallization or chromatography. The structures of the compounds obtained were confirmed by ^1H NMR and IR spectroscopy, mass-spectrometry, elemental analysis, and X-ray diffraction analysis.

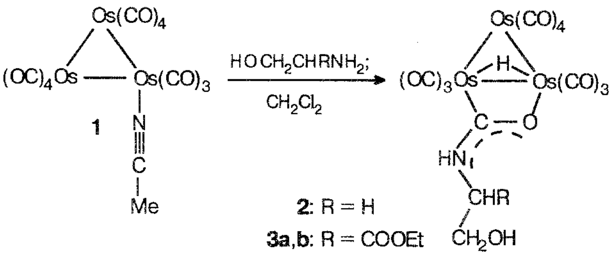
Key words: triosmium clusters; *L*-α-serine ethyl ester; ethanolamine; chiral cluster complexes; diastereomers.

Previously we reported that the interaction of $Os_3(CO)_{12}$ derivatives with *L*-α-cysteine ethyl ester, which has two reactive functional groups, differs fundamentally from their reactions with amines and esters of the simplest amino acids.[1] This stimulated us to study the reactions with the closest analog of cysteine, *L*-α-serine ethyl ester, and with ethanolamine, which contains similar functional groups. Though the coordination ability of an oxygen atom in complexes of transition metals in low oxidation states is lower than that of a sulfur atom and an amino group, organic compounds are readily bonded with trinuclear osmium clusters through the oxygen atom in the cases when chelate-type or bridging coordination is possible.[2] It should be emphasized that an optically active form of the serine ester was used, which allowed us to hope to prepare optically active clusters. In addition, the use of optically active ligands often makes it possible to reveal the peculiarities of the course of the reaction and of the structures of the resulting compounds.[3]

In the present work we used the same triosmium clusters, that had been used previously, as the starting complexes.[1] This makes it possible to compare the reactivities of polyfunctional compounds that differ in the presence of a hydroxy or thiol group.

The reaction of $Os_3(CO)_{11}(MeCN)$ (**1**) with amino alcohols $HOCH_2CHRNH_2$ (R = H, COOEt) occurs according to Scheme 1, which is characteristic of amines and esters of the simplest amino acids:[4] it involves carbonylation of the amino group and the resulting carboxamide ligand is coordinated at the Os_3 ring in a bridging fashion.

Scheme 1



The structure of the resulting compounds is indicated by the similarity of their IR spectra to the analogous spectrum[4] of the structurally characterized $(\mu\text{-}H)Os_3(CO)_{10}\{\mu\text{-}OCNHCH(Me)COOEt\}$ as well as by the data of NMR spectroscopy, mass spectrometry, and elemental analysis. The noncoordinated OH group manifests itself in both the IR and ^1H NMR spectra (see Experimental). The chemical shift of the μ-H groups is typical of triosmium clusters with bridging carboxylate ligands.[5]

It was found that the hydroxy group ranks far below the thiol group in its reactivity towards clusters similar to **1**, since in the reaction with **1** the cysteine ester is exclusively coordinated by the thiol group.[1] A comparison of ethanolamine with the *L*-α-serine ester (see Scheme 1) showed that the only effect of the carboxylate group is that the reactivity of the amino acid ester is somewhat lower than that of the amino alcohol; the carboxylate group exerts no effect on the reaction route.

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya*, No. 3, pp. 525—530, March, 1994.

The edge-type coordination of the asymmetric bridging ligand in complexes **2** and **3** leads to the disappearance of the symmetry plane and to the formation of chiral compounds. In the case of *L*-α-serine ethyl ester, a mixture of two diastereomers should be produced, which is confirmed by duplication of practically all of the signals in the ^{1}H NMR spectrum of complex **3** (Fig. 1). However, this pair of diastereomers could not be separated by chromatography, in contrast to the analogous complex $(\mu\text{-H})Os_3(CO)_{10}\{\mu\text{-OCNHCH(Me)COOEt}\}$ (see Ref. 6). This is probably due to strong interaction of the free hydroxyl group with silica gel. Therefore, we decided to coordinate the hydroxy group in compound **3** with the nearest osmium atom using Me_3NO, which we had used previously[1] to coordinate the free amino group in $(\mu\text{-H})Os_3(CO)_{10}\{\mu\text{-SCH}_2CH(NH_2)COOEt\}$. The possibility of such coordination from the stereochemical point of view was preliminarily shown with Dreiding models.

The reaction of cluster **3** with 1.5 equiv. of Me_3NO (Scheme 2) affords two new compounds in ~1:1 ratio, which we managed to isolate by chromatography, though their R_f are very close. The IR spectra of these complexes are practically identical in the region of CO stretching vibrations and their appearance is normal for the spectra of trinuclear osmium clusters of the type $(\mu\text{-H})Os_3(CO)_9(\mu\text{-X})(L)$ (X is a trielectron bridging ligand, L is an *n*-donor two-electron ligand).[4] The IR spectra of both compounds also exhibit bands corresponding to vibrations of the noncoordinated OH group.

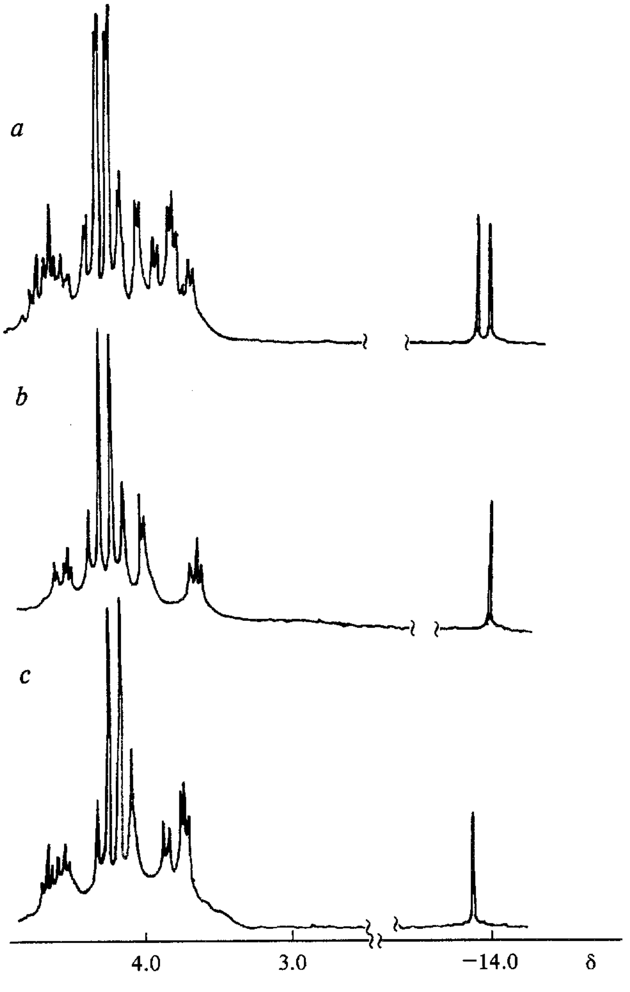
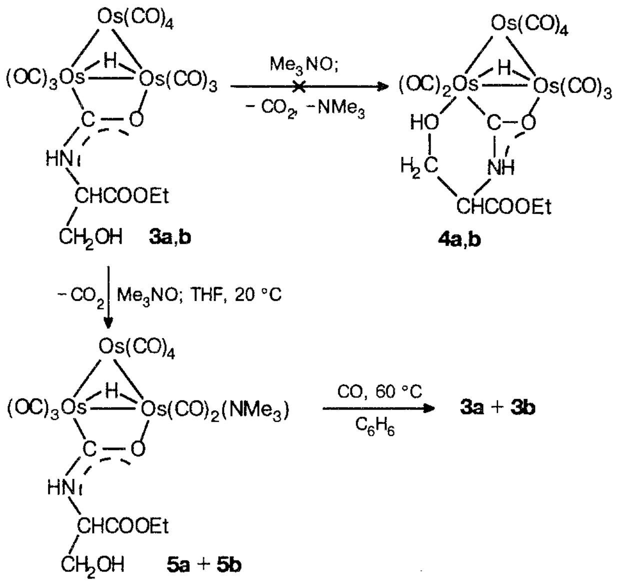


Fig. 1. Fragments of the ^{1}H NMR spectra ($CDCl_3$, TMS, 22 °C) of a mixture of diastereomers (*a*) and single diastereomers (*b*, *c*) of $(\mu\text{-H})Os_3(CO)_{10}\{\mu\text{-OCNHCH(COOEt)CH}_2OH\}$.

Scheme 2

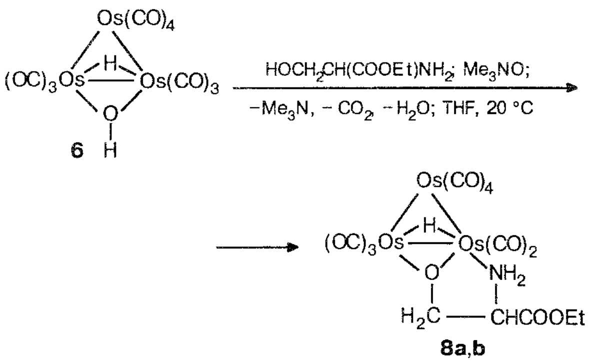


The ^{1}H NMR spectra of the starting and the resulting complexes differ little from one another. The main distinction is that the signals are not duplicated and an intense single peak (9 H) is exhibited at δ 2.3. The signal for the bridging hydrogen atom is shifted downfield by ~1.5 ppm. These data imply that, instead of the expected pair of diastereomers **4** with the coordinated OH group, a pair of diastereomers (**5a** + **5b**) has been formed in which the vacant coordination site is occupied by Me_3N produced in the reaction.

The trimethylamine ligand in clusters **5a** and **5b** is readily replaced by CO. As this takes place, the individual diastereomers **3a** and **3b**, having equal R_f in various eluents, form. The ^{1}H NMR spectra of these complexes, unlike that of the starting mixture of **3a**,**b** clusters do not exhibit duplicated signals, but each of them displays one high-field singlet whose chemical shift is identical to that in the spectrum of **3a,b**.

In the case of cluster complex $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-OH})$ (**6**), amines, phosphines, and other *n*-donor ligands in the presence of Me_3NO, substitute for one of the CO groups at the osmium atoms bonded through bridging ligands.[7] Therefore, the result[1] obtained previously in a study of the reaction of compound **6** with a cysteine ester in the presence of Me_3NO was somewhat unexpected: not only one of the CO groups but also the μ-OH ligand was replaced and compound $(\mu\text{-H})Os_3(CO)_9\{\mu,\eta^2\text{-}SCH_2CH(COOEt)NH_2\}$ (**7**) formed. Serine ethyl ester reacts with cluster **6** in a similar way, which is indicated by various physicochemical characteristics of the single resulting compound (Scheme 3).

Scheme 3

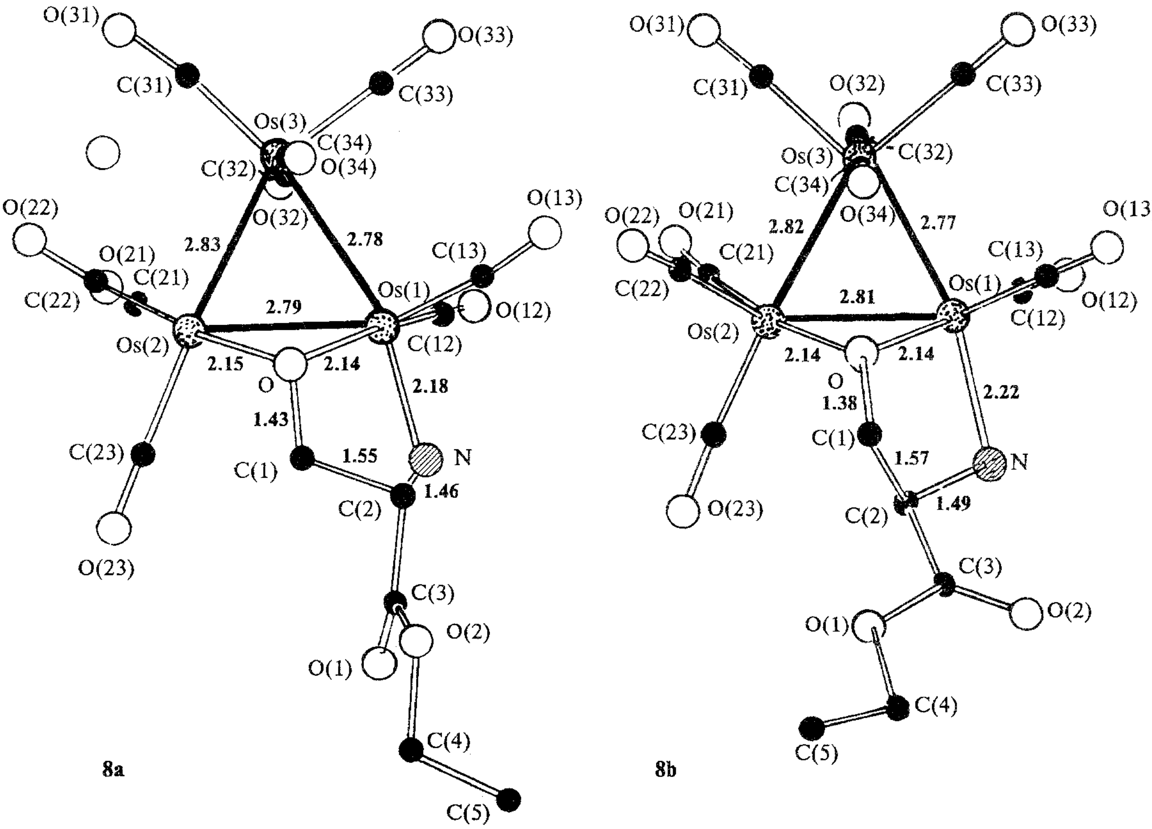


As in the case of *L*-α-cysteine ester, this reaction afforded a mixture of two diastereomers, **8a** and **8b**, which is confirmed by the fact that the ^1H NMR spectrum of the reaction product exhibits two hydride signals and a duplicated quartet of signals for the $O—C\underline{H}_2—CH_3$ methylene group. Crystallization from a hexane—CH_2Cl_2 mixture yielded two types of single crystals, which were separated manually. An X-ray structural investigation was carried out for each type of crystals.[8]

The structures of molecules **8a** and **8b** are given in Fig. 2. The serine ligands in both complexes are coordinated in a bidentate fashion along one of the edges of the Os_3 triangle through the practically symmetrical bridging oxygen atom and the terminal NH_2 group. As a result, a five-membered chelate-type ring is formed. A decrease in the Os(1)—Os(3) bond length as compared with the other two bond lengths, caused by the *trans*-effect of the NH_2 group, was noted.

The main distinctions between the structures **8a** and **8b** are that they have different conformations of the chelate ring (an "envelope" in the case of **8a** and a "twist"-form for **8b**) and different orientations of the carboxylate group with respect to the chelate ring.

The X-ray structural study of complexes **8a** and **8b** attests to a racemic nature of all of the single crystals studied, which implies that they contain amino acid ligands having a *D* configuration. In our opinion, *D*-amino acid is most likely to form from the *L*-isomer during its racemization upon bidentate-type coordination with the Os_3 ring. The presence of a *D* admixture in the commercial *L*-α-serine or racemization of *L*-amino



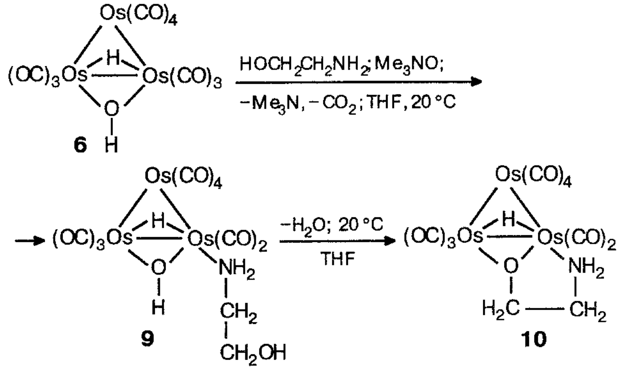
8a **8b**

Fig. 2. Structures of $(\mu\text{-H})Os_3(CO)_9\{\mu,\eta^2\text{-}OCNHCH(COOEt)NH_2\}$ diastereomers.

acid during esterification seems less probable, because the mass of the resulting single crystals of **8a** and **8b** comprises a substantial fraction of the amount used for crystallization (no less than 20 %) and the esterification is carried out in an acidic medium, which makes racemization practically impossible.

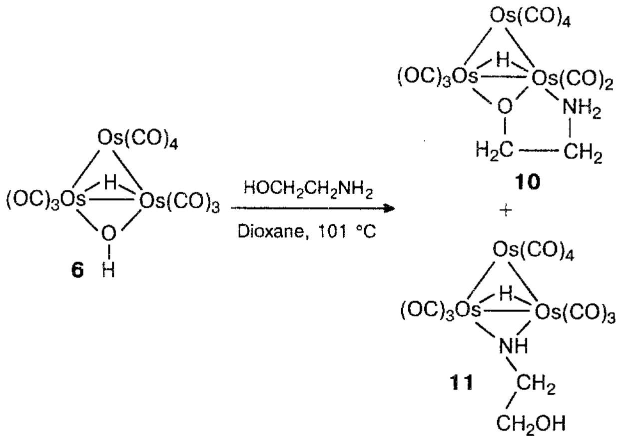
In contrast to Scheme 3, the reaction of $HOCH_2CH_2NH_2$ with compound **6** in the presence of Me_3NO initially affords an unstable compound, which decomposes when one attempts to isolate it by chromatography on silica gel. The region of the CO stretching vibrations in the IR spectrum recorded for this product without its isolation from the reaction mixture has an appearance typical[7] of $(\mu\text{-H})Os_3(CO)_9(\mu\text{-OH})(L)$ trinuclear osmium clusters and may probably be assigned to cluster **9**. When stored in solution, this complex is slowly converted into another cluster (**10**), which has a somewhat greater R_f and is stable under conditions of chromatographic isolation. The IR spectrum for this compound in the region of CO stretching vibrations is practically identical to that for complex **8**. Like the spectrum for compound **8**, that for the complex under consideration does not exhibit a band associated with the OH vibrations, and the bands of the NH_2-group vibrations are shifted by ~80 cm^{-1} with respect to those in the spectrum of free ethanolamine. These results, in combination with the data from the ^1H NMR spectra and elemental analysis, allowed us to assume that the reaction of cluster **6** with ethanolamine occurs according to Scheme 4.

Scheme 4



We also studied the thermal reaction of compound **6** with ethanolamine. Along with complex **10**, this reaction affords one more compound in about the same yield. The region of the stretching vibrations of CO groups in the IR spectrum of this compound is practically identical to that for the cluster $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-NHCHMeCOOEt})$, which has been prepared by a similar reaction of **6** with the ethyl ether of *L*-α-alanine.[3] The mass spectrum of the resulting complex exhibits a molecular-ion peak with *m/z* 917 corresponding to $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-NHCH_2CH_2OH})$. The ^1H NMR spectrum of the product is consistent with this structure (Scheme 5).

Scheme 5



The isolation of two products in this case is probably due to the presence of two reaction pathways: a) nucleophilic attack of the NH_2 group at an osmium atom with substitution for the μ-OH group to afford cluster **11**; and b) nucleophilic attack of the NH_2 group at the carbon atom of one of the CO groups and the substitution of a second ethanolamine molecule for the $OCNH_2CH_2CH_2OH$ moiety to give the intermediate complex **9**, which quickly transforms to cluster **10** under the reaction conditions.

The results obtained in this work once again confirm that the coordination of polyfunctional organic compounds by several metal sites in cluster-type complexes has a more complicated character than would have been expected from the simple combination of the reactivities of the separate functional groups incorporated in such ligands and is determined by both the nature of the organic ligand and the conditions in which the reaction is conducted as well as the type of cluster used.

Experimental

^1H NMR spectra were recorded on a Bruker SXP 4-100 spectrometer, IR spectra were obtained on a Specord IR-75 spectrophotometer. The electron impact mass spectra were obtained on an MX-1310 mass spectrometer at an ionizing voltage of 70 eV.

The starting compounds, $Os_3(CO)_{11}(MeCN)$ (see Ref. 9), $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-OH})$ (see Ref. 10), and $HOCH_2CH(NH_2)COOEt$ (see Ref. 11) were prepared by the previously described procedures.

All of the reactions were carried out under argon using freshly distilled solvents.

The reaction of $HOCH_2CH(NH_2)COOEt$ with $Os_3(CO)_{11}(MeCN)$. $Os_3(CO)_{11}(MeCN)$ (134 mg, 14.6 mmol) and *L*-α-serine ethyl ester (350 mg, 2.64 mmol) were dissolved in 15 mL of CH_2Cl_2. The solution was boiled for 2 h in an argon flow, completion of the reaction was determined by the disappearance of the TLC-spot of $Os_3(CO)_{11}(MeCN)$. The volume of the reaction mixture was brought to 8—10 mL, and the mixture was chromatographed on Silufol using a 3:2:1 benzene—hexane—diethyl ether mixture as the eluent to afford two fractions. The first fraction (31 mg) was the starting $Os_3(CO)_{11}(MeCN)$ and the second fraction (36 mg, 31.72 % on unreacted $Os_3(CO)_{11}(MeCN)$) was a mixture of diastereomers of $(\mu\text{-}H)Os_3(CO)_{10}\{\mu\text{-}OCNHCH(CH_2OH)COOEt\}$ (**3a**,**b**). IR (cyclohexane), $\nu CO/cm^{-1}$: 2107 w, 2068 s, 2054 m, 2023 s, 2008 s, 1983 m, 1976 m, 1746 w (COOEt); (CCl_4), ν/cm^{-1}: 1500 w (NC$\cdots$O); 3630 w (OH); 3427 w (NH); 3407 w (NH assoc.). ^1H NMR ($CDCl_3$, TMS), δ: 7.05 (m, 1 H, NH), 4.54 (m, 1 H, CH); 4.19 (m, 2 H, $O{-}C\underline{H}_2$); 3.72 (m, 2 H, $CH{-}C\underline{H}_2$); 2.09 (br.s, 1 H, OH); 1.25 (t, 3 H, $CH_2{-}C\underline{H}_3$); −14.31 and −14.39 (both s, 1 H, μ-H). Found (%): C, 19.24; H, 1.21; N, 1.09; Os, 55.47. $C_{14}H_{10}NO_{14}Os_3$. Calculated (%): C, 18.98; H, 1.09; N, 1.38; Os, 56.40. The mass spectrum contains a molecular-ion peak with *m/z* 1002 (for ^{192}Os) and peaks corresponding to the loss of 10 CO groups.

The reaction of $HOCH_2CH_2NH_2$ with $Os_3(CO)_{11}(MeCN)$. A mixture of $Os_3(CO)_{11}(MeCN)$ (122.7 mg, 0.133 mmol) and $HOCH_2CH_2NH_2$ (7.5 mg, 2.49 mmol) in 20 mL of CH_2Cl_2 was stirred for 5 h at room temperature. The solvent was evaporated, and the residue was chromatographed on silica gel using benzene as the eluent. Only one intense fraction was isolated. Yield of $(\mu\text{-}H)Os_3(CO)_{10}(\mu\text{-}OCNHCH_2CH_2OH)$ (**2**) was 116.6 mg (93 %). IR (hexane), $\nu CO/cm^{-1}$: 2109 w, 2068 s, 2056 m, 2023 s, 2010 s, 1995 m, 1986 m, 1978 m; (CCl_4) ν/cm^{-1}: 3642 w (OH); 3443 w (NH); 1505 w (NC$\cdots$O). ^1H NMR ($CDCl_3$, TMS), δ: 6.41 (br.s, 1 H, NH); 3.61 (m, 2 H, $O{-}C\underline{H}_2$); 3.16 (m, 2 H, $N{-}C\underline{H}_2$); 1.92 (s, 1 H, OH); −14.32 (s, 1 H, μ-H). Found (%): C, 20.26; H, 1.59; Os, 57.3. $C_{11}H_6NO_{12}Os_3 \cdot 2/3\ C_6H_{14}$. Calculated (%): C, 20.46; H, 1.69; Os, 57.23.

The reaction of claster 3 with Me_3NO. 1.5 mL of an alcoholic solution of $Me_3NO \cdot 2H_2O$ (11 mg, 0.099 mmol) was added dropwise over a period of 1 h to a solution of $(\mu\text{-}H)Os_3(CO)_{10}\{\mu\text{-}OCNHCH(CH_2OH)COOEt\}$ (**3**) (73 mg, 0.0722 mmol) in 8 mL of THF stirred at room temperature under reduced pressure. Then the solution was applied to Silufol plates without evaporation of the solvent and chromatographed using a 3:2:0.5 benzene—hexane—acetone mixture as the eluent. Two- or three-step elution afforded two fractions with close R_f, which were extracted from Silufol with diethyl ether to give 28 mg (37.26 %) of cluster **5a** and 25.4 mg (33.8 %) of cluster **5b**. **5a**: IR (cyclohexane), $\nu CO/cm^{-1}$: 2092 m, 2049 vs, 2015 s, 2002 s, 1994 s, 1979 m, 1970 m, 1926 m, 1748 w (COOEt); (CCl_4), ν/cm^{-1}: 3633 w (OH); 3440 w (NH); 3373 vw (NH assoc.). ^1H NMR (C_6D_6, TMS), δ: 6.92 (d, 1 H, NH); 4.71 (m, 1 H, $C\underline{H}$); 3.85 (q, 2 H, $O{-}C\underline{H}_2{-}CH_2$); 3.80 (m, 2 H, $C\underline{H}_2{-}OH$); 2.30 (s, 9 H, NMe_3); 1.32 (br.s., 1 H, OH); 0.87 (t, 3 H, $O{-}CH_2{-}C\underline{H}_3$); −12.87 (s, 1 H, μ-H). **5b**: IR (cyclohexane), $\nu CO/cm^{-1}$: 2093 m, 2051 vs, 2016 s, 2003 s, 1994 s, 1980 m, 1971 m, 1928 m, 1742 w (COOEt); (CCl_4), ν/cm^{-1}: 3633 w (OH); 3427 w (NH); 3367 vw (NH assoc.). ^1H NMR (C_6D_6, TMS), δ: 6.83 (d, 1 H, NH); 4.45 (m, 1 H, CH); 3.81 (q, 2 H, $O{-}C\underline{H}_2{-}CH_3$); 3.33 (m, 2 H, $C\underline{H}_2{-}OH$); 2.35 (s, 9 H, NMe_3); 0.84 (t, 3 H, $O{-}CH_2{-}C\underline{H}_3$); −12.87 (s, 1 H, μ-H).

Preparation of single diastereomers of $(\mu\text{-}H)Os_3(CO)_{10}\{\mu\text{-}OCNHCH(CH_2OH)COOEt\}$ (3a and 3b). Dry CO was bubbled through benzene solutions of diastereomers **5a** and **5b** (25—30 mg of either of the complexes in 10 mL of benzene) heated to 60 °C for 30 min. Both solutions were concentrated, and the solid residues were chromatographed in the same way as **3a**,**b**. **3a**: IR (cyclohexane), $\nu CO/cm^{-1}$: 2107 w, 2068 s, 2056 s, 2025 s, 2009 s, 1995 m, 1987 m, 1979 m, 1743 w (COOEt). ^1H NMR ($CDCl_3$, TMS) δ: 6.82 (d, 1 H, NH); 4.47 (m, 1 H, CH); 4.17 (q, 2 H, $O{-}C\underline{H}_2{-}CH_3$); 3.68 (m, 2 H, $C\underline{H}_2{-}OH$); 1.42 (br.s, 1 H, OH); 1.25 (t, 3 H, $CH_2{-}C\underline{H}_3$); −14.31 (s, 1 H, μ-H). **3b**: IR (cyclohexane), $\nu CO/cm^{-1}$): 2107 w, 2068 s, 2056 s, 2025 s, 2009 s, 1995 m, 1987 m, 1979 w, 1743 w (COOEt). ^1H NMR ($CDCl_3$, TMS), δ: 6.82 (d, 1 H, NH); 4.53 (m, 1 H, CH); 4.19 (q, 2 H, $O{-}C\underline{H}_2{-}CH_3$); 3.73 (m, 2 H, $C\underline{H}_2{-}OH$); 1.65 (br.s, 1 H, OH); 1.25 (t, 3 H, $O{-}CH_2{-}C\underline{H}_3$); −14.3 (s, 1 H, μ-H).

The reaction of $HOCH_2CH(NH_2)COOEt$ with $(\mu\text{-}H)Os_3(CO)_{10}(\mu\text{-}OH)$ (6). A mixture of cluster **6** (160 mg, 0.184 mmol) and *L*-α-serine ethyl ester (100 mg, 0.756 mmol) was dissolved in 10 mL of THF. 2.5 mL of an ethanolic solution of $Me_3NO \cdot 2H_2O$ (26 mg, 0.234 mmol) was added dropwise to the reaction mixture stirred at room temperature under reduced pressure over a period of 1 h. The resulting solution was applied to Silufol plates without concentration and chromatographed using a 3:2:1 hexane—benzene—acetone mixture as the eluent to give only one intense fraction with R_f 0.3. Yield 91 mg (51.75 %) of a mixture of $(\mu\text{-}H)Os_3(CO)_9\{\mu,\eta^2\text{-}OCH_2CH(COOEt)NH_2\}$ (**8a**,**b**). IR (cyclohexane), $\nu CO/cm^{-1}$: 2100 m, 2055 s, 2017 vs, 1996 s, 1971 m, 1930 m, 1740 w (COOEt); (CCl_4), ν/cm^{-1}: 3341 w (NH); 3284 w (NH). ^1H NMR ($CDCl_3$, TMS), δ: 4.23 and 3.73 (both m, 7 H, $N\underline{H}_2$, $O{-}C\underline{H}_2{-}CH_3$, $-C\underline{H}{-}C\underline{H}_2$); 1.28 (t, 3 H, $O{-}CH_2{-}C\underline{H}_3$); −10.71 (s, 1 H, μ-H). Found (%): C, 23.88; H, 2.47; Os, 47.69. $C_{14}H_{11}NO_{12}Os_3$. Calculated (%): C, 23.40; H, 2.44; Os, 46.86.

The reaction of $HOCH_2CH_2NH_2$ with $(\mu\text{-}H)Os_3(CO)_{10}(\mu\text{-}OH)$ (6). ***a.*** 0.2 mL of an ethanolic solution of $Me_3NO \cdot 2H_2O$ (5.9 mg, 0.053 mol) was added dropwise over a period of 1 h to a solution of cluster **6** (37.2 mg, 0.0428 mmol) and $HOCH_2CH_2NH_2$ (9.5 mg, 0.155 mol) in 5 mL of CH_2Cl_2, and the mixture was held for 2 h at room temperature. According to TLC, the mixture contained only one complex, which completely decomposed during attempts to isolate it by chromatography. IR spectrum of the reaction mixture ($\nu CO/cm^{-1}$): 2090 m, 2052 s, 2006 vs, 1989 m, 1968 sh, 1913 m. When the reaction mixture was held in the solution for a longer period, a new compound slowly formed, according to TLC. In THF this reaction occurs noticeably more quickly.

b. 1 mL of an ethanolic solution of $Me_3NO \cdot 2H_2O$ (12.6 mg, 0.114 mmol) was added dropwise over a period of 1 h to a solution of cluster **6** (39.5 mg, 0.0455 mmol) and $HOCH_2CH_2NH_2$ (7.6 g, 0.125 mmol) in 5 mL of THF. The solution was held for 15 h at room temperature, applied to Silufol plates without evaporation, and chromatographed in a 3:1 hexane—acetone system to give only one intense fraction, $(\mu\text{-}H)Os_3(CO)_9\{\mu,\eta^2\text{-}OCH_2CH_2NH_2\}$ (**10**) (26.6 mg, 66.2 %). IR (CCl_4), ν/cm^{-1}: 2099 m, 2056 s, 2017 vs, 1995 s, 1970 m, 1925 m (νCO); 3354 w, 3306 w (νNH). ^1H NMR (CD_2Cl_2, TMS), δ: 4.06 (br.s, 2 H, NH_2); 3.76 (m, 2 H, $O{-}C\underline{H}_2{-}CH_2$); 2.47 (m, 2 H, $N{-}C\underline{H}_2{-}CH_2$); −11.75 (s, 1 H, μ-H). Found (%): C, 15.19; H, 0.81; Os, 64.91. $C_{11}H_7NO_9Os_3$. Calculated (%): C, 14.93; H, 0.79; Os, 64.57.

c. A solution of cluster **6** (2.98 mg, 0.0343 mmol) and $HOCH_2CH_2NH_2$ (19 mg, 0.0311 mmol) in 8 mL of dioxane was boiled for 5 h and concentrated, and the residue was chromatographed on Silufol using a 3:1:1 hexane—benzene—ether mixture to afford two fractions, $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-}NHCH_2CH_2OH)$ (**11**) (13.75 mg, 43 %) and cluster **10** (11.65 mg, 37.6 %). Cluster **11**: IR (hexane), $\nu CO/cm^{-1}$: 2104 w, 2066 s, 2052 m, 2022 s, 2009 s, 1991 s, 1977 m; (CH_2Cl_2), ν/cm^{-1}: 3614 w (OH); 3360 w (NH). ^1H NMR ($CDCl_3$, TMS), δ: 4.93 (br.s, 1 H, NH); 3.71 (m, 2 H, $O-C\underline{H}_2-CH_2$); 2.95 (m, 2 H, $N-C\underline{H}_2-CH_2$); 1.55 (br.s, 1 H, OH); −14.93 (d, 1 H, μ-H). The mass spectrum exhibits a molecular ion peak with *m/z* 917 corresponding to $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-}NHCH_2CH_2OH)$, and peaks corresponding to the loss of 10 CO groups.

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Received August 2, 1993