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Syntheses and crystal structure of bridged Os_3-clusters with chiral 3-carane-type ligands

Vladimir A. Maksakov [a], Vladimir P. Kirin [a], Pavel A. Petukhov [b], Tatjana V. Rybalova [b], Yuri V. Gatilov [b], Alexey V. Tkachev [b],*

[a] *Institute of Inorganic Chemistry, Novosibirsk 630090, Russia*
[b] *Novosibirsk Institute of Organic Chemistry, Novosibirsk 630090, Russia*

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Abstract

Reaction of $(\mu\text{-H})_2Os_3(CO)_{10}$ cluster with (1*S*,3*S*,6*R*)-3-dimethylaminocarane-4-one *E*-oxime results in loss of dimethylamino group and formation of $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-ON=C<})$ cluster (43%). Treatment of cluster $Os_3(CO)_{11}(N{\equiv}CCH_3)$ with (1*S*,3*S*,6*R*)-3-mercaptocaran-4-one *E*-oxime gives cluster $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-S}\text{—}\cdots)$ in 86%, whose structure was proved by X-ray single crystal analysis. Decarbonylation of the later compound with $(CH_3)_3NO$ leads to a diastereomeric mixture of $(\mu\text{-H})Os_3(CO)_9(\mu,\eta^2\text{-}(S,N)\text{—}\cdots)$ clusters in 64% yield. © 2000 Published by Elsevier Science S.A. All rights reserved.

Keywords: Os_3 clusters; Osmium carbonyl clusters; Chiral ligand; Terpenoid

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1. Introduction

The main idea of the use of molecular clusters in catalysis is based on possibility of coordination of a organic molecule with several catalytic centers simultaneously by analogy with some metal-containing enzymes [1]. The distinctive feature of the metal cluster carbonyl complexes is strong interligand interactions, especially in cases of coordination of bulky polyfunctional organic molecules on several metal atoms of the same cluster. There are some examples of this effect that result in restriction of rotation around the single bond as in the cases of bridging coordination of amino acid esters with carbonyl cluster of Os [2] and stereospecific coordination of L-proline and L-oxyproline esters on triruthenium and triosmium clusters, respectively [3,4]. From this point of view different chiral terpene-based derivatives are of interest as potential ligands, many of them are easily prepared from widespread natural terpenoids. Cluster complexes with the ligands of this type have not been studied. In the present work we wish to report the reactions of cluster $(\mu\text{-H})_2Os_3(CO)_{10}$ (**1**) and $Os_3(CO)_{11}(NCCH_3)$ (**5**) with bifunctional carane-type derivatives-(1*S*,3*S*,6*R*)-3-dimethylaminocarane-4-one *E*-oxime (**2**) and (1*S*,3*S*,6*R*)-3-mercapthocaran-4-one *E*-oxime (**6**).

Clusters **1** and **5** possess high reactivity towards Lewis bases [5], and the reactions of Os_3-clusters with simple thiols, tertiary amines and oximes are known [5–7]. When treated with cluster **1**, simple oximes form chiral complexes due to μ-coordination by O and N atoms [7].

2. Results and discussion

The reaction of $(\mu\text{-H})_2Os_3(CO)_{10}$ with amino oxime **2** proceeds much more slowly as compared to the reactions of simple oximes. Spectral data of the main product **4** differ significantly from the data for the known Os_3 clusters with the $\mu,\eta^2\text{-}(O,N)$-coordinated oxime molecules [7]. At the same time, the set of bands in the region 1900–2150 cm^{-1} of the IR spectrum and chemical shift of the μ-H-ligand (−12.75 ppm) in ^{1}H-NMR spectrum of the resulting product **4** are typical for the clusters of the type $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-OR})$ [8]. ^{1}H-NMR spectrum of compound **4** (Table 1) is

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* Corresponding author.
E-mail address: atkachev@nioch.nsc.ru (A.V. Tkachev).

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practically identical to that of unsaturated oxime **3** [9]. ^{13}C-NMR spectrum of compound **4** (Table 2) is also very similar to the spectra of oxime **3** [9], except for the low-field region (169–182 ppm) having additional signals of 10 carbonyl carbons (total of five signals with relative intensities 1:1:3:2:3). Compound **4** might be formally regarded as a cluster, containing bridging α,β-unsaturated oxime **3**. The later compound is usually formed from amino oxime **2** on heating (> 90°C). However, the reaction of cluster $(\mu\text{-H})_2Os_3(CO)_{10}$ with the unsaturated oxime **3** under the same reaction conditions led only to decomposition products of the starting cluster due to the prolonged heating. Thus, the formation of complex **4** seems to proceed by elimination of dimethylamino group from an unstable intermediate complex of **1** with **2**. Cluster **4** is also quite unstable and slowly decomposed in a solution to form the known cluster $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-OH})$ [10] in 80% yield.

Reaction of mercaptho oxime **6** with cluster $Os_3(CO)_{11}(NCMe)$ proceeds smoothly at room temperature and gives a single crystalline orange coloured product **7** in 85% yield. The IR spectrum of the complex in the range of 2200–1800 cm^{-1} is similar to the spectra of clusters $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-SR})$ with symmetric coordination of bridged sulfur-containing groups [11]. The IR spectrum demonstrates also the presence of the oxime hydroxyl (3590 cm^{-1}) and the absence of the bond S–H. ^{1}H- and ^{13}C-NMR spectra of the complex

Table 1
^{1}H-NMR data for compounds **4**, **7** and **8**

	δH^i (ppm) (J, ± 0.1 Hz)			
i	**4**	**7** [a]	**8a** (major)	**8b** (minor)
1	1.24–1.06 m	0.94 ddd	0.90 ddd (9.7, 8.9, 6.2)	0.79 ddd (9.9, 8.9, 4.9)
2	6.07 d (5.3)	Hα: 2.06 dd	Hα: 2.47 dd (16.9, 9.7)	Hα: 3.03 dd (16.2, 9.9)
		Hβ: 1.65 dd	Hβ: 1.88 dd (16.9, 6.2)	Hβ: 2.18 dd (16.2, 4.9)
5	Hα: 2.89 d (19.5)	Hα: 2.67 dd	Hα: 3.55 dd (15.5, 8.3)	Hα: 3.41 dd (13.6, 8.8)
	Hβ: 2.61 dd (19.5, 8.0)	Hβ: 2.72 dd	Hβ: 1.42 dd (15.7, 7.6)	Hβ: 1.43 dd (13.6, 7.6)
6	1.24–1.06 m	1.04 ddd	0.74 ddd (8.9, 8.3, 7.6)	1.26 ddd (8.9, 8.8, 7.6)
8	0.77 s	0.77 s	0.97 s [b]	1.01 s [b]
9	1.07 s	1.05 s	1.01 s [b]	1.07 s [b]
10	1.83 br.s ($W_{1/2}$ = 4 Hz)	0.85 s	1.54 s	1.36 s
=NOH		8.35 s	6.73 br s ($W_{1/2}$ = 15 Hz)	6.80 br s ($W_{1/2}$ = 20 Hz)
μ-H	−12.76 s	−17.98 s	−15.65	−15.46

[a] Calculated couplings obtained by the iteration routine (± 0.3 Hz): $J_{H1-H6} = -9.0$, $J_{H1-H2\alpha} = 8.5$, $J_{H1-H2\beta} = 5.0$, $J_{H2\alpha-H2\beta} = -15.0$, $J_{H6-H5\alpha} = 7.0$, $J_{H6-H5\beta} = 1.5$, $J_{H5\alpha-H5\beta} = -16.0$.

[b] Assignments may have to be interchanged.

Table 2
^{13}C-NMR data for compounds **4**, **7** and **8**

	δC^i (ppm)			
i	**4**	**7**	**8a** (major)	**8b** (minor)
1	23.32	18.19 [a]	20.51	19.60
2	131.61	35.19	36.12	42.07
3	127.53	54.02	63.93	62.97
4	155.14	159.97	172.28 [b]	172.16 [b]
5	18.47	18.29	20.25	22.15
6	20.59	19.05 [a]	22.83	27.57
7	23.38	19.31	21.95	21.61
8	13.54	14.70	14.65	15.05
9	27.56	27.65	27.71	27.87
10	17.98	22.81	31.76	29.98
$Os_3(CO)_n$	182.08 (1C), 180.19 (1C), 176.17 (3C), 172.61 (2C), 169.88 (3C)	179.67 (2C), 175.16 (1C), 174.97 (1C), 173.59 (2C), 172.87 (1C), 171.72 (1C), 169.03 (1C), 168.53 (1C)	186.64 (1C), 181.60 (1C), 180.19 (1C), 175.88 (1C), 175.30 (1C), 174.93 (1C), 172.84 (1C), 172.73 (1C), 172.51 (1C)	186.87 (1C), 181.62 (1C), 180.28 (1C), 179.76 (1C), 175.53 (1C), 174.86 (1C), 174.21 (1C), 173.33 (1C), 172.87 (1C)

[a] Assignments may have to be interchanged.

[b] The signals can not be distinguished from those of carbonyl carbons.

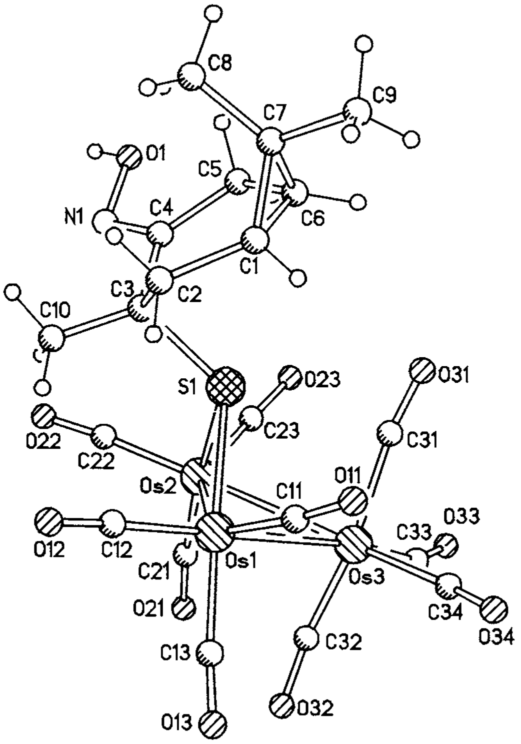
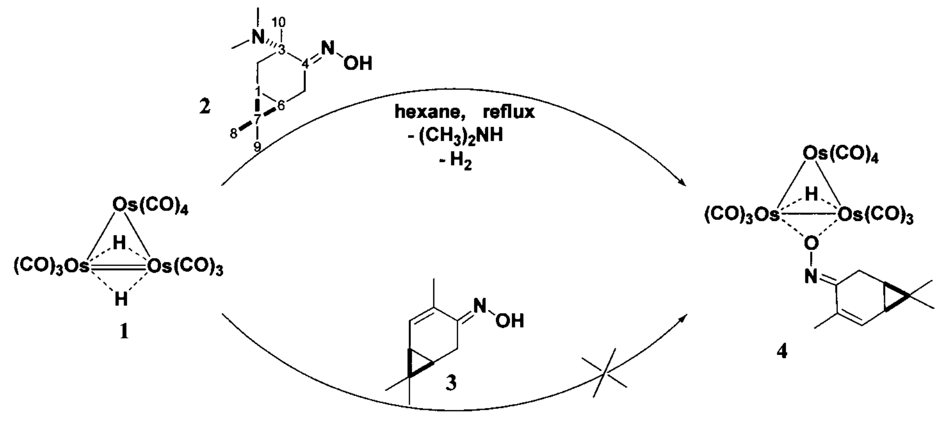
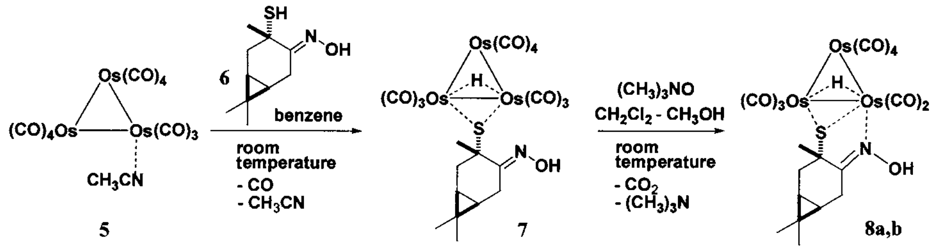


Fig. 1. Perspective view of one of the two crystallographically independent molecules **4** according to the X-ray analysis. The bridging hydride ligand (μ-H) is not shown. Selected bond lengths (Å) and angles (°): Os1–Os2 2.876(2), 2.871(3); Os1–Os3 2.871(2), 2.853(3); Os2–Os3 2.871(2), 2.867(2); Os1–S1 2.401(8), 2.390(7); Os2–S1 2.402(7), 2.385(8); Os3–Os1–S1 78.9(2), 79.3(2); Os3–Os2–S1 78.9(2), 79.1(2); Os1–S1–C3 117.5(9), 118.3(11); Os2–S1–C3 118.5(8), 119.4(13).

are shown in Tables 1 and 2, respectively. Structure of the new cluster **7** was established by X-ray analysis (Fig. 1, Scheme 1). Bond distances and bond angles for the organic ligands of two independent molecules are very close to the usual values. The first molecule has the conformation of the six-membered carbocycle, that can be characterized as the envelope-like form distorted towards boat. In this form, atoms C3 and C4 are out of the plane C2–C1–C6–C5 by 0.82(5) and 0.27(5) Å, respectively. The second molecule has the envelope-like conformation of the carbocycle, atom C3 being out of the plane C2–C1–C6–C5–C4 by 0.61(6) Å. The same difference for a pair of independent molecules was found in the crystals of 3-chloro-4-caranone *E*-oxime [9], whereas both independent molecules of 3-dimethylamino oxime **2** have the same envelope-like conformation [12]. Each pair of the independent molecules of the cluster **7** is joined into H-dimers by the hydrogen bonds O1-H···N1A $(-1+x,\ 1+y,\ z)$ 2.86(3) Å and N1···H-O1A $(-1+x,\ 1+y,\ z)$ 2.80(4) Å. There are no other intermolecular interactions in the crystal. Three atoms Os form a triangle with usual bond lengths Os–Os (2.853–2.876 Å), this interval is characteristic for a number of sulfur-containing clusters $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-SR})$: R = $CH_2S\{Os_3(\mu\text{-H})(CO)_{10}\}$ [13], $CHPh_2$ [14], Et [15], $(CH_2)_3S\{Os_3(\mu\text{-H})(CO)_{10}\}$ [16], CH=NC$_6$H$_4$-*p*-F [11], $CH(CH_2)_2N$ [17]. There is a slight difference between the bridged and unbridged Os–Os edges in the cluster **7**: the average length of the bridged Os–Os edge is 2.873(2) Å whereas the average length of the unbridged Os–Os edge is 2.865(7) Å. For 20 clusters of the type $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-SR})$ taken from the Cambridge Structural Database, the average lengths of the bridged and unbridged Os–Os edges are 2.861(12) and 2.854(13) Å, respectively. The bonds Os–S (2.385–2.402 Å) have also normal length as compared to the clusters listed above. Coordination of bulky organic ligand to two atoms Os results in steric strain and causes close contacts (3.29–3.35 Å) of methyl group C10 and two carbonyls C11 and C22 (normal contact is 3.42 Å [18]). As a result, the dihedral angle between the planes Os_3 and Os_2S as well as bond angles Os–S–C are larger than those usually observed. Thus, the dihedral angle is 81.1° {69.9° for the unstrained cluster $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-SH})$ [19]}. According to the Cambridge Structural Database [20], the maximum value of 82.2° was found for the complex $\{(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-SCH_2-})\}_2$ [21]. The bond angles Os1, 2-S1–C3 in the molecule **7** are increased to



Scheme 1.



Scheme 2.

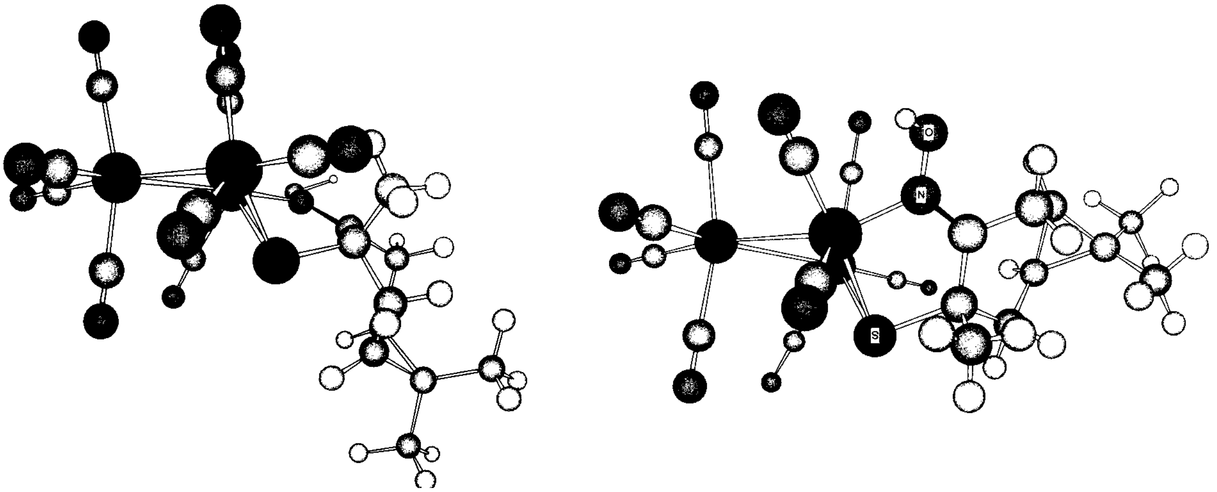


Fig. 2. Perspective view of a pair of diastereomeric clusters **8** having additional coordination of the oxime nitrogen with Os1 (left) and Os2 (right).

117° {103° for $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-}\eta^1\text{-SCH=NC}_6H_4\text{-}p\text{-F})$} [11].

Complex **7** contains a free oxime group that is also capable for coordination at the second step. This type of reaction has been observed when the cluster $(\mu\text{-H})Os_3(CO)_{10}\{\mu\text{-SCH}_2CH(CO_2Et)NH_2\}$ was treated with Me_3NO to give diastereomeric clusters $(\mu\text{-H})Os_3(CO)_9\{\mu,\eta^2\text{-}(S,N)\text{-SCH}_2CH(CO_2Et)NH_2\}$ [22,23]. Addition of a methanolic solution of Me_3NO to a solution of complex **7** in CH_2Cl_2 results in the fast formation of two new compounds **8a**,**b** in the ratio 3:1. The stereoisomeric complexes cannot be separated by chromatography, although careful crystallization of the mixture from 2:1 hexane–CH_2Cl_2 allowed the major component **8a** to be obtained. IR spectra of the diastereomeric complexes are similar to those reported for Os_3 clusters with μ,η^2-coordination of cysteine [22]. A relatively small shift of the band of the hydroxyl (3550 cm^{-1}) as compared to the starting cluster **7** (3590 cm^{-1}) lends support to the additional coordination by the nitrogen atom of the oxime rather than by the oxygen atom. ^1H- and ^{13}C-NMR spectra of the diastereomeric complexes **8a**,**b** are shown in Tables 1 and 2. There are considerable low-field shifts of the hydride hydrogen ($\Delta\delta H = 2.3$–2.5 ppm) in complexes **8a**,**b** as compared to the starting cluster **7**. The shift of this type is a typical result of the substitution of one of the carbonyls in the Os_3 clusters by a ligand [24,25]. Unusual up-field position of the oxime hydrogen (6.7–6.8 ppm) also proves the additional coordination to the oxime nitrogen. Values of the proton-proton couplings for diastereomers **8a**,**b** (Table 1) reinforce the additional coordination and indicates that both isomers have the same partially distorted boat-like conformation of the six-membered carbocycle (pairs of the couplings $J_{H1-H2\alpha,H2\beta}$ and $J_{H6-H5\alpha,H5\beta}$ are similar but not the same values) in contrast to the mononuclear η^2-N,N'-complex of dimethylamino oxime **2** with $PtCl_2$, having practically undistorted boat-like conformation of the carbocycle (pairs of the couplings $J_{H1-H2\alpha,H2\beta}$ and $J_{H6-H5\alpha,H5\beta}$ have the same values) [26]. Diastereomers **8a** and **8b** have quite different proton and carbon chemical shifts indicating different orientation of the C_{10}-terpenic moiety relative to the $Os_3(CO)_9$ fragment. Molecular models of two diastereomers **8a** and **8b** (additional coordination at Os1 or Os2 as compared to Fig. 1(Scheme 2)) are shown on Fig. 2. The models are optimized by molecular mechanics to bring the geometry of the fragment $(\mu\text{-H})Os_3(CO)_9(\mu,\eta^2L)$ to conformity with the known examples [27]. Unfortunately, in spite of significant distinctions in chemical shifts, we are not able to assign pair of diastereomers **8a**,**b** because it is impossible to take proper account of the anisotropy of all the neighboring groups.

Clusters **8a,b** are unstable and slowly decomposed in the solid state and rather quickly in a solution.

3. Experimental

3.1. General experimental procedures

All reactions and isolations of cluster complexes were carried out under inert atmosphere (argon). All the solvents used were freshly distilled. Commercial reagents were used without additional purification. Analytical and preparative thin layer chromatography (TLC) were performed on ready-made plates Silufol® (SILPEARL on aluminum foil, Czechoslovakia). IR spectra were obtained using a Specord IR-75 and Bruker IFS-66 spectrometers. Melting points were measured on a Kofler melting point apparatus. NMR spectra were recorded at room temperature on a Bruker AM 400 instrument (^{1}H 400.13 MHz, ^{13}C 100.61 MHz) for solutions in $CDCl_3$ ($4 \div 5 \times 10^{-2}$M) using the solvent signal as internal standard: δ_H 7.24 ppm and δ_C 76.90 ppm. A Palomat A (578 nm) and a Perkin-Elmer 241 (589 nm) polarimeters were used to measure optical rotation.

Starting cluster complexes were prepared by literature methods: $(\mu\text{-H})_2Os_3(CO)_{10}$ [28], $Os_3(CO)_{11}(NCMe)$ [29]. Starting monoterpenic derivatives were synthesized from demeric nitrosochloride of natural (+)-3-carene as described before: (1*S*,3*S*,6*R*)-3-*N*,*N*-Dimethylaminocaran-4-one *E*-oxime (**2**) with m.p. 118–119°C (MeCN) and $[\alpha]^{25}_{578}$ +105 (c 12.1, $CHCl_3$) [30], (1*S*,6*R*)-car-2-en-4-one *E*-oxime (**3**) with m.p. 97–98°C and $[\alpha]^{25}_{578}$ +361 (c 3.14, $CHCl_3$) [9], and (1*S*,3*S*,6*R*)-3-mercaptocaran-4-one *E*-oxime (**6**) with M.p. 67–69°C (MeCN) $[\alpha]^{30}_{578}$ +213 (c 3.08, $CHCl_3$) [31].

3.1.1. $(\mu\text{-}H)Os_3(CO)_{10}(\mu\text{-}ON{=}CCMe{=}CHCHCMe_2CHCH_2H_2)$ (**4**)

A mixture of $H_2Os_3(CO)_{10}$ (**1**) (0.24 g, 0.28 mmol) and dimethylamino oxime **2** (0.067 g, 0.32 mmol) in hexane (20 ml) was heated at reflux for 20 h. The solvent was removed at reduced pressure and the residue was chromatographed using a 2:1 mixture of hexane and benzene (v/v) as eluent. The intensive yellow fraction with $R_f = 0.5$ was extracted with diethyl ether to give 0.124 g of the title compound (43% calculating on the starting cluster) as yellow solid. $[\alpha]^{21}_{578}$ +24 (c 0.98, CCl_4–C_6D_6 4:1, v/v). IR (hexane, ν_{CO}, cm^{-1}): 2108 w, 2075 s, 2060 m, 2026 s, 2000 m, 1988 m, 1982 m, 1959 w.

3.1.2 $(\mu\text{-}H)Os_3(CO)_{10}$-$(\mu\text{-}S\text{-}CMeCH_2CHCMe_2CHCH_2C{=}NOH)$ (**7**)

A solution of $Os_3(CO)_{11}(NCMe)$ (**5**) (0.17 g, 0.19 mmol) and mercaptho–oxime **6** (0.039 g, 0.20 mmol) in benzene (20 ml) was stirred at room temperature (r.t.) for 3 h. The reaction mixture was concentrated at reduced pressure to the volume of ca. 10 ml and chromatographed using a 3:1 mixture of hexane and benzene (v/v) as eluent. The yellow fraction with $R_f =$ 0.25 was extracted with diethyl ether to give the title compound (0.166 g, 86% calculating on the starting cluster) as a yellow solid. Anal. Calc. for $C_{20}H_{17}O_{11}Os_3NS$: C, 22.87; H, 1.62; S, 3.04; Os, 54.36. Found: C, 23.1; H, 1.7; S, 2.9; Os, 54.7. $[M]^{22}_D$ +220.4 (*c* 0.76, CH_2Cl_2); MS, *m*/*z*: 1055 ($Os_3C_{20}NO_{11}SH_{17}$, ^{192}Os, M^+). IR (hexane, ν_{CO}, cm^{-1}): 2108 m, 2067 vs, 2059 s, 2024 s, 2020 s, 2000 s, 1989 m, 1984 m. IR (CCl_4, $\nu_{O–H}$, cm^{-1}): 3590 w.

3.1.3. $(\mu\text{-}H)Os_3(CO)_9(\mu\eta^2\text{-}(S,N)\text{-}S\text{-}CMeCH_2CHCMe_2CHCH_2C{=}NOH)$ (**8a + 8b**)

A solution of $Me_3NO \cdot 2H_2O$ (0.007 g, 0.07 mmol) in MeOH (4 ml) was added dropwise to a solution of complex **7** (0.06 g, 0.07 mmol) in CH_2Cl_2 (20 ml) and the resulting solution was stirred at r.t. for 0.5 h. The solvent was distilled off and the residue was chromatographed in 1:1 heptane–benzene (v/v). The intensively coloured yellow fraction with $R_f = 0.31$ was extracted with diethyl ether to give the title diastereomeric mixture (0.037 g, 64%) as yellow solid. $[\alpha]^{24}_{578}$ −5.2 (c 2.69, 1:1 $CHCl_3$–CCl_4, v/v). MS, *m*/*z*: 1027 ($Os_3C_{19}NO_{10}SH_{17}$, ^{192}Os). IR (hexane, ν_{CO}, cm^{-1}): 2094 m, 2055 s, 2017 vs, 1998 s, 1982 sh, 1977 m, 1936 m (hexane, ν_{CO}). IR (CCl_4, $\nu_{O–H}$, cm^{-1}): 3560 w.

3.2. X-ray crystallographic experiment

Intensity data for compound **7** were collected at 293 K on a Syntex-$P2_1$ diffractometer using graphite monochromated Cu–K_α radiation ($\lambda = 1.54178$ Å). After absorption correction, the structure was solved using SHELXS-86, refinement was carried out by full-matrix-least-squares procedure using SHELXL-93 (carbonyl oxygens treated isotropically and H atoms not located). The structure of the compound is shown on Fig. 1. Crystal data and structure refinement: θ range for data collection: from 2.02 to 60.01°. Index ranges: $0 \le h \le 8$, $0 \le k \le 18$, $0 \le l \le 49$. Reflections collected: 4943. Independent reflections: 4678 [$R_{int} =$ 0.0236]. Max and min transmission: 0.432 and 0.094. Data/restraints/parameters: 4676/0/549. Goodness-of-fit on F^2: 1.019. Final *R* indices [$I > 2\sigma(I)$]: $R_1 = 0.0553$, $wR_2 = 0.1163$. *R* indices (all data): $R_1 = 0.0820$, $wR_2 =$ 0.1367. Absolute structure parameter: −0.03(4). Extinction coefficient: 0.000025(5). Largest different peak and hole: 0.96 and −1.00 e $Å^{-3}$. Crystal size: $0.80 \times 0.05 \times 0.04$ mm^3, $C_{20}H_{17}NO_{11}Os_3S$, $M = 1050.01$, orthorhombic, space group $P2_12_12_1$, $a = 7.850(3)$,

$b = 16.178(8)$, $c = 43.74(2)$ Å, $V = 5555(4)$ Å^3, $Z = 8$, $D_{calc} = 2.511$ g cm^{-3}, $\mu = 26.541$ mm^{-1}, $F(000) = 3808$.

4. Supplementary material

Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 136461 for compound **7**. Copies of this information can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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