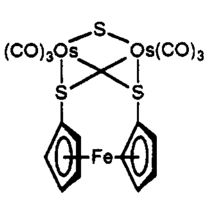

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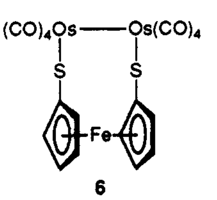

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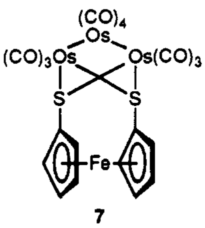

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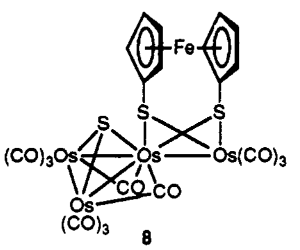

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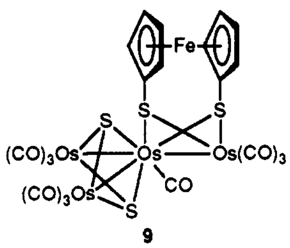

9

angle. The Os–S distances (average 2.354 Å) are similar to the average values of 2.405 and 2.390 Å found respectively in the open cluster compound $Os(\mu_3\text{-}S)_2(CO)_9$[16] and the closed cluster compound $Os_3(\mu\text{-}S)(\mu\text{-}H)_2(CO)_9$.[17] The Os–S–Os angles are faily acute; however, they lie in the range 65–75° reported for M–S–M angles in cluster compounds.[18]

The two cyclopentadienyl rings are eclipsed and nearly parallel, the dihedral angle between the two rings being only 5.91°. This small tilt is close to that observed in the ruthenium complex 4 (5.38°). The rings are virtually planar, while the two sulfur atoms (S(2) and S(3)) are displaced outward from the Cp ring planes (average 0.150 Å) again as in 4.

The semibridging carbonyls of 8 may well become more symmetrical in solution, because a strong absorption is seen in the bridging carbonyl region of the infrared spectrum of this compound.

Compound 9 proved to be very difficult to purify, and good microanalytical data were not obtained. The structure shown is based on strong spectroscopic evidence. First, the ^1H NMR spectrum is very similar to that of 8, showing four equal-intensity multiplets. Second, the mass spectrum indicates that 9 has one less CO group and one more sulfur atom than 8. The mass spectra of 8 and 9 show $Os_4(S)Fe(C_5H_4S)_2^+$ and $Os_4(S)_2Fe(C_5H_4S)_2^+$ ions after the loss of 11 and 10 carbonyls, respectively, and the ion $Os_4(CO)_8^+$ is present in the spectra of both 8 and 9. These data indicate that 8 and 9 are related as indicated.

Similar problems with purity were encountered with 5 and 7. The structures suggested for these two molecules seem reasonable on the basis of the spectroscopic data, particularly the mass spectrum and the ^1H NMR spectra, which indicate a symmetrical structure. The mass spectrum of 5 shows stepwise loss of six CO groups and one sulfur atom. The resulting ion $Fe(C_5H_4S)_2Os_2^+$ is also seen in the mass spectrum of 6, where the parent ion corresponds to the formulation $Fe(C_5H_4S)_2Os_2(CO)_8$. Microanalytical data for 6 support this, and a possible structure is as indicated.

The reaction of 1 with $Mn_2(CO)_{10}$ and $Co_2(CO)_8$ under mild conditions affords intractable solids. Similar results were obtained when ((dimethylamino)methyl)-1,2,3-trithia[3]ferrocenophane was reacted with a variety of transition-metal complexes such as $Ru_3(CO)_{12}$ and $Pd(COD)Cl_2$.[19]

Acknowledgment. We thank the Natural Sciences and Engineering Research Council of Canada and NATO for financial support.

Supplementary Material Available: Tables of bond lengths and angles, hydrogen atom parameters, anisotropic thermal parameters, torsion angles, intermolecular contacts, and least-squares planes for 4 and 8 (29 pages). Ordering information is given on any current masthead page.

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(16) Adams, R. D.; Horvath, I.; Segmuller, B. E.; Yang, L.-W. *Organometallics* **1983**, *2*, 145.

(17) Johnson, B. F. G.; Lewis, J.; Pippard, D.; Raithby, P. R.; Sheldrick, G. M.; Rouse, D. K. *J. Chem. Soc., Dalton Trans.* **1979**, 616.

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(19) Talaba, A. M.Sc. Thesis, University of British Columbia, 1991.

Additions and Corrections

Frank Bottomley* and Selami Karslioglu: Organometallic Oxides: Preparation and Properties of the Diamagnetic Trinuclear Cluster $\{[(\eta\text{-}C_5Me_5)Nb(\mu\text{-}Cl)(\mu\text{-}O)]_3\}^+$ and Related Chloride–Oxides of Niobium. **1992**, *11*, 326.

The crystal structure of $\{2^+\}_2\{Zn_6Cl_{14}^{2-}\}$ can be described in the monoclinic space group $C2/m$, with cell dimensions a = 23.592 (4) Å, b = 17.806 (2) Å, c = 11.117 (2) Å, and β = 105.97 (1)°. The cell is related to the triclinic $P\bar{1}$ used in the refinement by the transformation matrix −1,1,1, 0,−1,1, 100. The structure has been rerefined in $C2/m$ using new data (by Dr. Paul D. Boyle). The disorder in the $Zn_6Cl_{14}^{2-}$ anions and $\eta\text{-}C_5Me_5$ rings which was reported using $P\bar{1}$ remains in $C2/m$. The distances and angles in the $[Nb(\mu\text{-}Cl)(\mu\text{-}O)]_3$ core are identical within experimental error to those reported using $P\bar{1}$. Therefore, there is no change in the discussion or conclusions of the paper. Complete details of the $C2/m$ refinement are available as supplementary material.

Supplementary Material Available: Tables of crystal and refinement data, positional and thermal parameters, and bond distances and angles for the refinement in space group $C2/m$ and a table comparing dimensions of $\{2^+\}_2\{Zn_6Cl_{14}^{2-}\}$ in $P\bar{1}$ and $C2/m$ (7 pages). Ordering information is given on any current masthead page.