ν_{CO} are found[8] at 1915 and 1860 cm^{-1}.

The Ph_4P^+ salt of I also is obtained by a *remarkable*, $Mo(CO)_3$-induced rearrangement reaction. This reaction takes place upon heating (to ca. 60 °C for 20 min) a CH_3CN solution of $(CH_3CN)_3Mo(CO)_3$ and $(Ph_4P)_2(Fe_4S_4Cl_4)$[10] in a 4:3 molar ratio. After unexceptional workup, crystals of $(Ph_4P)_4[Fe_6S_6Cl_6(Mo(CO)_3)_2]$ (II), can be obtained in ~40% yield (based on $(Ph_4P)_2(Fe_3S_4Cl_4)$). The spectroscopic properties of II are identical with those of I.

Oxidation of II with 1 equiv of $(Cp)_2Fe^+$ in CH_2Cl_2 solution affords, after addition of ether, the crystalline $(Ph_4P)_3[Fe_6S_6Cl_6(Mo(CO)_3)_2]$ complex III. The IR spectrum of III contains two sharp CO vibrations (ν_{CO}, 1929, 1883 cm^{-1}) and in frozen CH_3CN solution (9 K) shows an EPR spectrum (g_x = 2.064). The EPR spectrum of III is very similar to the one observed for the $S = {}^1/_2$ $(Fe_6S_6Cl_6)^{3-}$ anion[9b] and suggests that III also is characterized by a $S = {}^1/_2$ ground state. The cyclic voltammetry traces of II and III in CH_3CN solution are identical and show reversible waves at −0.05 and −0.55 V (vs. SCE) that correspond to the 3−/4− and 4−/5− couples. The 3−/4− reduction for III occurs at nearly 350 mV more positive potential than the corresponding reduction for the $[Fe_6S_6(p\text{-}CH_3C_6H_4O)_6(Mo(CO)_3)_2]^{3-}$ trianion (−0.39 V). A similar trend was observed previously in the electrochemistry of the $[Fe_6S_6(L)_6]^{n-}$ prismanes.[9]

Single crystals of I and II were obtained by the slow diffusion of ether to CH_3CN solutions of these compounds. The crystal structures[11] of the centrosymmetric tetraanions in both I and II show virtually identical $[Fe_6Mo_2S_6]^{2+}$ cores (Figure 1). Selected structural parameters for these anions are compared (Table I) to corresponding parameters reported previously[9] for the $[Fe_6S_6]^{3+}$ and $[Fe_6S_6]^{4+}$ cores in the $[Fe_6S_6Cl_6]^{n-}$ clusters. Coordination of the $Mo(CO)_3$ units to the $[Fe_6S_6]$ central cage results in an elongation of the latter along the idealized $\bar{3}$ axis. Slight, but consistent, changes are apparent in the lengthening of the Fe–S bonds, the shortening of the Fe–Fe distances, and the concomitant closing of the Fe–S–Fe angles (Table I).

The $Mo(CO)_3$ fragment resembles the analogous unit in the 1,4,7-trithiacyclononane $Mo(CO)_3$ complex.[12] The Mo–C bond (1.95 (15) Å) and the IR spectrum (ν_{CO} absorptions at 1915 and 1783 cm^{-1}) in the latter are similar to those in I and II.

As stated previously,[8] the $[Fe_6Mo_2S_6]$ core cannot be considered as a satisfactory structural analogue for the core in the Fe/Mo/S center in nitrogenase. However, derivative clusters that contain the $[Fe_6MoS_6]$ and $[Fe_7MoS_6]$ units will satisfy a number of the necessary structural and stoichiometric requirements. The suggestion that a pentlandite-type $[Fe_7MoS_6]$ unit may be an appropriate model for the Fe/Mo/S center in nitrogenase has been advanced previously.[13] Presently we attempt to synthesize such units and are exploring the organometallic and coordination chemistry of the $[Fe_6S_6L_6]^{n-}$ "ligands".

Acknowledgment. This research was supported by a grant from the National Institutes of Health (GM-26671). We are indebted to Professor R. Sands for the preliminary EPR measurements.

Supplementary Material Available: Tables of structure factors and positional and thermal parameters (44 pages). Ordering information is given on any current masthead page.

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(11) Crystal and refinement data for $(Et_4N)_4Fe_6S_6Cl_6(Mo(CO)_3)_2 \cdot 2CH_3CN$ (I). a = 13.192 (2) Å, b = 16.504 (5) Å, c = 16.080 (4) Å, β = 97.97 (2)°; space group $P2_1/n$, Z = 2; d_{calcd} = 1.64 g/cm^3, d_{obsd} = 1.63 g/cm^3, μ = 19.7 cm^{-1}, $2\theta_{max}$ = 45° (Mo, $\lambda(K_\alpha)$, 0.71069 Å); unique reflections 4550, used in refinement $F_o^2 > 3\sigma(F_o^2)$, 3286; parameters 295. Final R = 0.046. For $(Ph_4P)_4Fe_6S_6Cl_6(Mo(CO)_3)_2 \cdot 2CH_3CN$ (II). a = 16.329 (6) Å, b = 16.253 (6) Å, c = 20.588 (5) Å, β = 94.97°; space group $P2_1/n$, Z = 2; d_{calcd} = 1.54 g/cm^3, d_{obsd} = 1.55 g/cm^3, μ = 13.2 cm^{-1}, $2\theta_{max}$ = 45° (Mo, $\lambda(K_\alpha)$, 0.71069 Å); unique relections 7167, used in refinement, $F_o^2 > 3\sigma(F_o^2)$, 5128; parameters 357. Final R = 0.06.

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$Os_4(CO)_{14}(PMe_3)$: An Irregular, Planar Cluster Showing Remarkable Nonrigidity

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Received August 5, 1985

One of the intriguing problems of current inorganic chemistry is the description of the bonding in transition-metal carbonyl clusters.[1,2] The polyhedral skeletal electron pair (PSEP) theory has had considerable success in rationalizing the many geometries found for these compounds.[1] However, Evans and Mingos have pointed out that PSEP theory is often unsuccessful in accounting for the structures of clusters with $M(CO)_4$ units and have extended the theory to encompass clusters with nonconical fragments.[3] Here we describe the synthesis and structure of $Os_4(CO)_{14}(PMe_3)$ that contains one $Os(CO)_3(PMe_3)$ and two $Os(CO)_4$ units. The asymmetry present in the structure is unexpected from PSEP theory or the extension of Evans and Mingos. We believe it is best rationalized in terms of three-center-two-electron metal–metal bonds. The compound also exhibits remarkable fluxional character in solution that may be attributable to its unusual bonding.

To a solution of $(Me_3P)(OC)_4OsOs_3(CO)_{11}$[4] in CH_2Cl_2 a solution of Me_3NO in MeOH was added dropwise until the absorption in the infrared spectrum at 2085 cm^{-1} had just disappeared. The resultant solution was filtered and evaporated to dryness. The product, $Os_4(CO)_{14}(PMe_3)$ (**1**), was recrystallized from hot hexane as air-stable, dark red crystals.[5] The structure of the compound[6] (Figure 1) consists of an almost planar Os_4 skeleton (the dihedral angle between the planes Os(1)–Os(2)–Os(3) and Os(1)–Os(3)–Os(4) is 177.10 (3)°). The periphery of the metal framework consists of two adjacent short Os–Os bonds (Os(1)–Os(2) = 2.779 (2) Å; Os(1)–Os(4) = 2.784 (2) Å) and two long Os–Os bonds (Os(2)–Os(3) = 3.013 (2) Å; Os(3)–Os(4) = 2.982 (2) Å). The short diagonal Os–Os vector is more typical of a normal Os–Os single bond (Os(1)–Os(3) = 2.935 (2) Å). (The average Os–Os bond length in $Os_3(CO)_{12}$ is 2.877 (3) Å.[7]) The structure found for **1** is unique for 62-electron tetranuclear clusters. Most clusters of this type adopt the butterfly geometry,[8] the structure most compatible with PSEP theory. We have also observed the butterfly configuration in a cluster closely related to **1**, namely, $Os_4(\mu\text{-}H)_2(CO)_{13}(PMe_3)$.[9] On the other hand

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(1) (a) Wade, K. In "Transition Metal Clusters"; Johnson, B. F. G., Ed.; Wiley: Chichester, England, 1980; p 193. (b) Johnson, B. F. G.; Benfield, R. E. *Top. Stereochem.* **1981**, *12*, 253. (c) Mingos, D. M. P. *Acc. Chem. Res.* **1984**, *17*, 311.

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(5) Anal. Calcd for $C_{17}H_9O_{14}POs_4$: C, 16.60; H, 0.74. Found: C, 16.84; H, 0.67. IR ν(CO) (hexane) 2116 (w), 2069 (s), 2046 (m), 2035 (vs), 2028 (sh), 2014 (m), 2007 (w), 1986 (vw), 1978 (m), 1969 (w), 1951 (vw), 1927 (w) cm^{-1}; MS, m/e 1202 ([M − CO]$^+$); ^{1}H NMR (C_6D_6) δ 0.95 (J_{P-H} = 10.2 Hz).

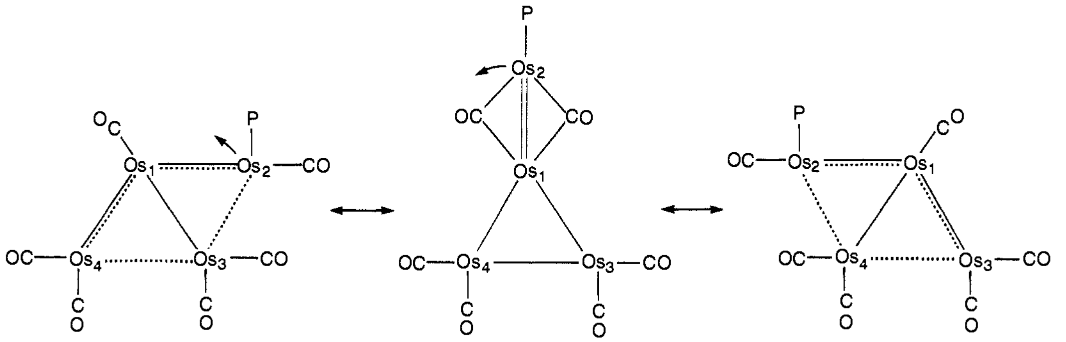
(6) X-ray diffraction data for $Os_4(CO)_{14}(PMe_3)$: M_r = 1230; monoclinic; space group $P2_1/a$; a = 13.525 (5) Å, b = 12.969 (6) Å, c = 14.825 (5) Å; β = 99.14 (3)°; V = 2567.54 Å^3; Z = 4, D_c = 3.185 g cm^{-3}; (an experimentally based absorption correction was applied); diffractometer, Enraf-Nonius CAD4F; radiation, Mo Kα, graphite monochromator ($\lambda(K\alpha_1)$ = 0.70930 Å); scan range = $3° \leq 2\theta \leq 45°$; reflections = 2424 with $I_0 \geq 2.5\sigma I_0$; R_f = 0.0385, R_W = 0.0479.

(7) Churchill, M. R.; DeBoer, B. G. *Inorg. Chem.* **1977**, *16*, 878.

(8) Some recent examples: (a) Carty, A. J.; MacLaughlin, S. A.; Wagner, J. V.; Taylor, N. J. *Organometallics* **1982**, *1*, 1013. (b) Johnson, B. F. G.; Lewis, J.; Nelson, W. J. H.; Puga, J.; Henrick, K.; McPartlin, M. *J. Chem. Soc., Dalton Trans.* **1983**, 1203. (c) Johnson, B. F. G.; Lewis, J.; Nelson, W. J. H.; Puga, J.; Raithby, P. R.; Whitmire, K. H. J. *Ibid.* **1983**, 1339. (d) Park, J. T.; Shapley, J. R.; Churchill, M. R.; Bueno, C. *Inorg. Chem.* **1984**, *23*, 4476. (e) Horwitz, C. P.; Holt, E. M.; Shriver, D. F. *J. Am. Chem. Soc.* **1985**, *107*, 281. See also: Bruce, M. I. In "Comprehensive Organometallic Chemistry"; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon Press: Oxford, 1982; Vol. 9, p 1395.

0002-7863/86/1508-0338$01.50/0

Scheme I



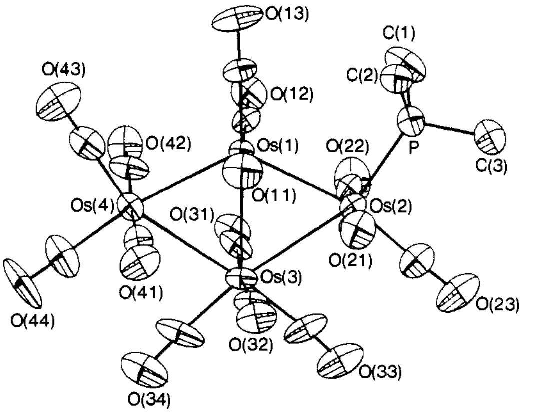
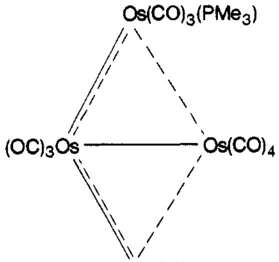


Figure 1. Molecular structure of $Os_4(CO)_{14}(PMe_3)$ (**1**).

$[Re_4(CO)_{16}]^{2-}$[10] and $HOs_3Re(CO)_{15}$,[11] which like **1** contain no bridging ligands, adopt a regular rhomboidal (flat butterfly) structure. In $Os_4(CO)_{13}(\mu_3\text{-}S)$ the osmium skeleton is also very nearly planar.[12]

Long and short Os–Os bonds have often been observed in osmium clusters[12,13] but the cause is not understood. We believe the Os–Os bond lengths in **1** are best rationalized in terms of three-center-two-electron metal–metal bonds (or two four-center-two-electron bonds) as shown. This leads to Os–Os bond



orders of 1.5 and 0.5 and achieves an 18-electron configuration for each osmium. Similar arguments may possibly be used to rationalize the bonding in other metal clusters where unusual bond lengths occur.

A ^{13}CO enriched sample of **1** in $CHFCl_2/CD_2Cl_2$ solution at −115 °C exhibited a ^{13}C NMR spectrum in the carbonyl region consistent with the structure found in the solid state: four resonances of relative intensity two (at δ 211.9, 202.0 (J_{P-C} = 8.6 Hz), 192.5, and 171.9) assigned to axial carbonyls, and six resonances of relative intensity one (at δ 192.2, 176.9, 174.7, 172.7, 171.0, and 166.4) assigned to equatorial carbonyls (Figure 2). However,

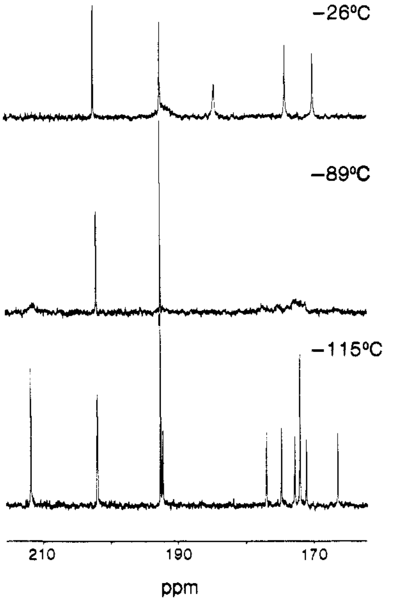


Figure 2. Variable-temperature, 100.6-MHz ^{13}C NMR spectra of **1** (^{13}CO enriched; $CHFCl_2/CD_2Cl_2$ solution).

by −89 °C eight of the signals had broadened and collapsed to the base line; the two remaining signals were due to axial carbonyls, one of which could be assigned to the axial carbonyls on osmium 2 by virtue of the phosphorus coupling. On further warming, four signals appeared at the average values of the original signals taken in pairs. An analysis of the spectra showed that the two axial resonances had coalesced together and the six equatorial peaks had collapsed in pairs (Figure 2).[14] This type of collapse is not consistent with any of the various mechanisms previously proposed to account for fluxionality in osmium clusters. For example, the 3-fold twist and bridge-terminal carbonyl exchange mechanisms cause axial and equatorial signals to average; a merry-go-round rearrangement produces a different isomer of **1**.[15] Furthermore, the process must involve bridging carbonyls in order to allow the equatorial carbonyl on Os(2) to exchange. The mechanism shown in Scheme I (axial carbonyls have been omitted) is provisionally proposed for the fluxional process; it correctly accounts for the observed changes.[16] Fluxional processes that involve rearrangement of a metal framework are very rare in metal cluster chemistry.[15] Such a rearrangement for **1** would,

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(10) Churchill, M. R.; Bau, R. *Inorg. Chem.* **1968**, *7*, 2606.

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(14) In the spectra shown in Figure 2: the signal at 202.0 ppm is a doublet; the most intense peak in the spectrum at −89 °C has lost intensity in the spectrum at −26 °C due to the onset of a second fluxional process; the shoulder to the immediate high field of this peak (in the spectrum at −26 °C) is due to the coalescence of the two axial signals.

(15) (a) Johnson, B. F. G.; Benfield, R. E. In "Transition Metal Clusters"; Johnson, B. F. G., Ed.; Wiley: Chichester, England, 1980; p 471. (b) Mann, B. E. In "Comprehensive Organometallic Chemistry"; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon Press: Oxford, 1982; Vol. 3, p 89.

(16) The mechanism predicts the following exchanges: C(13) with C(23), C(33) with C(43), C(34) with C(44), and C(31),C(32) with C(41),C(42). The axial carbons on Os(1) (C(11),C(12)) and Os(2) (C(21),C(22)) remain unaffected by the process.

however, be consistent with the presence of weak osmium–osmium bonds in the cluster.

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Supplementary Material Available: Tables of atomic coordinates, temperature factors, and bond lengths and angles for **1** (4 pages). Ordering information is given on any current masthead page.

Assembly of Vanadium-Iron-Sulfur Cubane Clusters from Mononuclear and Linear Trinuclear Reactants

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The spontaneous assembly from simple reactants of stable clusters containing the $MoFe_3S_4$ and WFe_3S_4 core units,[1] as well as their incorporation in a variety of double-[1-3] and single-cubane[1,4] structures, raises the issue of the scope of the generalized heterometal cluster core $MFe_3(\mu_3\text{-}S)_4$. The most likely new species would be isoelectronic with known clusters, all of which ultimately derive from $[MoS_4]^{2-}$ and $[WS_4]^{2-}$.[1] Consequently, initial investigations have involved systems based on $(NH_4)_3[VS_4]$,[5,6] which is the precursor to the "linear" trinuclear cluster $[VFe_2S_4Cl_4]^{3-}$ (**1**, Fe–V–Fe = 172.9 (1)°).[6] This species is particularly valuable synthetically because terminal chloride ligand substitution proceeds with retention of the VFe_2S_4 core structure,[6] as exemplified by the following preparation[8a] of a V–Fe–S cluster. A solution of $Li_2[Fe_2S_2(CO)_6]$[7] (2.9 mmol) in 100 mL of THF at –78 °C was treated with $(Et_4N)_3(\mathbf{1})$[6] (1.5 mmol) in 250 mL of acetonitrile. Addition of ether to the reaction mixture filtrate after a 16-h reaction at ~25 °C afforded black crystals of composition $(Et_4N)_3[VFe_6S_8(CO)_{12}]$[8b] (**3**, 31%; λ_{max} (ϵ_M) 316 (26 500), 416 (11 500), 526 (sh), 560 (12 100) nm (MeCN); ν_{CO} 1955, 1998, 2035 cm^{-1} (MeCN)). Diffraction-quality crystals have not been obtained. However, analytical data, the presence of a perturbed $[VS_4]^{3-}$ chromophore[6] in the UV/visible spectrum, and analogy to $[MoFe_3S_6(CO)_6]^{2-}$[2c] (**2**), which is prepared by the substitution of terminal chlorides in $[S_2MoS_2FeCl_2]^{2-}$ with $[Fe_2S_2(CO)_6]^{2-}$, establish structure **3**. While **3**, as **2**, may be subject to cubane cluster formation by oxidative decarbonylation,[2c] more direct routes were investigated.

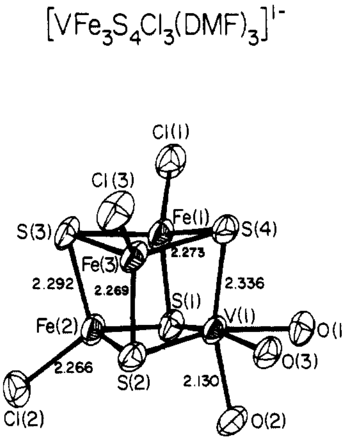
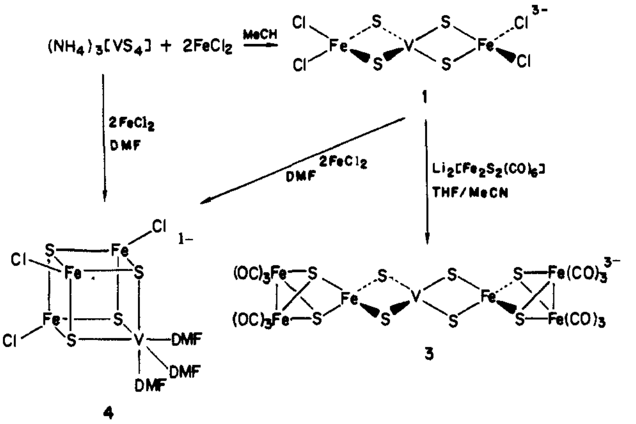


Figure 1. Structure of $[VFe_3S_4Cl_3(DMF)_3]^{1-}$ (**4**), showing 50% probability ellipsoids and mean values of bond distances under idealized trigonal symmetry. Of the DMF ligands, only the oxygen atoms are shown. Ranges of interatomic distances (Å): V–S, 2.331 (3)–2.340 (3); V–O, 2.112 (6)–2.145 (6); V–Fe, 2.771 (2)–2.781 (2); Fe–S(3), 2.287 (3)–2.301 (3); type Fe(3)–S(4), 2.265 (2)–2.281 (3); type Fe(3)–S(2), 2.263 (3)–2.276 (3); Fe–Cl, 2.260 (3)–2.275 (2).



A slurry of $(NH_4)_3[VS_4]$ (12 mmol) and equimolar Me_4NBr in 300 mL of DMF was allowed to react with 52 mmol of anhydrous $FeCl_2$,[8a] giving an intense red solution which slowly turned brown. After 16 h, workup of the reaction mixture as above afforded black crystalline $(Me_4N)[VFe_3S_4Cl_3(DMF)_3]\cdot 2DMF$[8b] (anion **4**, 50%). The absorption spectrum lacked UV/visible features characteristic of perturbed $[VS_4]^{3-}$, indicating reduction of V(V) and incorporation in the product. This compound crystallizes in monoclinic space group $P2_1/c$ with a = 12.479 (4) Å, b = 10.638 (3) Å, c = 29.422 (9) Å, β = 92.56 (2)°, and Z = 4. The crystal structure was solved and refined by standard procedures.[9] The stereochemistry of anion **4**, shown in Figure 1, is that of a cubane-type cluster whose $[VFe_3S_4]^{2+}$ core is isoelectronic and nearly isostructural with the $[MoFe_3S_4]^{3+}$ core found in numerous single and double cubanes,[1-4] including $[MoFe_3S_4Cl_3(al_2cat)(THF)]^{2-}$[4e] (**5**, al_2cat = 3,6-diallylcatecholate). The cluster closely approaches trigonal symmetry

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(8) (a) All reactions and manipulations were conducted under a pure dinitrogen atmosphere. (b) All new compounds gave satisfactory elemental analyses.

(9) Suitable crystals were obtained by diffusion of ether into a DMF solution. Diffraction data were collected at ~25 °C on a Nicolet P3F four-circle automated instrument with graphite-monochromatized Mo Kα radiation. An empirical absorption correction was applied. With use of 2889 unique data (5° ≤ 2θ ≤ 40°, $I \geq 3\sigma(I)$), the structure was solved by a combination of direct methods (MULTAN) and Fourier techniques and refined to $R(R_w)$ = 6.0 (7.9)%. The anion and cation were refined anisotropically, and the DMF solvate molecules were refined isotropically, by using block cascade least-squares refinement.

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