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3,3′-Dimethyl-4,4′-diphenyl-2,2′,5,5′-tetrathiafulvalenium Hexamolybdate, $2(C_{20}H_{16}S_4^+)[Mo_6O_{19}]^{2-}$

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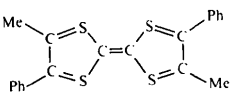
Abstract

The central C=C bond length [1.391 (9) Å] and the two types of C—S bond lengths [average 1.712 (6) and 1.735 (7) Å] of the DMDPh-TTF ($C_{20}H_{16}S_4$) molecule compare well with those observed for the fully oxidized species. This organic molecule has a *trans* configuration. The phenyl rings are not coplanar with the central TTF fragment. The dihedral angles observed between the phenyl rings and the central TTF core are 38.1 (2) and 50.9 (2)°. There are no interactions between the organic molecules since the intermolecular S···S contacts are greater than 4 Å. The shortest S···O contacts observed are: S(1)···O(6) 3.217 (5), S(2)···O(4) 3.296 (5) and S(3)···O(3) 3.298 (5) Å.

Comment

The crystal structure represented in Fig. 1 is built from one independent organic molecule (DMDPh-TTF) and one inorganic anion, $[Mo_6O_{19}]^{2-}$, having a



DMDPh-TTF

Lindquist type of structure (Lindquist, 1953). This anion is located at the origin of the lattice, with bond distances and bond angles within $[Mo_6O_{19}]^{2-}$ close to those observed previously for such a unit (Triki *et al.*, 1992; Allcock, Bissell & Shawl, 1973). The organic molecule has a *trans* configuration. The phenyl rings do not assume a coplanar configuration with respect to the central TTF fragment. The dihedral angle observed between the two phenyl rings is 91.4 (3)°, while those observed between the phenyl rings and the central TTF core are 38.1 (2) and 50.9 (2)°. These values are different from those (33.3 and 37.0°) observed in $(DMDPh\text{-}TTF)_2[Re_2Cl_8].2CH_2Cl_2$ (Fettouhi, Ouahab, Perrin, Grandjean & Fabre, 1991). The charge on the DMDPh-TTF moiety is assumed to be +1 as there are only two molecules per anionic unit. The central C=C bond length [1.391 (9) Å] and the two types of C—S bond lengths [average: 1.712 (6) and 1.735 (7) Å] of the organic molecule compare well with those observed for the fully oxidized species in $[(DMDPh\text{-}TTF)_2Re_2Cl_8].2CH_2Cl_2$ (Fettouhi *et al.*, 1991).

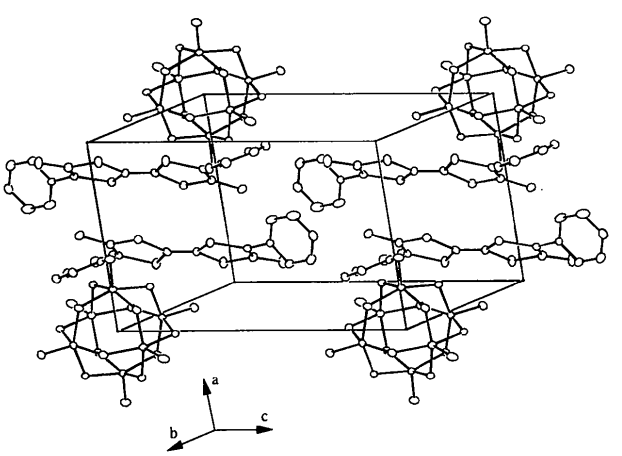


Fig. 1. Perspective view of the crystal structure, with four Mo_6O_{19} units omitted for clarity.

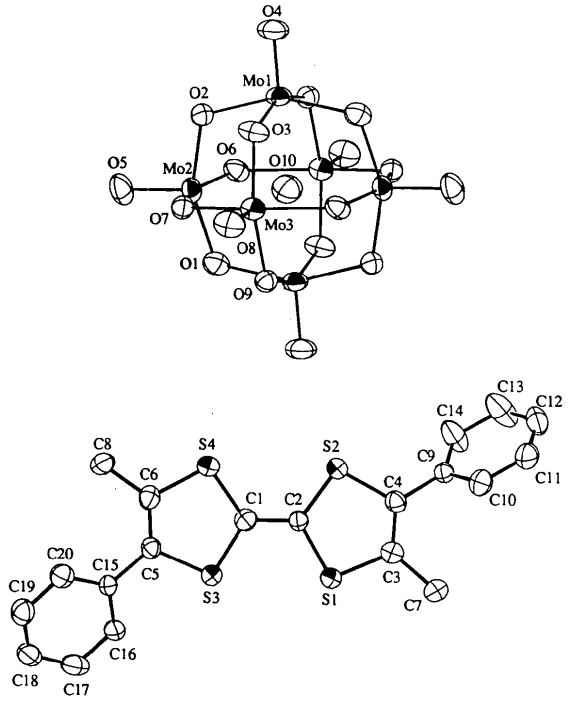


Fig. 2. Atom-numbering scheme.

Experimental

Crystal data

$2(C_{20}H_{16}S_4)[Mo_6O_{19}]$
M_r = 1648.84
Triclinic
$P\bar{1}$
a = 9.620 (3) Å
b = 11.051 (2) Å
c = 13.017 (3) Å
α = 113.10 (4)°
β = 96.80 (3)°
γ = 90.26 (2)°
V = 1261.9 Å^3
Z = 1
D_x = 2.170 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 7–12°
μ = 1.803 mm^{-1}
T = 293 K
Rhombohedral
0.3 × 0.2 × 0.2 mm
Black
Crystal source: electrocrystallization

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
T_{min} = 0.88, T_{max} = 1.18
4732 measured reflections
4150 independent reflections
2641 observed reflections [$I \geq 3\sigma(I)$]
R_{int} = 0.023
θ_{max} = 25°
$h = 0 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity variation: <2%

Refinement

Refinement on F
R = 0.028
wR = 0.037
S = 0.855
2641 reflections
353 parameters
H-atom parameters not refined
$w = 4F_o^2/[\sigma^2(I) + (0.07F_o^2)^2]$
$\Delta\rho_{max}$ = 0.582 e Å^{-3}
$\Delta\rho_{min}$ = −0.173 e Å^{-3}
Extinction correction: $|F_c|(1 + gI_c)^{-1}$
Extinction coefficient: $g = 3.623 \times 10^{-8}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

H atoms were included in the structure-factor calculations (C—H = 0.95 Å, B = 5 Å^2). All calculations were performed on a MicroVAX 3100 computer using the *SDP* programs (Frenz, 1985).

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = \frac{4}{3}\Sigma_i\Sigma_j\beta_{ij}a_i^*a_j^*$.

	x	y	z	B_{eq}
Mo(1)	0.04081 (5)	−0.12571 (5)	0.10689 (4)	2.43 (1)
Mo(2)	0.03848 (6)	−0.17903 (5)	−0.15835 (4)	2.54 (1)
Mo(3)	−0.23689 (5)	−0.06427 (5)	−0.03379 (4)	2.27 (1)
O(1)	0.0600 (4)	−0.2482 (4)	−0.0447 (3)	2.70 (9)
O(2)	−0.0045 (4)	0.0407 (4)	0.2139 (3)	2.75 (9)
O(3)	−0.1619 (4)	−0.1553 (4)	0.0548 (3)	3.00 (9)
O(4)	0.0662 (5)	−0.2204 (4)	0.1820 (3)	3.5 (1)
O(5)	0.0676 (5)	−0.3071 (5)	−0.2762 (4)	4.2 (1)
O(6)	−0.1609 (4)	−0.1933 (4)	−0.1602 (3)	2.7 (1)
O(7)	0.2224 (4)	−0.0947 (4)	−0.0994 (3)	2.48 (9)
O(8)	−0.4080 (4)	−0.1114 (4)	−0.0609 (4)	3.5 (1)
O(9)	0.2214 (4)	−0.0535 (4)	0.1136 (3)	2.33 (9)
O(10)	0.0	0.0	0.0	1.7 (1)
S(1)	0.2276 (2)	0.4885 (2)	0.1818 (1)	3.66 (4)
S(2)	0.3957 (2)	0.7330 (2)	0.2531 (1)	2.96 (4)
S(3)	0.2136 (2)	0.4429 (2)	−0.0696 (1)	3.44 (4)
S(4)	0.3997 (2)	0.6708 (2)	−0.0199 (1)	2.92 (4)
C(1)	0.3094 (6)	0.5758 (5)	0.0310 (5)	2.7 (1)
C(2)	0.3148 (6)	0.5992 (6)	0.1445 (5)	2.5 (1)
C(3)	0.2765 (7)	0.5709 (6)	0.3244 (5)	3.2 (2)
C(4)	0.3518 (6)	0.6862 (6)	0.3590 (5)	2.6 (1)
C(5)	0.2647 (6)	0.4661 (6)	−0.1844 (5)	2.6 (1)
C(6)	0.3490 (6)	0.5730 (6)	−0.1616 (5)	2.7 (1)
C(7)	0.2339 (8)	0.5021 (7)	0.3959 (6)	4.4 (2)
C(8)	0.4102 (7)	0.6166 (6)	−0.2435 (5)	3.4 (2)
C(9)	0.4006 (6)	0.7736 (5)	0.4770 (5)	2.4 (1)
C(10)	0.3023 (7)	0.8170 (7)	0.5524 (5)	3.5 (2)
C(11)	0.3457 (8)	0.8967 (7)	0.6640 (6)	4.0 (2)
C(12)	0.4828 (8)	0.9341 (7)	0.7005 (5)	3.8 (2)
C(13)	0.5793 (8)	0.8955 (9)	0.6276 (7)	5.6 (2)
C(14)	0.5369 (7)	0.8152 (9)	0.5138 (6)	5.1 (2)
C(15)	0.2065 (6)	0.3616 (6)	−0.2952 (4)	2.5 (1)
C(16)	0.2022 (7)	0.2301 (6)	−0.3078 (5)	3.0 (1)
C(17)	0.1465 (8)	0.1323 (6)	−0.4106 (6)	4.0 (2)
C(18)	0.0975 (7)	0.1672 (7)	−0.4988 (5)	3.5 (2)
C(19)	0.0994 (7)	0.2953 (7)	−0.4859 (5)	3.7 (2)
C(20)	0.1568 (7)	0.3953 (6)	−0.3838 (5)	3.4 (2)

Table 2. *Geometric parameters* (Å, °)

Mo(1)—Mo(2)	3.2641 (8)	Mo(1)—Mo(2^i)	3.2779 (8)
Mo(1)—Mo(3^i)	3.2906 (8)	Mo(2)—Mo(3)	3.2692 (7)
Mo(1)—Mo(3)	3.2840 (8)	Mo(2)—Mo(3^i)	3.2673 (6)
Mo(1)—O(1)	1.937 (4)	Mo(2)—O(6)	1.920 (4)
Mo(1)—O(2)	1.911 (4)	Mo(2)—O(7)	1.921 (4)
Mo(1)—O(3)	1.969 (4)	Mo(2)—O(10)	2.2994 (5)
Mo(1)—O(4)	1.691 (5)	Mo(3)—O(3)	1.892 (5)
Mo(1)—O(9)	1.886 (4)	Mo(3)—O(6)	1.931 (4)
Mo(1)—O(10)	2.3264 (6)	Mo(3)—O(7^i)	1.914 (3)
Mo(2)—O(1)	1.906 (5)	Mo(3)—O(8)	1.678 (4)
Mo(2)—O(2^i)	1.945 (5)	Mo(3)—O(9^i)	1.973 (5)
Mo(2)—O(5)	1.684 (4)	Mo(3)—O(10)	2.3225 (5)
S(1)—C(2)	1.729 (7)	C(6)—C(8)	1.51 (1)
S(1)—C(3)	1.720 (6)	C(9)—C(10)	1.393 (9)
S(2)—C(2)	1.696 (5)	C(9)—C(14)	1.354 (9)
S(2)—C(4)	1.744 (7)	C(10)—C(11)	1.383 (8)
S(3)—C(1)	1.707 (5)	C(11)—C(12)	1.35 (1)

S(3)—C(5)	1.737 (7)	C(12)—C(13)	1.36 (1)
S(4)—C(1)	1.723 (7)	C(13)—C(14)	1.40 (1)
S(4)—C(6)	1.740 (5)	C(15)—C(16)	1.396 (9)
C(1)—C(2)	1.391 (9)	C(15)—C(20)	1.38 (1)
C(3)—C(4)	1.347 (9)	C(16)—C(17)	1.391 (8)
C(3)—C(7)	1.50 (1)	C(17)—C(18)	1.38 (1)
C(4)—C(9)	1.474 (7)	C(18)—C(19)	1.36 (1)
C(5)—C(6)	1.341 (9)	C(19)—C(20)	1.400 (8)
C(5)—C(15)	1.491 (7)		
O(1)—Mo(1)—O(2)	152.7 (2)	O(6)—Mo(2)—O(7)	154.0 (1)
O(1)—Mo(1)—O(3)	84.6 (2)	O(6)—Mo(2)—O(10)	77.2 (1)
O(1)—Mo(1)—O(4)	102.7 (2)	O(7)—Mo(2)—O(10)	76.8 (1)
O(1)—Mo(1)—O(9)	87.7 (2)	O(3)—Mo(3)—O(6)	88.3 (2)
O(1)—Mo(1)—O(10)	76.3 (1)	O(3)—Mo(3)—O(7)	89.2 (2)
O(2)—Mo(1)—O(3)	85.8 (2)	O(3)—Mo(3)—O(8)	103.2 (2)
O(2)—Mo(1)—O(4)	104.3 (2)	O(3)—Mo(3)—O(9)	153.2 (2)
O(2)—Mo(1)—O(9)	89.4 (2)	O(3)—Mo(3)—O(10)	77.5 (1)
O(2)—Mo(1)—O(10)	76.6 (1)	O(6)—Mo(3)—O(7)	152.6 (2)
O(3)—Mo(1)—O(4)	102.4 (2)	O(6)—Mo(3)—O(8)	102.7 (2)
O(3)—Mo(1)—O(9)	153.2 (2)	O(6)—Mo(3)—O(9)	84.9 (2)
O(3)—Mo(1)—O(10)	76.0 (1)	O(6)—Mo(3)—O(10)	76.4 (1)
O(4)—Mo(1)—O(9)	104.4 (2)	O(7)—Mo(3)—O(8)	104.5 (2)
O(4)—Mo(1)—O(10)	178.2 (1)	O(7)—Mo(3)—O(9)	85.1 (2)
O(9)—Mo(1)—O(10)	77.2 (1)	O(7)—Mo(3)—O(10)	76.4 (1)
O(1)—Mo(2)—O(2)	154.2 (1)	O(8)—Mo(3)—O(9)	103.6 (2)
O(1)—Mo(2)—O(5)	104.0 (2)	O(8)—Mo(3)—O(10)	178.9 (2)
O(1)—Mo(2)—O(6)	88.4 (2)	O(9)—Mo(3)—O(10)	75.7 (1)
O(1)—Mo(2)—O(7)	87.6 (2)	Mo(1)—O(1)—Mo(2)	116.3 (2)
O(1)—Mo(2)—O(10)	77.6 (1)	Mo(1)—O(2)—Mo(2)	116.4 (2)
O(2)—Mo(2)—O(5)	101.8 (2)	Mo(1)—O(3)—Mo(3)	116.5 (2)
O(2)—Mo(2)—O(6)	86.6 (2)	Mo(2)—O(6)—Mo(3)	116.2 (2)
O(2)—Mo(2)—O(7)	85.9 (2)	Mo(2)—O(7)—Mo(3)	116.9 (2)
O(2)—Mo(2)—O(10)	76.6 (1)	Mo(1)—O(9)—Mo(3)	117.0 (2)
O(5)—Mo(2)—O(6)	103.3 (2)	Mo(1)—O(10)—Mo(2)	90.24 (2)
O(5)—Mo(2)—O(7)	102.6 (2)	Mo(1)—O(10)—Mo(3)	90.12 (2)
O(5)—Mo(2)—O(10)	178.3 (2)	Mo(2)—O(10)—Mo(3)	90.03 (2)
C(2)—S(1)—C(3)	95.3 (3)	S(4)—C(6)—C(5)	116.4 (5)
C(2)—S(2)—C(4)	95.6 (3)	S(4)—C(6)—C(8)	115.5 (4)
C(1)—S(3)—C(5)	96.1 (3)	C(5)—C(6)—C(8)	128.0 (5)
C(1)—S(4)—C(6)	95.8 (3)	C(4)—C(9)—C(10)	118.7 (5)
S(3)—C(1)—S(4)	115.0 (4)	C(4)—C(9)—C(14)	122.2 (6)
S(3)—C(1)—C(2)	120.6 (5)	C(10)—C(9)—C(14	119.0 (5)
S(4)—C(1)—C(2)	124.4 (4)	C(9)—C(10)—C(11)	119.8 (6)
S(1)—C(2)—S(2)	115.7 (4)	C(10)—C(11)—C(12)	120.5 (7)
S(1)—C(2)—C(1)	118.9 (4)	C(11)—C(12)—C(13)	120.4 (6)
S(2)—C(2)—C(1)	125.3 (5)	C(12)—C(13)—C(14)	119.8 (7)
S(1)—C(3)—C(4)	117.2 (6)	C(9)—C(14)—C(13)	120.3 (7)
S(1)—C(3)—C(7)	115.3 (4)	C(5)—C(15)—C(16)	119.3 (6)
C(4)—C(3)—C(7)	127.4 (5)	C(5)—C(15)—C(20)	120.0 (6)
S(2)—C(4)—C(3)	116.1 (4)	C(16)—C(15)—C(20)	120.7 (5)
S(2)—C(4)—C(9)	118.1 (5)	C(15)—C(16)—C(17)	119.6 (6)
C(3)—C(4)—C(9)	125.8 (7)	C(16)—C(17)—C(18)	119.2 (7)
S(3)—C(5)—C(6)	116.6 (4)	C(17)—C(18)—C(19)	121.2 (5)
S(3)—C(5)—C(15)	113.7 (5)	C(18)—C(19)—C(20)	120.6 (7)
C(6)—C(5)—C(15)	129.7 (6)	C(15)—C(20)—C(19)	118.7 (7)

Symmetry code: (i) $-x, -y, -z$.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71484 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1043]

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Barium (Cryptand 222B)$Cl_2.6H_2O$

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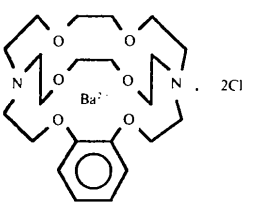
Abstract

In the title compound, (4,7,13,16,21,24-hexaoxa-1,10-diaza-5,6-benzobicyclo[8.8.8]hexacos-5-ene)barium dichloride hexahydrate, $[BaCl_2(C_{22}H_{36}N_2O_6)].6H_2O$, the Ba ion is surrounded by the cryptand molecule (222B) with the arm containing the fused benzene-ring substituent bent towards one of the other two cryptand arms, leaving two slightly larger open faces through which two additional water molecules are coordinated, making a total of ten atoms surrounding the Ba atom. The species could be more accurately represented as $[Ba(222B)(H_2O)_2]^{2+}.2Cl^-.4H_2O$

Comment

Crystals of the barium salt of cryptand 222B were prepared as a preliminary step in the development of a model using a bioconjugate of the 222B ligand, with the intent of exploring its potential in radioimmunotherapy. Previously, we have developed an HPLC (high-performance liquid chromatography) assay (Pettit, Swailes & Peterson, 1989) for studying the components in this system. The molecular structure of the title compound (I) and the labeling of the atoms is shown in Fig. 1.



(I)