

found in the five-coordinate  $N,N,N'$ -tris[2-(2'-pyridyl)ethyl]ethane-1,2-diaminenickel(II) perchlorate of 2.011–2.124 Å (Hoskins & Whillans, 1975).

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## Structure of 3,3'-Dimethyl-4,4'-diphenyl-2,2',5,5'-tetrathiafulvalenium Octachlorodirhenate–Dichloromethane (1/2)

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**Abstract.**  $2C_{20}H_{16}S_4^+ \cdot Re_2Cl_8^{2-} \cdot 2CH_2Cl_2$ ,  $M_r = 1595.10$ , triclinic,  $P\bar{1}$ ,  $a = 9.969$  (9),  $b = 10.656$  (8),  $c = 13.570$  (8) Å,  $\alpha = 105.01$  (6),  $\beta = 102.58$  (6),  $\gamma = 91.58$  (5)°,  $V = 1353.5$  Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.957$  g cm<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71073$  Å,  $\mu = 133.61$  cm<sup>-1</sup>,  $F(000) = 770$ ,  $T = 293$  K,  $R = 0.036$  for 2801 reflections with  $I \geq 6\sigma(I)$ . The methyl and phenyl groups are *trans* to each other with respect to the central tetrathiafulvalene part. The structure consists of isolated organic dimers. The eclipsed intradimer overlap of organic molecules implies short S...S contacts ranging from 3.47 to 3.52 Å. The Re—Re bond lengths in the  $Re_2Cl_8^{2-}$  dianion are close to those commonly observed for an Re—Re quadruple bond in such a unit.

**Experimental.** Dimethyldiphenyltetrathiafulvalene (DMDPhTTF) was prepared as described by Takamisawa & Hirai (1969). Its anodic oxidation ( $10^{-3}$  M) on a platinum-wire electrode under low constant current ( $I = 1.0$  μA) in a dichloromethane solution of the octachlorodirhenate tetrabutylammonium salt ( $10^{-2}$  M), which also acted as supporting electrolyte leads, produced blue needle crystals (1–2 mm long) after two weeks. A crystal of dimensions  $0.8 \times 0.1 \times 0.25$  mm was selected for the

intensity-data collections which were carried out on an Enraf–Nonius CAD-4 diffractometer equipped with graphite-monochromatized Mo  $K\alpha$  radiation. The cell dimensions were obtained and refined by least squares from setting angles of 25 centered reflections ( $\theta \leq 15^\circ$ ). 4759 reflections were recorded by a  $\theta$ - $2\theta$  scan in the range  $2 \leq 2\theta \leq 50^\circ$  and  $0 \leq h \leq 11$ ,  $-12 \leq k \leq 12$ ,  $-16 \leq l \leq 16$ . Three standard reflections measured every hour: no fluctuations in intensity. The intensities were corrected for Lorentz-polarization and absorption phenomena. The latter correction was performed using the *DIFABS* procedure (Walker & Stuart, 1983) (correction factors 0.783–1.300). The structure was solved by direct methods (*MULTAN84*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1984) and successive Fourier difference syntheses. 2801 independent reflections with  $I \geq 6\sigma(I)$ , after averaging the symmetry-related reflections ( $R_{int} = 0.036$ ), were used for the full-matrix least-squares anisotropic ( $\beta_{ij}$ ) refinements (on  $F$ ), secondary extinction refined,  $g = 3.75 \times 10^{-10}$ ,  $R = 0.036$ ,  $wR = 0.047$ ,  $w = 4F_o^2/[\sigma(I)^2 + (0.07F_o^2)^2]$ ,  $S = 1.12$ ,  $(\Delta/\sigma)_{max} = 0.01$ ,  $(\Delta\rho)_{max} = 1.28$  e Å<sup>-3</sup>. H atoms were placed at computed positions [ $d(C-H) = 1.0$  Å;  $B_{eq} = 5.0$  Å<sup>2</sup>] and not refined. The scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All the calculations were performed on a

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Table 1. Atomic coordinates and equivalent isotropic temperature factors

$$B_{eq} = (4/3) \sum_i \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Re1	-0.09416 (4)	-0.05211 (4)	0.00696 (3)	2.336 (7)
Cl1	-0.0617 (3)	-0.0209 (3)	0.1855 (2)	3.77 (6)
Cl2	-0.0374 (3)	-0.2636 (2)	-0.0017 (2)	4.00 (6)
Cl3	-0.2462 (3)	0.1086 (2)	0.0231 (2)	3.41 (6)
Cl4	-0.2195 (3)	-0.1329 (3)	-0.1647 (2)	3.72 (6)
S1	0.7200 (3)	0.6360 (2)	0.1566 (2)	3.18 (6)
S2	0.5599 (3)	0.5482 (2)	-0.1783 (2)	3.20 (6)
S3	0.7297 (3)	0.4070 (2)	-0.0517 (2)	3.09 (6)
S4	0.5545 (3)	0.7770 (2)	0.0279 (2)	3.20 (6)
C1	0.680 (1)	0.7905 (9)	0.2204 (7)	2.9 (2)
C2	0.741 (1)	0.834 (1)	0.3354 (8)	4.2 (3)
C3	0.868 (1)	0.797 (1)	0.374 (1)	5.8 (3)
C4	0.664 (1)	0.905 (1)	0.4002 (9)	5.3 (3)
C5	0.846 (2)	0.910 (1)	0.549 (1)	8.1 (5)
C6	0.924 (2)	0.836 (2)	0.483 (1)	8.1 (5)
C7	0.717 (2)	0.945 (1)	0.508 (1)	7.9 (5)
C8	0.6030 (9)	0.8553 (9)	0.1597 (7)	2.7 (2)
C9	0.691 (1)	0.3379 (9)	-0.1860 (7)	2.9 (2)
C10	0.612 (1)	0.4013 (9)	-0.2446 (7)	2.8 (2)
C11	0.561 (1)	0.3667 (9)	-0.3611 (7)	3.2 (2)
C12	0.431 (1)	0.398 (1)	-0.4042 (9)	4.7 (3)
C13	0.645 (1)	0.305 (1)	-0.4243 (9)	4.7 (3)
C14	0.384 (2)	0.362 (1)	-0.5134 (9)	6.4 (4)
C15	0.464 (2)	0.297 (1)	-0.5768 (9)	6.8 (4)
C16	0.595 (2)	0.272 (1)	-0.5345 (9)	6.2 (4)
C17	0.566 (1)	0.9941 (9)	0.1922 (8)	3.6 (2)
C18	0.6377 (9)	0.6398 (9)	0.0328 (7)	2.5 (2)
C19	0.640 (1)	0.5419 (9)	-0.0560 (7)	2.7 (2)
C20	0.750 (1)	0.211 (1)	-0.2217 (8)	4.2 (3)
C15	-0.1758 (6)	0.3541 (7)	0.3146 (5)	14.2 (2)
C16	0.0647 (6)	0.4777 (5)	0.2865 (7)	14.2 (3)
C21	-0.040 (2)	0.344 (2)	0.255 (1)	7.6 (5)

Table 2. Selected bond distances (Å) and bond angles (°) with e.s.d.'s in parentheses

Re1—Re1	2.2231 (6)	Re1—Cl1	2.306 (3)
Re1—Cl2	2.318 (3)	Re1—Cl3	2.317 (3)
Re1—Cl4	2.315 (2)	S1—C1	1.750 (9)
S1—C18	1.712 (9)	S2—C10	1.747 (9)
S2—C19	1.697 (9)	S3—C9	1.730 (9)
S3—C19	1.72 (1)	S4—C8	1.719 (9)
S4—C18	1.71 (1)	Cl1—C8	1.34 (1)
C9—C10	1.32 (1)	Cl18—C19	1.38 (1)
C21—C15	1.71 (2)	C21—C16	1.65 (2)
Re1—Re1—Cl1	103.14 (7)	Re1—Re1—Cl2	104.87 (8)
Re1—Re1—Cl3	103.00 (6)	Re1—Re1—Cl4	104.06 (8)
Cl1—Re1—Cl2	86.8 (1)	Cl1—Re1—Cl3	86.8 (1)
Cl1—Re1—Cl4	152.8 (1)	Cl2—Re1—Cl3	152.1 (1)
Cl2—Re1—Cl4	86.7 (1)	Cl3—Re1—Cl4	86.75 (9)
C1—S1—C18	95.9 (5)	C8—S4—C18	97.2 (4)
C9—S3—C19	95.9 (5)	C10—S2—C19	96.0 (5)
S1—C1—C8	116.3 (6)	S4—C8—C1	116.2 (7)
S1—C18—S4	114.3 (5)	S1—C18—C19	123.1 (8)
S2—C19—S3	114.8 (5)	S2—C19—C18	122.5 (8)

MicroVAX 3100 using the SDP programs (B. A. Frenz & Associates, Inc., 1985). Table 1\* gives the atomic coordinates and Table 2 selected bond distances and bond angles. Fig. 1 shows the intradimer overlap with the numbering scheme and Fig. 2 the crystal structure packing.

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54374 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Related literature.** The  $Re_2Cl_8$  unit has an eclipsed configuration with the following averaged bond lengths: Re—Re 2.2231 (6) and Re—Cl 2.314 (3) Å. These are in agreement with those observed in  $(Bu_4N)_2Re_2Cl_8$  (Cotton & Hall, 1977). The DMDPhTTF molecule has a *trans* configuration while the phenyl rings do not assume a planar conformation with the central TTF fragment. The dihedral angles are 33.3 and 37.0°. The molecule can be considered in the formal valence state  $DMDPhTTF^+$  as there are only two molecules per one anionic unit. The structure consists of isolated dimers, in which the overlap is of the ring-over-ring

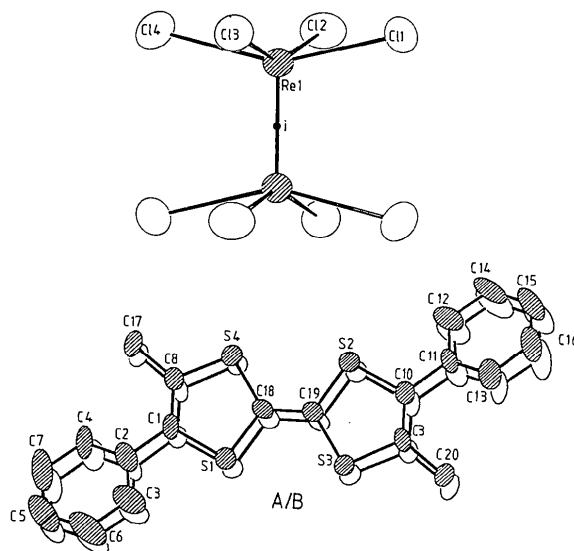


Fig. 1. Atomic numbering and intradimer overlap: A hatched circle  $(x, y, z)$  and B  $(1-x, 1-y, -z)$ .

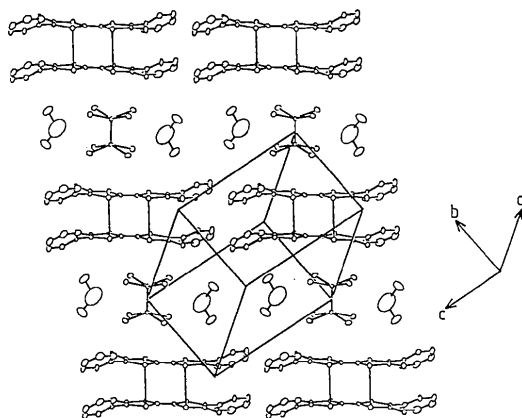


Fig. 2. Evidence of mixed stacks containing organic and inorganic blocks and cavities containing the solvent molecules. Intradimer interactions:  $S_1 - S_2^* = 3.476$  (4),  $S_3 - S_4^* = 3.521$  (4) Å (\* symmetry:  $1-x, 1-y, -z$ ).

type, piled up along the [110] direction. It can be described as mixed stacks of alternating organic and inorganic dimers (Fig. 2).

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## Structure of Dicarboxylbis( $\eta^2$ -dimethyl 7-acetyl-7-azabicyclo[2.2.1]hept-2-ene-2,3-dicarboxylate- $\kappa N$ )molybdenum(0)

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**Abstract.**  $[\text{Mo}(\text{C}_{24}\text{H}_{34}\text{N}_2\text{O}_{10})(\text{CO})_2]$ ,  $M_r = 662.51$ , monoclinic,  $C2/c$ ,  $a = 61.3502$  (41),  $b = 10.4370$  (4),  $c = 17.7387$  (14) Å,  $\beta = 99.742$  (6)°,  $V = 11194.5$  (18) Å<sup>3</sup>,  $Z = 16$ ,  $D_x = 1.57$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54056$  Å,  $\mu = 44.7$  cm<sup>-1</sup>,  $F(000) = 5472$ ,  $T = 298$  K,  $R = 0.046$  for 7459 reflections with  $I > 3\sigma(I)$ . There are two independent molecules per unit cell. In each molecule, two CO groups are *cis* to each other with Mo—CO distances from 1.955 (5) to 1.982 (7) Å. Two substituted double bonds are bound to the Mo atom [Mo—C from 2.193 (6) to 2.238 (6) Å]. The C—C distances for bound and non-bound double bonds are 1.437 (8) and 1.34 (1) Å, respectively. The Mo—N distances are 2.336 (4), 2.347 (5), 2.361 (5) and 2.339 (5) Å.

**Experimental.** The title compound,  $\text{C}_{26}\text{H}_{34}\text{MoN}_2\text{O}_{12}$ , was synthesized as reported by Sun & Chow (1988). Yellow crystals were grown from Et<sub>2</sub>O. A suitable prism crystal with dimensions 0.15 × 0.20 × 0.30 mm was mounted on a glass fiber with epoxy resin. Cell constants were derived from least-squares refinement of 25 reflections having  $50 < 2\theta < 52^\circ$ . Intensity data were collected at room temperature using the  $\theta/2\theta$  scanning technique on an Enraf-Nonius CAD-4 diffractometer with monochromated Cu  $K\alpha$  radiation. A total of 9595 reflections were measured with  $0 <$

$2\theta < 130^\circ$  ( $h = -72$  to  $70$ ,  $k = 0$  to  $12$ ,  $l = 0$  to  $20$ ), which were averaged to 9305 unique reflections with  $R_{\text{int}} = 0.019$ . Three standard reflections (17  $\bar{3}$  6,  $\bar{1}\bar{7}\bar{3}$   $\bar{6}$ ,  $\bar{6}$ 47) were measured every 7200 s and only small (< 3%) random variations were observed. Lorentz and polarization corrections were applied. An empirical absorption correction based on a series of  $\psi$  scans was applied to the data:  $T_{\text{min}} = 0.863$ ,  $T_{\text{max}} = 0.999$ . The space group,  $C2/c$ , was determined by systematic absences ( $hkl$ ,  $h + k = \text{odd}$ ;  $0k0$ ,  $k = \text{odd}$ ;  $h0l$ ,  $l = \text{odd}$ ).

The structure was solved by heavy-atom methods using *SHELXS86* (Sheldrick, 1986) which revealed two independent positions of Mo. The remaining non-H atoms were located in successive difference Fourier syntheses. H atoms were not included in calculations. Atomic scattering factors including anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The structure was refined with full-matrix least squares using *Personal SDP* (B. A. Frenz & Associates, Inc., 1989) on a 80386-based IBM compatible PC. All atoms were refined anisotropically.  $\sum w(|F_o| - |F_c|)^2$  was minimized, where  $w = 4F_o^2/[\sigma^2(I) + 0.01F_o^2]$ . A secondary-extinction coefficient refined to a value of  $\chi = 2.22 \times 10^{-7}$ , where the correction factor  $(1 + \chi I_c)^{-1}$  was applied to  $F_c$ . The final  $R = 0.046$ ,  $wR = 0.061$ ,  $R_{\text{all}} = 0.059$  and  $S = 3.137$  were obtained using 740 variables and 7459 observed

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