

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

Pt—N(1)	1.998 (5)	C(3)—N(7)	1.475 (7)	C(11)—C(12)	1.555 (6)
Pt—N(7)	2.019 (4)	C(4)—C(5)	1.532 (10)	C(11)—C(14)	1.591 (6)
Pt—O(8)	2.030 (4)	C(5)—C(6)	1.501 (10)	C(11)—C(16)	1.560 (10)
Pt—O(17)	1.998 (4)	C(6)—N(7)	1.492 (8)	C(12)—C(13)	1.529 (10)
N(1)—C(2)	1.486 (8)	O(8)—C(9)	1.318 (6)	C(13)—C(14)	1.546 (10)
C(2)—C(3)	1.512 (8)	C(9)—O(10)	1.224 (6)	O(15)—C(16)	1.255 (7)
C(3)—C(4)	1.535 (9)	C(9)—C(11)	1.482 (9)	C(16)—O(17)	1.258 (7)
N(1)—Pt—N(7)	84.4 (2)	O(8)—C(9)—C(11)	118.4 (4)		
N(1)—Pt—O(8)	175.7 (2)	O(10)—C(9)—C(11)	123.6 (4)		
N(1)—Pt—O(17)	92.1 (2)	C(9)—C(11)—C(12)	110.3 (5)		
N(7)—Pt—O(8)	92.6 (2)	C(9)—C(11)—C(14)	110.0 (6)		
N(7)—Pt—O(17)	176.3 (2)	C(9)—C(11)—C(16)	113.7 (4)		
O(8)—Pt—O(17)	90.9 (2)	C(12)—C(11)—C(14)	88.5 (3)		
N(1)—C(2)—C(3)	111.1 (5)	C(12)—C(11)—C(16)	118.0 (5)		
C(2)—C(3)—C(4)	115.7 (5)	C(14)—C(11)—C(16)	113.7 (6)		
C(2)—C(3)—N(7)	107.6 (4)	C(11)—C(12)—C(13)	89.4 (4)		
C(4)—C(3)—N(7)	103.4 (5)	C(12)—C(13)—C(14)	91.1 (5)		
C(3)—C(4)—C(5)	106.7 (5)	C(11)—C(14)—C(13)	87.5 (5)		
C(4)—C(5)—C(6)	102.9 (6)	C(11)—C(16)—O(15)	116.7 (5)		
C(5)—C(6)—N(7)	101.5 (5)	C(11)—C(16)—O(17)	121.1 (5)		
C(3)—N(7)—C(6)	106.7 (4)	O(15)—C(16)—O(17)	122.1 (5)		
O(8)—C(9)—O(10)	117.9 (5)				

— $|F_c|$)² minimized; $w = 1.0$ for $|F_o| < 119.26$, $w = (119.26/F_o)^2$ for $|F_o| \geq 119.26$. Final $R = 0.020$, $wR = 0.021$, $S = 1.88$ for 245 variables, secondary-extinction factor (g) $9.8 (1) \times 10^{-7}$ [$|F_o| = |F_c|/(1 + gI_o)$]; $\Delta/\sigma < 0.48$ for non-H atoms, largest peak in final ΔF map $+1.3 e \text{ \AA}^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf-Nonius SDP (Frenz, 1984), ORTEPII (Johnson, 1976). The structure of the title compound is shown in Fig. 1, and the crystal packing in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are

Acta Cryst. (1990). C46, 138–140

Bromodicarbonyl(η^3 -1-phenylallyl)bis(pyrazole)molybdenum(II)

BY F. ALBERT COTTON* AND RUDY L. LUCK

Laboratory for Molecular Structure and Bonding, Texas A&M University, College Station, TX 77843, USA

(Received 27 June 1989; accepted 31 July 1989)

Abstract. [MoBr(C₅H₉)(C₃H₄N₂)₂(CO)₂], $M_r = 485.19$, triclinic, $P\bar{1}$, $a = 8.149 (1)$, $b = 9.418 (1)$, $c = 12.917 (2) \text{ \AA}$, $\alpha = 79.91 (1)$, $\beta = 80.87 (1)$, $\gamma = 80.23 (1)^\circ$, $V = 953.4 (3) \text{ \AA}^3$, $Z = 2$, $D_x = 1.690 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$, $\mu = 27.640 \text{ cm}^{-1}$, $F(000) = 476$, $T = 292 \text{ K}$, $R = 0.0689$ for 1776 unique observed reflections. The complex can be considered as octahedral if one assumes that

given in Table 1, bond distances and angles are listed in Table 2.*

Related literature. The title compound is a low-toxicity antitumor Pt complex (Mitsui, Akamatsu, Koizumi, Tsuchiya, Tomita & Matsuno, 1987; Morikawa, Honda, Matsumoto, Endoh, Akamatsu, Mitsui & Koizumi, 1988). For the preparation of the compound see Morikawa, Honda & Endoh (1987).

* Lists of anisotropic thermal parameters, H-atom coordinates, torsion angles, least-squares planes, r.m.s. amplitudes of thermal vibration and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52209 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- FRENZ, B. A. (1984). *Enraf-Nonius Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- MITSUI, H., AKAMATSU, K., KOIZUMI, M., TSUCHIYA, M., TOMITA, E. & MATSUNO, T. (1987). Presented at the 15th Int. Congr. Chemotherapy, Istanbul.
- MORIKAWA, K., HONDA, M. & ENDOH, K. (1987). *Kokai Tokkyo Koho*, 61-129289 (Japanese patent pending).
- MORIKAWA, K., HONDA, M., MATSUMOTO, T., ENDOH, K., AKAMATSU, K., MITSUI, H. & KOIZUMI, M. (1988). *Yakugaku Zasshi*, **108**, 797–800.
- NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A24, 351–359.

the 1-phenylallyl ligand occupies only one coordination site. A pyrazolyl ligand is *trans* to the 1-phenylallyl ligand and the remaining ligands (one pyrazole, one bromide and two carbon monoxide ligands) can then be described as occupying equatorial positions with the two carbon monoxide ligands *cis* to each other. Bonds to Mo are Mo—Br = 2.756 (2), Mo—N(pyrazole *trans* to 1-phenylallyl) = 2.227 (11), Mo—N(pyrazole *cis* to 1-phenylallyl) = 2.266 (12), Mo—C(CO *trans* to Br) = 1.938 (15), Mo—C(CO *cis*

* To whom correspondence should be addressed.

to Br) = 1.974 (15), average Mo—C(η^3 -1-phenylallyl) = 2.35 (6) Å.

Experimental. The title compound, which can probably be synthesized directly by an adaptation of the published procedure (tom Dieck & Friedel, 1968) for related analogues, was obtained from a reaction using stoichiometric amounts of $[\text{Na}][\text{Mo}(\text{CO})_4\{(\text{C}_3\text{H}_4\text{N}_2)_2\text{BEt}_2\}]$ and $\text{PhCHCHCH}_2\text{Br}$ in benzene. The addition of hexane to this solution resulted in the formation of small crystals; one of an irregular geometry of dimensions $0.3 \times 0.1 \times 0.1$ mm was mounted on a glass fiber. The formation of the complex may be attributed to the presence of adventitious moisture in the reaction medium since this is known to effect the decomposition of the pyrazolylborate ligand. Cell constants were derived from least-squares refinement based on 25 reflections having $25 < 2\theta < 32^\circ$. Intensity data were collected at variable scan speeds ($3\text{--}20^\circ \text{min}^{-1}$), which depended on a pre-scan count with a skip option, using the moving-crystal/moving-counter technique with $4 \leq 2\theta \leq 45^\circ$ ($h = 0$ to 9, $k = -11$ to 11, $l = -14$ to 14) on a Nicolet P3/F equivalent diffractometer using graphite-monochromated $\text{Mo K}\alpha$ radiation. Three standard reflections ($05\bar{5}$, $12\bar{7}$, $51\bar{2}$), measured every 100 reflections, showed small ($< 0.2\%$) random variations. Data were corrected for Lorentz and polarization effects, and for absorption effects based on ψ scans using the empirical method of North, Phillips & Mathews (1968); $T_{\min} = 0.8529$, $T_{\max} = 0.9998$. 2533 data were collected and averaged to 1776 unique observed reflections ($F_o^2 > 3\sigma F_o^2$); $R_{\text{merge}}(F_o) = 0.083$. Scattering factors, including anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974). All computations were carried out using the *SDP/V*

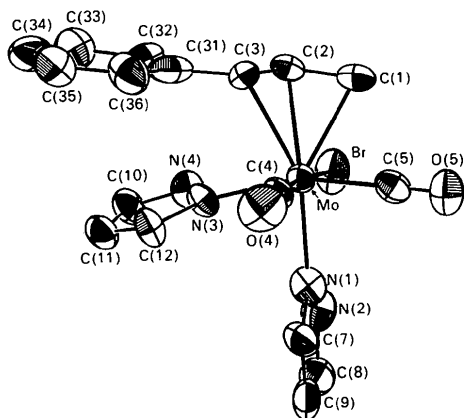


Fig. 1. An ORTEP drawing of $[\text{MoBr}(\eta^3\text{-CH}_2\text{CHCHPh})\text{(C}_3\text{H}_4\text{N}_2)_2\text{(CO)}_2]$ showing a partial atomic numbering scheme. Thermal ellipsoids have been drawn at the 50% probability level.

Table 1. Positional and equivalent isotropic thermal parameters and their e.s.d.'s for $[\text{MoBr}(\eta^3\text{-CH}_2\text{CHCHPh})(\text{C}_3\text{H}_4\text{N}_2)_2(\text{CO})_2]$

The equivalent isotropic displacement parameter is: $\frac{1}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + 2ab(\cos\gamma)B_{12} + 2ac(\cos\beta)B_{13} + 2bc(\cos\alpha)B_{23}]$.

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
Mo	0.8180 (2)	0.7638 (1)	0.3311 (1)	2.92 (2)
Br	0.9529 (3)	0.7823 (2)	0.5101 (1)	5.63 (5)
O(4)	0.704 (1)	0.713 (1)	0.1226 (9)	5.8 (3)
O(5)	0.674 (1)	0.473 (1)	0.4183 (9)	5.0 (3)
N(1)	1.059 (1)	0.619 (1)	0.2947 (9)	4.0 (3)
N(2)	1.190 (2)	0.592 (1)	0.351 (1)	4.8 (3)
N(3)	0.987 (1)	0.929 (1)	0.2490 (8)	3.3 (3)
N(4)	1.063 (1)	1.011 (1)	0.3001 (9)	3.9 (3)
C(1)	0.570 (2)	0.796 (2)	0.447 (1)	5.4 (4)
C(2)	0.615 (2)	0.935 (2)	0.393 (1)	4.8 (4)
C(3)	0.594 (2)	0.966 (2)	0.284 (1)	4.0 (4)
C(4)	0.740 (2)	0.735 (1)	0.203 (1)	3.1 (3)
C(5)	0.729 (2)	0.580 (2)	0.390 (1)	3.6 (3)
C(7)	1.109 (2)	0.545 (2)	0.213 (1)	4.4 (4)
C(8)	1.323 (2)	0.504 (2)	0.304 (2)	5.4 (5)
C(9)	1.279 (2)	0.471 (2)	0.217 (1)	4.6 (4)
C(10)	1.154 (2)	1.100 (2)	0.228 (1)	4.5 (4)
C(11)	1.144 (2)	1.074 (2)	0.127 (1)	4.3 (4)
C(12)	1.037 (2)	0.968 (2)	0.144 (1)	4.0 (4)
C(31)	0.635 (2)	1.099 (2)	0.210 (1)	4.7 (4)
C(32)	0.706 (2)	1.206 (2)	0.251 (1)	4.4 (4)
C(33)	0.737 (2)	1.337 (2)	0.177 (1)	5.4 (5)
C(34)	0.704 (2)	1.357 (2)	0.073 (1)	5.3 (4)
C(35)	0.637 (2)	1.250 (2)	0.038 (2)	7.0 (6)
C(36)	0.599 (2)	1.120 (2)	0.107 (1)	4.9 (4)

Table 2. Selected bond lengths (Å) and valence angles ($^\circ$) for $[\text{MoBr}(\eta^3\text{-CH}_2\text{CHCHPh})(\text{C}_3\text{H}_4\text{N}_2)_2(\text{CO})_2]$

Numbers in parentheses are estimated standard deviations in the least significant digits.

Mo—Br	2.756 (2)	Mo—C(4)	1.938 (15)	N(3)—N(4)	1.37 (2)
Mo—N(1)	2.227 (11)	Mo—C(5)	1.974 (15)	N(3)—C(12)	1.35 (2)
Mo—N(3)	2.266 (12)	O(4)—C(4)	1.18 (2)	C(1)—C(2)	1.45 (2)
Mo—C(1)	2.32 (2)	O(5)—C(5)	1.15 (2)	C(2)—C(3)	1.42 (2)
Mo—C(2)	2.259 (15)	N(1)—N(2)	1.35 (2)	C(3)—C(31)	1.49 (2)
Mo—C(3)	2.469 (13)	N(1)—C(7)	1.34 (2)		
Br—Mo—N(1)	83.4 (4)	C(1)—Mo—C(3)	60.4 (5)		
Br—Mo—N(3)	84.6 (3)	C(1)—Mo—C(4)	102.6 (6)		
Br—Mo—C(1)	81.4 (5)	C(1)—Mo—C(5)	66.6 (6)		
Br—Mo—C(2)	83.0 (5)	C(2)—Mo—C(3)	34.6 (6)		
Br—Mo—C(3)	114.5 (4)	C(2)—Mo—C(4)	102.6 (6)		
Br—Mo—C(4)	174.4 (4)	C(2)—Mo—C(5)	103.0 (6)		
Br—Mo—C(5)	95.3 (4)	C(3)—Mo—C(4)	71.0 (6)		
N(1)—Mo—N(3)	78.8 (4)	C(3)—Mo—C(5)	112.1 (5)		
N(1)—Mo—C(1)	145.8 (5)	C(4)—Mo—C(5)	82.9 (6)		
N(1)—Mo—C(2)	164.9 (6)	Mo—N(1)—N(2)	126. (1)		
N(1)—Mo—C(3)	153.0 (5)	Mo—N(1)—C(7)	129. (1)		
N(1)—Mo—C(4)	91.2 (5)	N(4)—N(3)—C(12)	106. (1)		
N(1)—Mo—C(5)	84.6 (5)	N(2)—N(1)—C(7)	105. (1)		
N(3)—Mo—C(1)	129.5 (5)	Mo—N(3)—N(4)	124.7 (7)		
N(3)—Mo—C(2)	93.5 (5)	Mo—N(3)—C(12)	129. (1)		
N(3)—Mo—C(3)	82.7 (4)	C(1)—C(2)—C(3)	115. (2)		
N(3)—Mo—C(4)	95.6 (5)	C(2)—C(3)—C(31)	125. (2)		
N(3)—Mo—C(5)	163.4 (5)	Mo—C(4)—O(4)	175. (1)		
C(1)—Mo—C(2)	36.8 (6)	Mo—C(5)—O(5)	176. (1)		

package of programs (B. A. Frenz & Associates, Inc., 1985). The structure was solved by direct methods and subjected to full-matrix least-squares refinement. All non-H atoms were refined anisotropically. $wR = \{[\sum w(|F_o| - |F_c|)^2] / [\sum w(F_o)^2]\}^{1/2}$ was minimized, where $w = \sigma^{-2}(|F_o|)^{-1}$; the final R value was 0.0689, $wR = 0.0959$ and $S = 1.933$ for 226 variables. The largest shift/e.s.d. in the final least-squares cycle was 0.07; the maximum residual electron density in the difference Fourier map was 1.275 e \AA^{-3} .

The molecule and the atomic labeling scheme are shown in Fig. 1. Final positional and equivalent isotropic thermal parameters are shown in Table 1;* some selected bond distances and angles are listed in Table 2.

Related literature. A review of similar complexes has been published by Davis & Kane-Maguire (1982). Structural work on related complexes has been reported by Graham, Akrigg & Sheldrick (1976, 1983, 1985) and by Graham & Fenn (1969, 1970).

These studies are supported by the National Science Foundation.

* Lists of structure factors, anisotropic thermal parameters, and complete intramolecular bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52161 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). **C46**, 140–142

Structure of (S₂N₂C—C₆H₄—CN₂S₂)²⁺·2SbF₆⁻·2C₆H₅CN

BY S. W. LIBLONG AND R. T. OAKLEY

*Guelph-Waterloo Centre for Graduate Work in Chemistry, Guelph Campus,
Department of Chemistry and Biochemistry, University of Guelph, Ontario, Canada N1G 2W1*

AND A. W. CORDES

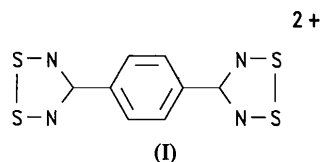
Department of Chemistry and Biochemistry, University of Arkansas, Fayetteville, AR 72701, USA

(Received 14 June 1989; accepted 28 July 1989)

Abstract. *p*-Di(1,2,3,5-dithiadiazolium-4-yl)benzene hexafluoroantimonate benzonitrile solvate, C₈H₄N₄S₄²⁺·2SbF₆⁻·2C₇H₅N, *M_r* = 962.1, monoclinic, *C*2/*c*, *a* = 11.578 (4), *b* = 22.122 (10), *c* = 13.672 (6) Å, β = 110.31 (3)°, *V* = 3284 (5) Å³, *Z* = 4, *D_x* = 1.95 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 19.9 cm⁻¹, *F*(000) = 1848, *T* = 293 K, *R* = 0.046 for 1374 reflections with *F_o*² > 3σ(*F_o*²). The cation is located on a crystallographic center of symmetry and two independent SbF₆⁻ anions are each located on twofold rotation axes. One of the anions is disordered. In the cation the terminal S₂N₂C rings are twisted 15.8° with respect to the phenylene ring. The S—S distance is 2.009 (4) Å and the average S—N, and C—N distances are 1.577 (9) and 1.34 (1) Å, respectively.

Experimental. Title salt (I) prepared by metathesis of [(S₂N₂C)C₆H₄(CN₂S₂)]²⁺·2Cl⁻ with NOSbF₆ in

PhCN. The dichloride salt itself was prepared by the reaction of (Me₃Si)₂N(Me₃SiN)C—C₆H₄—C(NSiMe₃)N(SiMe₃)₂ (Boere, Oakley & Reed, 1987) with excess SCl₂. Data crystal obtained by slow cooling of a benzonitrile solution. Yellow air-sensitive crystal approximately 0.40 × 0.40 × 0.40 mm was embedded in wax in a glass capillary. Intensities measured with an Enraf-Nonius CAD-4 diffractometer using ω-2θ scans of 8° min⁻¹ in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with 17 < 2θ < 20°. Absorption correction based on ψ scans varied from



References

- B. A. FRENZ & ASSOCIATES, INC. (1985). *SDP/V Structure Determination Package*. College Station, Texas, USA.
- DAVIS, R. & KANE-MAGUIRE, L. A. P. (1982). *Comprehensive Organometallic Chemistry*, edited by G. WILKINSON, F. G. A. STONE & E. W. ABEL, Vol. 3, pp. 1156–1159. Oxford: Pergamon Press.
- DIECK, H. TOM & FRIEDEL, H. (1968). *J. Organomet. Chem.* **14**, 375–385.
- GRAHAM, A. J., AKRIGG, D. & SHELDRICK, B. (1976). *Cryst. Struct. Commun.* **5**, 891–898.
- GRAHAM, A. J., AKRIGG, D. & SHELDRICK, B. (1983). *Acta Cryst.* **C39**, 192–194.
- GRAHAM, A. J., AKRIGG, D. & SHELDRICK, B. (1985). *Acta Cryst.* **C41**, 995–996.
- GRAHAM, A. J. & FENN, R. H. (1969). *J. Organomet. Chem.* **17**, 405–422.
- GRAHAM, A. J. & FENN, R. H. (1970). *J. Organomet. Chem.* **25**, 173–191.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.