Table 2. Bond distances (Å) and bond angles (°)

Sn—Br	2·615 (1)	P(1)—C(121)	1·842 (6)
Sn—Mn(1)	2·727 (1)	P(1)—C(131)	1·840 (5)
Sn—Mn(2)	2·718 (1)	P(2)—C(211)	1·863 (5)
Sn—Mn(3)	2·708 (1)	P(2)—C(221)	1·834 (6)
Mn(1)—C(1)	1·813 (7)	P(2)—C(231)	1·846 (4)
Mn(1)—C(2)	1·822 (7)	P(3)—C(311)	1·832 (5)
Mn(1)—C(3)	1·829 (7)	P(3)—C(321)	1·834 (4)
Mn(1)—C(4)	1·817 (7)	P(3)—C(331)	1·855 (5)
Mn(1)—P(1)	2·304 (2)	C(1)—O(1)	1·155 (9)
Mn(2)—C(5)	1·824 (8)	C(2)—O(2)	1·157 (9)
Mn(2)—C(6)	1·809 (8)	C(3)—O(3)	1·143 (8)
Mn(2)—C(7)	1·834 (8)	C(4)—O(4)	1·154 (9)
Mn(2)—C(8)	1·796 (8)	C(5)—O(5)	1·15 (1)
Mn(2)—P(2)	2·274 (2)	C(6)—O(6)	1·16 (1)
Mn(3)—C(9)	1·814 (6)	C(7)—O(7)	1·16 (1)
Mn(3)—C(10)	1·818 (6)	C(8)—O(8)	1·19 (1)
Mn(3)—C(11)	1·818 (8)	C(9)—O(9)	1·149 (7)
Mn(3)—C(12)	1·837 (7)	C(10)—O(10)	1·157 (8)
Mn(3)—P(3)	2·281 (2)	C(11)—O(11)	1·16 (1)
P(1)—C(111)	1·837 (5)	C(12)—O(12)	1·15 (1)

Br—Sn—Mn(1)	101·63 (3)	C(8)—Mn(2)—P(2)	94·3 (2)
Br—Sn—Mn(2)	102·21 (3)	Sn—Mn(3)—C(9)	89·2 (2)
Br—Sn—Mn(3)	100·03 (3)	Sn—Mn(3)—C(10)	83·4 (3)
Mn(1)—Sn—Mn(2)	117·95 (4)	Sn—Mn(3)—C(11)	93·0 (3)
Mn(1)—Sn—Mn(3)	116·73 (3)	Sn—Mn(3)—C(12)	79·7 (2)
Mn(2)—Sn—Mn(3)	114·09 (3)	Sn—Mn(3)—P(3)	170·49 (6)
Sn—Mn(1)—C(1)	85·0 (3)	C(9)—Mn(3)—C(10)	169·3 (3)
Sn—Mn(1)—C(2)	85·7 (2)	C(9)—Mn(3)—C(11)	85·7 (3)
Sn—Mn(1)—C(3)	80·4 (2)	C(9)—Mn(3)—C(12)	89·5 (3)
Sn—Mn(1)—C(4)	93·3 (3)	C(9)—Mn(3)—P(3)	94·6 (3)
Sn—Mn(1)—P(1)	167·05 (6)	C(10)—Mn(3)—C(11)	87·0 (3)
C(1)—Mn(1)—C(2)	167·5 (3)	C(10)—Mn(3)—C(12)	96·7 (3)
C(1)—Mn(1)—C(3)	94·4 (3)	C(10)—Mn(3)—P(3)	93·9 (3)
C(1)—Mn(1)—C(4)	85·6 (3)	C(11)—Mn(3)—C(12)	171·3 (3)
C(1)—Mn(1)—P(1)	95·6 (2)	C(11)—Mn(3)—P(3)	96·0 (3)
C(2)—Mn(1)—C(3)	92·2 (3)	C(12)—Mn(3)—P(3)	91·6 (2)
C(2)—Mn(1)—C(4)	86·6 (3)	Mn(1)—P(1)—C(111)	120·4 (1)
C(2)—Mn(1)—P(1)	95·4 (2)	Mn(1)—P(1)—C(121)	113·4 (2)
C(3)—Mn(1)—C(4)	173·6 (3)	Mn(1)—P(1)—C(131)	112·9 (2)
C(3)—Mn(1)—P(1)	86·7 (2)	C(111)—P(1)—C(121)	101·6 (2)
C(4)—Mn(1)—P(1)	99·7 (3)	C(111)—P(1)—C(131)	103·0 (2)
Sn—Mn(2)—C(5)	84·1 (2)	C(121)—P(1)—C(131)	103·6 (2)
Sn—Mn(2)—C(6)	92·4 (2)	Mn(2)—P(2)—C(211)	119·1 (2)
Sn—Mn(2)—C(7)	89·4 (2)	Mn(2)—P(2)—C(221)	110·7 (2)
Sn—Mn(2)—C(8)	79·9 (2)	Mn(2)—P(2)—C(231)	117·1 (2)
Sn—Mn(2)—P(2)	173·62 (7)	C(211)—P(2)—C(221)	105·2 (3)
C(5)—Mn(2)—C(6)	174·4 (3)	C(211)—P(2)—C(231)	100·2 (2)
C(5)—Mn(2)—C(7)	87·5 (3)	C(221)—P(2)—C(231)	102·6 (3)
C(5)—Mn(2)—C(8)	90·9 (4)	Mn(3)—P(3)—C(311)	112·8 (2)
C(5)—Mn(2)—P(2)	93·4 (2)	Mn(3)—P(3)—C(321)	115·2 (2)
C(6)—Mn(2)—C(7)	88·0 (4)	Mn(3)—P(3)—C(331)	118·3 (2)
C(6)—Mn(2)—C(8)	92·8 (4)	C(311)—P(3)—C(321)	105·6 (2)
C(6)—Mn(2)—P(2)	90·6 (2)	C(311)—P(3)—C(331)	102·8 (2)
C(7)—Mn(2)—C(8)	169·3 (3)	C(321)—P(3)—C(331)	100·4 (2)
C(7)—Mn(2)—P(2)	96·4 (2)		

Discussion. The molecular structure of the title compound is shown in Fig. 1; a stereoview of the molecule is given in Fig. 2. The positional parameters and the equivalent values of the anisotropic temperature factors U_{ij} [$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{23} \cos \alpha + 2U_{13} \times \cos \beta + 2U_{12} \cos \gamma)$] or the isotropic temperature factors U are given in the column headed U in Table 1. Bond lengths and angles are given in Table 2.

With the exception of the distance Sn—Mn(1) and a few angles which deviate a little more than 3σ, distances and angles in [SnBr{Mn(C$_{18}$H$_{15}$P)(CO)$_4$}$_3$] predominantly agree within 3σ with those in [SnCl{Mn-C$_{18}$H$_{15}$P)(CO)$_4$}$_3$] (Preut & Haupt, 1981). The change from Cl to Br brings about only slight changes in the molecule. The crystal structures are isomorphous. As in [SnCl{Mn(C$_{18}$H$_{15}$P)(CO)$_4$}$_3$] the Sn—Mn distances in the title compound are significantly different and shortened compared with those in [SnCl{Mn(CO)$_5$}$_3$] (Tsai, Flynn & Boer, 1967) [Sn—Mn: 2·720 (6), 2·746 (6), 2·753 (6), 2·703 (6), 2·745 (6), 2·758 (6) Å] and [SnBr{Mn(CO)$_5$}$_3$] (Haupt, Preut & Wolfes, 1978) [Sn—Mn: 2·739 (2), 2·722 (2), 2·758 (2) Å].

Intermolecular distances do not indicate interactions exceeding van der Waals forces.

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Tribromotris(4-methylpyridine)molybdenum(III)

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Abstract. [MoBr$_3$(C$_6$H$_7$N)$_3$], C$_{18}$H$_{21}$Br$_3$MoN$_3$, triclinic, $P\bar{1}$, $a = 9\cdot193$ (1), $b = 16\cdot379$ (2), $c = 17\cdot319$ (3) Å, $\alpha = 61\cdot82$ (2), $\beta = 80\cdot08$ (2), $\gamma = 75\cdot93$ (2)°, $Z = 4$, $D_o = 1\cdot81$ (3) (by flotation in dibromoethane/carbon tetrachloride), $D_c = 1\cdot836$ Mg m^{-3}; $R = 0\cdot079$ for 3408 reflections [$I > 5\sigma(I)$]. The

structure contains two independent [MoBr₃pic₃] [pic = 4-methylpyridine (4-picoline)] octahedra. The 4-methyl-pyridines within the octahedra occupy meridional positions. Average Mo—Br and Mo—N bonding distances are 2·57 (2) and 2·21 (3) Å. This is the second polymorphic form of this compound.

Introduction. MoX_3L_3 stoichiometry is predominant in Mo^{III} coordination chemistry. Several crystal structures are already known: $MoCl_3py_3$ (py = pyridine) (Brenčič 1974), $MoBr_3pic_3$ (Brenčič & Leban, 1978) as well as two 4-methylpyridine adducts of MoX_3pic_3, $MoCl_3pic_3 \cdot 0.5pic$ (Brenčič, & Leban, 1980) and $MoBr_3pic_3 \cdot 0.5pic$ (Brenčič, Leban & Slokar, 1980). All compounds contain MoX_3L_3 octahedra and have the ligands in the *trans* position.

The recrystallization of the crude product obtained from the reaction between $(NH_4)_2MoBr_5 \cdot H_2O$ and 4-methylpyridine from a 1:8 solution of 4-methyl-pyridine in methanol gave three crystalline products. Two of them were structurally identified as $MoBr_3pic_3$ (Brenčič & Leban, 1978) with the monoclinic space group and $MoBr_3pic_3 \cdot 0.5pic$ (Brenčič, Leban & Slokar, 1980). The minute amounts of the third product which was present in the form of needle-shaped crystals were separated by hand picking under the microscope; the only way to solve the problem of its chemical identity was crystal structure analysis.

The small crystal used for the structural analysis had the dimensions $0.15 \times 0.20 \times 0.35$ mm.

The unit-cell dimensions were determined from the least-squares analysis of 32 randomly chosen reflections for which 2θ values were measured on the diffractometer at 293 (2) K.

The intensity data were collected at the same temperature with an Enraf–Nonius CAD-4 diffractometer. Experimental conditions were: graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å), ω–2θ scan, $\theta_{min} = 1°$, $\theta_{max} = 25°$, 2θ scan width (°) = 0·9 + 0·3 tan θ, aperture (mm) = 2·5 + 0·9 tan θ, maximum scan time = 40 s, background: $\frac{1}{4}$ of the scan time at each of the scan limits; 5991 independent reflections were measured. Reflections with $I < 5\sigma(I)$ were rejected and the remaining 3408 were used for the solution and refinement of the structure. Structure factors on a relative scale were obtained by applying Lorentz and polarization corrections. No absorption correction was applied [μ(Mo $K\alpha = 5.8$ mm⁻¹)].

Multisolution Σ_2 sign expansion (Sheldrick, 1976) located molybdenum and bromine atoms. 4-Methyl-pyridine rings were found from a Fourier calculation using the $|F_o|$'s with signs taken from the $|F_c|$'s based on the heavy-atom positions. The structure factors included the real and imaginary components of anomalous scattering.

The function minimized in the least-squares calculations was: $\sum w(|F_o| - |F_c|)^2$. The empirical

Table 1. *Positional* ($\times 10^4$) *and thermal* ($\times 10^2$) *parameters for* Mo *and* Br *atoms with standard deviations*

$$B_{eq} = \frac{4}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	B_{eq} (Å²)
Mo(1)	6787 (2)	1996 (1)	2166 (1)	2·94 (11)
Br(11)	4970 (3)	3375 (2)	2352 (2)	4·17 (16)
Br(12)	7636 (3)	1328 (2)	3729 (2)	4·70 (16)
Br(13)	8530 (3)	660 (2)	1896 (2)	4·31 (16)
Mo(2)	−364 (2)	6865 (1)	2655 (1)	3·05 (12)
Br(21)	1805 (3)	5710 (2)	3606 (2)	4·05 (16)
Br(22)	482 (4)	6365 (2)	1436 (2)	6·51 (22)
Br(23)	−2644 (3)	8068 (2)	1841 (2)	4·77 (18)

Table 2. *Positional parameters* ($\times 10^4$) *and thermal parameters* ($\times 10^3$) *for* C *and* N *atoms with standard deviations*

The isotropic temperature factors are in the form
$$\exp[-B(\sin\theta/\lambda)^2].$$

	x	y	z	B (Å²)
N(11)	4932 (20)	1210 (12)	2651 (11)	3·7 (4)
C(11)	5190 (30)	290 (18)	3289 (16)	7·1 (7)
C(2)	4080 (29)	−249 (18)	3685 (16)	6·2 (7)
C(3)	2637 (26)	118 (16)	3403 (14)	4·6 (6)
C(4)	2453 (29)	1003 (17)	2713 (16)	5·1 (6)
C(5)	3423 (26)	1533 (16)	2382 (14)	4·8 (6)
C(6)	1381 (36)	−453 (22)	3819 (19)	6·5 (9)
N(12)	6091 (20)	2594 (12)	826 (11)	4·0 (4)
C(7)	5722 (23)	2088 (14)	518 (12)	4·0 (5)
C(8)	5212 (33)	2489 (20)	−316 (18)	5·8 (8)
C(9)	5212 (23)	3441 (14)	−892 (13)	3·7 (5)
C(10)	5579 (25)	3955 (15)	−579 (13)	4·1 (5)
C(11)	5980 (25)	3545 (15)	273 (13)	3·8 (5)
C(12)	4808 (35)	3895 (21)	−1855 (19)	8·2 (9)
N(13)	8586 (20)	2808 (12)	1653 (11)	3·9 (5)
C(13)	9537 (27)	2796 (16)	936 (14)	4·3 (6)
C(14)	10741 (28)	3347 (17)	589 (15)	5·0 (6)
C(15)	10857 (27)	3910 (16)	959 (14)	4·6 (6)
C(16)	9981 (30)	3910 (18)	1637 (17)	5·3 (7)
C(17)	8859 (28)	3346 (16)	2005 (15)	5·8 (6)
C(18)	12137 (30)	4487 (18)	574 (16)	6·0 (7)
N(21)	−1791 (20)	5745 (12)	3307 (11)	3·7 (4)
C(19)	−1134 (25)	4843 (15)	3448 (14)	3·7 (5)
C(20)	−1911 (29)	4113 (17)	3830 (16)	5·1 (7)
C(21)	−3499 (33)	4290 (20)	4121 (18)	6·9 (8)
C(22)	−4027 (30)	5212 (18)	3990 (16)	5·4 (7)
C(23)	−3117 (33)	5942 (20)	3590 (18)	6·2 (8)
C(24)	−4366 (45)	3521 (27)	4545 (25)	8·1 (9)
N(22)	−1075 (20)	7289 (12)	3705 (11)	3·8 (5)
C(25)	−1307 (26)	6656 (15)	4544 (14)	4·2 (6)
C(26)	1769 (28)	6907 (17)	5244 (15)	4·7 (7)
C(27)	−1804 (29)	7874 (17)	5035 (16)	5·2 (7)
C(28)	−1650 (25)	8481 (15)	4199 (13)	4·0 (5)
C(29)	−1316 (26)	8206 (16)	3539 (14)	4·1 (6)
C(30)	−2215 (37)	8191 (23)	5760 (20)	8·7 (9)
N(23)	1070 (23)	7874 (14)	2028 (13)	5·0 (5)
C(31)	2005 (29)	8065 (17)	2480 (16)	5·0 (7)
C(32)	2766 (31)	8780 (19)	2067 (17)	6·3 (8)
C(33)	2627 (38)	9442 (23)	1164 (21)	5·8 (10)
C(34)	1694 (33)	9325 (20)	718 (18)	6·6 (8)
C(35)	999 (30)	8584 (18)	1141 (17)	6·1 (7)
C(36)	3385 (36)	10269 (22)	771 (19)	9·5 (9)

Table 3. *Dimensions of the* $MoBr_3pic_3$ *octahedra with standard deviations in parentheses*

Bond lengths (Å)

Mo(1)–Br(11)	2·582 (3)	Mo(1)–N(11)	2·197 (20)
Mo(1)–Br(12)	2·579 (3)	Mo(1)–N(12)	2·195 (18)
Mo(1)–Br(13)	2·565 (4)	Mo(1)–N(13)	2·178 (20)
Mo(2)–Br(21)	2·574 (3)	Mo(2)–N(21)	2·251 (19)
Mo(2)–Br(22)	2·548 (5)	Mo(2)–N(22)	2·187 (22)
Mo(2)–Br(23)	2·579 (3)	Mo(2)–N(23)	2·242 (22)

Bond angles (°)

Br(11)–Mo(1)–Br(13)	176·7 (1)	Br(11)–Mo(1)–N(11)	88·4 (5)
Br(12)–Mo(1)–N(12)	178·6 (6)	Br(12)–Mo(1)–N(11)	92·0 (5)
N(11)–Mo(1)–N(13)	178·3 (6)	Br(13)–Mo(1)–N(11)	91·2 (5)
		N(12)–Mo(1)–N(11)	89·2 (7)
Br(11)–Mo(1)–Br(12)	90·4 (1)	Br(11)–Mo(1)–N(13)	90·8 (5)
Br(12)–Mo(1)–Br(13)	92·9 (1)	Br(12)–Mo(1)–N(13)	89·5 (5)
Br(13)–Mo(1)–N(12)	87·6 (5)	Br(13)–Mo(1)–N(13)	89·6 (5)
N(12)–Mo(1)–Br(11)	89·1 (5)	N(12)–Mo(1)–N(13)	89·3 (7)
Br(21)–Mo(2)–Br(23)	173·8 (2)	Br(21)–Mo(2)–N(21)	89·9 (4)
Br(22)–Mo(2)–N(22)	179·6 (3)	Br(22)–Mo(2)–N(21)	89·6 (6)
N(21)–Mo(2)–N(23)	179·0 (8)	Br(23)–Mo(2)–N(21)	89·7 (4)
		N(22)–Mo(2)–N(21)	90·4 (7)
Br(21)–Mo(2)–Br(22)	93·4 (1)	Br(21)–Mo(2)–N(23)	89·4 (5)
Br(22)–Mo(2)–Br(23)	92·8 (1)	Br(22)–Mo(2)–N(23)	91·1 (7)
Br(23)–Mo(2)–N(22)	87·6 (4)	Br(23)–Mo(2)–N(23)	91·0 (5)
N(22)–Mo(2)–Br(21)	86·2 (4)	N(22)–Mo(2)–N(23)	88·9 (8)

Br···Br and Br···N contacts (Å)

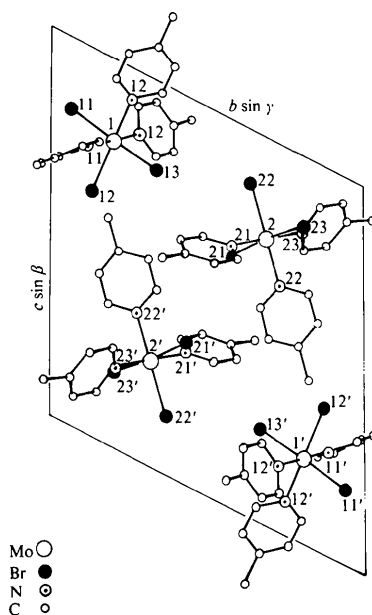|			
Br(11)–Br(12)	3·66	Br(21)–Br(22)	3·73
Br(11)–N(11)	3·34	Br(21)–N(21)	3·42
Br(11)–N(13)	3·40	Br(21)–N(23)	3·39
Br(11)–N(12)	3·36	Br(21)–N(22)	3·27
Br(12)–N(11)	3·45	Br(22)–N(21)	3·39
Br(12)–N(13)	3·36	Br(22)–N(23)	3·42
Br(12)–Br(13)	3·73	Br(22)–Br(23)	3·71
Br(13)–N(12)	3·31	Br(23)–N(22)	3·31
Br(13)–N(11)	3·41	Br(23)–N(21)	3·41
Br(31)–N(13)	3·35	Br(23)–N(23)	3·44
N(12)–N(13)	3·07	N(22)–N(23)	3·10
N(12)–N(11)	3·08	N(22)–N(21)	3·15

weighting scheme was $w = 2·182/\{[\sigma^2(F) + 0·004F^2]\}$. The final R values are: $R = \sum(|F_o| - |F_c|)/\sum|F_o| = 0·079$ and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum wF_o^2]^{1/2} = 0·085$. The final difference Fourier map showed only insignificant ripples around Mo and Br atoms. The average shift/error in the last cycle was 0·5.

All calculations were performed on the CDC Cyber-172 computer of RRC Ljubljana with the *SHELX*76 system of crystallographic programs (Sheldrick, 1976).

Atomic scattering factors for neutral atoms tabulated by Cromer & Mann (1968) and the values for f' and f'' for the anomalous-dispersion correction, compiled by Cromer (1965), were applied.

The positional and thermal parameters for Mo and Br atoms are listed in Table 1, and for the other atoms in Table 2.* Interatomic distances and angles are in Table 3. The packing of the molecules in the unit cell,

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Fig. 1. Projection of the structure along the a axis.

including the numbering scheme used for the $MoBr_3N_3$ skeleton, can be seen in Fig. 1.

Discussion. $MoBr_3pic_3$ is known in two polymorphic forms. The unit cell of the monoclinic form contains four equivalent $MoBr_3pic_3$ octahedra (Brenčič & Leban, 1978). The triclinic unit cell contains two pairs of unrelated $MoBr_3pic_3$ octahedra.

Average Mo–Br and Mo–N distances are 2·57 (1) and 2·21 (2) Å. All angles within the octahedra are close to 90 and 180°. The values are close to the ones found in the monoclinic $MoBr_3pic_3$ (Brenčič & Leban, 1978) and $MoBr_3pic_3$.0·5pic (Brenčič, Leban & Slokar, 1980). The short Mo(2)–N(22) bond length could be the result of neglecting the absorption effect. The volumes of the unit cells of the monoclinic (2205 Å³) and triclinic (2225 Å³) forms are equal within 1%. The constancy of the space occupied by $MoBr_3pic_3$ is evident.

All contacts between the octahedra are rather long. The shortest is the contact from $C(12)(x,y,z)$ of the first octahedron to $C(22)(-x, 1 - y, -z)$ of the second [3·41 (2) Å]. None of the carbon to bromine contacts between the octahedra is shorter than 3·65 Å.

Average C–C, C–C(CH₃), N–C distances and C–N–C, N–C–C, C–C–C angles of the 4-methylpyridine rings are 1·38 (3), 1·52 (4), 1·36 (3) Å, 117 (3), 122 (3) and 120 (9)°. All 4-methylpyridine planes, including the methyl carbon atom, are planar within 0·03 Å. They are oriented in a propeller fashion about the linear Br–Mo–Br axis. The planes of the *trans*-4-methylpyridines form angles close to 90°.

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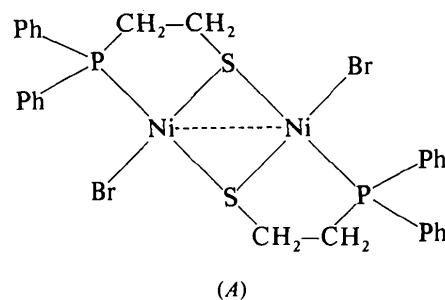# Dibromobis{μ-[2-(diphenylphosphino)ethanethiolato]-P,μ-S}-dinickel(II)*

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Abstract. $Ni_2Br_2(C_{14}H_{14}PS)_2$, $C_{28}H_{28}Br_2Ni_2P_2S_2$, $M_r = 767\cdot83$, monoclinic, $C2/c$, $a = 21\cdot748$ (2), $b = 9\cdot289$ (1), $c = 15\cdot473$ (1) Å, $\beta = 108\cdot041$ (5)°, $V = 2972$ Å^3, $Z = 4$, $D_m = 1\cdot69$, $D_x = 1\cdot716$ Mg m^{-3}, $R = 0\cdot043$ for 2029 observed reflexions. In the complex dibromo{μ-[2-(diphenylphosphino)ethanethiolato]-P,μ-S}-dinickel(II) the 2-(diphenylphosphino)ethanethiolate ion, $[(C_6H_5)_2PCH_2CH_2S]^-$, ps$^-$, acts as a bidentate ligand, the P and S atoms forming a five-membered non-planar ring (NiPC$_2$S). Br is coordinated to Ni cis to P. The S atoms bridge two NiBr(ps) units giving a dinuclear uncharged complex. A square plane is formed around each Ni by one Br, one P and two S atoms. The angle between these planes, which are connected by an S—S edge, is 100·7 (2)° giving a short Ni—Ni distance, 2·695 (2) Å, somewhat longer than the metallic Ni—Ni distance of 2·492 (12) Å [Pauling (1960). The Nature of the Chemical Bond. New York: Cornell Univ. Press]. All other interatomic distances and angles are normal. The compound is diamagnetic.

Introduction. The title compound (see schematic diagram A) in the form of dark-red crystals prepared by the late Professor G. Schwarzenbach (1965) was used for intensity measurements on a four-circle diffractometer (Enraf–Nonius CAD-4) at room temperature.

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* Structures of Complexes between Metal Halides and Phosphinothioethers or Related Ligands. XIII.

(A)

The cell parameters were refined from 49 accurately measured θ values. The space group, determined from Weissenberg photographs, is $C2/c$ or Cc. Data concerning the collection and reduction of the intensities are given in Table 1. Corrections were made for Lorentz–polarization and absorption effects. No systematic change was observed in the three standard reflexions which were measured every second hour. The positions of Br, Ni, P and S were determined with MULTAN (Germain, Main & Woolfson, 1971) and the C atoms from electron-density maps, space group $C2/c$ being assumed. The preliminary parameters were refined with SHELX (Sheldrick, 1976) minimizing $\sum w(|F_o| - |F_c|)^2$ with $w = K/[\sigma(F) + GF^2]$. The refined value $G = 0\cdot00022$ gave a satisfactory ΔR plot (Abrahams & Keve, 1971). The phenyl rings were treated as rigid groups (C—C 1·395 Å). The positions of the H atoms were geometrically generated at the end