Crystal Structures of Tris(diethyldithiocarbamato)-rhodium(III) and -arsenic(III)

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The crystal structures of the title compounds, $[Rh(CS_2{\cdot}NEt_2)_3]$, (I), and $[As(CS_2{\cdot}NEt_2)_3]$, (II) have been determined by *X*-ray diffraction from diffractometer data, and refined by least-squares to *R* 0.080 (I) and 0.058 (II) for 1 582 and 1 877 reflections. Crystals are monoclinic, space group $P2_1/a$, with $Z = 4$ in unit cells of dimensions: (I) $a = 18.07(3)$, $b = 8.276(3)$, $c = 15.80(1)$ Å, $\beta = 96.35(4)°$; (II) $a = 18.543(4)$, $b = 8.387(2)$, $c = 15.870(3)$ Å, $\beta = 101.05(2)°$. The compounds are isomorphous also with the manganese(III) derivative; whereas the MS_6 core in (I) is quite close to D_3 symmetry (mean Rh–S 2.36_8 Å), that of (II) is grossly distorted (as previously described) toward C_3 symmetry (mean As–S 2.84 and 2.34_9 Å). In (II), the usual equality in the ligand C–S distances is destroyed as a result (mean C–S 1.68 and 1.76 Å) and the surrounding angular geometry of the ligand also distorted. The arsenic has a close hydrogen contact (3.2 Å) along the pseudo three-fold axis from a neighbouring molecule.

RELATED studies[1] on the tris(diethyldithiocarbamato)-metal(III) derivatives, $[M(CS_2{\cdot}NEt_2)_3]$, have shown a widespread tendency for the derivatives to crystallize in a monoclinic unit cell *ca.* 14.9 × 10.3 × 18.0 Å, $\beta = 118°$. Subtle differences in crystal packing occur as the metal–sulphur distance *r* changes so that although a somewhat similar molecular array is common to all forms, we find that in derivatives with $r <$ *ca.* 2.36 Å (Co, Fe, Ir) the cell symmetry is $C2/c$, while if $r >$ *ca.* 2.43 Å it is $A2/a$ (Ga, In); in the intermediate range (*r* 2.36—2.40 Å) a $P2_1/c$ lattice is found (Fe, Cr, Ru). Exceptions to this are found in derivatives with electronically distorted MS_6 cores (Mn and As). In the course of our work the rhodium(III) derivative was examined and found to crystallize in two forms from chloroform–cyclohexane. One form appeared similar to that with the centred lattice, but photographic examination of the well-formed crystals showed symptoms of considerable disorder or thermal diffuse scattering and it was considered fruitless to pursue it further. A second form was isolated as thin small laths; on examination, this form proved to be similar in its cell dimensions to the Mn[2] and As[3] derivatives. Since solution of the structure showed no distortion of the MS_6 core comparable to the diverse and large distortions found for the Mn and As derivatives, it was considered to be of interest to compare the three derivatives. The structure of $[As(CS_2{\cdot}NEt_2)_3]$ has been reported previously,[3] but there were no details of the cell contents, so a redetermination was carried out. $[Rh(CS_2{\cdot}NEt_2)_3]$ (I) and $[As(CS_2{\cdot}NEt_2)_3]$ (II) were prepared as described previously[4,5] and recrystallized from chloroform–cyclohexane and acetone respectively yielding small elongated crystals. Crystal dimensions of the samples used in the crystallographic work were 0.07 × 0.12 × 0.07 mm (I) and 0.07 × 0.24 × 0.07 mm (II).

EXPERIMENTAL

Unit cells were calibrated by a least-squares fit of the angular parameters of 15 reflections centred in the counter aperture of a Syntex $P\bar{1}$ four-circle diffractometer. A unique set of data was gathered for both structures in the limit $2\theta < 40°$ by a conventional 2θ—θ scan (the limit being basically determined by the rather small crystals available), yielding 2 171 (I) and 2 277 (II) independent reflections of which 1 582 (I) and 1 877 (II) with $I > 2\sigma(I)$ were used in the structure solution and refinement; data for (II) were corrected for absorption.

Crystal Data [*values for* (II) *follow those for* (I)].—$C_{15}H_{30}N_3S_6M$, $M = 547.7$, 519.7, Monoclinic, $a = 18.07(3)$, 18.543(4); $b = 8.276(3)$, 8.387(2); $c = 15.80(1)$, 15.870(3) Å; $\beta = 96.35(4)$, $101.05(2)°$; $U = 2\,349(2)$, $2\,422(1)$ Å^3,

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[1] C. L. Raston and A. H. White, *J.C.S. Dalton*, to be published.

[2] P. C. Healy and A. H. White, *J.C.S. Dalton*, 1972, 1883.

[3] M. Colapietro, A. Domenicano, L. Scaramuzza, A. Vaciago, and L. Zambonelli, *Chem. Comm.*, 1968, 302.

[4] L. Malatesta, *Gazzetta*, 1938, **68**, 195.

[5] L. Malatesta, *Gazzetta*, 1939, **69**, 629.

$D_m = 1.57(1), 1.41(1)$; $Z = 4$; $D_c = 1.55, 1.41$; $F(000) = 1\,128, 1\,080$. Mo-K_α radiation, $\lambda = 0.710\,69$ Å; μ(Mo-K_α) = 12.2, 19.6 cm^{-1}. Space group $P2_1/a$, C_{2h}^5, No. 14. Neutral atom scattering factors,[6,7] those for arsenic, rhodium, and sulphur being corrected for anomalous dispersion ($\Delta f'$, $\Delta f''$).[8]

TABLE 1

Atom fractional cell (decimal point omitted from x, y, z) and thermal parameters ($\times 10^3$Å^2), with least-squares estimated standard deviations in parentheses. Values for (II) are given below those for (I)

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rh	3 760(1)	2 379(2)	2 472(1)	34(2)	34(2)	29(2)	−1(1)	−2(1)	−3(1)
As	37 921(8)	1 955(2)	2 591(9)	47(2)	37(2)	33(2)	4(1)	1(1)	0(1)
Ligand (1)									
S(1)	2 474(3)	2 052(8)	2 043(4)	42(4)	50(5)	32(4)	6(3)	2(3)	9(3)
	2 271(2)	1 636(5)	1 962(2)	53(3)	72(4)	47(3)	8(2)	13(2)	10(2)
S(2)	3 586(3)	2 882(8)	0 993(4)	36(4)	47(5)	38(4)	−2(3)	2(3)	5(3)
	3 493(2)	2 687(5)	1 140(2)	42(3)	49(3)	36(3)	3(2)	2(2)	3(2)
C(1)	266(1)	258(3)	106(1)	41(6)					
	2 551(7)	231(2)	1 093(8)	37(9)	52(11)	33(9)	−2(8)	4(7)	−6(8)
N	2 132(9)	280(2)	039(1)	35(5)					
	2 098(6)	259(1)	0 343(7)	43(7)	38(8)	34(7)	−5(6)	5(6)	−4(6)
C(2)	134(1)	236(3)	045(1)	55(7)					
	1 307(7)	229(2)	0 214(9)	34(9)	74(13)	70(11)	7(8)	7(8)	14(9)
C(3)	085(2)	383(4)	046(2)	80(9)					
	0 867(8)	374(2)	037(1)	54(11)	134(18)	60(11)	22(11)	9(9)	12(11)
C(4)	234(1)	342(3)	−045(1)	39(6)					
	2 340(7)	329(2)	−0 406(8)	61(9)	22(9)	46(9)	−12(8)	−1(8)	3(7)
C(5)	244(1)	208(3)	−107(1)	55(7)					
	2 457(8)	203(2)	−1 045(9)	66(11)	76(12)	57(10)	11(10)	11(9)	−2(9)
Ligand (2)									
S(1)	4 062(3)	−0 372(8)	2 318(4)	31(4)	38(5)	52(4)	1(4)	−1(3)	3(3)
	4 229(2)	−1 087(5)	2 136(2)	46(3)	40(3)	59(3)	−2(2)	3(2)	0(2)
S(2)	5 077(3)	2 225(8)	2 632(4)	36(4)	40(5)	47(4)	−3(3)	2(3)	2(5)
	5 054(2)	1 931(5)	2 548(2)	45(3)	37(3)	53(3)	1(2)	−1(2)	−7(2)
C(1)	499(1)	015(3)	253(1)	33(6)					
	5 046(8)	−016(2)	2 401(8)	62(12)	38(9)	35(9)	12(9)	6(8)	−1(8)
N	5 551(9)	−088(2)	254(1)	36(5)					
	5 682(6)	−089(1)	2 524(7)	43(8)	36(8)	54(8)	2(7)	11(6)	14(6)
C(2)	541(1)	−261(3)	246(1)	44(6)					
	5 708(8)	−267(2)	245(1)	58(11)	46(11)	87(12)	18(9)	−5(9)	11(9)
C(3)	542(2)	−322(4)	153(2)	86(9)					
	563(1)	−326(2)	153(1)	89(14)	78(14)	115(16)	−1(12)	7(12)	−39(13)
C(4)	634(1)	−025(3)	274(1)	38(6)					
	6 391(7)	−012(2)	2 771(9)	34(9)	68(12)	56(10)	8(8)	8(8)	18(9)
C(5)	658(1)	−036(3)	370(2)	58(7)					
	6 664(8)	−006(2)	373(1)	45(10)	98(15)	70(12)	−8(10)	3(9)	−10(11)
Ligand (3)									
S(1)	3 714(3)	2 461(8)	3 970(3)	52(4)	46(5)	35(4)	7(4)	−4(3)	1(4)
	3 940(3)	2 468(5)	4 426(3)	119(4)	48(3)	44(3)	3(3)	13(3)	9(2)
S(2)	3 667(3)	5 123(8)	2 841(3)	62(5)	43(5)	25(4)	5(4)	−2(3)	−2(3)
	3 857(2)	4 678(4)	2 946(2)	78(3)	37(3)	33(3)	4(2)	6(2)	−2(2)
C(1)	369(1)	454(3)	387(1)	32(6)					
	3 931(8)	438(2)	4 048(9)	49(10)	41(10)	41(10)	2(8)	7(8)	−3(8)
N	373(1)	557(2)	456(1)	51(6)					
	3 994(6)	566(1)	4 556(7)	47(8)	55(9)	34(7)	6(7)	3(6)	−13(7)
C(2)	379(1)	493(3)	544(1)	55(7)					
	4 048(8)	545(2)	5 486(8)	80(12)	70(12)	27(9)	1(10)	20(8)	−12(8)
C(3)	308(1)	513(4)	586(2)	71(8)					
	3 326(8)	552(2)	577(1)	100(14)	102(16)	56(11)	10(12)	38(10)	26(10)
C(4)	370(1)	737(3)	441(1)	53(7)					
	4 005(8)	729(2)	4 210(9)	52(10)	67(12)	62(11)	11(9)	14(8)	5(9)
C(5)	448(1)	810(3)	460(2)	70(8)					
	328(1)	799(3)	395(1)	94(16)	66(15)	104(18)	−16(13)	38(13)	−8(14)

The ligand numbering follows that given in C. L. Raston and A. H. White, *J.C.S. Dalton*, 1975, 2418.

The structures of (I) and (II) were determined by the heavy-atom method and refined by 9 × 9 block-diagonal least-squares; in the final stages of refinement, the parameters of the $MS_6C_3N_3$ inner molecular core were refined jointly to approximate to a full-matrix procedure. In (II), hydrogen atoms were located in a difference map and their positional parameters refined, isotropic thermal parameters U 0.10 Å^2 for the methyl and 0.07 Å^2 for the methylene hydrogens being fixed as invariants. For the remainder of the structures [excluding the non-RhS_6 atoms in (I), which are refined isotropically] anisotropic thermal parameters of the form $\exp(-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*))$ were employed. Refinement converged at R 0.080 (I), 0.058

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[6] D. T. Cromer and J. B. Mann, *Acta Cryst.*, 1968, **A24**, 321.

[7] R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

[8] D. T. Cromer, *Acta Cryst.*, 1965, **18**, 7.

TABLE 2

Interatomic distances (Å) and angles (°), with least squares estimated standard deviations in parentheses

(*a*) MS_6 core angles; values for (II) follow those for (I)

S(11)–M–S(21)	95.2(2), 98.1(1)	S(11)–M–S(22)	165.7(2), 157.2(1)
S(11)–M–S(31)	98.4(2), 105.3(1)	S(21)–M–S(32)	168.3(2), 159.9(1)
S(21)–M–S(31)	99.4(2), 114.5(1)	S(31)–M–S(12)	164.8(2), 154.8(1)
S(12)–M–S(22)	97.9(2), 91.4(1)	S(11)–M–S(32)	95.0(2), 100.3(1)
S(12)–M–S(32)	94.0(2), 88.4(1)	S(21)–M–S(12)	94.3(2), 90.7(1)
S(22)–M–S(32)	97.1(2), 90.5(2)	S(31)–M–S(22)	92.3(2), 97.3(1)

(*b*) Intraligand distances and angles

	(I)			(II)		
Ligand	(1)	(2)	(3)	(1)	(2)	(3)
M–S(1)	2.364(7)	2.360(7)	2.379(7)	2.819(4)	2.812(4)	2.904(4)
M–S(2)	2.360(6)	2.369(7)	2.355(7)	2.344(4)	2.354(4)	2.350(4)
S(1)–M–S(2)	73.6(2)	73.6(2)	73.4(2)	69.2(1)	69.4(1)	68.0(1)
S(1)–C(1)	1.69(2)	1.74(2)	1.73(2)	1.67(1)	1.68(1)	1.71(1)
S(2)–C(1)	1.71(2)	1.73(2)	1.70(2)	1.76(1)	1.77(1)	1.75(1)
M–S(1)	86.8(7)	88.3(7)	86.8(7)	79.1(4)	78.8(5)	78.2(5)
M–S(2)–C(1)	86.6(7)	88.2(7)	88.4(8)	92.4(4)	91.6(5)	95.2(5)
S(1)–C(1)–S(2)	113(1)	110(1)	111(1)	119.3(7)	118.5(9)	118.6(8)
C(1)–N	1.36(2)	1.32(3)	1.37(3)	1.34(2)	1.31(2)	1.34(2)
S(1)–C(1)–N	124(2)	124(2)	123(2)	124(1)	124(1)	123(1)
S(2)–C(1)–N	123(2)	126(2)	125(2)	117(1)	118(1)	118(1)
N–C(2)	1.50(3)	1.46(3)	1.49(3)	1.46(2)	1.49(2)	1.47(2)
N–C(4)	1.50(3)	1.51(3)	1.51(3)	1.47(2)	1.45(2)	1.47(2)
C(2)–N–C(4)	119(1)	121(2)	120(2)	114(1)	115(1)	119(1)
C(1)–N–C(2)	120(2)	120(2)	120(2)	122(1)	120(1)	119(1)
C(1)–N–C(4)	121(2)	119(2)	120(2)	123(1)	125(1)	122(1)
C(2)–C(3)	1.51(4)	1.56(4)	1.52(4)	1.51(2)	1.53(2)	1.49(2)
N–C(2)–C(3)	112(2)	113(2)	113(2)	114(1)	113(1)	114(1)
C(4)–C(5)	1.50(3)	1.54(3)	1.54(3)	1.51(2)	1.51(2)	1.44(3)
N–C(4)–C(5)	112(2)	110(2)	110(2)	112(1)	114(1)	114(1)
S(1) · · · S(2)	2.830(9)	2.833(9)	2.830(9)	2.957(6)	2.965(5)	2.972(6)

(*c*) Geometry about As · · · H(35c) [at x, $y - 1$, z = 0.332, −0.084, 0.375)] in (II)

H(35c) · · · As	3.2(2)	S(21)–As · · · H(35c)	67.0(3)
S(11)–As · · · H(35c)	77.0(2)	S(31)–As · · · H(35c)	61.0(3)
H(35c) · · · S(11)	3.7(2)	H(35c) · · · S(31)	3.1(2)
H(35c) · · · S(21)	3.3(2)		

(II), $R' = 0.086$ (I) and 0.061 (II) $[R' = (\Sigma w||F_o| - |F_c||^2/\Sigma w|F_o|^2)^{\frac{1}{2}}]$ structure (II) being rather more accurately determined than (I) generally because of the somewhat more intense data. A weighting scheme of the form $w = (\sigma^2(F_o) + n \times 10^{-4}(F_c)^2)^{-1}$ was found appropriate with $n = 5$ (I) and $n = 4$ (II).

Structure amplitudes and hydrogen atom parameters are deposited as Supplementary Publication No. SUP 21451 (19 pp., 1 microfiche).* Computation was carried out on our CDC 6 200 with a local variant of the *X*-Ray program system.[9] Results are shown in Tables 1—3 and Figures 1—3.

DISCUSSION

Both lattices consist of discrete molecules of $[M(CS_2 \cdot NEt_2)_3]$; the two complexes are isomorphous with each other and with the manganese(III) derivative, although specific and important differences between the three complexes are found, both in molecular geometry and in crystal packing. As in the manganese(III) derivative, denoted (III) hereafter, each molecule consists of a metal atom co-ordinated by three quasi-bidentate ligands. As usual, the S_2CNC_2 sections of the ligand are substantially planar, and in the present structural type we find that the ligand planes lie approximately parallel

TABLE 3

Equations of least-squares planes through the ligands (defined by the S_2CNC_2 fragment) in the form $pX + qY + rZ = s$, where in the orthogonal (Å) frame $X = ax + cz \cos \beta$, $Y = by$, and $Z = cz \sin \beta$. σ Å is the estimated standard deviation of the defining atoms, and χ^2 is given for 5 degrees of freedom. Values for (II) follow those for (I)

Ligand	(1)	(2)	(3)
10^4p	−1 583, −1 974	−1 553, −2 272	9 983, 9 932
10^4q	9 507, 9 322	−0 956, −1 119	0 155, −0 366
10^4r	2 666, 3 034	9 832, 9 674	0 563, 1 102
s	1.751, 1.459	2.539, 1.688	6.406, 6.586
σ	0.06, 0.03	0.02, 0.02	0.03, 0.01
χ^2	62.0, 44.3	4.83, 10.4	8.25, 7.57
M *	0.15, 0.06	0.10, 0.61	0.20, 0.00
Deviations (Å)			
S(1)	0.07, 0.03	−0.01, 0.00	−0.01, 0.02
S(2)	−0.07, −0.03	0.00, 0.02	0.03, −0.01
C(1)	−0.01, 0.00	0.03, −0.02	−0.03, −0.01
N	0.02, −0.02	−0.03, −0.02	0.00, 0.00
C(2)	−0.08, −0.03	0.01, 0.02	0.03, −0.02
C(3)	1.22, 1.34	−1.41, −1.35	−1.30, −1.38
C(4)	0.07, 0.04	0.01, 0.00	−0.02, 0.02
C(5)	−1.29, −1.32	1.46, 1.39	1.39, −1.29

Angles (°) between planes: (1)–(2), 78.7, 76.5; (1)–(3), 82.6, 78.7; (2)–(3), 84.2, 83.4.

Plane S(11)–(31), for (II): $0.5055X \times 0.7399Y - 0.4438Z = 1.485$ [ΔAs 1.09 Å]

Plane S(21)–(31), for (II): $0.5689X + 0.6957Y - 0.4389Z = 4.274$ [ΔAs −1.35 Å]

* M = metal.

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* For details, see Notice to Authors No. 7, *J.C.S. Dalton*, 1974, Index issue.

[9] '*X*-Ray System,' Technical Report TR 192, Computer Science Centre, University of Maryland, June 1972.

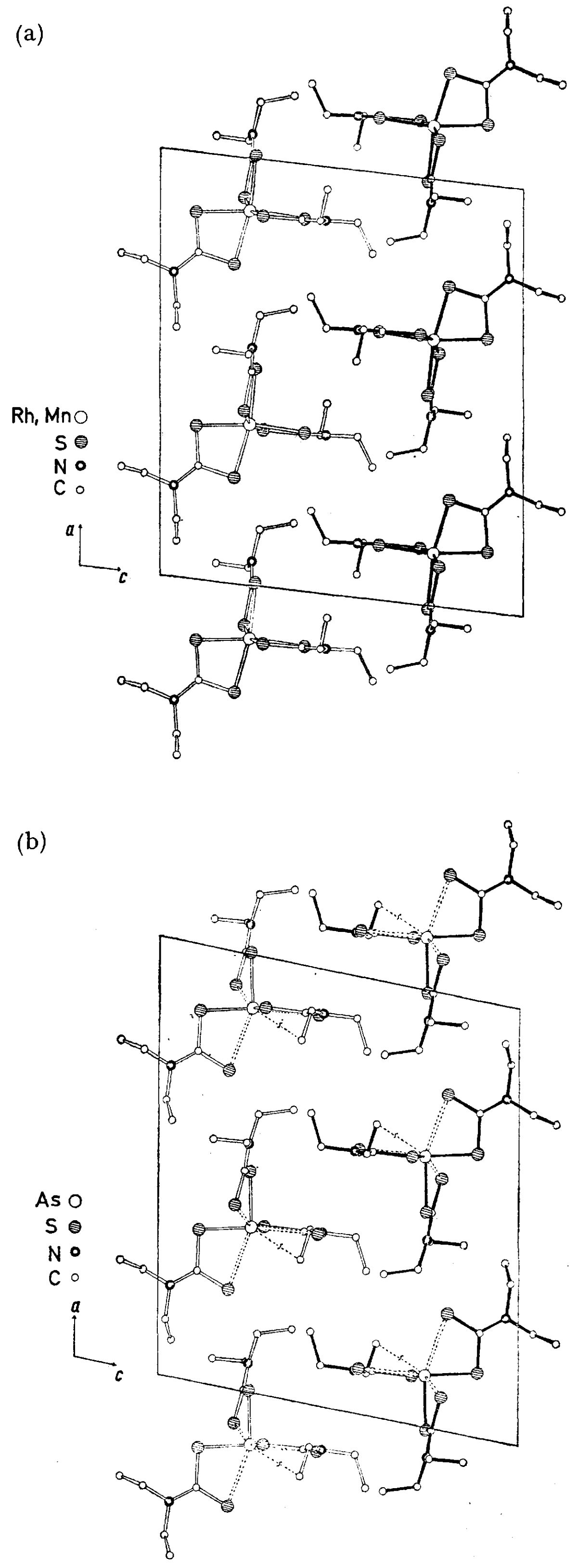


FIGURE 1 (a) Unit-cell contents of [Rh,Mn($CS_2{\cdot}NEt_2)_3$] compared with (b) unit cell contents of [As($CS_2{\cdot}NEt_2)_3$] (showing the H(35c) · · · As contact as a dotted line), both projected down *b*

to the unit-cell faces. Whereas usually in derivatives of the type M(dtc)$_3$ the overall molecular symmetry is a very good approximation to D_3, this is not so here. While in (I) and (III), the terminal methyl groups on the ligands adopt their more usual conformation, lying on alternate sides of the ligand plane, the intrinsic lack of symmetry other than rotation of such an isolated ligand is shown (Figure 2), with two of the ligand groups providing the blades of the three-bladed propeller configuration but the third co-ordinating as its mirror image. Because of the possibility of free rotation about the ethyl–nitrogen bonds this must be regarded as an essentially solid-state packing effect. In (II), the terminal ligand substituents are different yet again; in ligand (3), both terminal methyl groups lie on the same side of the ligand plane. The reason for this is discussed later.

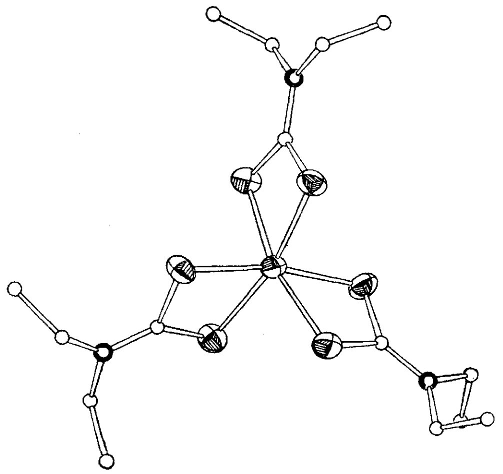


FIGURE 2 Solid-state conformation of [Rh,Mn($CS_2{\cdot}NEt_2)_3$]; 50% thermal ellipsoids of the RhS_6 core shown

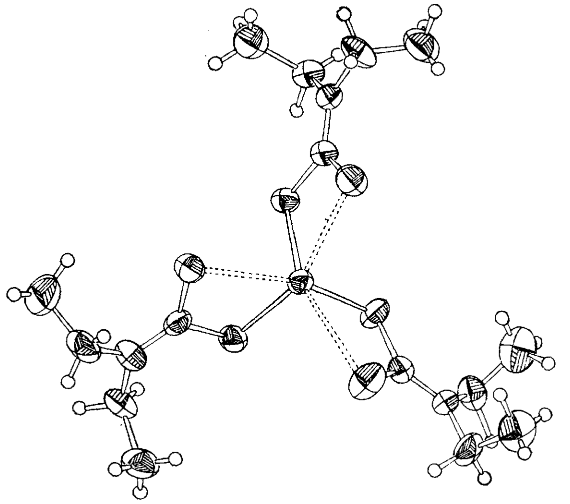
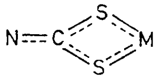


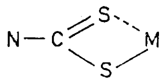
FIGURE 3 Solid-state conformation of [As($CS_2{\cdot}NEt_2)_3$]; 50% thermal ellipsoids are shown

Within the MS_6 cores, considerable differences are found. In (I), it is clear that although significant distortions from ideal D_3 symmetry occur (Table 2), they are, as shown by inequalities in the metal–sulphur distances and ligand ' bite ' angles, relatively trivial, no metal–sulphur distance deviating by $>2\sigma$ from the mean Rh–S distance (2.36_8 Å). In (III) relatively enormous differences have been found,[2] the manganese–sulphur distances ranging from 2.37 to 2.56 Å, and two of the distances being considerably longer than the remainder as a consequence of the pseudo-tetragonal distortion imposed on the intrinsic D_3 symmetry by Jahn–Teller distortion of the ^5E ground-state. In (II) also, considerable distortions are found but of an entirely different nature, as previously described,[3] the D_3 symmetry of the MS_6 core being degraded to C_3 by elongation of the metal–sulphur distances of one of the triangular faces normal to the three-fold axis so that different sets of distances are found; in one set no distance deviates by $>2\sigma$ from the mean (2.34_9 Å), while in the other two distances are close (2.81_2 and 2.81_9) and the third rather longer (2.90_4 Å). This observation is in substantial agreement with the previous description, and consistent with the postulation of a stereochemically active lone-pair. Again, this is essentially a solid-state phenomenon, since on the n.m.r. time-scale there is no evidence that the two ethyl groups within each ligand occupy electronically different sites.[10] A further unusual aspect of this array is that the abnormality observed in ligand (3), concerning the positioning of the terminal methyl groups, is clearly a consequence of the interaction of the hydrogen atoms [H(35c)] with the arsenic atom. This distance [H(35c) · · · As 3.2(2) Å] is comparable with the van der Waals estimate of 3.2 Å and arises out of an approach by H(35c) along the three-fold axis of the MS_6 core between those three sulphur atoms which have long metal–sulphur distances; this interaction provides further unusual circumstantial evidence for the presence of the stereochemically active lone-pair on the arsenic, and results in the reversal of the disposition of the terminal methyl C(35) into the stereochemically less favoured ligand disposition in which both terminal methyl groups lie on the same side of the ligand.

In (I) and (III), the ligand geometries are rather inaccurate, either as a consequence of small crystal size or the use of film data; in (II), however, it is apparent that the S_2CNC_2 ligand fragments are degraded from their usual intrinsic *mm* symmetry (found in derivatives in which the ligand is symmetrically bidentate) to *m* symmetry. This occurs by a disparity in the C–S distances; in all three ligands, the C–S distance associated with the short As–S bond is lengthened appreciably (mean 1.76 ± 0.01 Å), while that associated with the long As–S distance is shortened appreciably to 1.69 ± 0.02 Å (the usual value associated with the symmetrically chelated ligand is *ca.* 1.72 Å).[1] These values should be compared with the extreme ' unidentate ' case of the tetraethylthiuram disulphide,[11] $Et_2NCS_2{\cdot}CS_2NEt_2$, where C=S is 1.64, and C–S(·S) is 1.82 Å. Here, as in (II), a relief in strain at the ligand S–C–S angle takes place, the angle increasing from 112 in (I) to 119° in (II) (*cf.* 120° in $Et_2NCS_2{\cdot}CS_2NEt_2$) and accompanied by an increase in S · · · S distance. Clearly the co-ordination type is tending from (*a*) exemplified by (I) to (*b*) in (II) and the thiuram.



(*a*)



(*b*)

[5/587 *Received, 25th March,* 1975]

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[10] C. L. Raston and A. H. White, unpublished work.

[11] I. L. Karle, J. A. Estlin, and K. Britts, *Acta Cryst.*, 1967, **22**, 273.