SHORT-FORMAT PAPERS

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A Second Polymorph of Tris(*N*,*N*-diethyldithiocarbamato)manganese(III)

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Abstract. $[Mn(C_5H_{10}NS_2)_3]$, $M_r = 499{\cdot}7$, monoclinic, $I2/a$, $a = 17{\cdot}171\ (4)$, $b = 10{\cdot}188\ (2)$, $c = 14{\cdot}822\ (4)$ Å, $\beta = 112{\cdot}13\ (2)°$, $V = 2402\ (2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}40\ (1)$, $D_x = 1{\cdot}382$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}00$ mm^{-1}, $F(000) = 1045$, $T = 293\ (2)$ K, $R = 0{\cdot}023$ for 1762 observed reflections. A second polymorph of the title compound has been characterized in which the molecule is situated on a crystallographic twofold axis of symmetry. The Mn atom is octahedrally coordinated by three chelating dithiocarbamate ligands, two of which display slight asymmetry in their mode of coordination, Mn–S(2) 2·389 (1) and Mn–S(3) 2·582 (1) Å.

Experimental. $[Mn(S_2CNC_4H_{10})_3]$ prepared as in the literature (Golding, Healy, Newman, Sinn, Tennant & White, 1970); crystal 0·20 × 0·20 × 0·23 mm grown from acetone solution of the compound. Density measured in aq. $ZnBr_2$ solution. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromatized Mo $K\bar{\alpha}$ radiation; $\omega{:}2\theta$ scan technique. Cell parameters obtained from least-squares procedure (De Boer & Duisenberg, 1984) on 25 reflections ($11 \le \theta \le 17°$). Max. and min. transmission factors for analytical absorption correction 0·8560 and 0·8044 (Sheldrick, 1976). Total of 5349 reflections measured in range $1 \le \theta \le 25°$; $20 \le h \le 20$, $-1 \le k \le 12$, $-17 \le l \le 17$ with additional high-angle Friedel pairs. No significant variation in intensities of three standard reflections ($36\bar{1}$, $45\bar{1}$, 534) monitored every 3600 s X-ray exposure time. 2119 unique reflections ($R_{int} = 0{\cdot}020$), 1762 satisfied $I \ge 2{\cdot}5\sigma(I)$. Structure solved from Patterson method, full-matrix least-squares refinement of 116 parameters based on F

Table 1. *Fractional atomic coordinates and* B_{eq} (Å^2) *values*

$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3$.

	x	y	z	B_{eq}
Mn	0·25	−0·29538 (3)	0·0	2·86
S(1)	0·24162 (3)	−0·48190 (5)	−0·10012 (3)	3·72
C(1)	0·25	−0·5747 (2)	0·0	3·24
N(1)	0·25	−0·7043 (2)	0·0	3·95
C(2)	0·2596 (2)	−0·7791 (2)	0·0878 (2)	5·45
C(3)	0·1776 (2)	−0·8119 (3)	0·0963 (3)	8·47
S(2)	0·21858 (3)	−0·14829 (5)	−0·13462 (4)	3·67
S(3)	0·09229 (3)	−0·23665 (5)	−0·05997 (4)	3·70
C(4)	0·1157 (1)	−0·1428 (2)	−0·1419 (1)	2·90
N(2)	0·0593 (1)	−0·0680 (2)	−0·2084 (1)	3·24
C(5)	0·0789 (1)	0·0047 (2)	−0·2827 (1)	4·11
C(6)	0·0531 (2)	−0·0707 (3)	−0·3767 (2)	6·58
C(7)	−0·0276 (1)	−0·0561 (2)	−0·2133 (2)	4·42
C(8)	−0·0399 (2)	0·0637 (3)	−0·1620 (2)	6·98

Table 2. *Interatomic distances* (Å) *and angles* (°)

Mn–S(1)	2·382 (1)	Mn–S(2)	2·389 (1)
Mn–S(3)	2·582 (1)	S(1)–C(1)	1·719 (1)
S(2)–C(4)	1·730 (2)	S(3)–C(4)	1·709 (2)
N(1)–C(1)	1·320 (3)	N(2)–C(4)	1·330 (2)
N(1)–C(2)	1·463 (3)	C(2)–C(3)	1·498 (4)
N(2)–C(5)	1·469 (2)	C(5)–C(6)	1·503 (3)
N(2)–C(7)	1·471 (2)	C(7)–C(8)	1·495 (3)
S(1)–Mn–S(1′)	74·2 (1)	S(1)–Mn–S(2)	92·2 (1)
S(1)–Mn–S(3)	99·3 (1)	S(2)–Mn–S(3)	71·9 (1)
S(2)–Mn–S(2′)	102·3 (1)	S(3)–Mn–S(3′)	153·3 (1)
Mn–S(1)–C(1)	86·3 (1)	Mn–S(2)–C(4)	88·7 (1)
Mn–S(3)–C(4)	83·0 (1)	S(1)–C(1)–S(1′)	113·3 (1)
S(2)–C(4)–S(3)	116·5 (1)	S(1)–C(1)–N(1)	123·4 (1)
C(1)–N(1)–C(2)	121·4 (1)	C(2)–N(1)–C(2′)	117·2 (2)
N(1)–C(2)–C(3)	113·4 (2)	S(2)–C(4)–N(2)	120·4 (1)
S(3)–C(4)–N(2)	123·1 (1)	C(4)–N(2)–C(5)	122·4 (2)
C(4)–N(2)–C(7)	121·7 (2)	C(5)–N(2)–C(7)	115·9 (1)
N(2)–C(5)–C(6)	111·5 (2)	N(2)–C(7)–C(8)	112·2 (2)

Primed atoms are related by the twofold axis.

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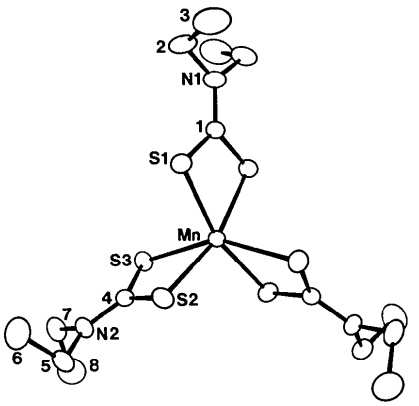

Fig. 1. The molecular structure of $[Mn(S_2CNC_4H_{10})_3]$; note that Mn, C(1) and N(1) all lie on a twofold axis. Atoms otherwise not indicated are C atoms.

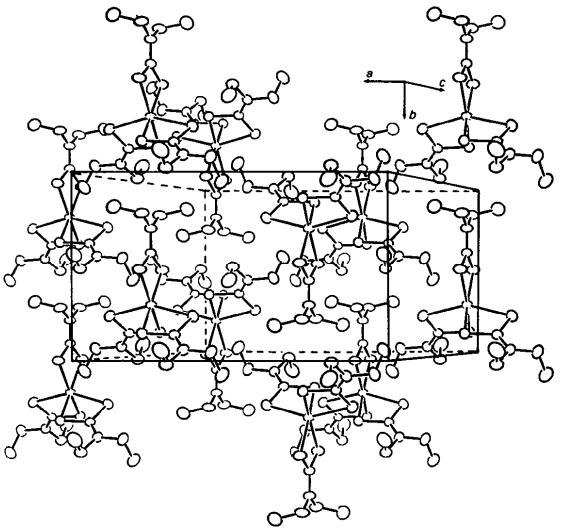

Fig. 2. Unit-cell contents for $[Mn(S_2CNC_4H_{10})_3]$.

(Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms, H atoms included at their calculated positions. At convergence $R = 0{\cdot}023$, $wR = 0{\cdot}026$, $w = 1{\cdot}08/[\sigma^2(F) + 0{\cdot}0003|F|^2]$, $S = 1{\cdot}5$, $(\Delta/\sigma)_{max} \leq 0{\cdot}002$, $(\Delta\rho)_{max} = 0{\cdot}17$, $(\Delta\rho)_{min} = -0{\cdot}15$ e Å^{-3}; no extinction correction. Scattering factors for H, C, N, and S given in *SHELX*76 (Sheldrick, 1976) and those for neutral Mn corrected for f' and f'' (Hamilton & Ibers, 1974). University of Adelaide's VAX VMS4·1 computer system with *SHELX*76 (Sheldrick, 1976). Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 is a perspective view of the molecule and Fig. 2 shows the unit-cell contents. The figures were drawn with *ORTEP*II (Johnson, 1971).

Related literature. The molecular geometry of $[Mn(S_2CNC_4H_{10})_3]$ is in essential agreement with that reported for the $P2_1/a$ crystal modification (Healy & White, 1972), the major difference being that in the $I2/a$ (C_{2h}^6, No. 15) polymorph the molecule is constrained to C_2 symmetry. The disparity in the Mn–S bond distances formed by two of the chelates has been ascribed (Healy & White, 1972) to a Jahn–Teller distortion.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and mean-plane data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42771 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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