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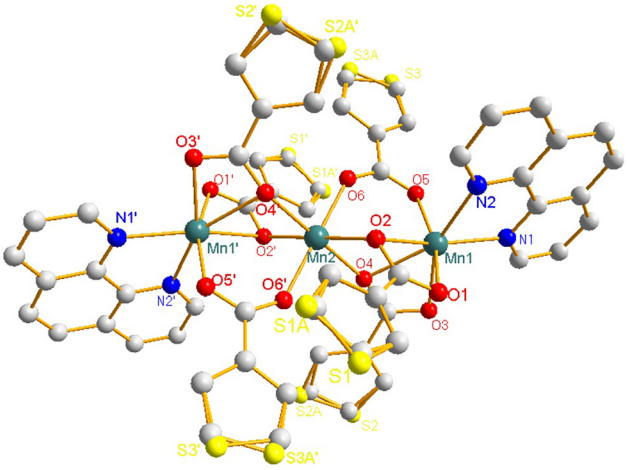
The crystal structure of hexakis(3-thiophenecarboxylato-κ^2O,O'')-bis(1,10-phenanthroline-κ^2N,N') trimanganese(II), $C_{54}H_{34}N_4O_{12}S_6Mn_3$



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Abstract

$C_{54}H_{34}N_4O_{12}S_6Mn_3$, monoclinic, $P2_1/c$ (no. 14), a = 11.0140(4) Å, b = 22.3222(9) Å, c = 11.2886(5) Å, β = 105.436(1)°, Z = 2, V = 2675.27(19) Å^3, $R_{gt}(F)$ = 0.0361, w$R_{ref}(F^2)$ = 0.0846, T = 273 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

3–Thiophenecarboxylic acid (6.0 mmol) was dissolved in 20 mL of anhydrous methanol. $Mn(CH_3COO)_2{\cdot}4H_2O$ (3.0 mmol) dissolved in 15 mL of anhydrous methanol was added dropwise to the above 3-thiophenecarboxylic acid solution and stirred for 4 h at 55 °C. Then 1,10-phenanthroline (2.0 mmol) was dissolved in 10 mL of anhydrous methanol was added dropwise to the above solution and stirred for 3 h at 55 °C cooled and filtered. The filtrate was left for slow evaporation at room temperature. The yellow block crystals were formed 16 days later. Yield: 39.7 %. Anal. Calcd. for $C_{54}H_{34}N_4O_{12}S_6Mn_3$: C, 50.35; H, 2.66; N, 4.35; S, 14.94; found: C, 50.31; H, 2.70; N, 4.39; S, 14.90.

Table 1: Data collection and handling.

Crystal:	Block, yellow
Size:	0.3 × 0.2 × 0.2 mm
Wavelength:	Mo $K\alpha$ radiation, l = 0.71073 Å
μ:	0.999 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.130°, >99.9 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18,414, 5917, 0.0212
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4961
$N(param)_{refined}$:	415
Programs:	Bruker [1], Olex2 [2]

2 Experimental details

A suitable crystal was selected and mounted on a Bruker Smart APEX—II CCD. Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Metal complexes are considered as promising anticancer drugs [3]. Thiophene carboxylates and its metal complexes have attracted extensive attention. Some of these metal complexes have excellent biological activities [4, 5]. We report herein a new trinuclear manganese metal complex constructed by 3-thiophenecarboxylate and 1,10-phenanthroline ligands. The title complex has a trinuclear structure, in

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

	x	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.73967 (3)	0.40694 (2)	0.14482 (3)	0.02828 (9)
Mn2	0.5000	0.5000	0.0000	0.02615 (10)
S1	0.95691 (19)	0.67057 (8)	0.0273 (3)	0.0514 (4)
S1A	0.8134 (7)	0.6943 (3)	0.0527 (4)	0.0555 (12)
S2	0.6314 (4)	0.3550 (2)	−0.4534 (3)	0.0647 (7)
S2A	0.5203 (9)	0.4195 (4)	−0.4609 (7)	0.0774 (14)
S3	0.2754 (5)	0.2953 (2)	0.3461 (6)	0.0670 (10)
S3A	0.1959 (2)	0.35109 (12)	0.3206 (3)	0.0621 (6)
O1	0.87238 (17)	0.47788 (7)	0.10875 (17)	0.0467 (4)
O2	0.68980 (14)	0.51176 (6)	0.12348 (15)	0.0371 (3)
O3	0.75166 (17)	0.36587 (8)	−0.03845 (14)	0.0466 (4)
O4	0.5896 (2)	0.42410 (8)	−0.05724 (17)	0.0572 (5)
O5	0.57984 (15)	0.36671 (8)	0.18492 (16)	0.0451 (4)
O6	0.45650 (16)	0.44745 (7)	0.13974 (15)	0.0418 (4)
N1	0.85287 (16)	0.32868 (8)	0.23992 (16)	0.0313 (4)
N2	0.81628 (18)	0.43196 (8)	0.35117 (17)	0.0362 (4)
C1	0.8661 (2)	0.27730 (10)	0.1860 (2)	0.0415 (5)
H1	0.8329	0.2741	0.1014	0.050*
C2	0.9275 (3)	0.22775 (11)	0.2505 (3)	0.0528 (7)
H2	0.9338	0.1922	0.2096	0.063*
C3	0.9780 (3)	0.23213 (12)	0.3739 (3)	0.0538 (7)
H3	1.0200	0.1996	0.4179	0.065*
C4	0.9670 (2)	0.28546 (11)	0.4347 (2)	0.0419 (6)
C5	0.90194 (19)	0.33307 (10)	0.3635 (2)	0.0320 (5)
C6	0.8849 (2)	0.38844 (10)	0.4220 (2)	0.0342 (5)
C7	0.9374 (2)	0.39500 (13)	0.5496 (2)	0.0469 (6)
C8	0.9154 (3)	0.44940 (14)	0.6029 (3)	0.0616 (8)
H8	0.9488	0.4559	0.6867	0.074*
C9	0.8454 (3)	0.49234 (14)	0.5317 (3)	0.0630 (8)
H9	0.8295	0.5283	0.5662	0.076*
C10	0.7971 (3)	0.48216 (12)	0.4059 (2)	0.0506 (6)
H10	0.7491	0.5121	0.3583	0.061*
C11	1.0188 (3)	0.29402 (15)	0.5642 (3)	0.0576 (8)
H11	1.0623	0.2628	0.6116	0.069*
C12	1.0058 (3)	0.34603 (16)	0.6181 (3)	0.0615 (8)
H12	1.0421	0.3505	0.7020	0.074*
C13	0.7980 (2)	0.52018 (10)	0.10373 (19)	0.0336 (5)
C14	0.8332 (2)	0.58191 (9)	0.07821 (17)	0.0321 (4)
C15	0.9443 (2)	0.59671 (10)	0.05037 (19)	0.0405 (5)
H15	1.0054	0.5688	0.0450	0.049*
H15A	1.0084	0.5707	0.0444	0.049*
C16	0.8193 (11)	0.6841 (5)	0.0531 (5)	0.059 (2)
H16	0.7850	0.7223	0.0499	0.070*
C16A	0.9406 (17)	0.6615 (9)	0.032 (3)	0.056 (3)
H16A	1.0045	0.6826	0.0109	0.067*
C17	0.7578 (2)	0.63207 (11)	0.0807 (2)	0.0423 (5)
H17	0.6797	0.6315	0.0977	0.051*
H17A	0.6799	0.6291	0.0977	0.051*
C18	0.6616 (2)	0.39456 (9)	−0.1044 (2)	0.0343 (5)
C19	0.6340 (2)	0.39290 (10)	−0.2403 (2)	0.0348 (5)
C20	0.6901 (3)	0.35426 (13)	−0.3045 (2)	0.0518 (6)
H20	0.7567	0.3295	−0.2659	0.062*
H20A	0.7506	0.3253	−0.2727	0.062*
C21	0.5322 (18)	0.4089 (8)	−0.4434 (15)	0.097 (8)
H21	0.4757	0.4258	−0.5114	0.116*
C21A	0.632 (2)	0.3691 (11)	−0.438 (2)	0.096 (15)
H21A	0.6570	0.3512	−0.5021	0.116*
C22	0.5417 (3)	0.42729 (14)	−0.3181 (3)	0.0619 (8)
H22	0.4941	0.4570	−0.2942	0.074*
H22A	0.4936	0.4542	−0.2869	0.074*
C23	0.4818 (2)	0.39585 (10)	0.18369 (19)	0.0315 (4)
C24	0.3880 (2)	0.36767 (10)	0.24012 (18)	0.0320 (4)
C25	0.3990 (3)	0.31041 (11)	0.2909 (2)	0.0457 (6)
H25	0.4655	0.2843	0.2937	0.055*
H25A	0.4654	0.2847	0.2910	0.055*
C26	0.2792 (2)	0.39597 (11)	0.2484 (2)	0.0439 (6)
H26	0.2577	0.4347	0.2202	0.053*
H26A	0.2547	0.4342	0.2188	0.053*
C27	0.2091 (12)	0.3628 (5)	0.3000 (14)	0.058 (2)
H27	0.1317	0.3750	0.3102	0.069*
C27A	0.3030 (12)	0.2960 (6)	0.3401 (16)	0.061 (2)
H27A	0.2973	0.2600	0.3797	0.074*

which three Mn(II) ions are connected by O atoms of 3-thiophenecarboxylates. In the complex, each Mn(II) ion is six-coordinated in a distorted octahedral environment, in which one Mn(II) ion is coordinated by six O atoms of the 3-thiophenecarboxylato ligand, whereas the other two Mn(II) ions are coordinated by four O atoms and two N atoms from the 3-thiophenecarboxylato ligand and 1,10-phenanthroline ligand, respectively. The bond distances of Mn–N are 2.2464 (17) and 2.3245(18) Å, bond lengths of Mn–O are 2.1204 (14)–2.465(2) Å, respectively, which are similar with the references [6, 7]. The complex is has a centrosymmetric structure, and the symmetry center is the Mn2(II) ion.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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