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Crystal structure of (acetic acid- $\kappa^1 O$)-bis(μ_2 -2-chlorobenzoato- $\kappa^2 O:O'$)-(2-chlorobenzoato- $\kappa^1 O$)-(μ^2 -hydroxy- $\kappa^2 O:O$)-bis(1,10-phenanthroline- $\kappa^2 N,N'$)dimanganese(II) – methanol (1/1), $C_{48}H_{37}Cl_3Mn_2N_4O_{10}$

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Abstract

$C_{48}H_{37}Cl_3Mn_2N_4O_{10}$, monoclinic, $P2_1/n$ (no. 14), $a = 12.759(3)$ Å, $b = 19.347(5)$ Å, $c = 18.945(5)$ Å, $\beta = 97.321(5)^\circ$, $V = 4638(2)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0432$, $wR_{ref}(F^2) = 0.1196$, $T = 298(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

2-Chlorobenzoic acid (312 mg, 2 mmol) and manganese(II) acetate (490 mg, 2 mmol), 1,10-phenanthroline (360 mg, 2 mmol) and 20 mL methanol were mixed and stirred for ca. 2 h. After the solution was cooled to room temperature, the solid was filtered off. The mixture solution was filtered and the filtration was distilled. The product was recrystallized

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.21 × 0.16 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.78 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX II,
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	23632, 8210, 0.034
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 5947
$N(param)_{refined}$:	611
Programs:	Bruker [1], SHELX [2]

in hexane/dichloromethane as the ratio of 1:1. The colorless block-shaped crystals were obtained by evaporation in air. Yield: 78% (base on Mn element), and elemental analysis: calc. for $C_{48}H_{37}Cl_3Mn_2N_4O_{10}$: C 55.22, H 3.38, N 5.37; found: C 55.31, H 3.24, N 5.15 (PERKIN ELMER MODEL 2400 SERIES II).

Experimental details

Hydrogen atoms were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2$ times $U_{eq}(C-aryl)$ and 1.5 times $U_{eq}(C, methyl)$ and (O, hydroxyl). All the H atoms were refined as riding on their parent atom.

Comment

During the last decades, carboxylate complexes have received much attention because of their interesting property in the field of biology and materials, especially many varieties of Mn complexes with the multi-carboxylic ligand and the co-ligand with N atoms are known [3–6].

The crystal structure shows two carboxylic ligands and one oxygen atom bridge two Mn atoms and two phen ligands chelate two Mn atoms, respectively. Acetic acid and a 2-chlorobenzoic acid group coordinate to one Mn atom, respectively. Both Mn atoms show six-coordinated octahedron geometry. For Mn1, the angle of O3-Mn1-O8 is 172.33(19)°, showing the geometry is distorted. Meanwhile, for Mn2, the angle of O2-Mn2-O5 is 174.69(10)°. The bond lengths of Mn–O are 2.115(2) Å – 2.216(2) Å and the bond lengths of Mn–N are from 2.236(3) Å to 2.298(3) Å. The bridged O atom is almost in the middle of two Mn atoms for the O7-Mn1 is 2.208(2) Å and O7-Mn2 is 2.216(2) Å.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
Mn1	0.20523(4)	0.47264(2)	0.85165(2)	0.03575(13)
Mn2	0.15541(4)	0.58569(2)	0.70399(2)	0.03679(14)
Cl1	−0.19920(9)	0.59268(6)	0.70077(5)	0.0739(3)
Cl2	0.17301(8)	0.63362(7)	1.02616(5)	0.0742(3)
Cl3	0.50473(12)	0.56410(7)	0.57292(7)	0.0980(4)
O1	0.03956(17)	0.48265(13)	0.82697(11)	0.0504(6)
O2	0.01812(17)	0.52257(13)	0.71572(11)	0.0481(6)
O3	0.22588(19)	0.57774(12)	0.88646(12)	0.0508(6)
O4	0.14383(19)	0.64231(12)	0.79929(11)	0.0497(6)
O5	0.29981(19)	0.64012(13)	0.68949(14)	0.0606(7)
O6	0.4279(2)	0.56666(16)	0.72816(17)	0.0828(10)
O7	0.25973(19)	0.50089(12)	0.74945(11)	0.0442(5)
O8	0.1931(2)	0.36978(12)	0.80336(12)	0.0551(6)
C47	0.1926(4)	0.26812(19)	0.7269(2)	0.0684(11)
H47A	0.214626	0.240125	0.767977	0.103*
H47B	0.232480	0.255574	0.689158	0.103*
H47C	0.118762	0.260813	0.711785	0.103*
O10	0.2501(3)	0.37935(14)	0.69913(13)	0.0778(9)
H10	0.258398	0.419582	0.712285	0.117*
O11	0.3327(4)	0.3285(3)	0.5837(2)	0.1364(16)
H11	0.297863	0.328190	0.617149	0.205*
N1	0.3692(2)	0.44502(14)	0.90795(13)	0.0412(6)
N2	0.1826(2)	0.43450(13)	0.96183(13)	0.0392(6)
N3	0.1520(2)	0.54944(14)	0.58823(12)	0.0399(6)
N4	0.0558(2)	0.66161(14)	0.63632(13)	0.0394(6)
C48	0.2120(3)	0.34512(19)	0.74594(19)	0.0516(9)
C1	−0.0146(2)	0.49522(17)	0.76819(16)	0.0388(7)
C2	−0.1285(2)	0.47158(18)	0.76208(15)	0.0418(8)
C3	−0.2142(3)	0.5099(2)	0.73229(17)	0.0520(9)
C4	−0.3177(3)	0.4843(3)	0.7290(2)	0.0762(14)
H4	−0.374983	0.511218	0.710290	0.091*
C5	−0.3328(4)	0.4194(3)	0.7537(2)	0.0848(15)
H5	−0.400887	0.401501	0.750349	0.102*
C6	−0.2492(4)	0.3802(3)	0.7834(2)	0.0797(14)
H6	−0.261087	0.336078	0.800149	0.096*
C7	−0.1487(3)	0.4055(2)	0.78855(18)	0.0612(11)
H7	−0.092764	0.378813	0.809818	0.073*
C8	0.1919(2)	0.63410(17)	0.86053(16)	0.0388(7)
C9	0.2159(2)	0.69787(17)	0.90450(16)	0.0402(7)
C10	0.2162(3)	0.70156(19)	0.97802(17)	0.0480(9)
C11	0.2487(3)	0.7609(3)	1.0157(2)	0.0675(12)
H11A	0.248624	0.762318	1.064764	0.081*
C12	0.2809(4)	0.8171(3)	0.9807(3)	0.0838(15)
H12	0.304596	0.856403	1.006090	0.101*
C13	0.2785(4)	0.8159(2)	0.9079(3)	0.0815(14)
H13	0.298648	0.854621	0.883912	0.098*
C14	0.2458(3)	0.7562(2)	0.8707(2)	0.0614(10)
H14	0.243950	0.755688	0.821430	0.074*
C15	0.3935(3)	0.6197(2)	0.69641(18)	0.0496(9)
C16	0.4705(3)	0.6644(2)	0.6642(2)	0.0537(9)
C17	0.5255(3)	0.6442(2)	0.6105(2)	0.0640(11)
C18	0.5977(4)	0.6892(3)	0.5832(3)	0.0869(16)
H18	0.634082	0.675699	0.545962	0.104*
C19	0.6123(4)	0.7536(3)	0.6135(3)	0.0956(17)
H19	0.660402	0.783533	0.596505	0.115*
C20	0.5596(4)	0.7750(3)	0.6668(4)	0.0966(17)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H20	0.572037	0.818936	0.685963	0.116*
C21	0.4879(3)	0.7325(2)	0.6930(3)	0.0847(15)
H21	0.450795	0.747743	0.729276	0.102*
C22	0.4602(3)	0.45006(19)	0.88202(18)	0.0502(9)
H22	0.459376	0.465790	0.835565	0.060*
C23	0.5575(3)	0.4331(2)	0.9205(2)	0.0598(10)
H23	0.619906	0.437294	0.900259	0.072*
C24	0.5584(3)	0.4102(2)	0.9886(2)	0.0626(11)
H24	0.622129	0.398576	1.015396	0.075*
C25	0.4645(3)	0.40406(19)	1.01817(18)	0.0528(9)
C26	0.4603(4)	0.3831(2)	1.0907(2)	0.0700(12)
H26	0.522460	0.371584	1.119362	0.084*
C27	0.3686(4)	0.3800(2)	1.1174(2)	0.0728(12)
H27	0.368283	0.366127	1.164388	0.087*
C28	0.2709(3)	0.39737(19)	1.07581(17)	0.0531(9)
C29	0.1729(4)	0.3956(2)	1.10126(18)	0.0615(11)
H29	0.168713	0.382220	1.147982	0.074*
C30	0.0844(3)	0.4131(2)	1.05837(19)	0.0596(10)
H30	0.019192	0.412688	1.075386	0.072*
C31	0.0920(3)	0.43165(18)	0.98848(18)	0.0489(8)
H31	0.030266	0.442626	0.958987	0.059*
C32	0.2720(3)	0.41780(16)	1.00472(16)	0.0404(8)
C33	0.3708(3)	0.42204(16)	0.97572(16)	0.0400(8)
C34	0.2038(3)	0.49763(19)	0.56379(18)	0.0532(9)
H34	0.250238	0.472593	0.595936	0.064*
C35	0.1927(3)	0.4782(2)	0.49233(19)	0.0622(11)
H35	0.230969	0.441214	0.477534	0.075*
C36	0.1259(3)	0.5139(2)	0.44517(18)	0.0594(10)
H36	0.116053	0.500770	0.397567	0.071*
C37	0.0713(3)	0.57081(19)	0.46773(16)	0.0481(9)
C38	0.0015(3)	0.6122(2)	0.42109(19)	0.0678(11)
H38	−0.010771	0.600834	0.373048	0.081*
C39	−0.0467(3)	0.6668(2)	0.4446(2)	0.0698(12)
H39	−0.091415	0.693170	0.412642	0.084*
C40	−0.0313(3)	0.68603(19)	0.51813(18)	0.0513(9)
C41	−0.0786(3)	0.7437(2)	0.5455(2)	0.0650(11)
H41	−0.124884	0.770947	0.515476	0.078*
C42	−0.0572(3)	0.7601(2)	0.6155(2)	0.0632(11)
H42	−0.086934	0.798980	0.633899	0.076*
C43	0.0105(3)	0.71701(19)	0.65903(19)	0.0532(9)
H43	0.024565	0.728068	0.707082	0.064*
C44	0.0361(2)	0.64609(16)	0.56594(16)	0.0377(7)
C45	0.0875(2)	0.58722(16)	0.54072(15)	0.0381(7)
C46	0.4403(5)	0.3285(5)	0.6096(4)	0.168(4)
H46A	0.463880	0.375207	0.618423	0.251*
H46B	0.479504	0.307767	0.575097	0.251*
H46C	0.451507	0.302589	0.653129	0.251*
H7A	0.3194(14)	0.514(2)	0.746(2)	0.072(14)*

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References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2004).

2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
3. Ma, C. B.; Wang, W. G.; Zhang, X. F.; Chen, C. N.; Liu, Q. T.; Zhu, H. P.; Liao, D. Z.; Li, L. C.: Molecular, one- and two-dimensional systems built from manganese(II) and phthalate/diimine ligands: syntheses, crystal structures and magnetic properties. *Eur. J. Inorg. Chem.* **2004** (2004) 3522–3532.
4. Mukhopadhyay, S.; Gandhi, B. A.; Kirk, M. L.; Armstrong, W. H.: A new class of oxo-bridged high-valent hexamanganese clusters supported by sterically hindered carboxylate ligands. *Inorg. Chem.* **42** (2003) 8171–8180.
5. Ma, C. B.; Chen, C. N.; Liu, Q. T.; Chen, F.; Liao, D. Z.; Li, L. C.; Sun, L. C.: Synthesis, structure and magnetic properties of a series of novel isophthalate-bridged manganese(II) polymers with double-layer or double-chain structures. *Eur. J. Inorg. Chem.* **2004** (2004) 3316–3325.
6. Xiao, H. P.; Hu, M. L.; Yuan, J. X.: Bis(4-bromo-isophthalato)-bis[bis(1,10-phenanthroline)-manganese(II)] tetrahydrate. *Acta Crystallogr.* **E61** (2005) m443–m445.