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Diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-κ^2N^3,O^4)-manganese(II) *N*,*N*-dimethylformamide disolvate

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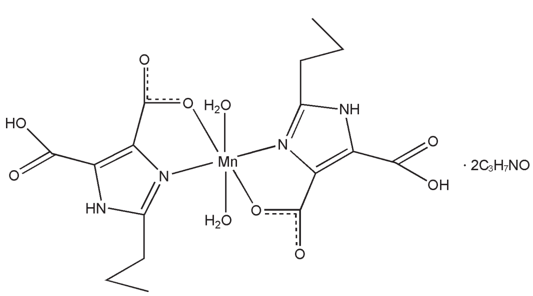
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.114; data-to-parameter ratio = 13.1.

In the title complex, $[Mn(C_8H_9N_2O_4)_2(H_2O)_2]\cdot 2C_3H_7NO$, the Mn^{II} atom, lying on an inversion centre, is six-coordinated by two *N*,*O*-bidentate 5-carboxy-2-propyl-1*H*-imidazole-4-carboxylate ligands and two water molecules in a distorted octahedral environment. In the crystal structure, the complex molecules and dimethylformamide solvent molecules are linked by N—H···O and O—H···O hydrogen bonds into a two-dimensional supramolecular network parallel to (001).

Related literature

For the potential uses and diverse structural types of complexes containing metals and *N*-heterocyclic carboxylic acids, see: Liang *et al.* (2002); Net *et al.* (1989); Nie *et al.* (2007); Song *et al.* (2010).



Experimental

Crystal data

$[Mn(C_8H_9N_2O_4)_2(H_2O)_2]\cdot 2C_3H_7NO$
M_r = 631.51
Triclinic, $P\overline{1}$
a = 7.3992 (8) Å
b = 9.4429 (11) Å
c = 11.1978 (13) Å
α = 76.591 (1)°
β = 87.927 (1)°
γ = 68.863 (1)°
V = 708.89 (14) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 273 K
0.32 × 0.25 × 0.21 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)) T_{min} = 0.847, T_{max} = 0.896
3653 measured reflections
2508 independent reflections
2131 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.114
S = 1.05
2508 reflections
191 parameters
27 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mn1—N1	2.1960 (18)	Mn1—O1	2.2530 (17)
Mn1—O1*W*	2.2036 (17)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O9^{i}	0.86	1.84	2.682 (3)	165
O3—H3···O2	0.82	1.65	2.471 (2)	176
O1*W*—H1*W*···O4^{ii}	0.85	1.92	2.764 (2)	170
O1*W*—H2*W*···O4^{iii}	0.84	2.11	2.927 (2)	164

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x, -y+1, -z$; (iii) $x-1, y+1, z$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge Guang Dong Ocean University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2265).

References

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supplementary materials

Acta Cryst. (2010). E**66**, m99 [**doi:10.1107/S1600536809054634**]

Diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-κ^2N^3,O^4)manganese(II) *N*,*N*-dimethylformamide disolvate

J.-B. Yan, S.-J. Li, W.-D. Song, H. Wang and D.-L. Miao

Comment

Structures of complexes containing metals and N-heterocyclic carboxylic acids have attracted much attention. The N-heterocyclic carboxylic acids can function as multidentate ligands, exhibiting diverse structrual types, and their metal complexes can be potentially used as functional materials (Liang *et al.*, 2002; Net *et al.*, 1989; Nie *et al.*, 2007). Recently, we have reported a new complex, poly[diaquabis(4-carboxy-2-propyl-1*H*-imidazole-5-carboxylato- $\kappa^3N^3,O^4:O^5$)calcium(II)] (Song *et al.*, 2010). In this paper, we report the synthesis and structure of a Mn^{II} complex obtained under hydrothermal conditions.

As illustrated in Fig. 1, the title complex molecule contains one Mn^{II} atom, lying on an inversion centre, one mono-deprotonated 5-carboxy-2-propyl-1*H*-imidazole-4-carboxylate ligand, one coordinated water molecule and one dimethylformamide solvent molecule in the asymmetric unit. The Mn^{II} atom is six-coordinated by two N,*O*-bidentate ligands and two water molecules in a distorted octahedral environment (Table 1). In the crystal structure, a two-dimensional supramolecular network is formed by N—H···O and O—H···O hydrogen bonds (Table 2 and Fig. 2).

Experimental

A mixture of $MnCl_2$ (0.5 mmol, 0.06 g) and 2-propyl-1*H*-imidazole-4,5-dicarboxylic acid (0.5 mmol, 0.99 g) in 15 ml of dimethylformamide solution was sealed in an autoclave equipped with a Teflon liner (20 ml) and then heated at 433 K for 4 d. Crystals of the title compound were obtained by slow evaporation of the solvent at room temperature.

Refinement

C– and N-bound H atoms were placed at calculated positions and refined as riding atoms, with C—H = 0.93 (CH), 0.97 (CH_2) and 0.96 (CH_3) Å and N—H = 0.86 Å and with U_{iso}(H) = 1.2(1.5 for methyl)U_{eq}(C, N). H atoms of water and carboxyl group were located in a difference Fourier map and refined as riding atoms, with U_{iso}(H) = 1.5U_{eq}(O).

Figures

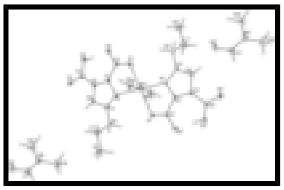

Fig. 1. Molecular structure of the title compound. Displacement ellipsoids are shown at the 30% probability level. [Symmetry code: (i) -*x*, 2 - *y*, -*z*.]

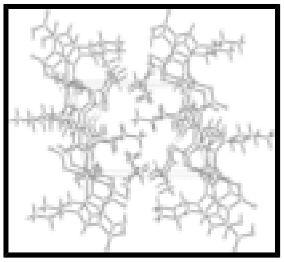

Fig. 2. A view of the two-dimensional network constructed by O—H···O and N—H···O hydrogen bonds (dashed lines).

Diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato- κ$^2N^3,O^4$)manganese(II) *N*,*N*-dimethylformamide disolvate

Crystal data

$[Mn(C_8H_9N_2O_4)_2H_2O)_2]\cdot 2C_3H_7NO$	$Z = 1$
$M_r = 631.51$	$F(000) = 331$
Triclinic, $P\overline{1}$	$D_x = 1.479$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, λ = 0.71073 Å
$a = 7.3992$ (8) Å	Cell parameters from 3600 reflections
$b = 9.4429$ (11) Å	θ = 1.4–28°
$c = 11.1978$ (13) Å	μ = 0.54 mm^{-1}
α = 76.591 (1)°	$T = 273$ K
β = 87.927 (1)°	Block, colourless
γ = 68.863 (1)°	0.32 × 0.25 × 0.21 mm
$V = 708.89$ (14) Å^3	

Data collection

Bruker APEXII CCD diffractometer	2131 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.025$
φ and ω scan	$\theta_{max} = 25.2°$, $\theta_{min} = 1.9°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996))	$h = -8\rightarrow 8$
$T_{min} = 0.847$, $T_{max} = 0.896$	$k = -9\rightarrow 11$
3653 measured reflections	$l = -13\rightarrow 12$
2508 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.114$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.120P]$ where $P = (F_o^2 + 2F_c^2)/3$

2508 reflections	$(\Delta/\sigma)_{max} < 0.001$
191 parameters	$\Delta\rho_{max} = 0.34$ e Å^{-3}
27 restraints	$\Delta\rho_{min} = -0.32$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.0000	1.0000	0.0000	0.03110 (18)
O1	−0.0548 (3)	0.92199 (19)	−0.16620 (15)	0.0379 (4)
O1W	−0.2795 (2)	0.9893 (2)	0.06711 (17)	0.0434 (4)
H1W	−0.2933	0.9113	0.0484	0.065*
H2W	−0.3672	1.0743	0.0329	0.065*
O2	0.0012 (3)	0.7160 (2)	−0.24509 (15)	0.0424 (4)
O3	0.1886 (3)	0.4324 (2)	−0.18355 (17)	0.0469 (5)
H3	0.1294	0.5271	−0.2021	0.070*
O4	0.3630 (3)	0.25664 (19)	−0.02155 (18)	0.0456 (5)
N1	0.1434 (3)	0.7447 (2)	0.04969 (16)	0.0279 (4)
N2	0.3073 (3)	0.4924 (2)	0.10599 (17)	0.0312 (4)
H2	0.3782	0.4030	0.1504	0.037*
C1	0.1335 (3)	0.6770 (2)	−0.0454 (2)	0.0261 (5)
C2	0.2340 (3)	0.5196 (3)	−0.0111 (2)	0.0285 (5)
C3	0.2498 (3)	0.6292 (3)	0.1400 (2)	0.0303 (5)
C4	0.0194 (3)	0.7798 (3)	−0.1592 (2)	0.0306 (5)
C5	0.2661 (3)	0.3927 (3)	−0.0755 (2)	0.0337 (5)
C6	0.2933 (4)	0.6449 (3)	0.2642 (2)	0.0410 (6)
H6A	0.2584	0.7548	0.2625	0.049*
H6B	0.4317	0.5939	0.2840	0.049*
C7	0.1855 (5)	0.5745 (4)	0.3637 (3)	0.0616 (8)
H7A	0.0477	0.6206	0.3409	0.074*
H7B	0.2268	0.4633	0.3688	0.074*
C8	0.2178 (5)	0.5986 (4)	0.4887 (3)	0.0648 (9)
H8A	0.3457	0.5307	0.5217	0.097*
H8B	0.1234	0.5753	0.5427	0.097*
H8C	0.2049	0.7052	0.4811	0.097*
O9	0.4607 (3)	−0.2429 (2)	0.7294 (2)	0.0626 (6)
N5	0.3789 (3)	0.0120 (3)	0.6354 (2)	0.0470 (6)
C17	0.4889 (4)	−0.1190 (4)	0.7092 (3)	0.0501 (7)
H17	0.5962	−0.1181	0.7492	0.060*
C18	0.2038 (5)	0.0185 (4)	0.5761 (3)	0.0702 (10)
H18A	0.0937	0.0641	0.6212	0.105*
H18B	0.2138	−0.0852	0.5745	0.105*
H18C	0.1880	0.0810	0.4936	0.105*
C19	0.4104 (7)	0.1561 (4)	0.6248 (4)	0.0879 (12)
H19A	0.5309	0.1353	0.6673	0.132*
H19B	0.3061	0.2259	0.6604	0.132*
H19C	0.4151	0.2033	0.5396	0.132*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0369 (3)	0.0180 (3)	0.0364 (3)	−0.0063 (2)	−0.0014 (2)	−0.0080 (2)
O1	0.0483 (10)	0.0222 (9)	0.0367 (9)	−0.0057 (8)	−0.0087 (7)	−0.0043 (7)
O1W	0.0411 (10)	0.0287 (9)	0.0616 (12)	−0.0120 (8)	0.0038 (8)	−0.0142 (8)
O2	0.0568 (11)	0.0346 (10)	0.0329 (9)	−0.0101 (9)	−0.0099 (8)	−0.0112 (8)
O3	0.0609 (12)	0.0307 (10)	0.0473 (11)	−0.0079 (9)	−0.0035 (9)	−0.0189 (8)
O4	0.0465 (10)	0.0218 (9)	0.0645 (12)	−0.0044 (8)	−0.0026 (9)	−0.0145 (8)
N1	0.0344 (10)	0.0210 (10)	0.0277 (10)	−0.0092 (8)	−0.0017 (8)	−0.0055 (8)
N2	0.0332 (10)	0.0188 (9)	0.0351 (11)	−0.0048 (8)	−0.0052 (8)	−0.0002 (8)
C1	0.0279 (11)	0.0210 (11)	0.0294 (11)	−0.0085 (9)	0.0007 (9)	−0.0064 (9)
C2	0.0288 (11)	0.0228 (12)	0.0341 (12)	−0.0087 (9)	0.0016 (9)	−0.0083 (9)
C3	0.0340 (12)	0.0234 (12)	0.0328 (12)	−0.0106 (10)	−0.0036 (9)	−0.0040 (9)
C4	0.0330 (12)	0.0272 (12)	0.0295 (12)	−0.0087 (10)	−0.0024 (9)	−0.0055 (10)
C5	0.0326 (12)	0.0267 (13)	0.0440 (14)	−0.0105 (10)	0.0066 (10)	−0.0138 (11)
C6	0.0512 (15)	0.0365 (14)	0.0354 (13)	−0.0164 (12)	−0.0098 (11)	−0.0062 (11)
C7	0.072 (2)	0.076 (2)	0.0455 (16)	−0.0325 (18)	0.0089 (15)	−0.0238 (15)
C8	0.069 (2)	0.077 (2)	0.0424 (16)	−0.0168 (19)	0.0026 (15)	−0.0191 (16)
O9	0.0712 (14)	0.0298 (11)	0.0699 (14)	−0.0062 (10)	−0.0216 (11)	0.0047 (10)
N5	0.0535 (13)	0.0309 (12)	0.0502 (13)	−0.0098 (11)	0.0019 (11)	−0.0065 (10)
C17	0.0472 (15)	0.0471 (18)	0.0500 (16)	−0.0084 (14)	−0.0051 (13)	−0.0129 (13)
C18	0.0575 (19)	0.058 (2)	0.073 (2)	−0.0073 (16)	−0.0120 (16)	0.0061 (17)
C19	0.134 (4)	0.050 (2)	0.089 (3)	−0.044 (2)	0.019 (3)	−0.0182 (19)

Geometric parameters (Å, °)

Mn1—N1	2.1960 (18)	C6—H6A	0.9700
Mn1—O1W	2.2036 (17)	C6—H6B	0.9700
Mn1—O1	2.2530 (17)	C7—C8	1.509 (4)
O1—C4	1.238 (3)	C7—H7A	0.9700
O1W—H1W	0.8509	C7—H7B	0.9700
O1W—H2W	0.8436	C8—H8A	0.9600
O2—C4	1.282 (3)	C8—H8B	0.9600
O3—C5	1.272 (3)	C8—H8C	0.9600
O3—H3	0.8200	O9—C17	1.229 (4)
O4—C5	1.240 (3)	N5—C17	1.313 (4)
N1—C3	1.326 (3)	N5—C19	1.440 (4)
N1—C1	1.379 (3)	N5—C18	1.452 (4)
N2—C3	1.347 (3)	C17—H17	0.9300
N2—C2	1.369 (3)	C18—H18A	0.9600
N2—H2	0.8600	C18—H18B	0.9600
C1—C2	1.367 (3)	C18—H18C	0.9600
C1—C4	1.480 (3)	C19—H19A	0.9600
C2—C5	1.481 (3)	C19—H19B	0.9600
C3—C6	1.491 (3)	C19—H19C	0.9600
C6—C7	1.516 (4)		

N1^{i}—Mn1—N1	180.0	O4—C5—C2	118.9 (2)
N1^{i}—Mn1—O1W	87.40 (7)	O3—C5—C2	116.6 (2)
N1—Mn1—O1W	92.60 (6)	C3—C6—C7	112.7 (2)
N1^{i}—Mn1—O1W^{i}	92.60 (6)	C3—C6—H6A	109.1
N1—Mn1—O1W^{i}	87.40 (7)	C7—C6—H6A	109.1
O1W—Mn1—O1W^{i}	180.0	C3—C6—H6B	109.1
N1^{i}—Mn1—O1^{i}	75.41 (6)	C7—C6—H6B	109.1
N1—Mn1—O1^{i}	104.59 (6)	H6A—C6—H6B	107.8
O1W—Mn1—O1^{i}	91.40 (6)	C8—C7—C6	113.4 (3)
O1W^{i}—Mn1—O1^{i}	88.60 (6)	C8—C7—H7A	108.9
N1^{i}—Mn1—O1	104.59 (6)	C6—C7—H7A	108.9
N1—Mn1—O1	75.41 (6)	C8—C7—H7B	108.9
O1W—Mn1—O1	88.60 (6)	C6—C7—H7B	108.9
O1W^{i}—Mn1—O1	91.40 (6)	H7A—C7—H7B	107.7
O1^{i}—Mn1—O1	180.0	C7—C8—H8A	109.5
C4—O1—Mn1	115.45 (14)	C7—C8—H8B	109.5
Mn1—O1W—H1W	107.7	H8A—C8—H8B	109.5
Mn1—O1W—H2W	107.5	C7—C8—H8C	109.5
H1W—O1W—H2W	112.0	H8A—C8—H8C	109.5
C5—O3—H3	109.5	H8B—C8—H8C	109.5
C3—N1—C1	105.96 (18)	C17—N5—C19	121.9 (3)
C3—N1—Mn1	141.35 (15)	C17—N5—C18	119.5 (3)
C1—N1—Mn1	112.53 (13)	C19—N5—C18	118.0 (3)
C3—N2—C2	108.51 (18)	O9—C17—N5	125.0 (3)
C3—N2—H2	125.7	O9—C17—H17	117.5
C2—N2—H2	125.7	N5—C17—H17	117.5
C2—C1—N1	109.72 (19)	N5—C18—H18A	109.5
C2—C1—C4	132.5 (2)	N5—C18—H18B	109.5
N1—C1—C4	117.81 (19)	H18A—C18—H18B	109.5
C1—C2—N2	105.37 (19)	N5—C18—H18C	109.5
C1—C2—C5	132.1 (2)	H18A—C18—H18C	109.5
N2—C2—C5	122.5 (2)	H18B—C18—H18C	109.5
N1—C3—N2	110.44 (19)	N5—C19—H19A	109.5
N1—C3—C6	125.5 (2)	N5—C19—H19B	109.5
N2—C3—C6	124.0 (2)	H19A—C19—H19B	109.5
O1—C4—O2	123.4 (2)	N5—C19—H19C	109.5
O1—C4—C1	118.6 (2)	H19A—C19—H19C	109.5
O2—C4—C1	117.9 (2)	H19B—C19—H19C	109.5
O4—C5—O3	124.4 (2)		

Symmetry codes: (i) $-x$, $-y+2$, $-z$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O9^{ii}	0.86	1.84	2.682 (3)	165
O3—H3···O2	0.82	1.65	2.471 (2)	176

O1W—H1W···O4^{iii}	0.85	1.92	2.764 (2)	170
O1W—H2W···O4^{iv}	0.84	2.11	2.927 (2)	164

Symmetry codes: (ii) −*x*+1, −*y*, −*z*+1; (iii) −*x*, −*y*+1, −*z*; (iv) *x*−1, *y*+1, *z*.

Fig. 1

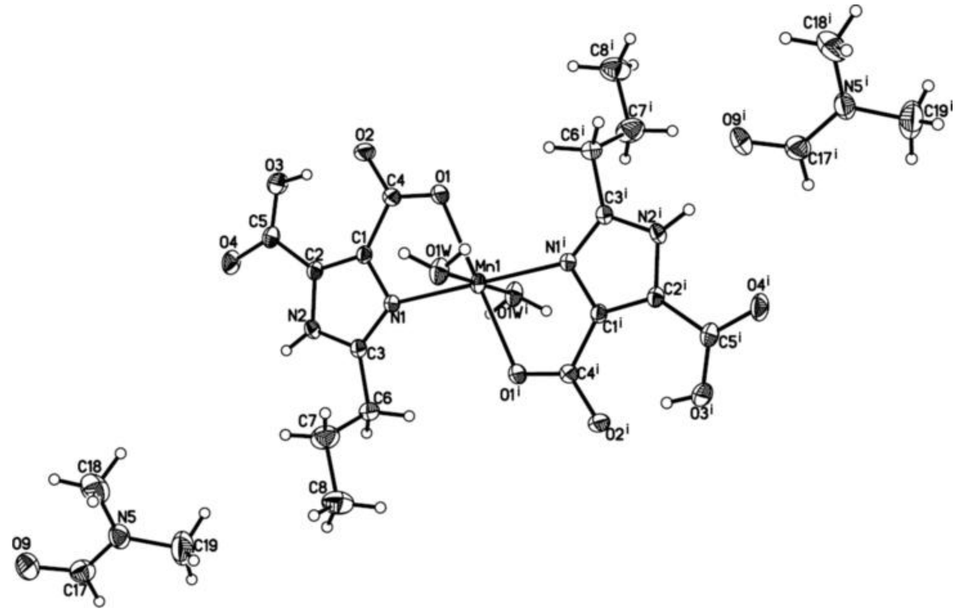
C18i
C8i
N5i
C7i
O9i
C19i
C6i
C17i
O2
O3
C4
O1
C3i
N2i
C5
O4
C2
C1
O1W
Mn1
N1i
N2
N1
O1Wi
C1i
C2i
O4i
C3
C5i
O1i
C4i
C6
C7
O2i
O3i
C18
C8
N5
O9
C19
C17

Fig. 2