

## Diaquabis(4-methylbenzoato- $\kappa$ O)bis-(nicotinamide- $\kappa$ N<sup>1</sup>)manganese(II)

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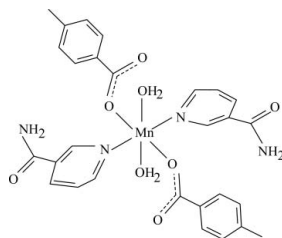
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.035;  $wR$  factor = 0.099; data-to-parameter ratio = 16.2.

In the mononuclear title complex,  $[\text{Mn}(\text{C}_8\text{H}_7\text{O}_2)_2(\text{C}_6\text{H}_6\text{N}_2\text{O})_2(\text{H}_2\text{O})_2]$ , the  $\text{Mn}^{\text{II}}$  ion is located on a crystallographic inversion center. The asymmetric unit contains one 4-methylbenzoate anion, one nicotinamide (NA) ligand and one coordinated water molecule. The four O atoms in the equatorial plane around the  $\text{Mn}^{\text{II}}$  ion form a slightly distorted square-planar arrangement, while the slightly distorted octahedral coordination is completed by the two pyridine N atoms of the NA ligands in the axial positions. The dihedral angle between the carboxylate group and the attached benzene ring is  $9.01(7)^\circ$ , while the pyridine and benzene rings are oriented at a dihedral angle of  $42.44(5)^\circ$ . In the crystal structure, intermolecular  $\text{O}-\text{H}\cdots\text{O}$ ,  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, and  $\text{O}-\text{H}\cdots\pi$  and  $\text{C}-\text{H}\cdots\pi$  interactions link the molecules into a two-dimensional network parallel to (001).

### Related literature

For niacin, see: Krishnamachari (1974), and for the nicotinic acid derivative  $N,N$ -diethylnicotinamide, see: Bigoli *et al.* (1972). For related structures, see: Hökelek *et al.* (1996, 2009*a,b,c*); Hökelek & Necefoğlu (1998); Necefoğlu *et al.* (2010).



### Experimental

#### Crystal data

$[\text{Mn}(\text{C}_8\text{H}_7\text{O}_2)_2(\text{C}_6\text{H}_6\text{N}_2\text{O})_2(\text{H}_2\text{O})_2]$   
 $M_r = 605.51$   
 Triclinic,  $P\bar{1}$   
 $a = 7.3289(2)$  Å  
 $b = 10.1768(3)$  Å  
 $c = 10.6292(3)$  Å  
 $\alpha = 66.852(2)^\circ$   
 $\beta = 78.232(4)^\circ$   
 $\gamma = 70.206(3)^\circ$   
 $V = 683.58(4)$  Å<sup>3</sup>  
 $Z = 1$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.54$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.38 \times 0.25 \times 0.19$  mm

#### Data collection

Bruker Kappa APEXII CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2005)  
 $T_{\text{min}} = 0.649$ ,  $T_{\text{max}} = 0.698$   
 11619 measured reflections  
 3297 independent reflections  
 3022 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.099$   
 $S = 1.08$   
 3297 reflections  
 204 parameters  
 3 restraints  
 H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.73$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.38$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

Mn1—O2	2.1036 (11)	Mn1—N1	2.2947 (13)
Mn1—O4	2.1924 (12)		

Table 2

Hydrogen-bond geometry (Å, °).

$Cg1$  and  $Cg2$  are the centroids of the  $C2-C7$  and  $N1/C9-C13$  rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2-H21\cdots O1^i$	0.88 (3)	1.97 (3)	2.8456 (19)	174 (2)
$N2-H22\cdots O3^{ii}$	0.86 (3)	2.09 (3)	2.952 (2)	172 (3)
$O4-H42\cdots O3^{iii}$	0.90 (3)	1.83 (3)	2.7071 (19)	164 (3)
$C11-H11\cdots O1^i$	0.93	2.33	3.200 (2)	156
$O4-H41\cdots Cg1^{iv}$	0.91 (2)	2.33 (2)	3.141 (2)	149 (3)
$C4-H4\cdots Cg2^{iv}$	0.93	2.80	3.490 (2)	132

Symmetry codes: (i)  $-x + 1, -y - 1, -z$ ; (ii)  $-x + 2, -y - 1, -z$ ; (iii)  $-x + 1, -y, -z$ ; (iv)  $-x + 1, -y + 2, -z$ .

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: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5069).

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**supplementary materials**

*Acta Cryst.* (2010). E66, m490-m491 [ doi:10.1107/S1600536810011815 ]

## Diaquabis(4-methylbenzoato- $\kappa O$ )bis(nicotinamide- $\kappa N^1$ )manganese(II)

H. Necefoglu, E. Çimen, B. Tercan, H. Dal and T. Hökelek

### Comment

As a part of our ongoing investigation on transition metal complexes of nicotinamide (NA), one form of niacin (Krishnamachari, 1974), and/or the nicotinic acid derivative *N,N*-diethylnicotinamide (DENA), an important respiratory stimulant (Bigoli *et al.*, 1972), the title compound was synthesized and its crystal structure is reported herein.

The title compound, (I), is a centrosymmetric mononuclear complex, consisting of two nicotinamide (NA) and two 4-methylbenzoate (PMB) ligands and two coordinated water molecules; the Mn<sup>II</sup> ion lies on a centre of symmetry (Fig. 1). The crystal structures of some PMB and/or NA complexes of Cu<sup>II</sup>, Co<sup>II</sup>, Ni<sup>II</sup>, Mn<sup>II</sup> and Zn<sup>II</sup> ions, [Cu(C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>14</sub>N<sub>2</sub>O)<sub>2</sub>], (II) (Hökelek *et al.*, 1996), [Co(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>(C<sub>7</sub>H<sub>4</sub>NO<sub>4</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>], (III) (Hökelek & Necefoglu, 1998), [Ni(C<sub>7</sub>H<sub>4</sub>ClO<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>], (IV) (Hökelek *et al.*, 2009a), [Ni(C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>], (V) (Necefoglu *et al.*, 2010), [Mn(C<sub>7</sub>H<sub>4</sub>ClO<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>14</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>], (VI) (Hökelek *et al.*, 2009b) and [Zn(C<sub>7</sub>H<sub>4</sub>BrO<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>], (VII) (Hökelek *et al.*, 2009c) have also been reported. In (II), the two benzoate ions are coordinated to the Cu atom as bidentate ligands, while in other structures all ligands being monodentate.

In the title compound, all ligands are monodentate. The four O atoms (O2, O4, and the symmetry-related atoms, O2', O4') in the equatorial plane around the Mn<sup>II</sup> atom form a slightly distorted square-planar arrangement, while the slightly distorted octahedral coordination is completed by the two N atoms of the NA ligands (N1, N1') in the axial positions (Fig. 1). The near equality of the C1—O1 [1.2466 (19) Å] and C1—O2 [1.2690 (18) Å] bonds in the carboxylate group indicates a delocalized bonding arrangement, rather than localized single and double bonds. The average Mn—O bond length is 2.148 (1) Å (Table 1). The dihedral angle between the planar carboxylate group (O1/O2/C1) and the benzene ring A (C2—C7) is 9.01 (7)°, while that between rings A and B (N1/C9—C13) is 42.44 (5)°.

In the crystal structure, intermolecular O—H $\cdots$ O, N—H $\cdots$ O and C—H $\cdots$ O hydrogen bonds (Table 2) link the molecules into a two-dimensional network parallel to the (001). Weak O—H $\cdots$  $\pi$  and C—H $\cdots$  $\pi$  interactions involving the pyridine and benzene rings are also observed (Table 2).

### Experimental

The title compound was prepared by the reaction of MnSO<sub>4</sub>·H<sub>2</sub>O (0.84 g, 5 mmol) in H<sub>2</sub>O (10 ml) and NA (1.22 g, 10 mmol) in H<sub>2</sub>O (10 ml) with sodium 4-methylbenzoate (1.58 g, 10 mmol) in H<sub>2</sub>O (150 ml). The mixture was filtered and set aside to crystallize at ambient temperature for one month, giving colourless single crystals.

### Refinement

H atoms of the NH<sub>2</sub> group (H21 and H22) and water molecules (H41 and H42) were located in a difference Fourier map and refined isotropically; the water H atoms were refined with O—H and H $\cdots$ H distance restraints. The remaining H atoms

## supplementary materials

were positioned geometrically with C–H = 0.93 and 0.96 Å, for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$ , where  $x = 1.5$  for methyl H and  $x = 1.2$  for aromatic H atoms.

### Figures

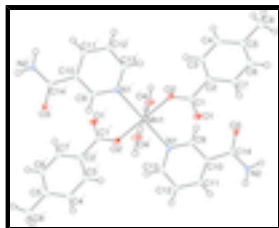


Fig. 1. The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Primed atoms are generated by the symmetry operator: (')  $-x, -y, -z$ .

### Diaquabis(4-methylbenzoato- $\kappa\text{O}$ )bis(nicotinamide- $\kappa\text{N}^1$ )manganese(II)

#### Crystal data

$[\text{Mn}(\text{C}_8\text{H}_7\text{O}_2)_2(\text{C}_6\text{H}_6\text{N}_2\text{O})_2(\text{H}_2\text{O})_2]$	$Z = 1$
$M_r = 605.51$	$F(000) = 315$
Triclinic, $P\bar{1}$	$D_x = 1.471 \text{ Mg m}^{-3}$
Hall symbol: $-P\ 1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 7.3289 (2) \text{ \AA}$	Cell parameters from 8087 reflections
$b = 10.1768 (3) \text{ \AA}$	$\theta = 2.5\text{--}28.4^\circ$
$c = 10.6292 (3) \text{ \AA}$	$\mu = 0.54 \text{ mm}^{-1}$
$\alpha = 66.852 (2)^\circ$	$T = 100 \text{ K}$
$\beta = 78.232 (4)^\circ$	Block, colourless
$\gamma = 70.206 (3)^\circ$	$0.38 \times 0.25 \times 0.19 \text{ mm}$
$V = 683.58 (4) \text{ \AA}^3$	

#### Data collection

Bruker Kappa APEXII CCD area-detector diffractometer	3297 independent reflections
Radiation source: fine-focus sealed tube graphite	3022 reflections with $I > 2\sigma(I)$
$\varphi$ and $\omega$ scans	$R_{\text{int}} = 0.028$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$\theta_{\text{max}} = 28.4^\circ$ , $\theta_{\text{min}} = 2.1^\circ$
$T_{\text{min}} = 0.649$ , $T_{\text{max}} = 0.698$	$h = -9 \rightarrow 9$
11619 measured reflections	$k = -11 \rightarrow 13$
	$l = -14 \rightarrow 14$

#### Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.099$

$S = 1.08$

3297 reflections

204 parameters

3 restraints

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.3059P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.73 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$$

### Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Mn1	0.0000	0.0000	0.0000	0.01276 (11)
O1	0.24237 (17)	-0.33733 (12)	0.25988 (12)	0.0207 (2)
O2	0.16077 (17)	-0.08763 (12)	0.17238 (12)	0.0201 (2)
O3	0.78404 (16)	-0.34130 (13)	-0.01469 (13)	0.0210 (3)
O4	0.18323 (19)	0.15102 (14)	-0.09400 (13)	0.0232 (3)
H41	0.208 (4)	0.190 (3)	-0.1860 (17)	0.064 (9)*
H42	0.218 (4)	0.205 (3)	-0.058 (2)	0.042 (7)*
N1	0.21166 (18)	-0.15932 (14)	-0.10409 (13)	0.0143 (3)
N2	0.84692 (19)	-0.50868 (14)	-0.11834 (14)	0.0167 (3)
H21	0.810 (3)	-0.555 (3)	-0.159 (2)	0.028 (5)*
H22	0.958 (4)	-0.545 (3)	-0.084 (2)	0.032 (6)*
C1	0.2620 (2)	-0.21470 (17)	0.24423 (15)	0.0151 (3)
C2	0.4196 (2)	-0.21314 (16)	0.31505 (15)	0.0145 (3)
C3	0.4331 (2)	-0.07942 (16)	0.31453 (15)	0.0164 (3)
H3	0.3426	0.0099	0.2714	0.020*
C4	0.5805 (2)	-0.07856 (18)	0.37770 (16)	0.0181 (3)
H4	0.5871	0.0115	0.3767	0.022*
C5	0.7191 (2)	-0.21079 (19)	0.44279 (16)	0.0190 (3)
C6	0.7063 (2)	-0.34413 (18)	0.44183 (16)	0.0203 (3)
H6	0.7979	-0.4333	0.4839	0.024*
C7	0.5589 (2)	-0.34546 (17)	0.37902 (16)	0.0177 (3)
H7	0.5528	-0.4355	0.3795	0.021*
C8	0.8783 (3)	-0.2097 (2)	0.51214 (18)	0.0257 (4)
H8A	0.8947	-0.1115	0.4763	0.039*
H8B	0.9977	-0.2797	0.4951	0.039*

## supplementary materials

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H8C	0.8437	-0.2371	0.6092	0.039*
C9	0.3975 (2)	-0.22336 (16)	-0.07444 (15)	0.0132 (3)
H9	0.4418	-0.1971	-0.0142	0.016*
C10	0.5280 (2)	-0.32707 (16)	-0.12893 (15)	0.0134 (3)
C11	0.4623 (2)	-0.36415 (17)	-0.22143 (16)	0.0170 (3)
H11	0.5453	-0.4328	-0.2602	0.020*
C12	0.2710 (2)	-0.29647 (18)	-0.25420 (17)	0.0198 (3)
H12	0.2239	-0.3182	-0.3164	0.024*
C13	0.1508 (2)	-0.19621 (17)	-0.19343 (16)	0.0172 (3)
H13	0.0221	-0.1523	-0.2153	0.021*
C14	0.7300 (2)	-0.39401 (16)	-0.08365 (15)	0.0142 (3)

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Mn1	0.01065 (17)	0.01054 (16)	0.01760 (17)	0.00071 (11)	-0.00334 (11)	-0.00758 (12)
O1	0.0252 (6)	0.0149 (5)	0.0241 (6)	-0.0030 (4)	-0.0062 (5)	-0.0092 (5)
O2	0.0216 (6)	0.0138 (5)	0.0243 (6)	0.0035 (4)	-0.0106 (5)	-0.0092 (4)
O3	0.0160 (5)	0.0184 (5)	0.0343 (6)	0.0021 (4)	-0.0096 (5)	-0.0174 (5)
O4	0.0308 (7)	0.0236 (6)	0.0233 (6)	-0.0146 (5)	0.0056 (5)	-0.0147 (5)
N1	0.0126 (6)	0.0117 (6)	0.0177 (6)	-0.0003 (4)	-0.0030 (5)	-0.0061 (5)
N2	0.0120 (6)	0.0146 (6)	0.0258 (7)	0.0020 (5)	-0.0057 (5)	-0.0125 (5)
C1	0.0149 (7)	0.0150 (7)	0.0155 (7)	0.0004 (5)	-0.0019 (5)	-0.0091 (6)
C2	0.0147 (7)	0.0137 (7)	0.0140 (6)	-0.0008 (5)	-0.0017 (5)	-0.0061 (5)
C3	0.0175 (7)	0.0129 (7)	0.0164 (7)	-0.0004 (5)	-0.0029 (6)	-0.0055 (6)
C4	0.0209 (8)	0.0181 (7)	0.0173 (7)	-0.0059 (6)	-0.0014 (6)	-0.0080 (6)
C5	0.0170 (7)	0.0245 (8)	0.0158 (7)	-0.0045 (6)	-0.0017 (6)	-0.0083 (6)
C6	0.0182 (7)	0.0177 (7)	0.0196 (7)	0.0026 (6)	-0.0066 (6)	-0.0053 (6)
C7	0.0203 (7)	0.0124 (7)	0.0186 (7)	-0.0006 (5)	-0.0041 (6)	-0.0056 (6)
C8	0.0199 (8)	0.0352 (10)	0.0240 (8)	-0.0067 (7)	-0.0061 (6)	-0.0114 (7)
C9	0.0137 (7)	0.0105 (6)	0.0160 (6)	-0.0017 (5)	-0.0027 (5)	-0.0060 (5)
C10	0.0118 (7)	0.0101 (6)	0.0174 (7)	-0.0007 (5)	-0.0031 (5)	-0.0051 (5)
C11	0.0146 (7)	0.0149 (7)	0.0224 (7)	0.0015 (5)	-0.0042 (6)	-0.0110 (6)
C12	0.0181 (8)	0.0194 (7)	0.0258 (8)	0.0016 (6)	-0.0092 (6)	-0.0142 (7)
C13	0.0137 (7)	0.0150 (7)	0.0229 (7)	0.0008 (5)	-0.0059 (6)	-0.0087 (6)
C14	0.0127 (7)	0.0113 (6)	0.0184 (7)	-0.0007 (5)	-0.0033 (5)	-0.0063 (5)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Mn1—O2	2.1036 (11)	C4—C3	1.387 (2)
Mn1—O2 <sup>i</sup>	2.1036 (11)	C4—C5	1.396 (2)
Mn1—O4	2.1924 (12)	C4—H4	0.93
Mn1—O4 <sup>i</sup>	2.1924 (12)	C5—C8	1.507 (2)
Mn1—N1	2.2947 (13)	C6—C5	1.396 (2)
Mn1—N1 <sup>i</sup>	2.2947 (13)	C6—C7	1.387 (2)
O1—C1	1.2466 (19)	C6—H6	0.93
O2—C1	1.2690 (18)	C7—H7	0.93
O3—C14	1.2464 (18)	C8—H8A	0.96

O4—H41	0.905 (16)	C8—H8B	0.96
O4—H42	0.906 (16)	C8—H8C	0.96
N1—C9	1.3386 (18)	C9—C10	1.391 (2)
N1—C13	1.345 (2)	C9—H9	0.93
N2—C14	1.3283 (19)	C11—C10	1.396 (2)
N2—H21	0.89 (2)	C11—C12	1.385 (2)
N2—H22	0.86 (2)	C11—H11	0.93
C1—C2	1.509 (2)	C12—H12	0.93
C2—C3	1.395 (2)	C13—C12	1.383 (2)
C2—C7	1.397 (2)	C13—H13	0.93
C3—H3	0.93	C14—C10	1.4956 (19)
O2—Mn1—O2 <sup>i</sup>	180.00 (4)	C3—C4—H4	119.5
O2—Mn1—O4	86.18 (5)	C5—C4—H4	119.5
O2 <sup>i</sup> —Mn1—O4	93.82 (5)	C4—C5—C8	120.94 (15)
O2—Mn1—O4 <sup>i</sup>	93.82 (5)	C6—C5—C4	118.20 (14)
O2 <sup>i</sup> —Mn1—O4 <sup>i</sup>	86.18 (5)	C6—C5—C8	120.86 (15)
O2—Mn1—N1 <sup>i</sup>	86.02 (4)	C5—C6—H6	119.5
O2 <sup>i</sup> —Mn1—N1 <sup>i</sup>	93.98 (4)	C7—C6—C5	120.91 (14)
O2—Mn1—N1	93.98 (4)	C7—C6—H6	119.5
O2 <sup>i</sup> —Mn1—N1	86.02 (4)	C2—C7—H7	119.6
O4—Mn1—O4 <sup>i</sup>	180.00 (5)	C6—C7—C2	120.74 (14)
O4—Mn1—N1 <sup>i</sup>	91.32 (4)	C6—C7—H7	119.6
O4 <sup>i</sup> —Mn1—N1 <sup>i</sup>	88.68 (4)	C5—C8—H8A	109.5
O4—Mn1—N1	88.68 (4)	C5—C8—H8B	109.5
O4 <sup>i</sup> —Mn1—N1	91.32 (4)	C5—C8—H8C	109.5
N1 <sup>i</sup> —Mn1—N1	180.00 (9)	H8A—C8—H8B	109.5
C1—O2—Mn1	136.86 (10)	H8A—C8—H8C	109.5
Mn1—O4—H41	122.2 (18)	H8B—C8—H8C	109.5
Mn1—O4—H42	129.2 (16)	N1—C9—C10	123.52 (13)
H42—O4—H41	107 (2)	N1—C9—H9	118.2
C9—N1—Mn1	121.52 (10)	C10—C9—H9	118.2
C9—N1—C13	117.56 (13)	C9—C10—C11	118.15 (13)
C13—N1—Mn1	120.88 (10)	C9—C10—C14	118.12 (13)
C14—N2—H21	124.4 (15)	C11—C10—C14	123.72 (13)
C14—N2—H22	113.3 (16)	C10—C11—H11	120.7
H21—N2—H22	121 (2)	C12—C11—C10	118.59 (14)
O1—C1—O2	125.89 (14)	C12—C11—H11	120.7
O1—C1—C2	118.59 (13)	C11—C12—H12	120.4
O2—C1—C2	115.51 (13)	C13—C12—C11	119.26 (14)
C3—C2—C1	120.90 (13)	C13—C12—H12	120.4
C3—C2—C7	118.51 (14)	N1—C13—C12	122.90 (14)
C7—C2—C1	120.56 (13)	N1—C13—H13	118.6
C2—C3—H3	119.7	C12—C13—H13	118.6
C4—C3—C2	120.59 (14)	O3—C14—N2	121.84 (14)
C4—C3—H3	119.7	O3—C14—C10	119.46 (13)
C3—C4—C5	121.05 (14)	N2—C14—C10	118.71 (13)



## supplementary materials

O4—Mn1—O2—C1	-124.12 (16)	O2—C1—C2—C7	-169.99 (14)
O4 <sup>i</sup> —Mn1—O2—C1	55.88 (16)	C1—C2—C3—C4	-178.83 (14)
N1 <sup>i</sup> —Mn1—O2—C1	144.28 (16)	C7—C2—C3—C4	-0.8 (2)
N1—Mn1—O2—C1	-35.72 (16)	C1—C2—C7—C6	178.70 (15)
O2—Mn1—N1—C9	-23.31 (12)	C3—C2—C7—C6	0.6 (2)
O2 <sup>i</sup> —Mn1—N1—C9	156.69 (12)	C5—C4—C3—C2	0.2 (2)
O2—Mn1—N1—C13	154.04 (12)	C3—C4—C5—C6	0.4 (2)
O2 <sup>i</sup> —Mn1—N1—C13	-25.96 (12)	C3—C4—C5—C8	-179.60 (15)
O4—Mn1—N1—C9	62.76 (11)	C5—C6—C7—C2	0.0 (2)
O4 <sup>i</sup> —Mn1—N1—C9	-117.24 (11)	C7—C6—C5—C4	-0.6 (2)
O4—Mn1—N1—C13	-119.89 (12)	C7—C6—C5—C8	179.46 (15)
O4 <sup>i</sup> —Mn1—N1—C13	60.11 (12)	N1—C9—C10—C11	1.2 (2)
Mn1—O2—C1—O1	-26.6 (3)	N1—C9—C10—C14	-177.98 (13)
Mn1—O2—C1—C2	153.01 (12)	C12—C11—C10—C9	-0.1 (2)
Mn1—N1—C9—C10	176.18 (11)	C12—C11—C10—C14	179.01 (15)
C13—N1—C9—C10	-1.3 (2)	C10—C11—C12—C13	-0.8 (2)
Mn1—N1—C13—C12	-177.22 (13)	N1—C13—C12—C11	0.8 (3)
C9—N1—C13—C12	0.2 (2)	O3—C14—C10—C9	-9.9 (2)
O1—C1—C2—C3	-172.31 (14)	O3—C14—C10—C11	170.95 (15)
O1—C1—C2—C7	9.7 (2)	N2—C14—C10—C9	170.04 (14)
O2—C1—C2—C3	8.0 (2)	N2—C14—C10—C11	-9.1 (2)

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

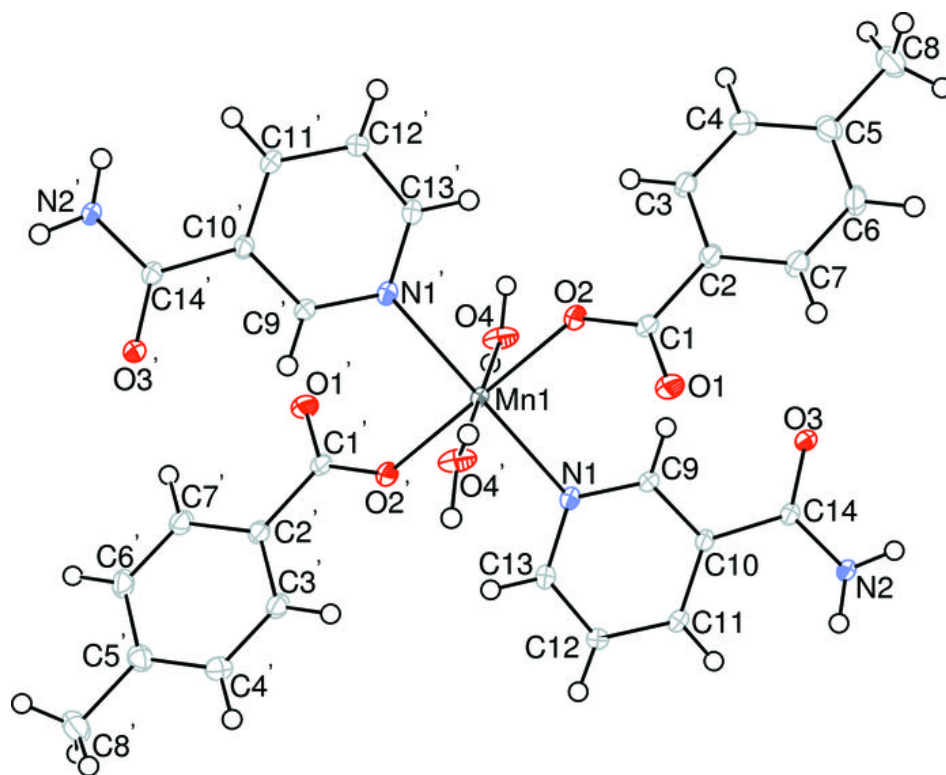
### Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ )

Cg1 and Cg2 are the centroids of the C2-C7 and N1/C9-C13 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H21 $\cdots$ O1 <sup>ii</sup>	0.88 (3)	1.97 (3)	2.8456 (19)	174 (2)
N2—H22 $\cdots$ O3 <sup>iii</sup>	0.86 (3)	2.09 (3)	2.952 (2)	172 (3)
O4—H42 $\cdots$ O3 <sup>iv</sup>	0.90 (3)	1.83 (3)	2.7071 (19)	164 (3)
C11—H11 $\cdots$ O1 <sup>ii</sup>	0.93	2.33	3.200 (2)	156
O4—H41 $\cdots$ Cg1 <sup>v</sup>	0.91 (2)	2.33 (2)	3.141 (2)	149 (3)
C4—H4 $\cdots$ Cg2 <sup>v</sup>	0.93	2.80	3.490 (2)	132

Symmetry codes: (ii)  $-x+1, -y-1, -z$ ; (iii)  $-x+2, -y-1, -z$ ; (iv)  $-x+1, -y, -z$ ; (v)  $-x+1, -y+2, -z$ .

Fig. 1



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## Structure Reports

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# *N*-(2,3-Dimethylphenyl)-2,4-dimethylbenzenesulfonamide

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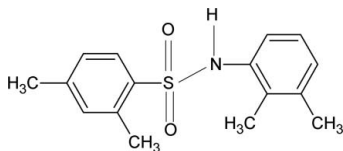
Received 27 March 2010; accepted 28 March 2010

 Key indicators: single-crystal X-ray study;  $T = 299$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.058;  $wR$  factor = 0.161; data-to-parameter ratio = 16.4.

The asymmetric unit of the title compound,  $\text{C}_{16}\text{H}_{19}\text{NO}_2\text{S}$ , contains two independent molecules: the dihedral angles between the sulfonyl and anilino benzene rings in the two molecules are 41.5 (1) and 43.8 (1)°. The independent molecules are linked into a dimer by a pair of intermolecular N—H···O hydrogen bonds.

## Related literature

For the preparation of the title compound, see: Savitha & Gowda (2006). For our studies of the effect of substituents on the structures of *N*-(aryl)arylsulfonamides, see: Gowda *et al.* (2009*a,b,c*). For related structures, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006).



## Experimental

### Crystal data

$\text{C}_{16}\text{H}_{19}\text{NO}_2\text{S}$   
 $M_r = 289.38$   
 Triclinic,  $P\bar{1}$   
 $a = 8.3643$  (7) Å  
 $b = 10.975$  (1) Å

$c = 16.996$  (2) Å  
 $\alpha = 83.034$  (9)°  
 $\beta = 80.100$  (7)°  
 $\gamma = 81.796$  (9)°  
 $V = 1513.7$  (3) Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.22$  mm<sup>-1</sup>

$T = 299$  K  
 $0.34 \times 0.30 \times 0.20$  mm

### Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector  
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)  
 $T_{\min} = 0.931$ ,  $T_{\max} = 0.958$   
 11164 measured reflections  
 6136 independent reflections  
 4196 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$   
 $wR(F^2) = 0.161$   
 $S = 1.03$   
 6136 reflections  
 375 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.66$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.41$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···O3	0.83 (3)	2.15 (3)	2.952 (3)	161 (3)
N2—H2N···O1	0.79 (3)	2.22 (3)	2.982 (3)	164 (3)

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5070).

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