

Tetraaquis[4-(methylamino)benzoato- κ O]nickel(II)

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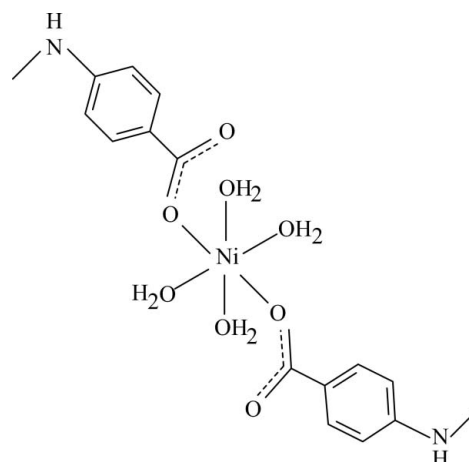
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.023; wR factor = 0.061; data-to-parameter ratio = 15.5.

The title complex, $[\text{Ni}(\text{C}_8\text{H}_8\text{NO}_2)_2(\text{H}_2\text{O})_4]$, is centrosymmetric with the Ni^{II} ion located on a centre of symmetry. It contains two 4-(methylamino)benzoate (PMAB) anions and four coordinated water molecules. The four O atoms in the equatorial plane around the Ni^{II} ion form a slightly distorted square-planar arrangement, while the slightly distorted octahedral coordination is completed by two O atoms of the PMAB anions in the axial positions. In the crystal structure, intermolecular $\text{O}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{N}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into a three-dimensional network.

Related literature

For structure–function–coordination relationships of the arylcarboxylate ion in transition-metal complexes of benzoic acid derivatives, see: Nadzhafov *et al.* (1981); Shnulin *et al.* (1981). For studies of transition-metal complexes with biochemical model systems, see: Antolini *et al.* (1982). For the coordination modes of benzoic acid derivatives, see: Chen & Chen (2002); Amiraslanov *et al.* (1979); Hauptmann *et al.* (2000). For related structures, see: Hökelek *et al.* (2009a,b); Necefoğlu *et al.* (2010); Sertçelik *et al.* (2009).



Experimental

Crystal data

$[\text{Ni}(\text{C}_8\text{H}_8\text{NO}_2)_2(\text{H}_2\text{O})_4]$
 $M_r = 431.06$
Monoclinic, $P2_1/n$
 $a = 7.5466$ (2) Å
 $b = 6.1811$ (2) Å
 $c = 19.4802$ (3) Å
 $\beta = 98.628$ (3)°

$V = 898.40$ (4) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 1.13$ mm⁻¹
 $T = 100$ K
 $0.35 \times 0.25 \times 0.13$ mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\text{min}} = 0.718$, $T_{\text{max}} = 0.863$

8475 measured reflections
2242 independent reflections
2085 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.061$
 $S = 1.06$
2242 reflections
145 parameters
6 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Selected bond lengths (Å).

Ni1—O2	2.0537 (10)	Ni1—O4	2.0772 (10)
Ni1—O3	2.0662 (10)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
N1—H1 ⁱ ⋯O4 ⁱ	0.85 (2)	2.55 (2)	3.3902 (16)	170 (2)
O3—H31 ⁱ ⋯N1 ⁱⁱ	0.91 (2)	1.97 (2)	2.8780 (16)	172 (2)
O3—H32 ⁱ ⋯O1 ⁱⁱⁱ	0.87 (2)	1.90 (2)	2.7131 (15)	155 (2)
O4—H41 ⁱ ⋯O1 ^{iv}	0.96 (2)	1.68 (2)	2.6240 (14)	165 (3)
O4—H42 ⁱ ⋯O1 ⁱⁱⁱ	0.92 (2)	2.11 (2)	2.8781 (15)	139 (2)
C8—H8A ⁱ ⋯O3 ^v	0.96	2.43	3.2583 (18)	144
C8—H8C ⁱ ⋯O1 ^{vi}	0.96	2.47	3.4105 (19)	168

Symmetry codes: (i) $-x - \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, y - 1, z$; (iv) $-x, -y + 1, -z$; (v) $x - \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (vi) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5081).

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supplementary materials

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Tetraaquabis[4-(methylamino)benzoato- κ O]nickel(II)

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Comment

The structure-function-coordination relationships of the arylcarboxylate ion in transition metal complexes of benzoic acid derivatives change, depending on the nature and position of the substituent groups on the benzene ring, the nature of the additional ligand molecule or solvent, and the medium of synthesis (Nadzhafov *et al.*, 1981; Shnulin *et al.*, 1981). Transition metal complexes with biochemical molecules frequently show interesting physical and/or chemical properties, as a result they may find applications in biological systems (Antolini *et al.*, 1982). Some benzoic acid derivatives, such as 4-aminobenzoic acid, have been extensively reported in coordination chemistry, as bifunctional organic ligands, due to varieties of their coordination modes (Chen & Chen, 2002; Amiraslanov *et al.*, 1979; Hauptmann *et al.*, 2000). The title compound was synthesized and its crystal structure is reported herein.

The title compound is a monomeric complex, with the Ni^{II} ion on a centre of symmetry. It contains two 4-(methylamino)benzoate (PMAB) ligands and four coordinated water molecules. The PMAB ligands are monodentate. The four O atoms (O3, O4, and the symmetry-related atoms O3' and O4') in the equatorial plane around the Ni atom form a slightly distorted square-planar arrangement, while the slightly distorted octahedral coordination is completed by the carboxylate O atoms (O2 and O2') of the symmetry related PMAB ligands (Fig. 1 and Table 1).

The C1—O1 [1.2761 (17) Å] and C1—O2 [1.2561 (18) Å] bonds in the carboxylate groups may be compared with the corresponding distances: 1.263 (4) and 1.249 (4) Å in [Ni(C₈H₅O₃)₂(C₁₀H₁₄N₂O)₂(H₂O)₂] (Sertçelik *et al.*, 2009), 1.267 (3) and 1.258 (3) Å in [Ni(C₇H₄ClO₂)₂(C₆H₆N₂O)₂(H₂O)₂] (Hökelek *et al.*, 2009a), 1.2616 (17) and 1.2435 (18) Å in [Ni(C₇H₄ClO₂)₂(C₁₀H₁₄N₂O)₂(H₂O)₂] (Hökelek *et al.*, 2009b), and 1.2678 (17) and 1.2654 (17) Å in [Ni(C₈H₇O₂)₂(C₆H₆N₂O)₂(H₂O)₂] (Necefoglu *et al.*, 2010).

The Ni atom is displaced out of the least-square plane of the carboxylate group (O1/C1/O2) by 0.0728 (1) Å. On the other hand, O1, O2, N1 and C1 atoms are 0.0061 (11), 0.0549 (10), -0.0636 (13) and 0.0163 (13) Å away from the plane of the benzene ring A (C2—C7), respectively.

In the crystal structure, intermolecular O—H \cdots O, O—H \cdots N, N—H \cdots O and C—H \cdots O hydrogen bonds (Table 2) link the molecules into a three-dimensional network.

Experimental

The title compound was prepared by the reaction of Ni(SO₄).6(H₂O) (1.31 g, 5 mmol) in H₂O (50 ml) and sodium 4-(methylamino)benzoate (1.74 g, 10 mmol) in H₂O (50 ml). The mixture was filtered and set aside to crystallize at ambient temperature for one week, giving green single crystals.

Refinement

H atoms of NH group and water molecules were located in difference maps and refined isotropically; the O–H and H···H distances in the water molecules were restrained to 0.95 (2) Å and 1.46 (4) Å, respectively. The remaining H atoms were positioned geometrically with C–H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.2$ for aromatic H and $x = 1.5$ for methyl H atoms.

Figures

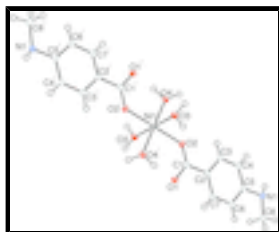


Fig. 1. The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The primed atoms are generated by the symmetry operation $(-x, 1 - y, -z)$.

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Crystal data

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$M_r = 431.06$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 7.5466$ (2) Å

$b = 6.1811$ (2) Å

$c = 19.4802$ (3) Å

$\beta = 98.628$ (3)°

$V = 898.40$ (4) Å³

$Z = 2$

$F(000) = 452$

$D_x = 1.594$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5176 reflections

$\theta = 2.7\text{--}28.4^\circ$

$\mu = 1.13$ mm⁻¹

$T = 100$ K

Block, green

$0.35 \times 0.25 \times 0.13$ mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\text{min}} = 0.718$, $T_{\text{max}} = 0.863$

8475 measured reflections

2242 independent reflections

2085 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\text{max}} = 28.4^\circ$, $\theta_{\text{min}} = 2.1^\circ$

$h = -10 \rightarrow 10$

$k = -8 \rightarrow 8$

$l = -25 \rightarrow 26$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.023$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.061$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.3955P]$
2242 reflections	where $P = (F_o^2 + 2F_c^2)/3$
145 parameters	$(\Delta/\sigma)_{\max} = 0.001$
6 restraints	$\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.0000	0.5000	0.0000	0.01094 (9)
O1	0.12315 (14)	0.95272 (17)	0.08266 (5)	0.0166 (2)
O2	-0.00352 (13)	0.63206 (17)	0.09653 (5)	0.0153 (2)
O3	0.23373 (13)	0.34113 (17)	0.03994 (5)	0.0150 (2)
H31	0.327 (2)	0.418 (3)	0.0632 (10)	0.031 (5)*
H32	0.220 (3)	0.227 (3)	0.0651 (11)	0.044 (7)*
O4	-0.15665 (13)	0.25462 (17)	0.03223 (5)	0.0146 (2)
H41	-0.163 (4)	0.167 (4)	-0.0089 (11)	0.070 (9)*
H42	-0.090 (3)	0.178 (4)	0.0679 (10)	0.046 (7)*
N1	-0.05050 (17)	1.0577 (2)	0.39282 (6)	0.0155 (2)
H1	-0.133 (3)	0.984 (3)	0.4069 (11)	0.022 (5)*
C1	0.04946 (17)	0.8156 (2)	0.11841 (7)	0.0130 (3)
C2	0.02308 (17)	0.8804 (2)	0.19011 (7)	0.0126 (3)
C3	-0.05536 (18)	0.7378 (2)	0.23236 (7)	0.0149 (3)
H3	-0.0902	0.6007	0.2159	0.018*
C4	-0.08175 (18)	0.7983 (2)	0.29847 (7)	0.0156 (3)
H4	-0.1342	0.7015	0.3260	0.019*

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C5	-0.03016 (18)	1.0039 (2)	0.32435 (7)	0.0137 (3)
C6	0.04875 (18)	1.1470 (2)	0.28224 (7)	0.0147 (3)
H6	0.0840	1.2840	0.2987	0.018*
C7	0.07450 (18)	1.0852 (2)	0.21607 (7)	0.0142 (3)
H7	0.1269	1.1817	0.1885	0.017*
C8	-0.0564 (2)	1.2874 (3)	0.41124 (7)	0.0182 (3)
H8A	-0.0838	1.3009	0.4576	0.027*
H8B	-0.1473	1.3589	0.3795	0.027*
H8C	0.0578	1.3526	0.4087	0.027*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.01233 (13)	0.00929 (13)	0.01089 (13)	-0.00098 (9)	0.00076 (9)	0.00030 (8)
O1	0.0221 (5)	0.0123 (5)	0.0157 (5)	-0.0032 (4)	0.0035 (4)	0.0001 (4)
O2	0.0192 (5)	0.0124 (5)	0.0140 (4)	-0.0025 (4)	0.0015 (4)	-0.0013 (4)
O3	0.0151 (5)	0.0128 (5)	0.0163 (5)	-0.0011 (4)	-0.0001 (4)	0.0012 (4)
O4	0.0162 (5)	0.0130 (5)	0.0144 (4)	-0.0015 (4)	0.0011 (4)	0.0005 (4)
N1	0.0159 (6)	0.0170 (6)	0.0143 (5)	-0.0006 (5)	0.0043 (4)	0.0006 (5)
C1	0.0118 (6)	0.0125 (6)	0.0138 (6)	0.0023 (5)	-0.0009 (4)	0.0013 (5)
C2	0.0115 (5)	0.0122 (6)	0.0134 (6)	0.0004 (5)	-0.0003 (4)	-0.0003 (5)
C3	0.0145 (6)	0.0122 (6)	0.0171 (6)	-0.0014 (5)	-0.0002 (5)	-0.0003 (5)
C4	0.0159 (6)	0.0151 (7)	0.0160 (6)	-0.0013 (5)	0.0026 (5)	0.0033 (5)
C5	0.0107 (6)	0.0158 (7)	0.0141 (6)	0.0021 (5)	0.0001 (5)	0.0008 (5)
C6	0.0143 (6)	0.0132 (6)	0.0163 (6)	-0.0014 (5)	0.0017 (5)	-0.0014 (5)
C7	0.0142 (6)	0.0134 (7)	0.0151 (6)	-0.0013 (5)	0.0021 (5)	0.0005 (5)
C8	0.0196 (6)	0.0185 (7)	0.0172 (6)	-0.0005 (6)	0.0055 (5)	-0.0018 (5)

Geometric parameters (\AA , $^\circ$)

Ni1—O2	2.0537 (10)	C2—C3	1.3969 (19)
Ni1—O2 ⁱ	2.0537 (10)	C3—H3	0.93
Ni1—O3	2.0662 (10)	C4—C3	1.3839 (19)
Ni1—O3 ⁱ	2.0662 (10)	C4—C5	1.401 (2)
Ni1—O4	2.0772 (10)	C4—H4	0.93
Ni1—O4 ⁱ	2.0772 (10)	C6—C5	1.3987 (19)
O1—C1	1.2761 (17)	C6—H6	0.93
O2—C1	1.2561 (18)	C7—C2	1.396 (2)
O3—H31	0.909 (16)	C7—C6	1.3858 (19)
O3—H32	0.873 (17)	C7—H7	0.93
O4—H41	0.964 (17)	C8—N1	1.466 (2)
O4—H42	0.926 (17)	C8—H8A	0.96
N1—C5	1.4052 (18)	C8—H8B	0.96
N1—H1	0.85 (2)	C8—H8C	0.96
C2—C1	1.4948 (18)		
O2—Ni1—O3	88.38 (4)	O2—C1—O1	123.87 (13)
O2 ⁱ —Ni1—O3	91.62 (4)	O2—C1—C2	118.61 (12)
O2—Ni1—O2 ⁱ	180.00 (2)	C3—C2—C1	120.60 (13)

O2—Ni1—O3 ⁱ	91.62 (4)	C7—C2—C1	120.83 (12)
O2 ⁱ —Ni1—O3 ⁱ	88.38 (4)	C7—C2—C3	118.57 (12)
O2—Ni1—O4	85.83 (4)	C2—C3—H3	119.6
O2 ⁱ —Ni1—O4	94.17 (4)	C4—C3—C2	120.71 (13)
O2—Ni1—O4 ⁱ	94.17 (4)	C4—C3—H3	119.6
O2 ⁱ —Ni1—O4 ⁱ	85.83 (4)	C3—C4—C5	120.60 (13)
O3 ⁱ —Ni1—O3	180.00 (5)	C3—C4—H4	119.7
O3—Ni1—O4	91.81 (4)	C5—C4—H4	119.7
O3 ⁱ —Ni1—O4	88.19 (4)	C4—C5—N1	119.48 (13)
O3—Ni1—O4 ⁱ	88.19 (4)	C6—C5—N1	121.60 (13)
O3 ⁱ —Ni1—O4 ⁱ	91.81 (4)	C6—C5—C4	118.84 (13)
O4—Ni1—O4 ⁱ	180.00 (5)	C5—C6—H6	119.9
C1—O2—Ni1	128.50 (9)	C7—C6—C5	120.18 (13)
Ni1—O3—H31	119.5 (15)	C7—C6—H6	119.9
Ni1—O3—H32	115.3 (16)	C2—C7—H7	119.4
H31—O3—H32	106.7 (19)	C6—C7—C2	121.11 (13)
Ni1—O4—H41	96.8 (18)	C6—C7—H7	119.4
Ni1—O4—H42	109.2 (15)	N1—C8—H8A	109.5
H42—O4—H41	107 (2)	N1—C8—H8B	109.5
C5—N1—C8	118.22 (12)	N1—C8—H8C	109.5
C5—N1—H1	111.6 (14)	H8A—C8—H8B	109.5
C8—N1—H1	113.0 (13)	H8A—C8—H8C	109.5
O1—C1—C2	117.52 (12)	H8B—C8—H8C	109.5
O3—Ni1—O2—C1	98.00 (11)	C7—C2—C1—O2	-177.98 (12)
O3 ⁱ —Ni1—O2—C1	-82.00 (11)	C1—C2—C3—C4	-179.27 (12)
O4—Ni1—O2—C1	-170.07 (12)	C7—C2—C3—C4	0.1 (2)
O4 ⁱ —Ni1—O2—C1	9.93 (12)	C5—C4—C3—C2	0.0 (2)
Ni1—O2—C1—O1	-2.6 (2)	C3—C4—C5—N1	-176.96 (13)
Ni1—O2—C1—C2	176.51 (8)	C3—C4—C5—C6	-0.1 (2)
C8—N1—C5—C4	-159.10 (13)	C7—C6—C5—N1	176.96 (13)
C8—N1—C5—C6	24.13 (19)	C7—C6—C5—C4	0.2 (2)
C3—C2—C1—O1	-179.47 (12)	C6—C7—C2—C1	179.34 (12)
C3—C2—C1—O2	1.36 (19)	C6—C7—C2—C3	0.0 (2)
C7—C2—C1—O1	1.19 (19)	C2—C7—C6—C5	-0.1 (2)

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

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O4 ⁱⁱ	0.85 (2)	2.55 (2)	3.3902 (16)	170 (2)
O3—H31 \cdots N1 ⁱⁱⁱ	0.91 (2)	1.97 (2)	2.8780 (16)	172 (2)
O3—H32 \cdots O1 ^{iv}	0.87 (2)	1.90 (2)	2.7131 (15)	155 (2)
O4—H41 \cdots O1 ⁱ	0.96 (2)	1.68 (2)	2.6240 (14)	165 (3)
O4—H42 \cdots O1 ^{iv}	0.92 (2)	2.11 (2)	2.8781 (15)	139 (2)
C8—H8A \cdots O3 ^v	0.96	2.43	3.2583 (18)	144

supplementary materials

C8—H8C \cdots O1^{vi}

0.96

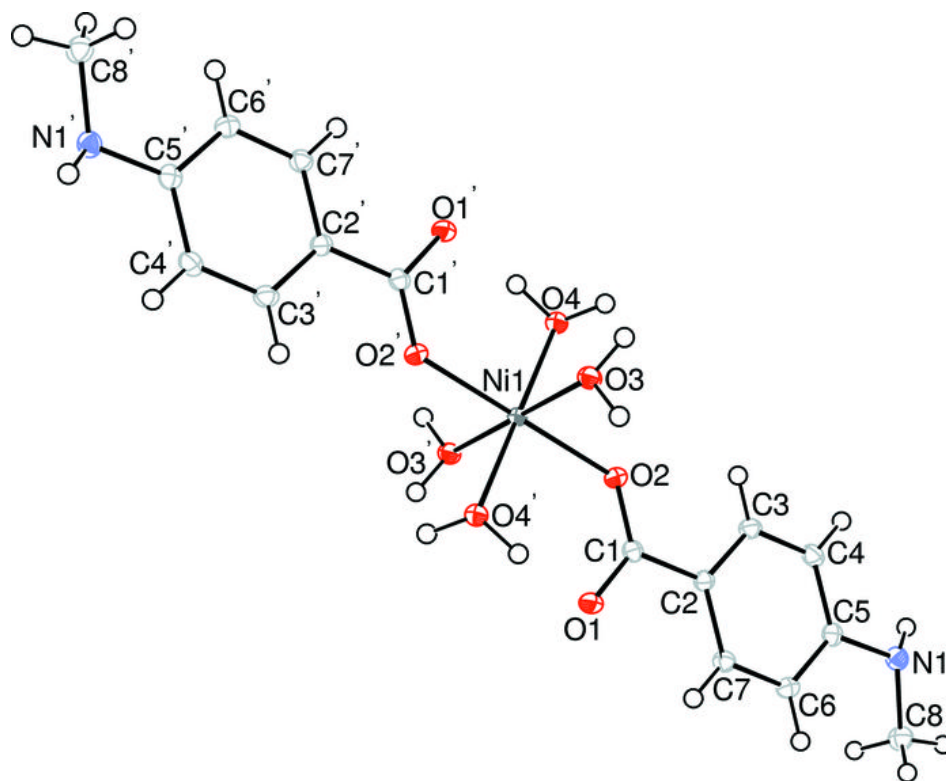
2.47

3.4105 (19)

168

Symmetry codes: (ii) $-x-1/2, y+1/2, -z+1/2$; (iii) $-x+1/2, y-1/2, -z+1/2$; (iv) $x, y-1, z$; (i) $-x, -y+1, -z$; (v) $x-1/2, -y+3/2, z+1/2$; (vi) $-x+1/2, y+1/2, -z+1/2$.

Fig. 1



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Structure Reports

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N'-Acetyl-5-amino-1-methyl-1*H*-pyrazole-4-carbohydrazonamide dihydrate

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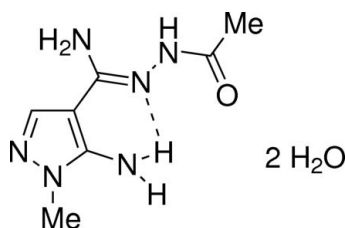
Received 22 April 2010; accepted 26 April 2010

 Key indicators: single-crystal X-ray study; $T = 223$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.052; wR factor = 0.141; data-to-parameter ratio = 13.9.

In the title compound, $\text{C}_7\text{H}_{12}\text{N}_6\text{O}\cdot 2\text{H}_2\text{O}$, the *Z* configuration of the hydrazone fragment is stabilized by an intramolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bond involving one of the amino groups. In the crystal structure, the hydrazonamide molecules are connected *via* intermolecular $\text{N}-\text{H}\cdots\text{O}=\text{C}$ hydrogen bonds, forming *C*(7) chains running along [010]. The chains form sheets parallel to the (101). The chains are cross-linked by water molecules to form a three-dimensional hydrogen-bonded network.

Related literature

For bioactive pyrazoles, see: Elguero *et al.* (2002); Lamberth (2007). For the use of pyrazoles as synthons in heterocyclic chemistry, see: Schenone *et al.* (2007); Dolzhenko *et al.* (2008). For the use of pyrazoles in metal-organic chemistry, see: Mukherjee (2000); Halcrow (2009). For the crystal structures of related 5-amino-1*H*-pyrazole-4-carboxylic acid derivatives, see: Zia-ur-Rehman *et al.* (2008, 2009); Caruso *et al.* (2009). For the crystal structure of *N'*-acetyl-2-phenylethylhydrazonamide, see: Ianelli *et al.* (2001). For the graph-set analysis of hydrogen bonding, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_7\text{H}_{12}\text{N}_6\text{O}\cdot 2\text{H}_2\text{O}$	$\gamma = 110.810$ (2)°
$M_r = 232.26$	$V = 562.75$ (11) Å ³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.5496$ (9) Å	Mo $K\alpha$ radiation
$b = 7.6208$ (9) Å	$\mu = 0.11$ mm ⁻¹
$c = 11.2518$ (13) Å	$T = 223$ K
$\alpha = 102.645$ (2)°	$0.45 \times 0.12 \times 0.10$ mm
$\beta = 101.440$ (2)°	

Data collection

Bruker SMART APEX CCD diffractometer	3963 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	2548 independent reflections
$T_{\min} = 0.953$, $T_{\max} = 0.989$	2174 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.141$	$\Delta\rho_{\text{max}} = 0.31$ e Å ⁻³
$S = 1.05$	$\Delta\rho_{\text{min}} = -0.26$ e Å ⁻³
2548 reflections	
183 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}2\text{W}-\text{H}4\text{W}\cdots\text{N}1^i$	0.87 (3)	2.04 (3)	2.884 (2)	162 (3)
$\text{O}2\text{W}-\text{H}3\text{W}\cdots\text{O}1$	0.86 (3)	2.11 (3)	2.885 (2)	150 (3)
$\text{O}1\text{W}-\text{H}2\text{W}\cdots\text{O}2\text{W}^{ii}$	0.89 (3)	1.93 (3)	2.824 (2)	175 (3)
$\text{O}1\text{W}-\text{H}1\text{W}\cdots\text{N}5$	0.81 (3)	2.24 (3)	2.982 (2)	153 (3)
$\text{N}6-\text{H}6\text{N}\cdots\text{O}1\text{W}^{iii}$	0.84 (2)	2.07 (2)	2.905 (2)	177 (2)
$\text{N}4-\text{H}42\cdots\text{O}1\text{W}^{iii}$	0.88 (2)	2.14 (3)	2.995 (2)	165 (2)
$\text{N}4-\text{H}41\cdots\text{O}1^{\text{iv}}$	0.81 (2)	2.08 (2)	2.874 (2)	169 (2)
$\text{N}3-\text{H}32\cdots\text{N}5$	0.86 (2)	2.18 (2)	2.791 (2)	128 (2)
$\text{N}3-\text{H}31\cdots\text{O}2\text{W}^{\text{v}}$	0.83 (2)	2.27 (2)	3.082 (2)	163 (2)

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5086).

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