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{4,4′-Dimethyl-2,2′-[(2,2-dimethylpropane-1,3-diyl)bis(nitrilomethanylylidene)]diphenolato}nickel(II) monohydrate

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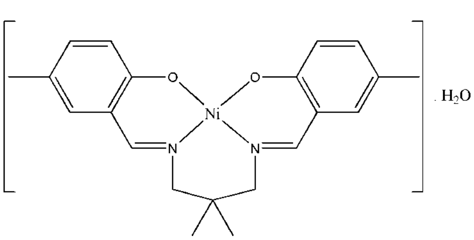
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.083; data-to-parameter ratio = 20.5.

In the title compound, $[Ni(C_{21}H_{24}N_2O_2)]\cdot H_2O$, both the complex molecule and the water molecule lie on a twofold rotation axis. The Ni^{II} ion is coordinated in a distorted square-planar geometry by the tetradentate ligand. The dihedral angle between the two symmetry-related benzene rings is 47.12 (8)°. In the crystal, pairs of symmetry-related O—H···O hydrogen bonds form $R_2^2(6)$ ring motifs. In addition, there are weak intermolecular C—H···O hydrogen bonds, and π–π stacking interactions with a centroid–centroid distance of 3.4760 (8) Å.

Related literature

For related structures, see for example: Fun *et al.* (2008); Kargar *et al.* (2008, 2011); Rayati *et al.* (2011); Kia *et al.* (2010). For standard bond lengths, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$[Ni(C_{21}H_{24}N_2O_2)]\cdot H_2O$
M_r = 413.15
Monoclinic, $C2/c$
a = 13.3333 (4) Å
b = 15.9424 (5) Å
c = 9.9965 (3) Å
β = 104.736 (1)°
V = 2055.01 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.97 mm^{-1}
T = 296 K
0.25 × 0.12 × 0.08 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.794, T_{max} = 0.927
17468 measured reflections
2557 independent reflections
2131 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.06
2557 reflections
125 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1···O1^{i}	0.98	1.92	2.781 (2)	145
C3—H3A···O1W^{ii}	0.93	2.55	3.477 (2)	173

Symmetry codes: (i) $-x, y, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: LH5396).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *APEX2* , *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fun, H.-K., Kia, R. & Kargar, H. (2008). *Acta Cryst.* E**64**, o1895–o1896.
Kargar, H., Fun, H.-K. & Kia, R. (2008). *Acta Cryst.* E**64**, m1541–m1542.
Kargar, H., Kia, R., Pahlavani, E. & Tahir, M. N. (2011). *Acta Cryst.* E**67**, o614.
Kia, R., Kargar, H., Tahir, M. N. & Kianoosh, F. (2010). *Acta Cryst.* E**66**, o2296.
Rayati, S., Ghaemi, A. & Notash, B. (2011). *Acta Cryst.* E**67**, m448.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2012). E**68**, m82 [**doi:10.1107/S1600536811054262**]

{4,4'-Dimethyl-2,2'-[(2,2-dimethylpropane-1,3-diyl)bis(nitrilomethanylylidene)]diphenolato}nickel(II) monohydrate

H. Kargar, R. Kia, Z. Sharafi and M. N. Tahir

Comment

In continuation of our work on the crystal structures of a Schiff base ligands and complexes (Fun *et al.*, 2008; Kargar *et al.*, 2008,2011; Rayati *et al.,* 2011; Kia *et al.,* 2010), we have determined the X-ray structure of the title compound.

The molecular structure of the title compound is ahown in Fig. 1. The asymmetric unit comprises half of Schiff base complex and half a water molecule. The Ni^{II} ion, the central carbon atom of the diamine segment (C10) and the O atom of water molecule lie on a two-fold rotation axis. The coordination geometry of Ni1 is distorted square-planar formed by the tetradentate ligand. The bond lengths (Allen *et al.,* 1987) and angles are within the normal ranges and are comparable to related structures (Fun *et al.* 2008; Kargar *et al.* 2008; Rayati *et al.,* 2011). The dihedral angle between the two symmetry related benzene rings is 47.12 (8)°. A pair of symmetry related intermolecular O—H···O hydrogen bonds form an $R^2_2(6)$ ring motif (Bernstein *et al.*, 1995). In the crystal, molecules are linked through weak intermolecular C—H···O interactions. The crystal structure is further stabilized by intermolecular π–π interactions [$Cg1\cdots Cg1^{iii}$ = 3.4760 (8)Å; (iii) -*x*, 1 - *y*, 1 - *z*; *Cg*1 is the centroid of the Ni1/O1/C1/C6/C8/N1 ring].

Experimental

The title compound was synthesized by adding bis(5-methylsalicylaldehyde)-2,2-dimethyl-1,3-propanediimine (2 mmol) to a solution of nickel(II) chloride hexahydrate (2.1 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for half an hour. The resultant solution was filtered. Yellow single crystals of the title compound suitable for *X*-ray structure determination were recrystallized from an ethanol solution of the title compound by slow evaporation of the solvent at room temperature over several days.

Refinement

Hydrogen atoms bonded to C atoms were positioned geometrically with C—H = 0.93-0.97 Å and included in a riding model approximation with U_{iso} (H) = 1.2 or 1.5 U_{eq} (C) The unique water H atom was located in a difference Fourier map and then constrained to ride to the parent atom with U_{iso} (H) = 1.5 U_{eq} (O). A rotating group model was used only for the benzene- substituent methyl group.

Figures

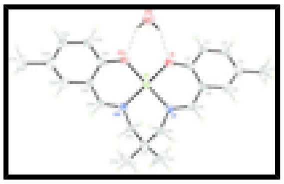

Fig. 1. The molecular structure of the title compound, showing 40% probability displacement ellipsoids. The dashed lines show hydrogen bonds [symmetry code: (A) -x, y, -z+1/2].

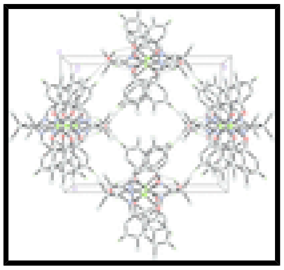

Fig. 2. A partial packing diagram of the title compound viewed approximately along the *c*-axis showing molecules linked through intermolecular hydrogen bonds (dashed lines). Only the H atoms involved in the interactions are shown.

{4,4'-Dimethyl-2,2'-[(2,2-dimethylpropane-1,3- diyl)bis(nitrilomethanylylidene)]diphenolato}nickel(II) monohydrate

Crystal data

$[Ni(C_{21}H_{24}N_2O_2)]\cdot H_2O$	$F(000) = 872$
$M_r = 413.15$	$D_x = 1.335$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 3245 reflections
$a = 13.3333$ (4) Å	$\theta = 2.8$–$27.8°$
$b = 15.9424$ (5) Å	$\mu = 0.97$ mm^{-1}
$c = 9.9965$ (3) Å	$T = 296$ K
$\beta = 104.736$ (1)°	Block, red
$V = 2055.01$ (11) Å^3	0.25 × 0.12 × 0.08 mm
$Z = 4$	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	2557 independent reflections
Radiation source: fine-focus sealed tube	2131 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.040$
φ and ω scans	$\theta_{max} = 28.3°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -17 \rightarrow 16$
$T_{min} = 0.794$, $T_{max} = 0.927$	$k = -21 \rightarrow 21$
17468 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods

Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.031$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.083$	H-atom parameters constrained
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
2557 reflections	$(\Delta/\sigma)_{max} < 0.001$
125 parameters	$\Delta\rho_{max} = 0.21$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.31$ e Å^{-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 > 2sigma(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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.0000	0.489837 (15)	0.2500	0.03589 (11)
O1	0.09186 (9)	0.57316 (6)	0.34800 (11)	0.0457 (3)
N1	0.04523 (10)	0.40475 (8)	0.39178 (12)	0.0414 (3)
C1	0.16634 (12)	0.56212 (9)	0.46203 (15)	0.0401 (3)
C2	0.23779 (14)	0.62692 (10)	0.50786 (17)	0.0497 (4)
H2A	0.2346	0.6747	0.4537	0.060*
C3	0.31279 (14)	0.62162 (12)	0.63132 (18)	0.0567 (5)
H3A	0.3589	0.6660	0.6581	0.068*
C4	0.32147 (14)	0.55163 (13)	0.71719 (17)	0.0550 (4)
C5	0.25490 (14)	0.48675 (11)	0.67168 (18)	0.0487 (4)
H5A	0.2604	0.4390	0.7265	0.058*
C6	0.17758 (13)	0.48878 (9)	0.54427 (17)	0.0410 (3)
C7	0.40114 (17)	0.54821 (18)	0.85526 (19)	0.0817 (7)
H7A	0.4556	0.5101	0.8494	0.123*
H7B	0.3687	0.5292	0.9252	0.123*
H7C	0.4297	0.6031	0.8788	0.123*
C8	0.11388 (13)	0.41566 (10)	0.50691 (15)	0.0431 (4)
H8A	0.1230	0.3725	0.5714	0.052*
C9	−0.01669 (14)	0.32780 (10)	0.36982 (16)	0.0488 (4)
H9A	−0.0895	0.3426	0.3512	0.059*
H9B	0.0006	0.2949	0.4542	0.059*
C10	0.0000	0.27358 (15)	0.2500	0.0564 (7)
C11	0.0973 (2)	0.21930 (15)	0.2981 (2)	0.1038 (9)

H11A	0.1569	0.2548	0.3287	0.156*
H11B	0.1057	0.1851	0.2226	0.156*
H11C	0.0903	0.1840	0.3729	0.156*
O1W	0.0000	0.72531 (12)	0.2500	0.0992 (8)
H1	−0.0556	0.6878	0.2007	0.149*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.03715 (18)	0.03024 (16)	0.03666 (16)	0.000	0.00274 (11)	0.000
O1	0.0462 (7)	0.0353 (6)	0.0472 (6)	−0.0002 (5)	−0.0032 (5)	0.0020 (5)
N1	0.0448 (8)	0.0367 (7)	0.0424 (7)	−0.0023 (6)	0.0110 (6)	0.0012 (5)
C1	0.0364 (8)	0.0408 (8)	0.0418 (8)	0.0035 (6)	0.0075 (6)	−0.0040 (6)
C2	0.0470 (10)	0.0449 (9)	0.0540 (9)	−0.0035 (7)	0.0069 (8)	−0.0022 (7)
C3	0.0434 (10)	0.0635 (12)	0.0586 (10)	−0.0101 (8)	0.0047 (8)	−0.0118 (9)
C4	0.0391 (10)	0.0794 (13)	0.0432 (9)	−0.0003 (9)	0.0042 (7)	−0.0047 (9)
C5	0.0428 (10)	0.0604 (11)	0.0413 (8)	0.0055 (8)	0.0076 (7)	0.0051 (7)
C6	0.0373 (9)	0.0449 (9)	0.0390 (8)	0.0040 (7)	0.0065 (7)	−0.0010 (6)
C7	0.0590 (14)	0.121 (2)	0.0537 (11)	−0.0122 (13)	−0.0074 (10)	−0.0005 (12)
C8	0.0472 (10)	0.0411 (8)	0.0408 (8)	0.0029 (7)	0.0109 (7)	0.0055 (6)
C9	0.0603 (11)	0.0394 (9)	0.0494 (9)	−0.0103 (8)	0.0189 (8)	0.0006 (7)
C10	0.0806 (19)	0.0355 (12)	0.0563 (14)	0.000	0.0236 (14)	0.000
C11	0.159 (3)	0.0726 (15)	0.0929 (16)	0.0629 (16)	0.0553 (17)	0.0332 (13)
O1W	0.1069 (18)	0.0426 (11)	0.1201 (18)	0.000	−0.0225 (15)	0.000

Geometric parameters (Å, °)

Ni1—O1	1.901 (1)	C5—H5A	0.9300
Ni1—O1^{i}	1.9010 (10)	C6—C8	1.435 (2)
Ni1—N1^{i}	1.9436 (12)	C7—H7A	0.9600
Ni1—N1	1.9436 (12)	C7—H7B	0.9600
O1—C1	1.3194 (17)	C7—H7C	0.9600
N1—C8	1.2870 (19)	C8—H8A	0.9300
N1—C9	1.4638 (19)	C9—C10	1.539 (2)
C1—C2	1.401 (2)	C9—H9A	0.9700
C1—C6	1.415 (2)	C9—H9B	0.9700
C2—C3	1.379 (2)	C10—C11	1.532 (2)
C2—H2A	0.9300	C10—C11^{i}	1.532 (2)
C3—C4	1.394 (3)	C10—C9^{i}	1.539 (2)
C3—H3A	0.9300	C11—H11A	0.9600
C4—C5	1.364 (3)	C11—H11B	0.9600
C4—C7	1.513 (2)	C11—H11C	0.9600
C5—C6	1.421 (2)	O1W—H1	0.9818
O1—Ni1—O1^{i}	91.34 (6)	C4—C7—H7A	109.5
O1—Ni1—N1^{i}	154.58 (6)	C4—C7—H7B	109.5
O1^{i}—Ni1—N1^{i}	94.14 (5)	H7A—C7—H7B	109.5
O1—Ni1—N1	94.14 (5)	C4—C7—H7C	109.5

O1^{i}—Ni1—N1	154.58 (6)	H7A—C7—H7C	109.5
N1^{i}—Ni1—N1	91.48 (7)	H7B—C7—H7C	109.5
C1—O1—Ni1	126.63 (9)	N1—C8—C6	125.58 (15)
C8—N1—C9	119.54 (13)	N1—C8—H8A	117.2
C8—N1—Ni1	125.16 (11)	C6—C8—H8A	117.2
C9—N1—Ni1	114.64 (10)	N1—C9—C10	113.57 (13)
O1—C1—C2	118.94 (14)	N1—C9—H9A	108.9
O1—C1—C6	123.87 (14)	C10—C9—H9A	108.9
C2—C1—C6	117.16 (15)	N1—C9—H9B	108.9
C3—C2—C1	121.69 (16)	C10—C9—H9B	108.9
C3—C2—H2A	119.2	H9A—C9—H9B	107.7
C1—C2—H2A	119.2	C11—C10—C11^{i}	111.2 (3)
C2—C3—C4	121.84 (17)	C11—C10—C9^{i}	106.43 (11)
C2—C3—H3A	119.1	C11^{i}—C10—C9^{i}	110.61 (11)
C4—C3—H3A	119.1	C11—C10—C9	110.61 (11)
C5—C4—C3	117.16 (16)	C11^{i}—C10—C9	106.43 (11)
C5—C4—C7	121.55 (19)	C9^{i}—C10—C9	111.63 (18)
C3—C4—C7	121.28 (19)	C10—C11—H11A	109.5
C4—C5—C6	122.98 (17)	C10—C11—H11B	109.5
C4—C5—H5A	118.5	H11A—C11—H11B	109.5
C6—C5—H5A	118.5	C10—C11—H11C	109.5
C1—C6—C5	119.02 (15)	H11A—C11—H11C	109.5
C1—C6—C8	123.49 (15)	H11B—C11—H11C	109.5
C5—C6—C8	117.48 (14)		
O1^{i}—Ni1—O1—C1	163.48 (15)	C7—C4—C5—C6	178.41 (17)
N1^{i}—Ni1—O1—C1	−93.95 (16)	O1—C1—C6—C5	−174.24 (15)
N1—Ni1—O1—C1	8.32 (13)	C2—C1—C6—C5	4.2 (2)
O1—Ni1—N1—C8	1.34 (13)	O1—C1—C6—C8	5.0 (2)
O1^{i}—Ni1—N1—C8	−100.61 (16)	C2—C1—C6—C8	−176.58 (15)
N1^{i}—Ni1—N1—C8	156.53 (16)	C4—C5—C6—C1	−2.0 (3)
O1—Ni1—N1—C9	171.99 (11)	C4—C5—C6—C8	178.70 (16)
O1^{i}—Ni1—N1—C9	70.04 (15)	C9—N1—C8—C6	−177.90 (15)
N1^{i}—Ni1—N1—C9	−32.82 (8)	Ni1—N1—C8—C6	−7.7 (2)
Ni1—O1—C1—C2	169.47 (11)	C1—C6—C8—N1	5.6 (3)
Ni1—O1—C1—C6	−12.1 (2)	C5—C6—C8—N1	−175.14 (16)
O1—C1—C2—C3	175.28 (15)	C8—N1—C9—C10	−117.17 (16)
C6—C1—C2—C3	−3.2 (2)	Ni1—N1—C9—C10	71.61 (15)
C1—C2—C3—C4	−0.1 (3)	N1—C9—C10—C11	82.3 (2)
C2—C3—C4—C5	2.4 (3)	N1—C9—C10—C11^{i}	−156.78 (16)
C2—C3—C4—C7	−177.33 (18)	N1—C9—C10—C9^{i}	−36.00 (9)
C3—C4—C5—C6	−1.3 (3)		

Symmetry codes: (i) $-x$, y, $-z+1/2$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

O1W—H1···O1^{ii}	0.98	1.92	2.781 (2)	145
C3—H3A···O1W^{iii}	0.93	2.55	3.477 (2)	173

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

Fig. 1

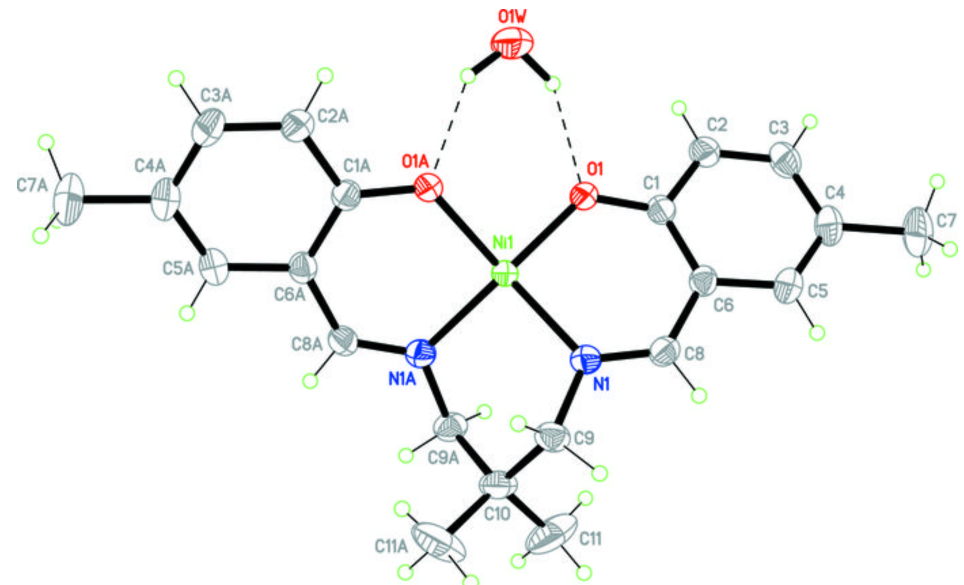
O1W
C3A
C2A
C2
C3
C4A
C1A
O1A
O1
C1
C4
C7A
C7
Ni1
C5A
C6A
C6
C5
C8A
C8
N1A
N1
C9
C9A
C10
C11A
C11

Fig. 2