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Aqua(dimethylglyoxime-κ^2N,N')(3,5-dinitro-2-oxidobenzoato-κ^2O,O')nickel(II)

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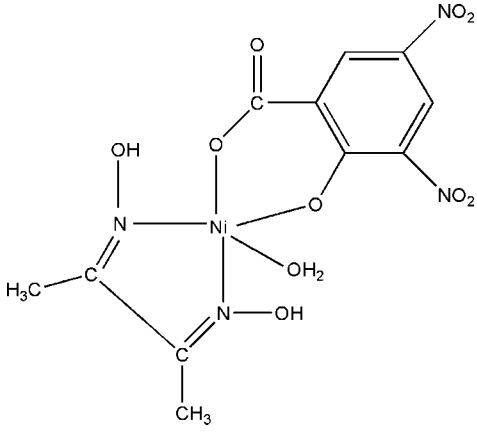
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.026; wR factor = 0.073; data-to-parameter ratio = 11.8.

In the mononuclear title complex, $[Ni(C_7H_2N_2O_7)(C_4H_8N_2O_2)(H_2O)]$, the nickel(II) ion is five-coordinated in a distorted square-pyramidal geometry by two N atoms from a dimethylglyoxime molecule, two O atoms from a 2-oxido-3,5-dinitrobenzoate anion and one O atom from a water molecule. Strong hydrogen bonds connect discrete adjacent molecules, forming a ribbon parallel to the b axis.

Related literature

For a related complex with the same structure as the title compound, see: Liu & Wen (2007).



Experimental

Crystal data

$[Ni(C_7H_2N_2O_7)(C_4H_8N_2O_2)(H_2O)]$
M_r = 418.94
Monoclinic, $P2_1/c$
a = 12.476 (2) Å
b = 7.0786 (14) Å
c = 17.929 (4) Å
β = 103.014 (3)°
V = 1542.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.32 mm^{-1}
T = 293 (2) K
0.28 × 0.26 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
9276 measured reflections
2824 independent reflections
2426 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.073
S = 1.07
2824 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O8—H8···O7	0.82	2.15	2.7427 (19)	129
O8—H8···O4	0.82	2.22	2.981 (2)	155
O9—H9···O10^{i}	0.82	1.86	2.675 (2)	171
O10—H10D···O5^{ii}	0.83	1.86	2.690 (2)	177
O10—H10E···O6^{i}	0.83	1.91	2.6695 (19)	152
O10—H10E···O5^{i}	0.83	2.63	3.370 (2)	150

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2265).

References

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  doi:10.1107/S1600536807056681

supplementary materials

Acta Cryst. (2007). E**63**, m3002 [**doi:10.1107/S1600536807056681**]

Aqua(dimethylglyoxime-κ^2N,N')(3,5-dinitro-2-oxidobenzoato-κ^2O,O')nickel(II)

Y.-Q. Liu

Comment

The title complex, [NiC~11Ñ~4~ O~10~H~12~], is a mononuclear nickel(II) compound. The nickel(II) ion is five-coordinated in a distorted pyramidal geometry by two N atoms from one Dimethylglyoxime molecule, two O atoms from one 2-hydroxy-3,5-dinitrobenzoic acid molecule and one O atom from a water molecule (Fig. 1). The water molecule appears to be weakly coordinated to the Ni atom with a rather long Ni—O bond length of 2.365 (1) Å. The Ni atom is located slightly above the basal N3, N4, O6, O7 square plane by 0.0988 (8) Å

Strong hydrogen bonds connect discrete adjacent molecule building a ribbonn parallel to the *b* axis (Table 1).

Experimental

A mixture of Dimethylglyoxime(0.116 g,0.001 mol), 2-hydroxy-3,5-dinitrobenzoic acid(0.228 g,0.001 mol) and $Ni(NO_3)_2{\cdot}6H_2O$ (0.290 g, 0.001 mol) in the More ratio of 1:1:1 was added to 15 ml me thanol, The mixture was heated at 408 K or so for two days in a closed steel tomb with a linner, crystal was obtained after it cooled down untouched in the air.

Refinement

All H atoms attached to C atoms and hydroxyl O atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.96 Å (methyl) and O—H = 0.82Å with $U_{iso}(H) = 1.2U_{eq}(C_{aromatic}$ or $O_{hydroxyl})$. H atoms of water molecule were located in difference Fourier maps and included in the subsequent refinement using restraints (O—H= 0.82 (1)Å and H···H= 1.35 (2) Å) with $U_{iso}(H) = 1.5U_{eq}(O)$. In the last stage of refinement, they were treated as riding on the O atom.

Figures

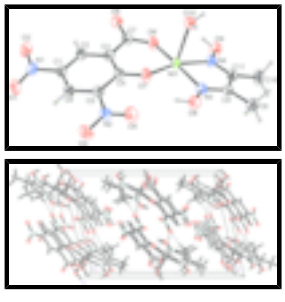

Fig. 1. Molecular view of compound (I) showing 30% probability displacement ellipsoids and the atom numbering scheme. H atoms are shown as small spheres of arbitrary radii.

Aqua(dimethylglyoxime-κ^2*N*,*N*')(3,5-dinitro-2-oxidobenzoato- κ^2*O*,*O*')nickel(II)

Crystal data

$[Ni(C_7H_2N_2O_7)(C_4H_8N_2O_2)(H_2O)]$	F_{000} = 856.0
M_r = 418.94	D_x = 1.804 Mg m^{-3}
Monoclinic, *P*2(1)/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 7541 reflections
a = 12.476 (2) Å	θ = 0.9–28.3º
b = 7.0786 (14) Å	μ = 1.32 mm^{-1}
c = 17.929 (4) Å	*T* = 293 (2) K
β = 103.014 (3)º	Block, blue
V = 1542.7 (5) Å^3	0.28 × 0.26 × 0.22 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2426 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.019
Monochromator: graphite	θ_{max} = 25.5º
T = 293(2) K	θ_{min} = 2.3º
φ and ω scans	*h* = −14→15
Absorption correction: none	*k* = −8→8
9276 measured reflections	*l* = −21→21
2824 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.026	H-atom parameters constrained
$wR(F^2)$ = 0.073	$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.2949P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max}$ = 0.003
2824 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
239 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.38531 (16)	0.8249 (3)	−0.15675 (10)	0.0431 (4)
C2	0.29061 (16)	0.8484 (3)	−0.12964 (11)	0.0429 (4)
H2	0.2494	0.9584	−0.1411	0.051*
C3	0.25681 (15)	0.7100 (3)	−0.08576 (10)	0.0389 (4)
C4	0.31731 (15)	0.5365 (3)	−0.07013 (10)	0.0377 (4)
C5	0.41420 (15)	0.5225 (3)	−0.09925 (10)	0.0379 (4)
C6	0.44881 (16)	0.6656 (3)	−0.14101 (10)	0.0410 (4)
H6	0.5137	0.6539	−0.1580	0.049*
C7	0.15838 (16)	0.7567 (3)	−0.05465 (11)	0.0434 (4)
C8	0.2461 (2)	−0.0792 (3)	0.17163 (15)	0.0625 (6)
H8A	0.2909	−0.1448	0.1429	0.094*
H8B	0.1793	−0.1485	0.1692	0.094*
H8C	0.2855	−0.0681	0.2240	0.094*
C9	0.21937 (16)	0.1128 (3)	0.13860 (11)	0.0441 (5)
C10	0.1053 (2)	0.2006 (4)	0.23813 (14)	0.0690 (7)
H10A	0.0894	0.3166	0.2612	0.104*
H10B	0.1610	0.1325	0.2736	0.104*
H10C	0.0397	0.1253	0.2250	0.104*
C11	0.14518 (16)	0.2430 (3)	0.16752 (11)	0.0461 (5)
Ni1	0.19075 (2)	0.41717 (3)	0.035803 (14)	0.04002 (10)
N1	0.41838 (17)	0.9713 (3)	−0.20418 (10)	0.0561 (5)
N2	0.48544 (14)	0.3559 (2)	−0.08555 (9)	0.0455 (4)
N3	0.25549 (13)	0.1823 (2)	0.08265 (9)	0.0438 (4)
N4	0.11866 (14)	0.3919 (2)	0.12644 (10)	0.0460 (4)
O1	0.50896 (16)	0.9592 (3)	−0.21874 (10)	0.0769 (5)
O2	0.35279 (17)	1.0994 (3)	−0.22726 (12)	0.0866 (6)
O3	0.57504 (13)	0.3662 (3)	−0.10271 (11)	0.0724 (5)
O4	0.45619 (13)	0.2136 (2)	−0.05635 (10)	0.0664 (5)
O5	0.10174 (13)	0.89514 (19)	−0.08015 (10)	0.0573 (4)
O6	0.13486 (12)	0.6563 (2)	−0.00039 (9)	0.0524 (4)
O7	0.28663 (13)	0.39915 (19)	−0.03220 (9)	0.0517 (4)
O8	0.32368 (13)	0.0709 (2)	0.05091 (9)	0.0546 (4)
H8	0.3413	0.1281	0.0157	0.082*
O9	0.05069 (15)	0.5146 (2)	0.15476 (9)	0.0638 (4)
H9	0.0265	0.5949	0.1224	0.096*
O10	0.04723 (11)	0.24631 (18)	−0.04489 (8)	0.0496 (3)
H10D	0.0660	0.1377	−0.0548	0.074*
H10E	−0.0077	0.2383	−0.0261	0.074*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0520 (12)	0.0435 (11)	0.0355 (9)	−0.0072 (9)	0.0136 (8)	0.0006 (8)
C2	0.0471 (11)	0.0367 (10)	0.0453 (10)	0.0010 (8)	0.0112 (9)	0.0015 (8)
C3	0.0413 (10)	0.0338 (9)	0.0437 (10)	0.0015 (8)	0.0137 (8)	−0.0008 (7)
C4	0.0418 (11)	0.0338 (9)	0.0399 (9)	0.0002 (8)	0.0144 (8)	−0.0021 (7)
C5	0.0396 (10)	0.0376 (9)	0.0373 (9)	0.0016 (8)	0.0108 (8)	−0.0057 (8)
C6	0.0418 (11)	0.0480 (11)	0.0363 (9)	−0.0042 (9)	0.0155 (8)	−0.0067 (8)
C7	0.0452 (11)	0.0310 (9)	0.0572 (12)	0.0029 (8)	0.0178 (9)	−0.0011 (8)
C8	0.0626 (15)	0.0570 (14)	0.0707 (15)	0.0121 (11)	0.0208 (12)	0.0219 (11)
C9	0.0389 (11)	0.0460 (11)	0.0468 (11)	0.0001 (8)	0.0087 (9)	0.0061 (8)
C10	0.0776 (17)	0.0758 (17)	0.0639 (14)	0.0064 (13)	0.0372 (13)	0.0187 (12)
C11	0.0435 (11)	0.0496 (11)	0.0480 (11)	0.0010 (9)	0.0163 (9)	0.0065 (9)
Ni1	0.04354 (17)	0.03448 (15)	0.04867 (16)	0.00837 (10)	0.02439 (12)	0.00723 (10)
N1	0.0639 (13)	0.0591 (12)	0.0465 (10)	−0.0081 (10)	0.0147 (9)	0.0104 (8)
N2	0.0438 (10)	0.0474 (9)	0.0486 (9)	0.0070 (8)	0.0171 (8)	−0.0047 (7)
N3	0.0400 (9)	0.0443 (9)	0.0497 (9)	0.0077 (7)	0.0159 (7)	0.0035 (7)
N4	0.0463 (10)	0.0469 (10)	0.0503 (9)	0.0057 (7)	0.0223 (8)	0.0013 (7)
O1	0.0749 (12)	0.0903 (13)	0.0777 (11)	−0.0063 (10)	0.0429 (10)	0.0231 (10)
O2	0.0819 (13)	0.0832 (14)	0.0964 (14)	0.0058 (10)	0.0239 (11)	0.0487 (11)
O3	0.0478 (10)	0.0778 (11)	0.1014 (13)	0.0172 (8)	0.0373 (9)	0.0104 (10)
O4	0.0708 (11)	0.0463 (9)	0.0928 (12)	0.0153 (8)	0.0411 (9)	0.0107 (8)
O5	0.0529 (9)	0.0391 (8)	0.0841 (11)	0.0139 (6)	0.0245 (8)	0.0084 (7)
O6	0.0531 (9)	0.0430 (8)	0.0719 (9)	0.0112 (7)	0.0366 (8)	0.0088 (7)
O7	0.0581 (9)	0.0377 (8)	0.0708 (10)	0.0111 (6)	0.0387 (8)	0.0115 (6)
O8	0.0564 (9)	0.0518 (9)	0.0622 (9)	0.0179 (7)	0.0271 (7)	0.0063 (7)
O9	0.0776 (11)	0.0594 (10)	0.0662 (10)	0.0232 (9)	0.0413 (9)	0.0076 (8)
O10	0.0461 (8)	0.0407 (7)	0.0676 (9)	0.0087 (6)	0.0245 (7)	0.0004 (7)

Geometric parameters (Å, °)

C1—C6	1.370 (3)	C10—C11	1.492 (3)
C1—C2	1.385 (3)	C10—H10A	0.9600
C1—N1	1.458 (3)	C10—H10B	0.9600
C2—C3	1.380 (3)	C10—H10C	0.9600
C2—H2	0.9300	C11—N4	1.286 (3)
C3—C4	1.436 (3)	Ni1—O6	1.8889 (14)
C3—C7	1.495 (3)	Ni1—O7	1.8949 (14)
C4—O7	1.293 (2)	Ni1—N3	1.9538 (16)
C4—C5	1.424 (3)	Ni1—N4	2.0347 (17)
C5—C6	1.385 (3)	Ni1—O10	2.3654 (14)
C5—N2	1.464 (2)	N1—O1	1.219 (2)
C6—H6	0.9300	N1—O2	1.229 (3)
C7—O5	1.234 (2)	N2—O3	1.227 (2)
C7—O6	1.291 (2)	N2—O4	1.228 (2)
C8—C9	1.490 (3)	N3—O8	1.374 (2)
C8—H8A	0.9600	N4—O9	1.387 (2)

C8—H8B	0.9600	O8—H8	0.8200
C8—H8C	0.9600	O9—H9	0.8200
C9—N3	1.286 (3)	O10—H10D	0.8340
C9—C11	1.480 (3)	O10—H10E	0.8300
C6—C1—C2	121.71 (18)	H10A—C10—H10C	109.5
C6—C1—N1	118.59 (18)	H10B—C10—H10C	109.5
C2—C1—N1	119.69 (18)	N4—C11—C9	114.23 (17)
C3—C2—C1	120.61 (18)	N4—C11—C10	124.0 (2)
C3—C2—H2	119.7	C9—C11—C10	121.76 (19)
C1—C2—H2	119.7	O6—Ni1—O7	94.41 (6)
C2—C3—C4	120.23 (17)	O6—Ni1—N3	174.35 (7)
C2—C3—C7	115.97 (17)	O7—Ni1—N3	87.68 (6)
C4—C3—C7	123.76 (16)	O6—Ni1—N4	99.08 (6)
O7—C4—C5	121.14 (17)	O7—Ni1—N4	164.71 (6)
O7—C4—C3	122.75 (17)	N3—Ni1—N4	78.24 (7)
C5—C4—C3	116.11 (17)	O6—Ni1—O10	94.48 (6)
C6—C5—C4	122.77 (17)	O7—Ni1—O10	94.16 (6)
C6—C5—N2	115.37 (17)	N3—Ni1—O10	90.60 (6)
C4—C5—N2	121.86 (17)	N4—Ni1—O10	91.93 (6)
C1—C6—C5	118.48 (18)	O1—N1—O2	124.0 (2)
C1—C6—H6	120.8	O1—N1—C1	118.2 (2)
C5—C6—H6	120.8	O2—N1—C1	117.80 (19)
O5—C7—O6	120.53 (18)	O3—N2—O4	122.07 (17)
O5—C7—C3	119.13 (18)	O3—N2—C5	117.59 (17)
O6—C7—C3	120.31 (16)	O4—N2—C5	120.32 (16)
C9—C8—H8A	109.5	C9—N3—O8	117.52 (16)
C9—C8—H8B	109.5	C9—N3—Ni1	118.49 (14)
H8A—C8—H8B	109.5	O8—N3—Ni1	123.02 (12)
C9—C8—H8C	109.5	C11—N4—O9	113.77 (16)
H8A—C8—H8C	109.5	C11—N4—Ni1	115.24 (14)
H8B—C8—H8C	109.5	O9—N4—Ni1	130.84 (12)
N3—C9—C11	113.05 (17)	C7—O6—Ni1	128.55 (12)
N3—C9—C8	124.9 (2)	C4—O7—Ni1	126.33 (12)
C11—C9—C8	122.05 (19)	N3—O8—H8	109.5
C11—C10—H10A	109.5	N4—O9—H9	109.5
C11—C10—H10B	109.5	Ni1—O10—H10D	113.0
H10A—C10—H10B	109.5	Ni1—O10—H10E	111.5
C11—C10—H10C	109.5	H10D—O10—H10E	108.6

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H8···O7	0.82	2.15	2.7427 (19)	129
O8—H8···O4	0.82	2.22	2.981 (2)	155
O9—H9···O10^{i}	0.82	1.86	2.675 (2)	171
O10—H10D···O5^{ii}	0.83	1.86	2.690 (2)	177
O10—H10E···O6^{i}	0.83	1.91	2.6695 (19)	152
O10—H10E···O5^{i}	0.83	2.63	3.370 (2)	150

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

Fig. 1

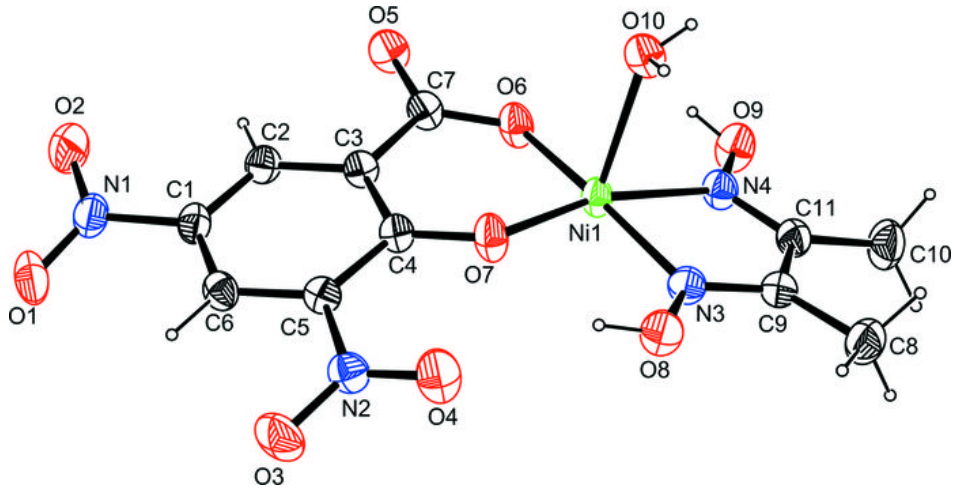

Fig. 2