Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

cyclo-Tetrakis[μ-*N*-(2-hydroxybenzoyl)-*N*′-(2-hydroxy-3-methoxybenzylidene)hydrazinato(2−)]tetranickel(II) *N*,*N*-dimethylformamide tetrasolvate

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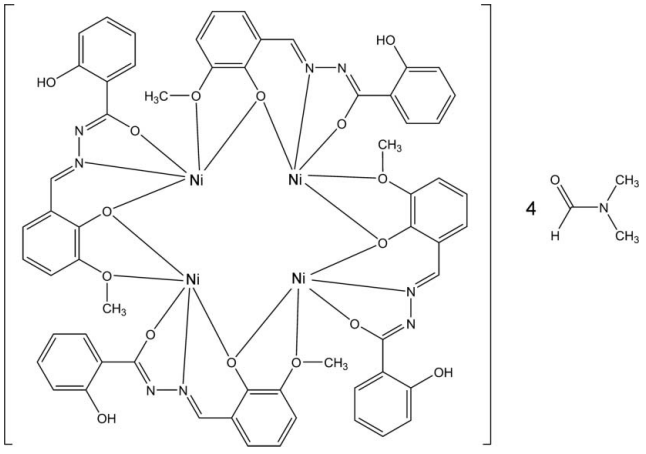
Received 19 September 2007; accepted 2 October 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.041; wR factor = 0.148; data-to-parameter ratio = 14.3.

The title compound, $[Ni_4(C_{15}H_{12}N_2O_4)_4].4C_3H_7NO$, is isostructural with its Co^{II} and Zn^{II} analogues. The compound contains *N*-(2-hydroxybenzoyl)-*N*-(2-hydroxy-3-methoxybenzylidene)-hydrazine anions and Ni^{II} cations linked into a tetrameric complex about a position of $\overline{4}$ point symmetry. Each Ni^{II} cation is pentacoordinate with a distorted square-based pyramidal geometry. The ligand exhibits an intramolecular O—H···N hydrogen bond.

Related literature

For the analogous Co^{II} and Zn^{II} complexes, see: Gao *et al.* (2007*a*,*b*).



Experimental

Crystal data

$[Ni_4(C_{15}H_{12}N_2O_4)_4]\cdot4C_3H_7NO$
M_r = 1664.29
Tetragonal, $I4_1/a$
a = 24.1627 (5) Å
c = 13.0428 (5) Å
V = 7614.9 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.05 mm^{-1}
T = 293 (2) K
0.44 × 0.32 × 0.28 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.641, T_{max} = 0.742
19316 measured reflections
3554 independent reflections
2243 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.148
S = 1.00
3554 reflections
248 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···N3	0.82	1.86	2.579 (5)	145

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

The authors thank Tonghua Teachers College for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2239).

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  doi:10.1107/S1600536807048325

supplementary materials

Acta Cryst. (2007). E**63**, m2662 [**doi:10.1107/S1600536807048325**]

cyclo-Tetrakis[μ-*N*-(2-hydroxybenzoyl)-*N*'-(2-hydroxy-3-methoxybenzylidene)hydrazinato(2-)]tetranickel(II) *N*,*N*-dimethylformamide tetrasolvate

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Comment

The title compound, $[Ni_4(C_{15}H_{12}N_2O_4)_4]\cdot 4C_3H_7NO$, is isostructural with its Co^{II} (Gao *et al.*, 2007*a*) and Zn^{II} (Gao *et al.*, 2007*b*) analogues. As shown in Figure 1, each Ni^{II} cation is penta-coordinated by four O atoms and one N atom, forming a distorted square-based pyramidal geometry. The Ni—O bond lengths are in the range 1.936 (3)–2.277 (3) Å, and Ni—N = 1.951 (3) Å. The Ni^{II} cations are linked into tetrameric complexes about positions of $\overline{4}$ point symmetry by four *N*-(2-hydroxybenzoyl)-*N*-(2-hydroxy-3-methoxybenzylidene)-hydrazine anions (Figure 2). The ligand exhibits an intramolecular O—H···N hydrogen bond.

Experimental

A mixture of nickel(II) acetate (0.5 mmol) and *N*-(2-hydroxybenzoyl)-*N*-(2-hydroxy-3-methoxybenzylidene)-hydrazine in DMF (35 ml) was refluxed for 1 h then filtered. The filtrate was evaporated in the open flask to give green, block-shaped crystals in a yield of 22%. Elemental analysis calculated: C 51.32, H 4.51, N 9.98%; found: C 51.23, H 4.59, N 9.92%.

Refinement

H atoms were generated geometrically and refined as riding atoms with C—H = 0.93Å or 0.96 Å, U_{iso}(H)= 1.2 or 1.5U_{eq}(C), and O—H = 0.82 Å, U_{iso}(H)= 1.5 U_{eq}(O).

Figures

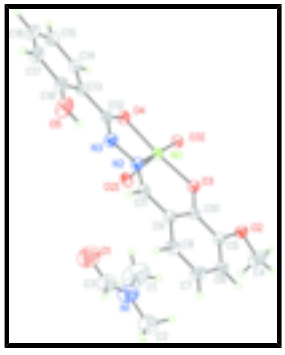

Fig. 1. The asymmetric unit of the title compound, showing displacement ellipsoids at 50% probability for non-H atoms. Atoms labeled with the subscript I are generated by the symmetry operator $y + 1/4, -x + 3/4, -z + 3/4$.

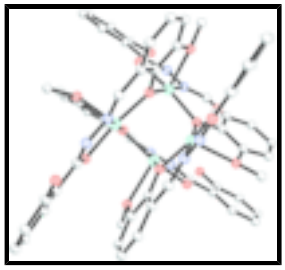

Fig. 2. Tetrameric complex formed about the position of $\bar{4}$ point symmetry.

cyclo-Tetrakis[μ-_N_-(2-hydroxybenzoyl)-_N_'-(2-hydroxy-3-\ methoxybenzylidene)hydrazinato(2-)]tetranickel(II) _N,N_-dimethylformamide tetrasolvate

Crystal data

$[Ni_4(C_{15}H_{12}N_2O_4)_4]\cdot 4C_3H_7NO$	$Z = 4$
$M_r = 1664.29$	$F_{000} = 3456$
Tetragonal, $I4_1/a$	$D_x = 1.452$ Mg m^{-3}
Hall symbol: -I 4ad	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 24.1627$ (5) Å	Cell parameters from 3554 reflections
$b = 24.1627$ (10) Å	$\theta = 1.7$–25.6º
$c = 13.0428$ (5) Å	$\mu = 1.05$ mm^{-1}
$\alpha = 90$º	$T = 293$ (2) K
$\beta = 90$º	Block, green
$\gamma = 90$º	0.44 × 0.32 × 0.28 mm
$V = 7614.9$ (5) Å^3	

Data collection

Bruker APEXII CCD diffractometer	3554 independent reflections
Radiation source: fine-focus sealed tube	2243 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.062$
$T = 293$(2) K	$\theta_{max} = 25.6$º
φ and ω scans	$\theta_{min} = 1.7$º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -29\rightarrow26$
$T_{min} = 0.641$, $T_{max} = 0.742$	$k = -26\rightarrow29$
19316 measured reflections	$l = -15\rightarrow15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters not refined
$wR(F^2) = 0.148$	$w = 1/[\sigma^2(F_o^2) + (0.0789P)^2 + 9.3105P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
3554 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
248 parameters	$\Delta\rho_{min} = -0.27$ 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}
Ni1	0.51343 (2)	0.18333 (2)	0.46561 (3)	0.0409 (2)
C1	0.7168 (5)	0.1344 (6)	0.4322 (8)	0.216 (6)
H1A	0.6779	0.1285	0.4422	0.324*
H1B	0.7332	0.1016	0.4039	0.324*
H1C	0.7222	0.1648	0.3859	0.324*
C2	0.7902 (4)	0.1806 (4)	0.5196 (7)	0.143 (3)
H2A	0.8041	0.1883	0.5871	0.215*
H2B	0.7810	0.2147	0.4861	0.215*
H2C	0.8181	0.1616	0.4807	0.215*
C3	0.7206 (4)	0.1337 (4)	0.6169 (7)	0.115 (2)
H3	0.7379	0.1475	0.6753	0.138*
C4	0.6877 (3)	0.3233 (3)	0.2908 (5)	0.125 (3)
H4A	0.7052	0.3548	0.3216	0.187*
H4B	0.6749	0.3329	0.2234	0.187*
H4C	0.7138	0.2935	0.2861	0.187*
C5	0.65107 (17)	0.29037 (18)	0.4539 (3)	0.0507 (10)
C6	0.69529 (19)	0.3068 (2)	0.5121 (4)	0.0635 (13)
H6	0.7229	0.3284	0.4828	0.076*
C7	0.6993 (2)	0.2913 (2)	0.6157 (4)	0.0703 (14)
H7	0.7296	0.3027	0.6543	0.084*
C8	0.6593 (2)	0.2599 (2)	0.6597 (4)	0.0662 (13)
H8	0.6619	0.2509	0.7289	0.079*
C9	0.61399 (17)	0.24100 (17)	0.6018 (3)	0.0500 (10)
C10	0.60984 (16)	0.25627 (17)	0.4954 (3)	0.0458 (10)
C11	0.57372 (18)	0.20921 (17)	0.6556 (3)	0.0519 (10)
H11	0.5778	0.2048	0.7260	0.062*
C12	0.45681 (17)	0.13432 (17)	0.6187 (3)	0.0502 (10)

C13	0.41377 (18)	0.10458 (18)	0.6752 (4)	0.0546 (11)
C14	0.3737 (2)	0.07678 (19)	0.6201 (5)	0.0713 (14)
H14	0.3753	0.0761	0.5488	0.086*
C15	0.3308 (2)	0.0498 (2)	0.6711 (7)	0.096 (2)
H15	0.3037	0.0309	0.6345	0.116*
C16	0.3291 (3)	0.0515 (3)	0.7802 (7)	0.101 (2)
H16	0.3000	0.0344	0.8142	0.121*
C17	0.3682 (3)	0.0772 (3)	0.8349 (6)	0.095 (2)
H17	0.3664	0.0772	0.9062	0.115*
C18	0.4115 (2)	0.1039 (2)	0.7853 (4)	0.0702 (14)
N1	0.7419 (3)	0.1467 (3)	0.5271 (5)	0.122 (2)
N2	0.53184 (15)	0.18618 (14)	0.6112 (3)	0.0504 (9)
N3	0.49535 (15)	0.15758 (15)	0.6751 (3)	0.0550 (9)
O1	0.6796 (3)	0.1047 (3)	0.6286 (5)	0.169 (3)
O2	0.64209 (12)	0.30661 (13)	0.3519 (2)	0.0618 (8)
O3	0.56721 (11)	0.24110 (12)	0.4338 (2)	0.0483 (7)
O4	0.45500 (12)	0.13623 (12)	0.5176 (2)	0.0553 (8)
O5	0.44927 (19)	0.12902 (19)	0.8454 (3)	0.0966 (13)
H5	0.4730	0.1436	0.8094	0.145*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0523 (3)	0.0493 (3)	0.0210 (3)	−0.0025 (2)	−0.0030 (2)	0.0027 (2)
C1	0.232 (12)	0.307 (16)	0.110 (9)	−0.076 (12)	0.016 (8)	−0.032 (9)
C2	0.118 (6)	0.146 (7)	0.166 (9)	0.000 (6)	0.011 (6)	0.018 (6)
C3	0.103 (6)	0.138 (7)	0.103 (6)	0.006 (5)	0.008 (5)	−0.011 (5)
C4	0.104 (5)	0.188 (8)	0.082 (5)	−0.057 (5)	0.002 (4)	0.050 (5)
C5	0.055 (3)	0.059 (3)	0.038 (2)	−0.001 (2)	−0.0010 (19)	0.0006 (19)
C6	0.058 (3)	0.073 (3)	0.059 (3)	−0.013 (2)	−0.005 (2)	0.002 (2)
C7	0.064 (3)	0.084 (3)	0.063 (3)	−0.019 (3)	−0.016 (3)	0.001 (3)
C8	0.069 (3)	0.087 (3)	0.042 (3)	−0.004 (3)	−0.016 (2)	0.002 (2)
C9	0.055 (2)	0.057 (3)	0.037 (2)	−0.0041 (19)	−0.0067 (19)	0.0004 (19)
C10	0.049 (2)	0.053 (2)	0.036 (2)	0.0001 (18)	−0.0048 (18)	−0.0029 (18)
C11	0.063 (3)	0.064 (3)	0.030 (2)	−0.003 (2)	−0.012 (2)	0.0046 (19)
C12	0.059 (3)	0.052 (2)	0.040 (2)	0.007 (2)	0.000 (2)	0.0116 (19)
C13	0.059 (3)	0.051 (2)	0.054 (3)	0.005 (2)	0.003 (2)	0.014 (2)
C14	0.062 (3)	0.063 (3)	0.089 (4)	0.000 (2)	0.002 (3)	0.021 (3)
C15	0.066 (3)	0.079 (4)	0.145 (7)	−0.009 (3)	−0.002 (4)	0.031 (4)
C16	0.083 (4)	0.088 (4)	0.132 (7)	−0.001 (4)	0.032 (4)	0.040 (4)
C17	0.100 (5)	0.095 (4)	0.092 (5)	0.008 (4)	0.040 (4)	0.030 (4)
C18	0.077 (3)	0.076 (3)	0.058 (3)	0.007 (3)	0.018 (3)	0.017 (3)
N1	0.102 (4)	0.171 (6)	0.092 (5)	−0.004 (4)	0.018 (4)	−0.010 (4)
N2	0.060 (2)	0.061 (2)	0.0303 (19)	−0.0001 (17)	−0.0012 (16)	0.0080 (16)
N3	0.061 (2)	0.071 (2)	0.0333 (19)	−0.0056 (19)	−0.0004 (17)	0.0076 (17)
O1	0.149 (5)	0.217 (7)	0.143 (6)	−0.022 (5)	0.024 (5)	0.015 (5)
O2	0.0626 (19)	0.080 (2)	0.0431 (18)	−0.0146 (16)	0.0003 (14)	0.0138 (16)
O3	0.0555 (16)	0.0626 (17)	0.0268 (14)	−0.0049 (13)	−0.0038 (12)	0.0038 (13)

O4	0.0681 (19)	0.0615 (18)	0.0364 (17)	−0.0058 (14)	−0.0051 (14)	0.0039 (13)
O5	0.123 (3)	0.124 (3)	0.043 (2)	−0.027 (3)	0.015 (2)	0.009 (2)

Geometric parameters (Å, °)

Ni1—O4	1.936 (3)	C7—H7	0.930
Ni1—O3	1.952 (3)	C8—C9	1.408 (6)
Ni1—N2	1.951 (3)	C8—H8	0.930
Ni1—O3^{i}	2.022 (3)	C9—C11	1.425 (6)
Ni1—O2^{i}	2.277 (3)	C9—C10	1.440 (6)
C1—N1	1.410 (11)	C10—O3	1.357 (5)
C1—H1A	0.960	C11—N2	1.292 (5)
C1—H1B	0.960	C11—H11	0.930
C1—H1C	0.960	C12—N3	1.313 (5)
C2—N1	1.429 (10)	C12—O4	1.320 (5)
C2—H2A	0.960	C12—C13	1.463 (6)
C2—H2B	0.960	C13—C14	1.380 (7)
C2—H2C	0.960	C13—C18	1.438 (7)
C3—O1	1.224 (9)	C14—C15	1.393 (7)
C3—N1	1.318 (10)	C14—H14	0.930
C3—H3	0.930	C15—C16	1.423 (10)
C4—O2	1.418 (6)	C15—H15	0.930
C4—H4A	0.960	C16—C17	1.337 (9)
C4—H4B	0.960	C16—H16	0.930
C4—H4C	0.960	C17—C18	1.389 (7)
C5—C6	1.369 (6)	C17—H17	0.930
C5—O2	1.404 (5)	C18—O5	1.347 (6)
C5—C10	1.401 (6)	N2—N3	1.396 (5)
C6—C7	1.406 (7)	O2—Ni1^{ii}	2.277 (3)
C6—H6	0.930	O3—Ni1^{ii}	2.022 (3)
C7—C8	1.356 (7)	O5—H5	0.820
O4—Ni1—O3	168.64 (12)	C8—C9—C10	119.2 (4)
O4—Ni1—N2	81.15 (13)	C11—C9—C10	124.4 (4)
O3—Ni1—N2	91.71 (13)	O3—C10—C5	118.1 (4)
O4—Ni1—O3^{i}	97.99 (12)	O3—C10—C9	123.7 (4)
O3—Ni1—O3^{i}	88.71 (11)	C5—C10—C9	118.2 (4)
N2—Ni1—O3^{i}	177.17 (13)	N2—C11—C9	123.1 (4)
O4—Ni1—O2^{i}	89.95 (12)	N2—C11—H11	118.4
O3—Ni1—O2^{i}	100.67 (12)	C9—C11—H11	118.4
N2—Ni1—O2^{i}	107.47 (13)	N3—C12—O4	124.6 (4)
O3^{i}—Ni1—O2^{i}	75.18 (10)	N3—C12—C13	115.6 (4)
N1—C1—H1A	109.5	O4—C12—C13	119.8 (4)
N1—C1—H1B	109.5	C14—C13—C18	119.2 (5)
H1A—C1—H1B	109.5	C14—C13—C12	118.4 (4)
N1—C1—H1C	109.5	C18—C13—C12	122.4 (4)
H1A—C1—H1C	109.5	C13—C14—C15	120.0 (6)
H1B—C1—H1C	109.5	C13—C14—H14	120.0

N1—C2—H2A	109.5	C15—C14—H14	120.0
N1—C2—H2B	109.5	C14—C15—C16	119.0 (6)
H2A—C2—H2B	109.5	C14—C15—H15	120.5
N1—C2—H2C	109.5	C16—C15—H15	120.5
H2A—C2—H2C	109.5	C17—C16—C15	121.8 (6)
H2B—C2—H2C	109.5	C17—C16—H16	119.1
O1—C3—N1	124.3 (9)	C15—C16—H16	119.1
O1—C3—H3	117.8	C16—C17—C18	119.9 (7)
N1—C3—H3	117.8	C16—C17—H17	120.1
O2—C4—H4A	109.5	C18—C17—H17	120.1
O2—C4—H4B	109.5	O5—C18—C17	116.6 (6)
H4A—C4—H4B	109.5	O5—C18—C13	123.4 (4)
O2—C4—H4C	109.5	C17—C18—C13	120.0 (6)
H4A—C4—H4C	109.5	C3—N1—C1	124.2 (8)
H4B—C4—H4C	109.5	C3—N1—C2	121.1 (8)
C6—C5—O2	124.4 (4)	C1—N1—C2	114.4 (8)
C6—C5—C10	120.8 (4)	C11—N2—N3	116.1 (3)
O2—C5—C10	114.8 (4)	C11—N2—Ni1	129.1 (3)
C5—C6—C7	120.6 (4)	N3—N2—Ni1	114.8 (3)
C5—C6—H6	119.7	C12—N3—N2	109.0 (3)
C7—C6—H6	119.7	C5—O2—C4	119.5 (4)
C8—C7—C6	120.4 (4)	C5—O2—Ni1^{ii}	111.2 (2)
C8—C7—H7	119.8	C4—O2—Ni1^{ii}	122.3 (3)
C6—C7—H7	119.8	C10—O3—Ni1	125.0 (3)
C7—C8—C9	120.6 (4)	C10—O3—Ni1^{ii}	120.2 (2)
C7—C8—H8	119.7	Ni1—O3—Ni1^{ii}	113.04 (13)
C9—C8—H8	119.7	C12—O4—Ni1	110.3 (3)
C8—C9—C11	116.3 (4)	C18—O5—H5	109.5

Symmetry codes: (i) *y*+1/4, −*x*+3/4, −*z*+3/4; (ii) −*y*+3/4, *x*−1/4, −*z*+3/4.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···N3	0.82	1.86	2.579 (5)	145

Fig. 1

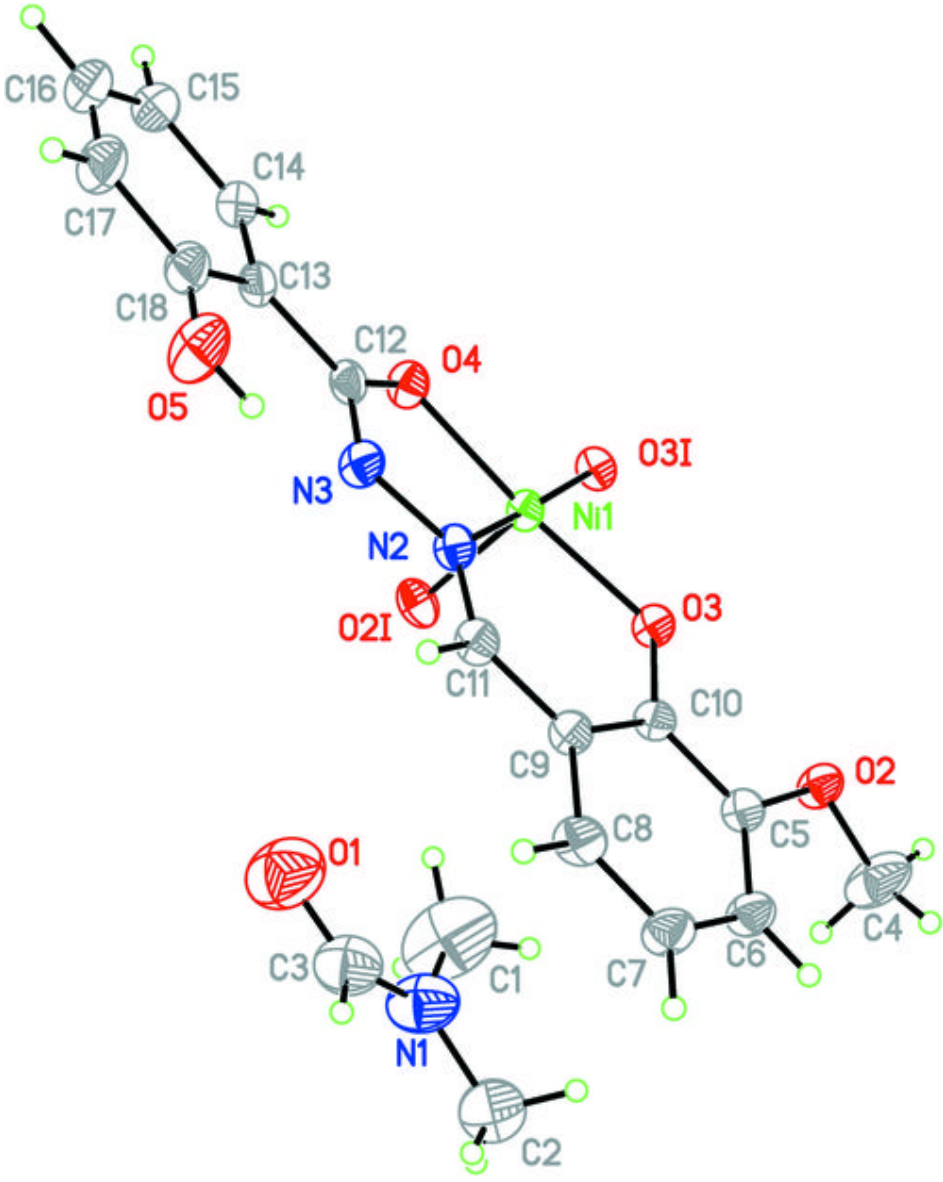
C16
C15
C14
C17
C13
C18
C12
O4
O5
O3I
N3
Ni1
N2
O2I
O3
C11
C10
C9
O2
C8
C5
O1
C4
C3
C1
C7
C6
N1
C2

Fig. 2