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Bis(*tert*-butyl-3-oxobutanoato)dipyridine-nickel(II) benzene solvate

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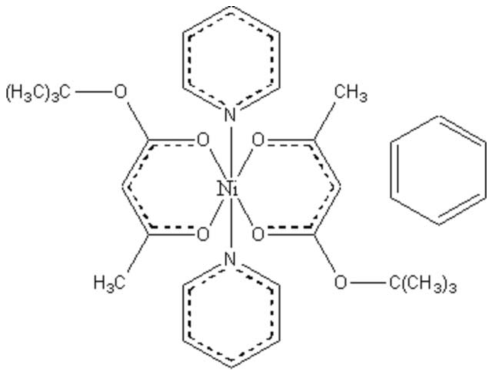
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.004 Å; R factor = 0.031; wR factor = 0.086; data-to-parameter ratio = 15.9.

The title compound, $[Ni(C_8H_{13}O_3)_2(C_5H_5N)_2]\cdot C_6H_6$, a potential metal–organic chemical vapour deposition (MOCVD) precursor, crystallizes with half a molecule of the complex and half a molecule of benzene in the asymmetric unit, both molecules being centrosymmetric. The Ni atom is at the origin (0, 0, 0), while the centroid of the benzene solvent molecule is at another centre of symmetry ($0, \frac{1}{2}, \frac{1}{2}$). The Ni atom has a distorted octahedral environment, with four O atoms from the bidentate *tert*-butyl-3-oxobutanoate ligand units in the axial positions and the two N atoms of the pyridine ligands at the apical positions. C—H$\cdots\pi$ contacts [3.021 (4) Å] between solvent benzene and the pyridine ligand, along with π–π stacking interactions [3.896 (1) Å] between adjacent pyridine ligands, result in a packing motif along the c axis.

Related literature

For a background to chemical vapour deposition (CVD) and the synthesis of MOCVD precursors, see: Williams (1989), Dharmaprakash *et al.* (2006, and references therein); Neelgund *et al.* (2007).



Experimental

Crystal data

$[Ni(C_8H_{13}O_3)_2(C_5H_5N)_2]\cdot C_6H_6$
M_r = 609.37
Triclinic, $P\overline{1}$
a = 8.3748 (11) Å
b = 8.8057 (12) Å
c = 12.5949 (17) Å
α = 99.205 (2)°
β = 93.504 (2)°
γ = 117.338 (2)°
V = 804.81 (19) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 292 (2) K
0.54 × 0.43 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.722, T_{max} = 0.876
8193 measured reflections
3031 independent reflections
2913 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.10
3031 reflections
191 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2312).

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supplementary materials

Acta Cryst. (2007). E**63**, m1604   [**doi:10.1107/S1600536807021496**]

Bis(*tert*-butyl-3-oxobutanoato)dipyridinenickel(II) benzene solvate

S. K. Nayak, **A. Jena**, **G. M. Neelgund**, **S. A. Shivashankar** and **T. N. G. Row**

Comment

Based on the methodology suggested for making thin films via chemical vapour deposition (CVD) by Williams (1989), several metal-organic (MO) CVD precursors have been synthesized and characterized (Dharmaprakash *et al.*, 2006; Neelgund *et al.*, 2007). In our attempts to synthesize less volatile hydrate precursors, the title compound has been prepared and its structure determined by X-ray diffraction.

The Ni atom is six co-ordinated with the participation of the ketonic O atoms of two bidentate *tert*-butyl-3-oxobutanoate ligands in the basal plane and with the N atoms of the two pyridine molecules in *anti* positions to each other, resulting in a distorted octahedral geometry [Fig. 1]. The Ni atom and solvent benzene molecule lie on the inversion centres at (0 0 0) and (0 1/2 1/2) respectively. Selected bond Ni—O/Ni—N distances are Ni1—O1 [2.024 (1) Å], Ni1—O2 [2.062 (1) Å] and Ni1—N1 [2.104 (1) Å].

Pairs of centrosymmetrically related pyridine ligands are involved in intermolecular π···π interactions [Cg···Cg = 3.896 (1) Å C g = centroid of the pyridine ring (N1/C1/C2/C3/C4/C5), symmetry code (-x, 1-y, -z)], with each such pair linked to a solvent benzene molecule via C18—H18···π interactions [H18···Cg = 3.021 (4) Å, symmetry code (-x+1, -y, -z)], stabilizing the crystal packing in (I) [Fig. 2]. The supramolecular assembly is thus brought by an infinite chain of C—H···π and π···π interactions along the c-axis [Fig. 2].

Experimental

The compound was synthesized by dissolving $Ni(NO_3)_2.6H_2O$ (2.9 g, 10 mmol) in 30% ethanol (30 ml). To this solution, (3.26 ml, 20 mmol) *tert*-butyl-3-oxobutanoate was added, maintaining the pH at 6.5 by adding KOH dissolved in 30% ethanol and the mixture was stirred for 30 minutes at 4 °C. The precipitate formed was filtered off, suction-dried and recrystallized to obtain block shaped crystals from a mixture of pyridine and benzene (2:1 v/v).

Refinement

Hydrogen atoms were fixed geometrically and treated as riding atoms, with C—H distance of 0.93Å (Csp^2), 0.96Å (Csp^3) and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for other H atoms. The disordered benzene solvent was subjected to restrained refinement using DFIX 1.400 0.002 command for the distances C16—C17 and C16—C18 respectively.

Figures

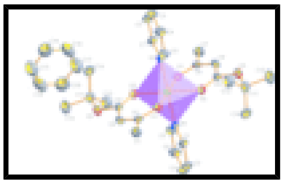

Fig. 1. The asymmetric unit of (I) showing 30% probability dispacement ellipsoids of (I). The octahedral coordination at the Ni atom is shown shaded. Hydrogen atoms are omitted for clarity.

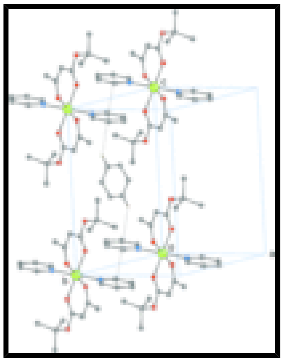

Fig. 2. : Molecular packing of (I) showing the infinite chain of C–H···π and π···π interactions.

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Crystal data

$[Ni(C_8H_{13}O_3)_2(C_5H_5N)_2]\cdot C_6H_6$

$M_r = 609.37$

Triclinic, $P\overline{1}$

Hall symbol: -P 1

$a = 8.3748\ (11)$ Å

$b = 8.8057\ (12)$ Å

$c = 12.5949\ (17)$ Å

$\alpha = 99.205\ (2)$°

$\beta = 93.504\ (2)$°

$\gamma = 117.338\ (2)$°

$V = 804.81\ (19)$ Å^3

$Z = 1$

$F_{000} = 324$

$D_x = 1.257$ Mg m^{-3}

Melting point: 124 K

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

Cell parameters from 622 reflections

$\theta = 0.9–28.0$°

$\mu = 0.65$ mm^{-1}

$T = 292\ (2)$ K

Block, pale blue

0.54 × 0.43 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 292(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{min} = 0.722$, $T_{max} = 0.876$

8193 measured reflections

3031 independent reflections

2913 reflections with $I > 2\sigma(I)$

$R_{int} = 0.020$

$\theta_{max} = 25.7$°

$\theta_{min} = 2.7$°

$h = -10\rightarrow10$

$k = -10\rightarrow10$

$l = -15\rightarrow15$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.1788P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.086$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.10$	$\Delta\rho_{max} = 0.27$ e Å^{-3}
3031 reflections	$\Delta\rho_{min} = -0.32$ e Å^{-3}
191 parameters	Extinction correction: none
2 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.0000	0.0000	0.0000	0.03488 (11)
O1	0.23256 (15)	0.17167 (15)	−0.04472 (10)	0.0424 (3)
O2	0.14118 (16)	0.04802 (16)	0.15216 (10)	0.0437 (3)
N1	0.06342 (19)	−0.20147 (18)	−0.05506 (12)	0.0405 (3)
O3	0.16353 (19)	0.06159 (19)	0.33340 (10)	0.0563 (3)
C9	0.0652 (2)	0.0073 (2)	0.23278 (14)	0.0415 (4)
C6	0.2545 (2)	0.1808 (2)	−0.14322 (15)	0.0411 (4)
C8	−0.1222 (3)	−0.0968 (2)	0.23407 (15)	0.0482 (4)
H8	−0.1596	−0.1099	0.3016	0.058*
C5	−0.0671 (3)	−0.3621 (2)	−0.10124 (16)	0.0500 (4)
H5	−0.1875	−0.3844	−0.1075	0.060*
C1	0.2349 (2)	−0.1730 (3)	−0.04644 (16)	0.0496 (4)
H1	0.3272	−0.0617	−0.0136	0.059*
C11	0.3635 (3)	0.1579 (3)	0.35310 (17)	0.0591 (5)
C2	0.2816 (3)	−0.3003 (3)	−0.08382 (19)	0.0611 (5)
H2	0.4028	−0.2755	−0.0770	0.073*

C4	−0.0311 (3)	−0.4962 (3)	−0.1399 (2)	0.0661 (6)
H4	−0.1256	−0.6070	−0.1716	0.079*
C7	0.4466 (3)	0.3003 (3)	−0.15884 (19)	0.0603 (5)
H7A	0.4866	0.4157	−0.1164	0.090*
H7B	0.4496	0.3050	−0.2344	0.090*
H7C	0.5256	0.2563	−0.1357	0.090*
C12	0.4337 (4)	0.3310 (3)	0.3181 (2)	0.0760 (7)
H12A	0.4097	0.3104	0.2402	0.114*
H12B	0.5624	0.3991	0.3423	0.114*
H12C	0.3736	0.3933	0.3497	0.114*
C14	0.4380 (4)	0.0438 (4)	0.2969 (2)	0.0771 (7)
H14A	0.3888	−0.0649	0.3209	0.116*
H14B	0.5682	0.1029	0.3148	0.116*
H14C	0.4041	0.0210	0.2196	0.116*
C3	0.1455 (4)	−0.4647 (3)	−0.1313 (2)	0.0699 (6)
H3	0.1728	−0.5536	−0.1574	0.084*
C13	0.4045 (4)	0.1869 (4)	0.4768 (2)	0.0907 (9)
H13A	0.3537	0.2568	0.5107	0.136*
H13B	0.5338	0.2461	0.4994	0.136*
H13C	0.3519	0.0760	0.4980	0.136*
C16	0.9918 (6)	0.3375 (5)	0.4840 (3)	0.1279 (14)
H16	0.9844	0.2273	0.4716	0.153*
C17	0.9913 (8)	0.4253 (8)	0.5856 (3)	0.155 (2)
H17	0.9879	0.3752	0.6457	0.186*
C18	1.0046 (8)	0.4301 (8)	0.4024 (3)	0.155 (2)
H18	1.0105	0.3795	0.3330	0.186*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02993 (16)	0.03302 (17)	0.03890 (18)	0.01287 (12)	0.00500 (11)	0.00705 (12)
O1	0.0350 (6)	0.0391 (6)	0.0473 (7)	0.0129 (5)	0.0073 (5)	0.0088 (5)
O2	0.0375 (6)	0.0467 (7)	0.0421 (6)	0.0167 (5)	0.0035 (5)	0.0089 (5)
N1	0.0383 (7)	0.0375 (7)	0.0453 (8)	0.0177 (6)	0.0073 (6)	0.0088 (6)
O3	0.0547 (8)	0.0633 (9)	0.0406 (7)	0.0211 (7)	0.0011 (6)	0.0088 (6)
C9	0.0478 (9)	0.0370 (8)	0.0395 (9)	0.0212 (8)	0.0037 (7)	0.0058 (7)
C6	0.0393 (9)	0.0341 (8)	0.0550 (10)	0.0197 (7)	0.0149 (7)	0.0130 (7)
C8	0.0506 (10)	0.0490 (10)	0.0448 (10)	0.0216 (9)	0.0145 (8)	0.0137 (8)
C5	0.0447 (10)	0.0395 (9)	0.0582 (11)	0.0151 (8)	0.0078 (8)	0.0062 (8)
C1	0.0402 (9)	0.0475 (10)	0.0613 (11)	0.0215 (8)	0.0073 (8)	0.0104 (8)
C11	0.0554 (12)	0.0583 (12)	0.0532 (11)	0.0224 (10)	−0.0076 (9)	0.0045 (9)
C2	0.0577 (12)	0.0687 (14)	0.0743 (14)	0.0420 (11)	0.0181 (10)	0.0206 (11)
C4	0.0702 (14)	0.0413 (10)	0.0772 (14)	0.0224 (10)	0.0109 (11)	0.0008 (10)
C7	0.0438 (10)	0.0610 (12)	0.0724 (14)	0.0174 (9)	0.0196 (10)	0.0244 (11)
C12	0.0670 (15)	0.0547 (13)	0.0864 (17)	0.0168 (11)	−0.0042 (13)	0.0073 (12)
C14	0.0720 (15)	0.0850 (17)	0.0809 (16)	0.0476 (14)	−0.0065 (13)	0.0095 (13)
C3	0.0892 (17)	0.0598 (13)	0.0805 (16)	0.0514 (13)	0.0237 (13)	0.0125 (11)
C13	0.0869 (19)	0.106 (2)	0.0571 (14)	0.0357 (17)	−0.0196 (13)	0.0033 (14)

C16	0.115 (3)	0.143 (4)	0.121 (3)	0.069 (3)	−0.003 (2)	0.000 (3)
C17	0.229 (6)	0.184 (5)	0.093 (3)	0.126 (5)	0.039 (3)	0.042 (3)
C18	0.217 (6)	0.206 (6)	0.078 (2)	0.131 (5)	0.037 (3)	0.019 (3)

Geometric parameters (Å, °)

Ni1—O1	2.0243 (11)	C2—C3	1.370 (3)
Ni1—O1^{i}	2.0243 (11)	C2—H2	0.9300
Ni1—O2^{i}	2.0626 (12)	C4—C3	1.368 (3)
Ni1—O2	2.0626 (12)	C4—H4	0.9300
Ni1—N1	2.1041 (14)	C7—H7A	0.9600
Ni1—N1^{i}	2.1041 (14)	C7—H7B	0.9600
O1—C6	1.275 (2)	C7—H7C	0.9600
O2—C9	1.245 (2)	C12—H12A	0.9600
N1—C5	1.332 (2)	C12—H12B	0.9600
N1—C1	1.333 (2)	C12—H12C	0.9600
O3—C9	1.353 (2)	C14—H14A	0.9600
O3—C11	1.470 (3)	C14—H14B	0.9600
C9—C8	1.411 (3)	C14—H14C	0.9600
C6—C8^{i}	1.380 (3)	C3—H3	0.9300
C6—C7	1.512 (2)	C13—H13A	0.9600
C8—C6^{i}	1.380 (3)	C13—H13B	0.9600
C8—H8	0.9300	C13—H13C	0.9600
C5—C4	1.371 (3)	C16—C17	1.387 (6)
C5—H5	0.9300	C16—C18	1.391 (6)
C1—C2	1.375 (3)	C16—H16	0.9300
C1—H1	0.9300	C17—C18^{ii}	1.243 (6)
C11—C12	1.512 (3)	C17—H17	0.9300
C11—C14	1.517 (3)	C18—C17^{ii}	1.243 (6)
C11—C13	1.529 (3)	C18—H18	0.9300
O1—Ni1—O1^{i}	180.00 (7)	C3—C2—C1	118.5 (2)
O1—Ni1—O2^{i}	90.70 (5)	C3—C2—H2	120.8
O1^{i}—Ni1—O2^{i}	89.30 (5)	C1—C2—H2	120.8
O1—Ni1—O2	89.30 (5)	C3—C4—C5	119.1 (2)
O1^{i}—Ni1—O2	90.70 (5)	C3—C4—H4	120.4
O2^{i}—Ni1—O2	180.00 (8)	C5—C4—H4	120.4
O1—Ni1—N1	89.06 (5)	C6—C7—H7A	109.5
O1^{i}—Ni1—N1	90.94 (5)	C6—C7—H7B	109.5
O2^{i}—Ni1—N1	89.84 (5)	H7A—C7—H7B	109.5
O2—Ni1—N1	90.16 (5)	C6—C7—H7C	109.5
O1—Ni1—N1^{i}	90.94 (5)	H7A—C7—H7C	109.5
O1^{i}—Ni1—N1^{i}	89.06 (5)	H7B—C7—H7C	109.5
O2^{i}—Ni1—N1^{i}	90.16 (5)	C11—C12—H12A	109.5
O2—Ni1—N1^{i}	89.84 (5)	C11—C12—H12B	109.5
N1—Ni1—N1^{i}	180.00 (7)	H12A—C12—H12B	109.5

C6—O1—Ni1	123.90 (11)	C11—C12—H12C	109.5
C9—O2—Ni1	123.11 (11)	H12A—C12—H12C	109.5
C5—N1—C1	117.60 (16)	H12B—C12—H12C	109.5
C5—N1—Ni1	120.88 (12)	C11—C14—H14A	109.5
C1—N1—Ni1	121.52 (12)	C11—C14—H14B	109.5
C9—O3—C11	122.78 (15)	H14A—C14—H14B	109.5
O2—C9—O3	120.71 (16)	C11—C14—H14C	109.5
O2—C9—C8	127.34 (16)	H14A—C14—H14C	109.5
O3—C9—C8	111.94 (15)	H14B—C14—H14C	109.5
O1—C6—C8^{i}	126.91 (16)	C4—C3—C2	119.06 (19)
O1—C6—C7	114.90 (16)	C4—C3—H3	120.5
C8^{i}—C6—C7	118.18 (17)	C2—C3—H3	120.5
C6^{i}—C8—C9	125.26 (17)	C11—C13—H13A	109.5
C6^{i}—C8—H8	117.4	C11—C13—H13B	109.5
C9—C8—H8	117.4	H13A—C13—H13B	109.5
N1—C5—C4	122.65 (19)	C11—C13—H13C	109.5
N1—C5—H5	118.7	H13A—C13—H13C	109.5
C4—C5—H5	118.7	H13B—C13—H13C	109.5
N1—C1—C2	123.11 (18)	C17—C16—C18	113.6 (4)
N1—C1—H1	118.4	C17—C16—H16	123.2
C2—C1—H1	118.4	C18—C16—H16	123.2
O3—C11—C12	111.15 (18)	C18^{ii}—C17—C16	120.9 (4)
O3—C11—C14	109.53 (18)	C18^{ii}—C17—H17	119.5
C12—C11—C14	112.4 (2)	C16—C17—H17	119.5
O3—C11—C13	101.87 (19)	C17^{ii}—C18—C16	125.4 (4)
C12—C11—C13	110.6 (2)	C17^{ii}—C18—H18	117.3
C14—C11—C13	110.8 (2)	C16—C18—H18	117.3

O2^{i}—Ni1—O1—C6	−16.16 (13)	C11—O3—C9—C8	174.11 (17)
O2—Ni1—O1—C6	163.84 (13)	Ni1—O1—C6—C8^{i}	9.6 (2)
N1—Ni1—O1—C6	73.67 (13)	Ni1—O1—C6—C7	−171.77 (12)
N1^{i}—Ni1—O1—C6	−106.33 (13)	O2—C9—C8—C6^{i}	3.9 (3)
O1—Ni1—O2—C9	163.87 (13)	O3—C9—C8—C6^{i}	−175.27 (17)
O1^{i}—Ni1—O2—C9	−16.13 (13)	C1—N1—C5—C4	−0.4 (3)
N1—Ni1—O2—C9	−107.07 (13)	Ni1—N1—C5—C4	178.80 (16)
N1^{i}—Ni1—O2—C9	72.93 (13)	C5—N1—C1—C2	0.7 (3)
O1—Ni1—N1—C5	−144.94 (14)	Ni1—N1—C1—C2	−178.42 (16)
O1^{i}—Ni1—N1—C5	35.06 (14)	C9—O3—C11—C12	62.2 (2)
O2^{i}—Ni1—N1—C5	−54.24 (14)	C9—O3—C11—C14	−62.7 (2)
O2—Ni1—N1—C5	125.76 (14)	C9—O3—C11—C13	179.96 (19)
O1—Ni1—N1—C1	34.18 (14)	N1—C1—C2—C3	−0.6 (3)
O1^{i}—Ni1—N1—C1	−145.82 (14)	N1—C5—C4—C3	−0.2 (3)
O2^{i}—Ni1—N1—C1	124.88 (14)	C5—C4—C3—C2	0.3 (4)
O2—Ni1—N1—C1	−55.12 (14)	C1—C2—C3—C4	0.0 (4)
Ni1—O2—C9—O3	−171.36 (12)	C18—C16—C17—C18^{ii}	2.4 (10)
Ni1—O2—C9—C8	9.6 (2)	C17—C16—C18—C17^{ii}	−2.6 (10)

C11—O3—C9—O2	−5.1 (3)

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*+2, −*y*+1, −*z*+1.

Fig. 1

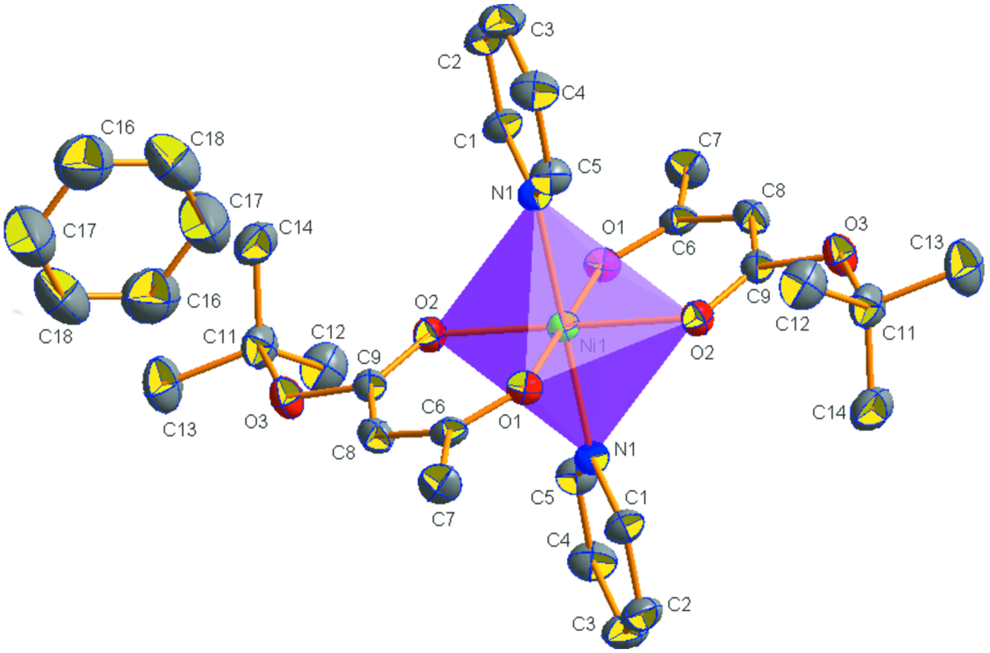

Fig. 2