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# Tris(pyridin-2-ylmethanol)nickel(II) hexafluoridophosphate trifluoroacetate

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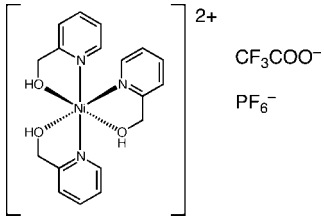
Key indicators: single-crystal X-ray study; $T$ = 200 K; mean $\sigma$(C–C) = 0.003 Å; disorder in solvent or counterion; $R$ factor = 0.033; $wR$ factor = 0.088; data-to-parameter ratio = 15.3.

In the crystal structure of the title complex, $[Ni(C_6H_7NO)_3](PF_6)(C_2F_3O_2)$, the $Ni^{II}$ ion is in a slightly distorted octahedral $NiO_3N_3$ coordination geometry with each of the three N and three O atoms in a meridional coordination. In the crystal, the complex molecules and the trifluoroacetate anions are connected *via* O—H···O hydrogen bonding into layers parallel to the *ab* plane.

## Related literature

For related complexes, see: Ito & Onaka (2004); Kermagoret & Braunstein (2008).




## Experimental

### Crystal data

$[Ni(C_6H_7NO)_3](PF_6)(C_2F_3O_2)$
$M_r$ = 644.08
Triclinic, $P\overline{1}$
$a$ = 9.6381 (2) Å
$b$ = 11.9668 (4) Å
$c$ = 11.9892 (3) Å
$\alpha$ = 109.950 (1)°
$\beta$ = 95.348 (1)°
$\gamma$ = 101.411 (1)°
$V$ = 1254.60 (6) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.94 mm$^{-1}$
$T$ = 200 K
0.40 × 0.30 × 0.20 mm

### Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Rigaku, 1995)
$T_{min}$ = 0.813, $T_{max}$ = 1.000
12517 measured reflections
5736 independent reflections
5234 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.088
$S$ = 1.05
5736 reflections
376 parameters
9 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.45 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Ni1—O1 | 2.0461 (12) | Ni1—N1 | 2.0662 (14) |
| Ni1—N2 | 2.0601 (14) | Ni1—O3 | 2.0714 (12) |
| Ni1—O2 | 2.0647 (12) | Ni1—N3 | 2.0769 (14) |
| O1—Ni1—N1 | 78.11 (5) | O3—Ni1—N3 | 78.09 (5) |
| N2—Ni1—O2 | 78.53 (5) | | |

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···O4$^{i}$ | 0.87 | 1.76 | 2.6003 (19) | 162.5 |
| O2—H2···O5$^{ii}$ | 0.92 | 1.77 | 2.6965 (18) | 175.8 |
| O3—H3···O5$^{iii}$ | 0.98 | 1.65 | 2.6267 (18) | 173.8 |

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

Data collection: *RAPID-AUTO* (Rigaku, 2002); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Yadokari-XG* (Wakita, 2001; Kabuto *et al.*, 2009), *ORTEP-3* for Windows (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *Yadokari-XG* and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2249).

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# supplementary materials

*Acta Cryst.* (2011). E**67**, m1632 [ **doi:10.1107/S160053681104431X** ]

# Tris(pyridin-2-ylmethanol)nickel(II) hexafluoridophosphate trifluoroacetate

**T. Hamaguchi, T. Nagata, S. Kawata and I. Ando**

## Comment

The crystal structure of the title compound is composed of $[Ni^{II}(C_6H_7ON)_3]^{2+}$ cations, hexafluorophosphate and trifluoroacetate anions. The $Ni^{II}$ ion is in a slightly distorted octahedral coordination, comprising three N atoms and three O atoms from three pyridine-2-methanol ligands (Fig. 1 and Table 1). The three N and three O atoms make a meridional $NiO_3N_3$ coordination and the mean bite angle of the pyridine-2-methanol ligand amount to 78.2 (2) °.

In the crystal structure the complexes are connected via O—H···O hydrogen bonding between the hydroxy H atoms of the pyridine-2-methanol ligand and the O atoms of the trifluoroacetate anion into layers that are parallel to the a/b plane. (Fig. 2 and 3 and Table 2).

## Experimental

A solution of $NiSO_4.6H_2O$ (0.5 mmol) in $H_2O$ (1 ml) was added to the solution of pyridine-2-methanol (1.5 mmol) in $H_2O$ (3 ml). Afterwards $NH_4PF_6$ (6.0 mmol) and $CF_3COONa$ (2.5 mmol) were added to the resulting blue solution. The resulting pale blue precipitate was collected. The crude product was purified by recrystallization from acetone and water. The blue prism-like crystals were obtained a few days later on slow evaporation of the solvent.

## Refinement

The O–H H atoms were located in a difference Fourier map and the coordinates were fixed. Their $U_{iso}$(H) values were set to 1.5$U_{eq}$(O). Other H atoms were placed at calculated positions and were treated as riding on the parent C atoms, with C–H = 0.93 (CH) and 0.97 ($CH_2$) Å and with $U_{iso}$(H) = 1.2$U_{eq}$(C). Three F atoms in $CF_3COO$ anions are rotationally disordered between three positions. The two parts of lower occupation were refined only isotropic (sof. 0.6:0.24:0.16).

## Figures

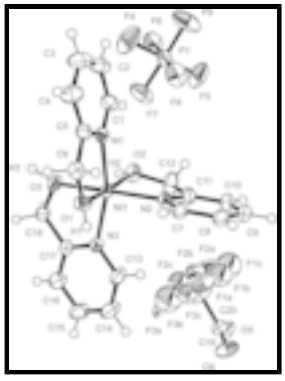

Fig. 1. *ORTEP* drawing for the title complex with labeling showing 50% probability displacement ellipsoids. Please note: The trifluoroacetate anion is disordered.

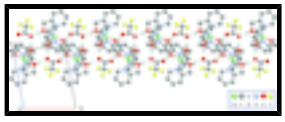

Fig. 2. Crystal structure of the title compound view along the b-axis. The C-H H atoms, the $PF_6$ anions and the disordered F atoms with lower occupation of the trifluoroacetate anions are omitted for clarity. O—H···O hydrogen bonding is shown as dashed blue lines.

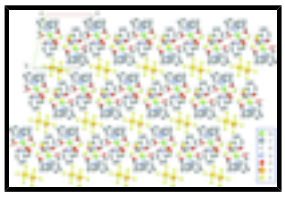

Fig. 3. Crystal structure of the title compound with view along the c-axis. The C-H H atoms and the disordered F atoms with lower occupation of the trifluoroacetate anions are omitted for clarity.

**Tris(pyridin-2-ylmethanol)nickel(II) hexafluoridophosphate trifluoroacetate**

*Crystal data*

| | |
|---|---|
| $[Ni(C_6H_7NO)_3](PF_6)(C_2F_3O_2)$ | $V = 1254.60\ (6)$ Å$^3$ |
| $M_r = 644.08$ | $Z = 2$ |
| Triclinic, $P\overline{1}$ | $F(000) = 652$ |
| Hall symbol: -P 1 | $D_x = 1.705$ Mg m$^{-3}$ |
| $a = 9.6381\ (2)$ Å | Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å |
| $b = 11.9668\ (4)$ Å | $\mu = 0.94$ mm$^{-1}$ |
| $c = 11.9892\ (3)$ Å | $T = 200$ K |
| $\alpha = 109.950\ (1)°$ | Block, blue |
| $\beta = 95.348\ (1)°$ | 0.40 × 0.30 × 0.20 mm |
| $\gamma = 101.411\ (1)°$ | |

*Data collection*

| | |
|---|---|
| Rigaku R-AXIS RAPID diffractometer | 5234 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.018$ |
| ω scans | $\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$ |
| Absorption correction: multi-scan (*ABSCOR*; Rigaku, 1995) | $h = -12\rightarrow11$ |
| $T_{min} = 0.813$, $T_{max} = 1.000$ | $k = -15\rightarrow15$ |
| 12517 measured reflections | $l = -15\rightarrow15$ |
| 5736 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.033$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.088$ | H-atom parameters constrained |
| $S = 1.05$ | $w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.5841P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 5736 reflections | $(\Delta/\sigma)_{max} = 0.001$ |

376 parameters

9 restraints

$\Delta\rho_{max} = 0.50$ e Å$^{-3}$

$\Delta\rho_{min} = -0.45$ 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 $F^2 > \sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Ni1 | 0.18393 (2) | 0.369025 (18) | 0.808996 (17) | 0.02212 (7) | |
| O1 | 0.00845 (13) | 0.39356 (12) | 0.88953 (12) | 0.0304 (3) | |
| H1 | −0.0596 | 0.4198 | 0.8609 | 0.046* | |
| O2 | 0.35252 (13) | 0.32008 (12) | 0.72559 (12) | 0.0337 (3) | |
| H2 | 0.4425 | 0.3733 | 0.7547 | 0.051* | |
| O3 | 0.33165 (14) | 0.49798 (11) | 0.95482 (11) | 0.0316 (3) | |
| H3 | 0.3431 | 0.5104 | 1.0408 | 0.047* | |
| N1 | 0.16982 (15) | 0.24566 (12) | 0.89561 (13) | 0.0257 (3) | |
| N2 | 0.07694 (15) | 0.24685 (13) | 0.64160 (13) | 0.0264 (3) | |
| N3 | 0.20297 (15) | 0.52595 (13) | 0.76794 (12) | 0.0255 (3) | |
| C1 | 0.26677 (19) | 0.17914 (16) | 0.90061 (18) | 0.0338 (4) | |
| H4 | 0.3381 | 0.1775 | 0.8509 | 0.041* | |
| C2 | 0.2656 (2) | 0.11372 (19) | 0.9755 (2) | 0.0447 (5) | |
| H5 | 0.3343 | 0.0670 | 0.9765 | 0.054* | |
| C3 | 0.1636 (3) | 0.1169 (2) | 1.0488 (2) | 0.0530 (6) | |
| H6 | 0.1624 | 0.0740 | 1.1025 | 0.064* | |
| C4 | 0.0627 (2) | 0.1836 (2) | 1.0432 (2) | 0.0458 (5) | |
| H7 | −0.0093 | 0.1864 | 1.0924 | 0.055* | |
| C5 | 0.06813 (18) | 0.24611 (15) | 0.96467 (16) | 0.0286 (3) | |
| C6 | −0.04137 (18) | 0.31692 (16) | 0.95294 (17) | 0.0297 (4) | |
| H8 | −0.1347 | 0.2596 | 0.9086 | 0.036* | |
| H9 | −0.0553 | 0.3674 | 1.0338 | 0.036* | |
| C7 | −0.06644 (19) | 0.20268 (16) | 0.60926 (16) | 0.0306 (4) | |
| H10 | −0.1254 | 0.2263 | 0.6681 | 0.037* | |
| C8 | −0.1309 (2) | 0.12438 (17) | 0.49383 (18) | 0.0377 (4) | |
| H11 | −0.2324 | 0.0945 | 0.4734 | 0.045* | |
| C9 | −0.0448 (2) | 0.09037 (18) | 0.40852 (18) | 0.0418 (5) | |
| H12 | −0.0866 | 0.0372 | 0.3282 | 0.050* | |
| C10 | 0.1021 (2) | 0.13424 (19) | 0.44087 (17) | 0.0400 (4) | |
| H13 | 0.1628 | 0.1117 | 0.3833 | 0.048* | |

| | | | | | |
|---|---|---|---|---|---|
| C11 | 0.1602 (2) | 0.21193 (17) | 0.55887 (16) | 0.0317 (4) | |
| C12 | 0.3203 (2) | 0.2616 (2) | 0.59846 (19) | 0.0488 (6) | |
| H14 | 0.3690 | 0.1939 | 0.5724 | 0.059* | |
| H15 | 0.3558 | 0.3211 | 0.5607 | 0.059* | |
| C13 | 0.1246 (2) | 0.53539 (18) | 0.67400 (16) | 0.0345 (4) | |
| H16 | 0.0528 | 0.4656 | 0.6217 | 0.041* | |
| C14 | 0.1440 (2) | 0.6415 (2) | 0.6504 (2) | 0.0446 (5) | |
| H17 | 0.0860 | 0.6451 | 0.5838 | 0.054* | |
| C15 | 0.2493 (2) | 0.7428 (2) | 0.7254 (2) | 0.0459 (5) | |
| H18 | 0.2664 | 0.8168 | 0.7102 | 0.055* | |
| C16 | 0.3291 (2) | 0.73469 (18) | 0.82255 (19) | 0.0375 (4) | |
| H19 | 0.4015 | 0.8034 | 0.8757 | 0.045* | |
| C17 | 0.30264 (17) | 0.62549 (15) | 0.84201 (15) | 0.0255 (3) | |
| C18 | 0.38677 (18) | 0.61428 (15) | 0.94787 (16) | 0.0282 (3) | |
| H20 | 0.4892 | 0.6238 | 0.9393 | 0.034* | |
| H21 | 0.3805 | 0.6801 | 1.0230 | 0.034* | |
| P1 | 0.59552 (5) | 0.02974 (4) | 0.74358 (4) | 0.03228 (11) | |
| F4 | 0.5841 (2) | 0.0626 (2) | 0.88218 (15) | 0.0891 (6) | |
| F5 | 0.61092 (18) | 0.00030 (18) | 0.60798 (13) | 0.0725 (5) | |
| F6 | 0.76632 (13) | 0.04945 (12) | 0.77770 (13) | 0.0494 (3) | |
| F7 | 0.61897 (16) | 0.17125 (12) | 0.76545 (17) | 0.0680 (5) | |
| F8 | 0.42616 (14) | 0.01136 (14) | 0.71068 (15) | 0.0597 (4) | |
| F9 | 0.57607 (18) | −0.10966 (14) | 0.7258 (2) | 0.0789 (5) | |
| F1A | 0.2162 (3) | 0.4114 (5) | 0.3777 (4) | 0.0633 (9) | 0.60 |
| F2A | 0.4293 (6) | 0.4201 (7) | 0.3364 (5) | 0.0959 (18) | 0.60 |
| F3A | 0.3715 (5) | 0.5861 (4) | 0.4479 (2) | 0.0956 (15) | 0.60 |
| F1B | 0.2326 (11) | 0.3584 (9) | 0.3289 (9) | 0.075 (3)* | 0.24 |
| F2B | 0.4556 (11) | 0.4729 (9) | 0.3644 (10) | 0.048 (2)* | 0.24 |
| F3B | 0.3095 (15) | 0.5496 (11) | 0.4460 (11) | 0.097 (4)* | 0.24 |
| F1C | 0.3378 (19) | 0.3456 (13) | 0.2889 (13) | 0.091 (4)* | 0.16 |
| F2C | 0.4376 (13) | 0.5252 (12) | 0.4082 (13) | 0.063 (3)* | 0.16 |
| F3C | 0.2240 (13) | 0.4706 (12) | 0.4110 (11) | 0.054 (3)* | 0.16 |
| O4 | 0.15453 (15) | 0.51056 (17) | 0.21720 (16) | 0.0494 (4) | |
| O5 | 0.38100 (14) | 0.53211 (14) | 0.18470 (12) | 0.0372 (3) | |
| C19 | 0.28069 (19) | 0.50937 (18) | 0.23870 (16) | 0.0320 (4) | |
| C20 | 0.3227 (3) | 0.4746 (3) | 0.3470 (2) | 0.0605 (7) | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.01984 (11) | 0.02370 (12) | 0.02007 (11) | 0.00408 (8) | 0.00167 (7) | 0.00581 (8) |
| O1 | 0.0279 (6) | 0.0369 (7) | 0.0343 (7) | 0.0153 (5) | 0.0108 (5) | 0.0175 (6) |
| O2 | 0.0225 (6) | 0.0426 (7) | 0.0292 (6) | 0.0059 (5) | 0.0058 (5) | 0.0058 (5) |
| O3 | 0.0354 (7) | 0.0307 (6) | 0.0223 (6) | −0.0020 (5) | −0.0054 (5) | 0.0102 (5) |
| N1 | 0.0229 (6) | 0.0228 (6) | 0.0273 (7) | 0.0037 (5) | −0.0003 (5) | 0.0064 (5) |
| N2 | 0.0275 (7) | 0.0248 (7) | 0.0227 (7) | 0.0042 (5) | 0.0015 (5) | 0.0055 (5) |
| N3 | 0.0244 (7) | 0.0279 (7) | 0.0217 (7) | 0.0032 (5) | 0.0020 (5) | 0.0085 (5) |
| C1 | 0.0264 (8) | 0.0289 (9) | 0.0426 (10) | 0.0080 (7) | 0.0000 (7) | 0.0099 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.0370 (10) | 0.0365 (10) | 0.0634 (14) | 0.0103 (8) | −0.0038 (9) | 0.0243 (10) |
| C3 | 0.0543 (13) | 0.0514 (13) | 0.0652 (15) | 0.0095 (10) | 0.0025 (11) | 0.0402 (12) |
| C4 | 0.0453 (12) | 0.0497 (12) | 0.0522 (13) | 0.0087 (9) | 0.0137 (10) | 0.0313 (11) |
| C5 | 0.0263 (8) | 0.0252 (8) | 0.0304 (8) | 0.0019 (6) | 0.0023 (6) | 0.0085 (7) |
| C6 | 0.0247 (8) | 0.0284 (8) | 0.0344 (9) | 0.0035 (6) | 0.0079 (7) | 0.0105 (7) |
| C7 | 0.0280 (8) | 0.0312 (9) | 0.0293 (9) | 0.0016 (7) | −0.0013 (7) | 0.0117 (7) |
| C8 | 0.0383 (10) | 0.0316 (9) | 0.0357 (10) | −0.0020 (7) | −0.0101 (8) | 0.0131 (8) |
| C9 | 0.0574 (13) | 0.0314 (9) | 0.0267 (9) | 0.0082 (9) | −0.0084 (8) | 0.0040 (8) |
| C10 | 0.0544 (12) | 0.0372 (10) | 0.0248 (9) | 0.0169 (9) | 0.0052 (8) | 0.0044 (8) |
| C11 | 0.0346 (9) | 0.0300 (9) | 0.0269 (8) | 0.0094 (7) | 0.0046 (7) | 0.0051 (7) |
| C12 | 0.0325 (10) | 0.0682 (15) | 0.0309 (10) | 0.0091 (9) | 0.0099 (8) | 0.0007 (10) |
| C13 | 0.0352 (9) | 0.0379 (10) | 0.0258 (8) | 0.0003 (7) | −0.0039 (7) | 0.0132 (7) |
| C14 | 0.0460 (11) | 0.0492 (12) | 0.0394 (11) | 0.0012 (9) | −0.0067 (9) | 0.0269 (10) |
| C15 | 0.0483 (12) | 0.0403 (11) | 0.0511 (13) | 0.0000 (9) | −0.0021 (10) | 0.0282 (10) |
| C16 | 0.0339 (9) | 0.0326 (9) | 0.0414 (10) | −0.0022 (7) | −0.0023 (8) | 0.0159 (8) |
| C17 | 0.0210 (7) | 0.0298 (8) | 0.0247 (8) | 0.0049 (6) | 0.0050 (6) | 0.0092 (7) |
| C18 | 0.0257 (8) | 0.0256 (8) | 0.0283 (8) | 0.0018 (6) | −0.0021 (6) | 0.0080 (7) |
| P1 | 0.0319 (2) | 0.0294 (2) | 0.0314 (2) | 0.00634 (18) | 0.00592 (18) | 0.00648 (19) |
| F4 | 0.0957 (13) | 0.1473 (19) | 0.0426 (9) | 0.0587 (13) | 0.0304 (9) | 0.0368 (11) |
| F5 | 0.0719 (10) | 0.1113 (14) | 0.0344 (7) | 0.0355 (10) | 0.0129 (7) | 0.0187 (8) |
| F6 | 0.0357 (6) | 0.0454 (7) | 0.0592 (8) | 0.0106 (5) | 0.0001 (5) | 0.0114 (6) |
| F7 | 0.0577 (9) | 0.0331 (7) | 0.1023 (13) | 0.0123 (6) | −0.0049 (8) | 0.0157 (8) |
| F8 | 0.0328 (7) | 0.0575 (8) | 0.0740 (10) | 0.0086 (6) | 0.0046 (6) | 0.0086 (7) |
| F9 | 0.0600 (9) | 0.0381 (7) | 0.1392 (17) | 0.0055 (7) | 0.0159 (10) | 0.0374 (9) |
| F1A | 0.0651 (18) | 0.089 (3) | 0.061 (2) | 0.0150 (19) | 0.0181 (15) | 0.059 (2) |
| F2A | 0.082 (3) | 0.167 (5) | 0.110 (4) | 0.085 (4) | 0.032 (3) | 0.105 (4) |
| F3A | 0.075 (2) | 0.159 (4) | 0.0224 (12) | −0.018 (3) | −0.0068 (13) | 0.0261 (17) |
| O4 | 0.0281 (7) | 0.0754 (11) | 0.0610 (10) | 0.0183 (7) | 0.0077 (6) | 0.0425 (9) |
| O5 | 0.0281 (6) | 0.0558 (8) | 0.0263 (6) | 0.0041 (6) | 0.0028 (5) | 0.0171 (6) |
| C19 | 0.0276 (8) | 0.0416 (10) | 0.0291 (9) | 0.0093 (7) | 0.0032 (7) | 0.0158 (8) |
| C20 | 0.0362 (11) | 0.114 (2) | 0.0521 (14) | 0.0214 (13) | 0.0112 (10) | 0.0533 (16) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Ni1—O1 | 2.0461 (12) | C10—C11 | 1.389 (3) |
| Ni1—N2 | 2.0601 (14) | C10—H13 | 0.9500 |
| Ni1—O2 | 2.0647 (12) | C11—C12 | 1.506 (3) |
| Ni1—N1 | 2.0662 (14) | C12—H14 | 0.9900 |
| Ni1—O3 | 2.0714 (12) | C12—H15 | 0.9900 |
| Ni1—N3 | 2.0769 (14) | C13—C14 | 1.374 (3) |
| O1—C6 | 1.421 (2) | C13—H16 | 0.9500 |
| O1—H1 | 0.8716 | C14—C15 | 1.381 (3) |
| O2—C12 | 1.417 (2) | C14—H17 | 0.9500 |
| O2—H2 | 0.9235 | C15—C16 | 1.377 (3) |
| O3—C18 | 1.422 (2) | C15—H18 | 0.9500 |
| O3—H3 | 0.9822 | C16—C17 | 1.384 (3) |
| N1—C5 | 1.340 (2) | C16—H19 | 0.9500 |
| N1—C1 | 1.352 (2) | C17—C18 | 1.501 (2) |
| N2—C11 | 1.342 (2) | C18—H20 | 0.9900 |

| | | | |
|---|---|---|---|
| N2—C7 | 1.346 (2) | C18—H21 | 0.9900 |
| N3—C17 | 1.340 (2) | P1—F5 | 1.5697 (15) |
| N3—C13 | 1.347 (2) | P1—F9 | 1.5777 (15) |
| C1—C2 | 1.377 (3) | P1—F7 | 1.5881 (14) |
| C1—H4 | 0.9500 | P1—F4 | 1.5915 (16) |
| C2—C3 | 1.376 (4) | P1—F8 | 1.5960 (14) |
| C2—H5 | 0.9500 | P1—F6 | 1.6087 (13) |
| C3—C4 | 1.385 (3) | F1A—C20 | 1.308 (4) |
| C3—H6 | 0.9500 | F2A—C20 | 1.316 (4) |
| C4—C5 | 1.387 (3) | F3A—C20 | 1.417 (5) |
| C4—H7 | 0.9500 | F1B—C20 | 1.420 (10) |
| C5—C6 | 1.503 (2) | F2B—C20 | 1.284 (11) |
| C6—H8 | 0.9900 | F3B—C20 | 1.255 (12) |
| C6—H9 | 0.9900 | F1C—C20 | 1.505 (14) |
| C7—C8 | 1.380 (3) | F2C—C20 | 1.188 (11) |
| C7—H10 | 0.9500 | F3C—C20 | 1.279 (12) |
| C8—C9 | 1.381 (3) | O4—C19 | 1.223 (2) |
| C8—H11 | 0.9500 | O5—C19 | 1.249 (2) |
| C9—C10 | 1.376 (3) | C19—C20 | 1.536 (3) |
| C9—H12 | 0.9500 | | |
| O1—Ni1—N2 | 98.09 (6) | N2—C11—C10 | 121.78 (18) |
| O1—Ni1—O2 | 172.33 (5) | N2—C11—C12 | 117.35 (16) |
| O1—Ni1—N1 | 78.11 (5) | C10—C11—C12 | 120.87 (18) |
| N2—Ni1—O2 | 78.53 (5) | O2—C12—C11 | 109.50 (16) |
| N2—Ni1—N1 | 97.35 (6) | O2—C12—H14 | 109.8 |
| O2—Ni1—N1 | 95.41 (6) | C11—C12—H14 | 109.8 |
| O1—Ni1—O3 | 95.00 (5) | O2—C12—H15 | 109.8 |
| N2—Ni1—O3 | 166.15 (6) | C11—C12—H15 | 109.8 |
| O2—Ni1—O3 | 89.01 (5) | H14—C12—H15 | 108.2 |
| N1—Ni1—O3 | 89.74 (5) | N3—C13—C14 | 122.91 (17) |
| O1—Ni1—N3 | 93.77 (5) | N3—C13—H16 | 118.5 |
| N2—Ni1—N3 | 96.49 (6) | C14—C13—H16 | 118.5 |
| O2—Ni1—N3 | 93.45 (6) | C13—C14—C15 | 118.77 (18) |
| N1—Ni1—N3 | 164.81 (6) | C13—C14—H17 | 120.6 |
| O3—Ni1—N3 | 78.09 (5) | C15—C14—H17 | 120.6 |
| C6—O1—Ni1 | 118.49 (10) | C16—C15—C14 | 118.86 (18) |
| C6—O1—H1 | 112.8 | C16—C15—H18 | 120.6 |
| Ni1—O1—H1 | 122.2 | C14—C15—H18 | 120.6 |
| C12—O2—Ni1 | 115.90 (11) | C15—C16—C17 | 119.37 (18) |
| C12—O2—H2 | 114.4 | C15—C16—H19 | 120.3 |
| Ni1—O2—H2 | 118.1 | C17—C16—H19 | 120.3 |
| C18—O3—Ni1 | 117.12 (10) | N3—C17—C16 | 122.05 (16) |
| C18—O3—H3 | 106.7 | N3—C17—C18 | 117.36 (15) |
| Ni1—O3—H3 | 130.7 | C16—C17—C18 | 120.58 (15) |
| C5—N1—C1 | 118.61 (16) | O3—C18—C17 | 110.11 (13) |
| C5—N1—Ni1 | 116.13 (11) | O3—C18—H20 | 109.6 |
| C1—N1—Ni1 | 124.57 (13) | C17—C18—H20 | 109.6 |
| C11—N2—C7 | 118.69 (15) | O3—C18—H21 | 109.6 |
| C11—N2—Ni1 | 115.61 (12) | C17—C18—H21 | 109.6 |

| | | | |
|---|---|---|---|
| C7—N2—Ni1 | 125.69 (12) | H20—C18—H21 | 108.2 |
| C17—N3—C13 | 118.01 (15) | F5—P1—F9 | 90.77 (11) |
| C17—N3—Ni1 | 116.35 (11) | F5—P1—F7 | 90.50 (11) |
| C13—N3—Ni1 | 125.64 (12) | F9—P1—F7 | 178.09 (10) |
| N1—C1—C2 | 122.16 (19) | F5—P1—F4 | 178.33 (12) |
| N1—C1—H4 | 118.9 | F9—P1—F4 | 90.26 (12) |
| C2—C1—H4 | 118.9 | F7—P1—F4 | 88.43 (12) |
| C3—C2—C1 | 119.20 (19) | F5—P1—F8 | 90.97 (9) |
| C3—C2—H5 | 120.4 | F9—P1—F8 | 91.58 (9) |
| C1—C2—H5 | 120.4 | F7—P1—F8 | 89.83 (8) |
| C2—C3—C4 | 119.1 (2) | F4—P1—F8 | 90.31 (10) |
| C2—C3—H6 | 120.5 | F5—P1—F6 | 89.37 (8) |
| C4—C3—H6 | 120.5 | F9—P1—F6 | 88.81 (8) |
| C3—C4—C5 | 119.1 (2) | F7—P1—F6 | 89.77 (7) |
| C3—C4—H7 | 120.5 | F4—P1—F6 | 89.35 (9) |
| C5—C4—H7 | 120.5 | F8—P1—F6 | 179.48 (9) |
| N1—C5—C4 | 121.87 (17) | O4—C19—O5 | 129.17 (18) |
| N1—C5—C6 | 117.09 (15) | O4—C19—C20 | 115.85 (17) |
| C4—C5—C6 | 121.03 (17) | O5—C19—C20 | 114.97 (16) |
| O1—C6—C5 | 108.67 (14) | F2C—C20—F3C | 111.5 (9) |
| O1—C6—H8 | 110.0 | F3B—C20—F2B | 102.8 (7) |
| C5—C6—H8 | 110.0 | F1A—C20—F2A | 109.8 (4) |
| O1—C6—H9 | 110.0 | F1A—C20—F3A | 104.4 (3) |
| C5—C6—H9 | 110.0 | F2A—C20—F3A | 106.6 (4) |
| H8—C6—H9 | 108.3 | F3B—C20—F1B | 108.1 (7) |
| N2—C7—C8 | 122.40 (18) | F2B—C20—F1B | 110.5 (6) |
| N2—C7—H10 | 118.8 | F2C—C20—F1C | 102.0 (9) |
| C8—C7—H10 | 118.8 | F3C—C20—F1C | 107.2 (8) |
| C7—C8—C9 | 118.65 (18) | F2C—C20—C19 | 118.3 (6) |
| C7—C8—H11 | 120.7 | F3B—C20—C19 | 113.8 (6) |
| C9—C8—H11 | 120.7 | F3C—C20—C19 | 113.1 (6) |
| C10—C9—C8 | 119.43 (18) | F2B—C20—C19 | 113.3 (5) |
| C10—C9—H12 | 120.3 | F1A—C20—C19 | 115.0 (2) |
| C8—C9—H12 | 120.3 | F2A—C20—C19 | 113.6 (3) |
| C9—C10—C11 | 119.03 (19) | F3A—C20—C19 | 106.5 (3) |
| C9—C10—H13 | 120.5 | F1B—C20—C19 | 108.2 (4) |
| C11—C10—H13 | 120.5 | F1C—C20—C19 | 103.1 (6) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O4$^{i}$ | 0.87 | 1.76 | 2.6003 (19) | 162.5 |
| O2—H2···O5$^{ii}$ | 0.92 | 1.77 | 2.6965 (18) | 175.8 |
| O3—H3···O5$^{iii}$ | 0.98 | 1.65 | 2.6267 (18) | 173.8 |

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

**Fig. 1**

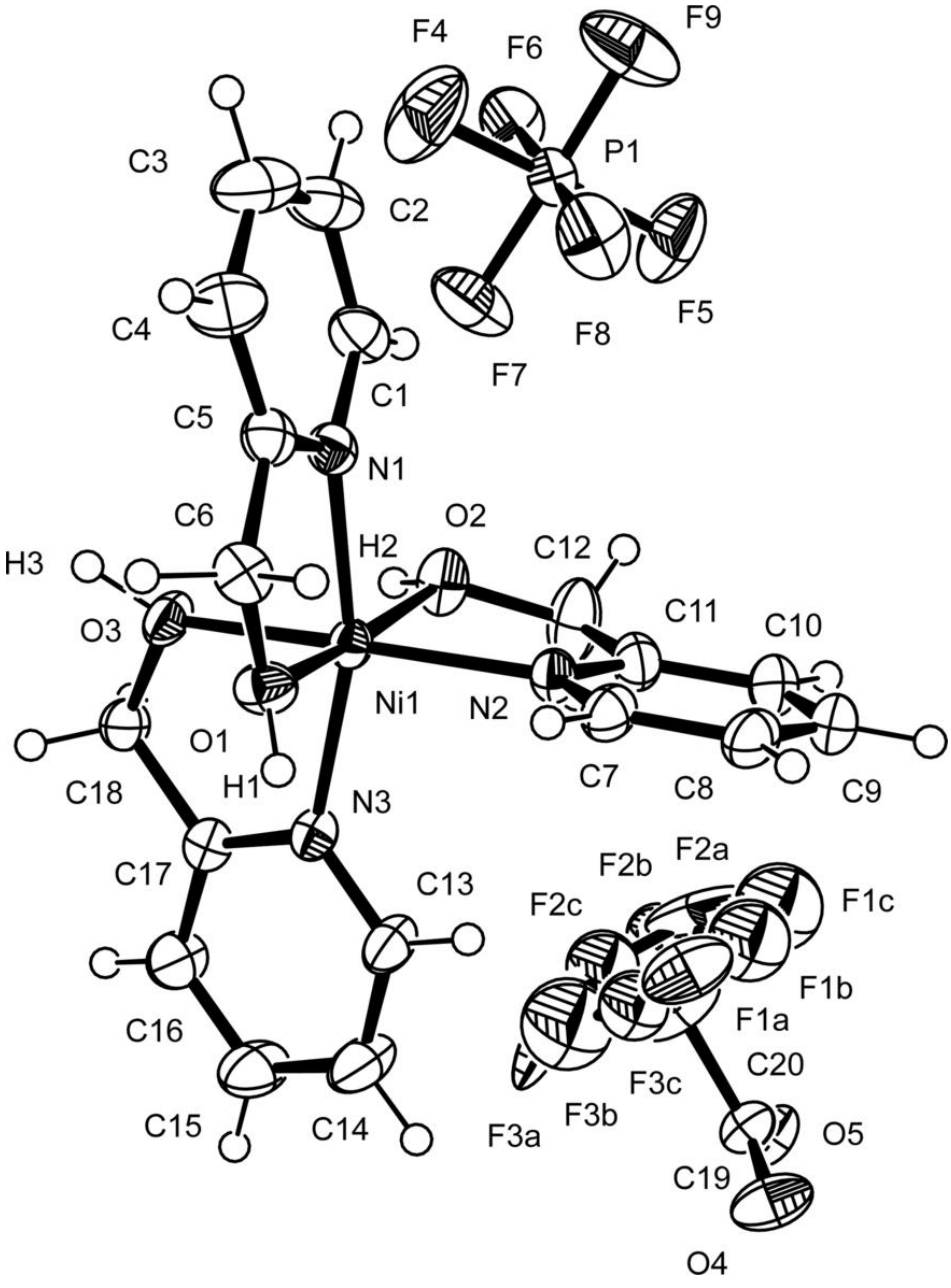
F4
F6
F9
P1
C3
C2
F5
C4
F8
F7
C1
C5
N1
C6
O2
H2
C12
H3
C11
C10
O3
Ni1
N2
O1
C18
H1
C7
C8
C9
N3
F2a
F2b
C17
C13
F2c
F1c
F1b
C16
F1a
C20
F3c
F3b
C15
C14
F3a
O5
C19
O4

**Fig. 2**

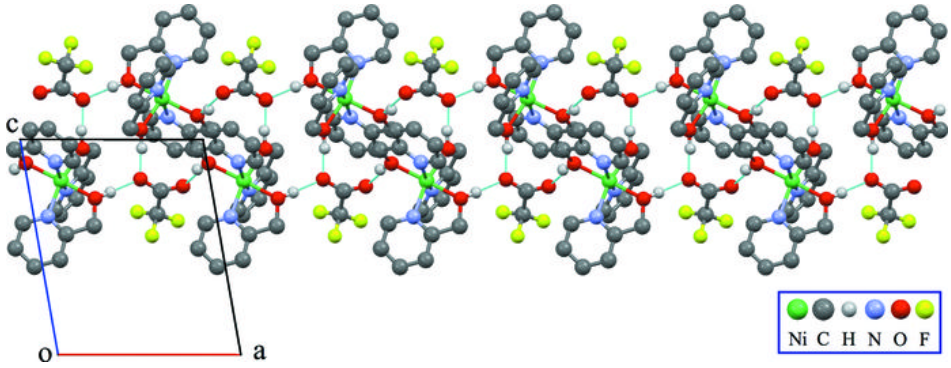

**Fig. 3**