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Crystal structure of an unknown solvate of bis(tetra-*n*-butylammonium) [*N*,*N*′-(4-trifluoromethyl-1,2-phenylene)bis(oxamato)-κ^4O,N,N',O']nickelate(II)

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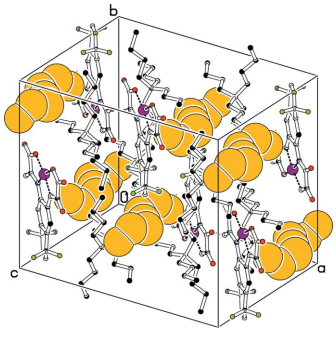
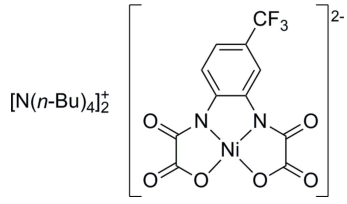
In the title compound, $[N(C_4H_9)_4]_2[Ni(C_{11}H_3F_3N_2O_6)]$ or $[N(n\text{-Bu})_4]_2$-[Ni(topbo)] [*n*-Bu = *n*-butyl and topbo = 4-trifluoromethyl-1,2-phenylenebis(oxamate)], the Ni^{2+} cation is coordinated by two deprotonated amido N atoms and two carboxylate O atoms, setting up a slightly distorted square-planar coordination environment. The $[Ni(topbo)]^{2-}$ anion lies on a twofold rotation axis. Due to an incompatibility with the point-group symmetry of the complete molecule, orientational disorder of the CF_3 group is observed. The tetrahedral ammonium cations and the anion are linked by weak intermolecular C—H···O and C—H···F hydrogen-bonding interactions into a three-dimensional network. A region of electron density was treated with the SQUEEZE procedure in *PLATON* [Spek (2015). *Acta Cryst*. C**71**, 9–18] following unsuccessful attempts to model it as plausible solvent molecule(s). The given chemical formula and other crystal data do not take into account the unknown solvent molecule.

1. Chemical context

Oxamate-bridged polymetallic complexes are of interest in the discipline of supramolecular magnetism as they exhibit diverse supramolecular architectures and magnetic properties (Pardo *et al.*, 2008; Kahn, 1987, 2000) and have been synthesized by, for example, Ruiz *et al.* (1997*a*,*b*), Berg *et al.* (2002), Martín *et al.* (2002) and Ottenwaelder *et al.* (2005). Over the last decade, we have been interested in the synthesis of bis(oxamates) and bis(oxamate) complexes (Rüffer *et al.*, 2007*a*,*b*, 2008, 2009; Eya'ane Meva *et al.*, 2012), as well as their deposition as thin films (Bräuer *et al.*, 2006, 2008, 2009). In order to optimize the deposition conditions and to increase the thin-film quality, the monometallic title compound, bis(tetra-*n*-butylammonium) [*N*,*N*′-(4-trifluoromethyl-1,2-phenylene)bis(oxamato)-κ^4O,N,N',O']nickelate(II), (I), was prepared. The complex includes four sites of coordination and a CF_3 group which provides a good solubility in organic solvents.





2. Structural commentary

The asymmetric unit of compound (I) contains one $[N(n\text{-Bu})_4]^+$ cation and half of the complex anion $[Ni(topbo)]^{2-}$

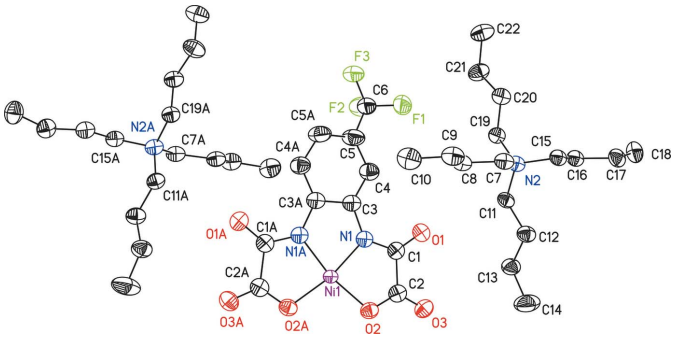


Figure 1
The molecular components of (I) drawn with displacement ellipsoids at the 50% probability level. H atoms were omitted for clarity. Only one disordered part of the –CF_3 group is shown. [Symmetry code: (A) $-x+2$, y, $-z+\frac{3}{2}$.]

(Fig. 1). The anion possesses point-group symmetry 2. This imposes orientational disorder of the CF_3 group, which lies on both sides of the twofold rotation axis with 0.5 occupancy. The anion is essentially planar (root-mean-square deviation 0.145 Å), the highest deviation from planarity being observed for C6 [0.440 (5) Å]. The Ni^{2+} cation is coordinated by two deprotonated amido N atoms and two carboxylate O atoms, resulting in a slightly distorted square-planar coordination geometry. In agreement with related nickel compounds, the Ni—N bonds are significantly shorter than the Ni—O bonds, which is due to the stronger donicity of the amido nitrogens (Fettouhi *et al.*, 1996; Rüffer *et al.*, 2007*a*,*b*, 2008; Abdulmalic *et al.*, 2013; Milek *et al.*, 2013). Compared to the respective nickel complex without the CF_3 group (Abdulmalic *et al.*, 2013), compound (I) exhibits longer Ni—N and Ni—O bonds. It is instructive to note that for other complexes, the presence of electron-withdrawing substituents at the benzene moiety, *e.g.* Cl, NO_2, causes a shortening of the Ni—N and Ni—O bonds (Fettouhi *et al.*, 1996; Rüffer *et al.*, 2008).

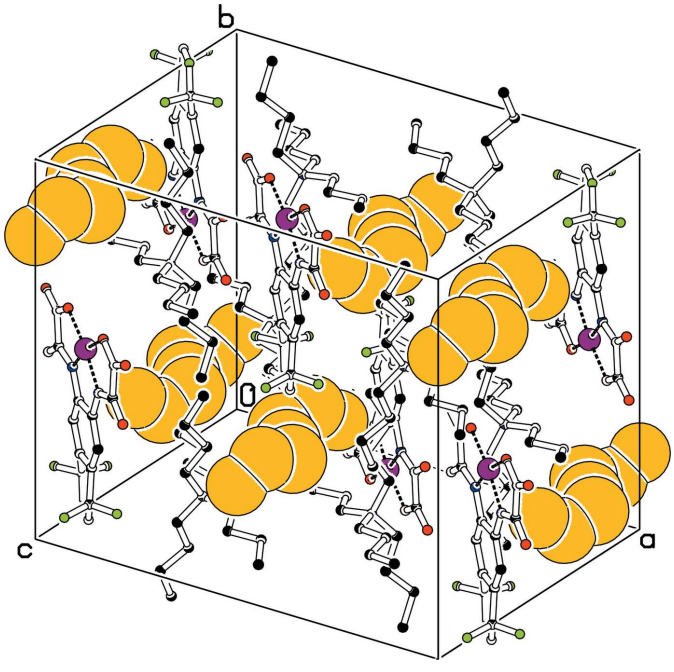


Figure 2
Packing diagram of compound (I), with voids in the structure represented by yellow spheres [drawn using the CAVITYPLOT routine in *PLATON* (Spek, 2009)]. H atoms are omitted for clarity. Color code: black (C), blue (N), red (O), green (F), purple (Ni).

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O1	0.97	2.42	3.347 (2)	160
C11—H11B···O1^{i}	0.97	2.40	3.368 (2)	172
C15—H15A···O2^{ii}	0.97	2.56	3.529 (2)	174
C17—H17A···O2^{iii}	0.97	2.41	3.333 (3)	159
C19—H19A···O3^{i}	0.97	2.55	3.441 (2)	152
C21—H21B···F1	0.97	2.29	3.208 (4)	156

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

3. Supramolecular features

Five weak C—H···O and one weak C—H···F hydrogen bonds (Steiner, 2002) are observed in the crystal structure of (I) (Table 1), which connect the $[N(n\text{-Bu})_4]^+$ cations and the $[Ni(topbo)]^{2-}$ anion, forming a three-dimensional network. A packing diagram is shown in Fig. 2.

4. Synthesis and crystallization

4-Trifluoromethyl-1,2-phenylenebis(ethyl oxamate) was prepared from ethyl oxalyl chloride and 4-trifluoromethyl-1,2-phenylenediamine in analogy to Cervera *et al.* (1998). To a solution of 4-trifluoromethyl-1,2-phenylenediamine (0.4 g, 2.22 mmol) dissolved in tetrahydrofuran (50 ml) was added dropwise *via* a dropping funnel a solution of ethyl oxalyl chloride (5.05 g, 4.45 mmol) in tetrahydrofuran (25 ml) within 20 min. The resulting mixture was refluxed for 30 min at 343 K, filtrated and concentrated to about one third on a rotary evaporator. The careful addition of water resulted in the precipitation of a brown solid which was filtered off and dried in air.

To a solution of 4-trifluoromethyl-1,2-phenylenebis(ethyl oxamate) (0.4 g, 1.06 mmol) in ethanol (40 ml) was added dropwise under stirring $[N(n\text{-Bu})_4]OH$ (2.76 g, 4.25 mmol, 40 wt-% aqueous solution) in water (20 ml); the resulting mixture was refluxed for 30 min. After cooling to room temperature, an aqueous solution (20 ml) of $NiCl_2{\cdot}6H_2O$ (0.25 g, 1.05 mmol) was added dropwise under stirring. The yellow solution was filtered, concentrated to a volume of 20 ml on a rotatory evaporator, and extracted with dichloromethane (100 ml). The organic layer was separated, washed with water (3 x 25 ml) dried over Na_2SO_4 and concentrated to a volume of 10 ml. The title compound was precipitated by adding Et_2O (100 ml). The yellow solid was filtered off, washed with Et_2O and dried in air. Single crystals were obtained by the slow diffusion of Et_2O into a saturated solution of the title compound in CH_2Cl_2/thf (1:1).

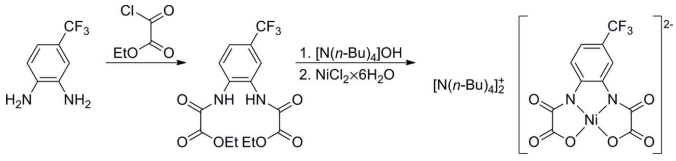


Figure 3
Scheme representing the synthesis of compound (I).

The overall synthetic procedure is schematically shown in Fig. 3.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bonded H atoms were placed in calculated positions and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) and a C—H distance of 0.93 Å for aromatic and 0.97 Å for methylene protons as well as U_{iso}(H) = 1.5U_{eq}(C) and a C—H distance of 0.96 Å for methyl protons.

A small region of electron density at a distance of 1.6–3.7 Å from the trifluoromethyl group indicates the presence of a disordered solvent molecule. All attempts to model a disordered tetrahydrofuran, dichloromethane or diethyl ether molecule (solvents used for crystallization) failed. Therefore, the solvent contributions have been removed using the SQUEEZE procedure in *PLATON* (Spek, 2015). SQUEEZE calculated a void volume of approximately 310 Å^3 occupied by 24 electrons per unit cell. Fig. 2 shows the positions of the voids within the unit cell.

Table 2
Experimental details.

Crystal data	
Chemical formula	$(C_{16}H_{36}N)_2[Ni(C_{11}H_3F_3N_2O_6)]$
M_r	859.78
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	110
a, *b*, *c* (Å)	19.5285 (3), 17.3370 (3), 14.1484 (3)
β (°)	92.136 (2)
V (Å^3)	4786.83 (15)
Z	4
Radiation type	Cu *K*α
μ (mm^{-1})	1.06
Crystal size (mm)	0.10 × 0.08 × 0.06
Data collection	
Diffractometer	Oxford Gemini S
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min}, T_{max}	0.807, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15600, 3545, 3142
R_{int}	0.023
θ_{max} (°)	60.5
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.564
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.036, 0.102, 1.09
No. of reflections	3545
No. of parameters	277
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.20

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2006), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2013* (Sheldrick, 2015*b*), *SHELXTL* (Sheldrick, 2008), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), *PLATON* (Spek, 2009), *publCIF* (Westrip, 2010) and *SQUEEZE* (Spek, 2015).

Acknowledgements

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supporting information

Acta Cryst. (2015). E**71**, 578-581 [doi:10.1107/S205698901500835X]

Crystal structure of an unknown solvate of bis(tetra-*n*-butylammonium) [*N*,*N*′-(4-trifluoromethyl-1,2-phenylene)bis(oxamato)-κ^4*O*,*N*,*N*′,*O*′]nickelate(II)

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Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015*b*); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012), *publCIF* (Westrip, 2010) and *SQUEEZE* (Spek, 2015).

Bis(tetra-*n*-butylammonium) [*N*,*N*′-(4-trifluoromethyl-1,2-phenylene)bis(oxamato)-κ^4*O*,*N*,*N*′,*O*′]nickelate(II)

Crystal data

$(C_{16}H_{36}N)_2[Ni(C_{11}H_3F_3N_2O_6)]$
M_r = 859.78
Monoclinic, *C*2/*c*
a = 19.5285 (3) Å
b = 17.3370 (3) Å
c = 14.1484 (3) Å
β = 92.136 (2)°
V = 4786.83 (15) Å^3
Z = 4
F(000) = 1856
D_x = 1.193 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 5954 reflections
θ = 4.5–60.4°
μ = 1.06 mm^{-1}
T = 110 K
Block, orange
0.1 × 0.08 × 0.06 mm

Data collection

Oxford Gemini S diffractometer
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.807, T_{max} = 1.000
15600 measured reflections
3545 independent reflections
3142 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 60.5°, θ_{min} = 3.4°
h = −21→21
k = −19→19
l = −15→15
2 standard reflections every 25 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 1.09
3545 reflections
277 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 2.8029P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.88594 (10)	0.35368 (11)	0.65544 (14)	0.0356 (5)	
C2	0.88021 (10)	0.44362 (12)	0.65187 (14)	0.0370 (5)	
C3	0.96883 (10)	0.25942 (11)	0.72161 (15)	0.0371 (4)	
C4	0.94040 (11)	0.18979 (12)	0.69174 (16)	0.0454 (5)	
H4	0.9011	0.1893	0.6525	0.055*	
C5	0.97086 (14)	0.12088 (13)	0.72067 (18)	0.0566 (6)	
H5	0.9519	0.0743	0.7004	0.068*	0.5
C7	0.68930 (10)	0.16654 (11)	0.76809 (14)	0.0363 (4)	
H7A	0.6668	0.2105	0.7958	0.044*	
H7B	0.6729	0.1207	0.7993	0.044*	
C8	0.76537 (10)	0.17349 (11)	0.79037 (14)	0.0386 (5)	
H8A	0.7821	0.2220	0.7661	0.046*	
H8B	0.7895	0.1319	0.7600	0.046*	
C9	0.77901 (12)	0.16984 (13)	0.89696 (15)	0.0466 (5)	
H9A	0.7528	0.2100	0.9267	0.056*	
H9B	0.7631	0.1206	0.9201	0.056*	
C10	0.85411 (13)	0.17956 (14)	0.92587 (17)	0.0567 (6)	
H10A	0.8596	0.1768	0.9935	0.085*	
H10B	0.8699	0.2288	0.9045	0.085*	
H10C	0.8803	0.1393	0.8979	0.085*	
C11	0.68357 (10)	0.23650 (11)	0.61193 (14)	0.0356 (4)	
H11A	0.7321	0.2469	0.6224	0.043*	
H11B	0.6756	0.2277	0.5447	0.043*	
C12	0.64405 (11)	0.30812 (11)	0.63902 (16)	0.0412 (5)	
H12A	0.6507	0.3180	0.7062	0.049*	
H12B	0.5955	0.3006	0.6252	0.049*	
C13	0.67014 (11)	0.37655 (12)	0.58245 (17)	0.0466 (5)	
H13A	0.7196	0.3798	0.5912	0.056*	
H13B	0.6596	0.3679	0.5157	0.056*	
C14	0.63858 (16)	0.45253 (15)	0.6120 (2)	0.0789 (9)	
H14A	0.6558	0.4935	0.5739	0.118*	
H14B	0.6503	0.4623	0.6774	0.118*	
H14C	0.5897	0.4498	0.6032	0.118*	
C15	0.59008 (10)	0.14707 (11)	0.66235 (14)	0.0358 (4)	
H15A	0.5822	0.1001	0.6975	0.043*	
H15B	0.5684	0.1889	0.6956	0.043*	
C16	0.55475 (10)	0.13920 (11)	0.56594 (14)	0.0378 (5)	
H16A	0.5698	0.0922	0.5358	0.045*	
H16B	0.5668	0.1825	0.5265	0.045*	

C17	0.47760 (10)	0.13688 (13)	0.57613 (16)	0.0445 (5)	
H17A	0.4669	0.0999	0.6248	0.053*	
H17B	0.4622	0.1872	0.5966	0.053*	
C18	0.43874 (11)	0.11520 (15)	0.48520 (17)	0.0532 (6)	
H18A	0.3905	0.1147	0.4958	0.080*	
H18B	0.4529	0.0649	0.4653	0.080*	
H18C	0.4483	0.1522	0.4370	0.080*	
C19	0.70443 (10)	0.09850 (10)	0.61375 (14)	0.0348 (4)	
H19A	0.6871	0.0961	0.5487	0.042*	
H19B	0.7526	0.1121	0.6128	0.042*	
C20	0.69831 (11)	0.01936 (11)	0.65738 (15)	0.0390 (5)	
H20A	0.6509	0.0027	0.6530	0.047*	
H20B	0.7124	0.0217	0.7238	0.047*	
C21	0.74277 (12)	−0.03850 (12)	0.60717 (17)	0.0497 (6)	
H21A	0.7284	−0.0410	0.5409	0.060*	
H21B	0.7901	−0.0214	0.6111	0.060*	
C22	0.73766 (14)	−0.11826 (13)	0.65073 (17)	0.0544 (6)	
H22A	0.7661	−0.1535	0.6176	0.082*	
H22B	0.6909	−0.1356	0.6461	0.082*	
H22C	0.7527	−0.1161	0.7161	0.082*	
N1	0.94347 (8)	0.33319 (9)	0.70205 (12)	0.0356 (4)	
N2	0.66664 (8)	0.16218 (9)	0.66403 (11)	0.0338 (4)	
O1	0.84134 (7)	0.31211 (8)	0.61726 (10)	0.0410 (3)	
O2	0.92921 (7)	0.48170 (7)	0.69564 (10)	0.0391 (3)	
O3	0.83119 (7)	0.47330 (8)	0.60942 (10)	0.0445 (4)	
C6	0.9520 (2)	0.0478 (2)	0.6727 (4)	0.0499 (11)	0.5
F1	0.88379 (14)	0.04371 (15)	0.6729 (3)	0.0760 (9)	0.5
F2	0.96828 (17)	0.03869 (15)	0.5806 (2)	0.0698 (8)	0.5
F3	0.97597 (13)	−0.01539 (13)	0.7165 (2)	0.0581 (7)	0.5
Ni1	1.0000	0.41486 (2)	0.7500	0.02432 (15)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0395 (11)	0.0420 (11)	0.0257 (11)	0.0005 (9)	0.0080 (8)	0.0002 (8)
C2	0.0439 (11)	0.0423 (11)	0.0253 (11)	0.0022 (9)	0.0080 (9)	0.0001 (9)
C3	0.0452 (10)	0.0352 (10)	0.0312 (11)	0.0002 (8)	0.0084 (8)	0.0005 (8)
C4	0.0555 (13)	0.0409 (12)	0.0397 (13)	−0.0012 (9)	−0.0016 (10)	−0.0032 (9)
C5	0.0773 (16)	0.0352 (11)	0.0563 (15)	−0.0023 (11)	−0.0124 (12)	−0.0044 (11)
C7	0.0501 (11)	0.0329 (10)	0.0264 (11)	−0.0015 (8)	0.0098 (9)	−0.0003 (8)
C8	0.0516 (12)	0.0328 (10)	0.0318 (12)	−0.0029 (8)	0.0068 (9)	−0.0016 (8)
C9	0.0641 (14)	0.0428 (12)	0.0328 (13)	−0.0020 (10)	0.0028 (10)	0.0022 (9)
C10	0.0729 (16)	0.0570 (14)	0.0396 (14)	−0.0056 (12)	−0.0062 (11)	0.0000 (11)
C11	0.0407 (10)	0.0342 (10)	0.0322 (12)	−0.0057 (8)	0.0073 (8)	0.0031 (8)
C12	0.0496 (12)	0.0362 (11)	0.0384 (13)	−0.0024 (9)	0.0106 (9)	0.0017 (9)
C13	0.0521 (12)	0.0381 (11)	0.0505 (14)	−0.0008 (9)	0.0113 (10)	0.0080 (10)
C14	0.097 (2)	0.0411 (14)	0.101 (3)	0.0092 (14)	0.0333 (18)	0.0174 (14)
C15	0.0406 (11)	0.0334 (10)	0.0342 (12)	−0.0024 (8)	0.0120 (8)	−0.0008 (8)

C16	0.0434 (11)	0.0360 (10)	0.0348 (12)	−0.0025 (8)	0.0108 (9)	−0.0011 (8)
C17	0.0429 (11)	0.0479 (12)	0.0435 (13)	−0.0054 (9)	0.0114 (9)	0.0027 (10)
C18	0.0421 (12)	0.0666 (15)	0.0510 (15)	−0.0062 (10)	0.0040 (10)	0.0046 (11)
C19	0.0404 (10)	0.0342 (10)	0.0303 (11)	−0.0004 (8)	0.0087 (8)	−0.0038 (8)
C20	0.0500 (12)	0.0366 (10)	0.0309 (12)	−0.0019 (9)	0.0079 (9)	−0.0030 (8)
C21	0.0631 (14)	0.0442 (12)	0.0427 (14)	0.0123 (10)	0.0124 (11)	0.0008 (10)
C22	0.0783 (16)	0.0412 (12)	0.0437 (14)	0.0138 (11)	0.0028 (12)	−0.0010 (10)
N1	0.0391 (9)	0.0356 (9)	0.0324 (10)	−0.0001 (7)	0.0047 (7)	−0.0002 (7)
N2	0.0427 (9)	0.0326 (8)	0.0266 (9)	−0.0030 (7)	0.0096 (7)	−0.0009 (6)
O1	0.0432 (8)	0.0456 (8)	0.0344 (8)	−0.0040 (6)	0.0030 (6)	−0.0010 (6)
O2	0.0435 (7)	0.0365 (7)	0.0376 (8)	0.0027 (6)	0.0048 (6)	−0.0005 (6)
O3	0.0485 (8)	0.0476 (8)	0.0373 (9)	0.0078 (7)	0.0012 (7)	0.0029 (6)
C6	0.050 (3)	0.037 (2)	0.062 (3)	0.0017 (19)	0.004 (2)	−0.004 (2)
F1	0.0477 (16)	0.0465 (15)	0.134 (3)	−0.0011 (12)	0.0005 (16)	−0.0231 (17)
F2	0.100 (2)	0.0521 (16)	0.0572 (19)	0.0026 (15)	−0.0005 (16)	−0.0133 (14)
F3	0.0630 (17)	0.0351 (13)	0.076 (2)	0.0013 (11)	0.0042 (12)	0.0005 (12)
Ni1	0.0293 (2)	0.0226 (2)	0.0215 (3)	0.000	0.00539 (16)	0.000

Geometric parameters (Å, º)

C1—O1	1.239 (2)	C14—H14B	0.9600
C1—N1	1.330 (3)	C14—H14C	0.9600
C1—C2	1.564 (3)	C15—C16	1.512 (3)
C2—O3	1.224 (2)	C15—N2	1.517 (2)
C2—O2	1.300 (2)	C15—H15A	0.9700
C3—C4	1.388 (3)	C15—H15B	0.9700
C3—N1	1.396 (2)	C16—C17	1.519 (3)
C3—C3^{i}	1.433 (4)	C16—H16A	0.9700
C4—C5	1.389 (3)	C16—H16B	0.9700
C4—H4	0.9300	C17—C18	1.516 (3)
C5—C5^{i}	1.383 (5)	C17—H17A	0.9700
C5—H5	0.9300	C17—H17B	0.9700
C7—C8	1.512 (3)	C18—H18A	0.9600
C7—N2	1.523 (2)	C18—H18B	0.9600
C7—H7A	0.9700	C18—H18C	0.9600
C7—H7B	0.9700	C19—C20	1.511 (3)
C8—C9	1.523 (3)	C19—N2	1.519 (2)
C8—H8A	0.9700	C19—H19A	0.9700
C8—H8B	0.9700	C19—H19B	0.9700
C9—C10	1.517 (3)	C20—C21	1.520 (3)
C9—H9A	0.9700	C20—H20A	0.9700
C9—H9B	0.9700	C20—H20B	0.9700
C10—H10A	0.9600	C21—C22	1.519 (3)
C10—H10B	0.9600	C21—H21A	0.9700
C10—H10C	0.9600	C21—H21B	0.9700
C11—C12	1.519 (3)	C22—H22A	0.9600
C11—N2	1.527 (2)	C22—H22B	0.9600
C11—H11A	0.9700	C22—H22C	0.9600

C11—H11B	0.9700	N1—Ni1	1.9047 (16)
C12—C13	1.529 (3)	O2—Ni1	1.9407 (13)
C12—H12A	0.9700	C6—F1	1.333 (5)
C12—H12B	0.9700	C6—F3	1.335 (5)
C13—C14	1.519 (3)	C6—F2	1.361 (6)
C13—H13A	0.9700	F3—F3^{i}	1.308 (5)
C13—H13B	0.9700	Ni1—N1^{i}	1.9047 (16)
C14—H14A	0.9600	Ni1—O2^{i}	1.9408 (13)

O1—C1—N1	128.92 (18)	C16—C15—H15B	108.2
O1—C1—C2	121.17 (17)	N2—C15—H15B	108.2
N1—C1—C2	109.91 (16)	H15A—C15—H15B	107.3
O3—C2—O2	124.63 (18)	C15—C16—C17	109.73 (16)
O3—C2—C1	119.25 (18)	C15—C16—H16A	109.7
O2—C2—C1	116.12 (16)	C17—C16—H16A	109.7
C4—C3—N1	127.00 (19)	C15—C16—H16B	109.7
C4—C3—C3^{i}	119.50 (12)	C17—C16—H16B	109.7
N1—C3—C3^{i}	113.51 (11)	H16A—C16—H16B	108.2
C3—C4—C5	119.7 (2)	C18—C17—C16	113.12 (18)
C3—C4—H4	120.1	C18—C17—H17A	109.0
C5—C4—H4	120.1	C16—C17—H17A	109.0
C5^{i}—C5—C4	120.69 (13)	C18—C17—H17B	109.0
C5^{i}—C5—H5	119.7	C16—C17—H17B	109.0
C4—C5—H5	119.7	H17A—C17—H17B	107.8
C8—C7—N2	116.96 (15)	C17—C18—H18A	109.5
C8—C7—H7A	108.1	C17—C18—H18B	109.5
N2—C7—H7A	108.1	H18A—C18—H18B	109.5
C8—C7—H7B	108.1	C17—C18—H18C	109.5
N2—C7—H7B	108.1	H18A—C18—H18C	109.5
H7A—C7—H7B	107.3	H18B—C18—H18C	109.5
C7—C8—C9	109.72 (16)	C20—C19—N2	114.92 (16)
C7—C8—H8A	109.7	C20—C19—H19A	108.5
C9—C8—H8A	109.7	N2—C19—H19A	108.5
C7—C8—H8B	109.7	C20—C19—H19B	108.5
C9—C8—H8B	109.7	N2—C19—H19B	108.5
H8A—C8—H8B	108.2	H19A—C19—H19B	107.5
C10—C9—C8	113.21 (18)	C19—C20—C21	110.68 (17)
C10—C9—H9A	108.9	C19—C20—H20A	109.5
C8—C9—H9A	108.9	C21—C20—H20A	109.5
C10—C9—H9B	108.9	C19—C20—H20B	109.5
C8—C9—H9B	108.9	C21—C20—H20B	109.5
H9A—C9—H9B	107.8	H20A—C20—H20B	108.1
C9—C10—H10A	109.5	C22—C21—C20	111.33 (18)
C9—C10—H10B	109.5	C22—C21—H21A	109.4
H10A—C10—H10B	109.5	C20—C21—H21A	109.4
C9—C10—H10C	109.5	C22—C21—H21B	109.4
H10A—C10—H10C	109.5	C20—C21—H21B	109.4
H10B—C10—H10C	109.5	H21A—C21—H21B	108.0

C12—C11—N2	116.55 (16)	C21—C22—H22A	109.5
C12—C11—H11A	108.2	C21—C22—H22B	109.5
N2—C11—H11A	108.2	H22A—C22—H22B	109.5
C12—C11—H11B	108.2	C21—C22—H22C	109.5
N2—C11—H11B	108.2	H22A—C22—H22C	109.5
H11A—C11—H11B	107.3	H22B—C22—H22C	109.5
C11—C12—C13	108.66 (17)	C1—N1—C3	129.06 (17)
C11—C12—H12A	110.0	C1—N1—Ni1	116.48 (13)
C13—C12—H12A	110.0	C3—N1—Ni1	114.46 (13)
C11—C12—H12B	110.0	C15—N2—C19	111.27 (14)
C13—C12—H12B	110.0	C15—N2—C7	105.92 (14)
H12A—C12—H12B	108.3	C19—N2—C7	111.10 (14)
C14—C13—C12	112.47 (19)	C15—N2—C11	111.66 (14)
C14—C13—H13A	109.1	C19—N2—C11	105.63 (14)
C12—C13—H13A	109.1	C7—N2—C11	111.37 (14)
C14—C13—H13B	109.1	C2—O2—Ni1	112.72 (12)
C12—C13—H13B	109.1	F1—C6—F3	106.8 (4)
H13A—C13—H13B	107.8	F1—C6—F2	105.4 (4)
C13—C14—H14A	109.5	F3—C6—F2	105.0 (4)
C13—C14—H14B	109.5	F3^{i}—F3—C6	124.5 (2)
H14A—C14—H14B	109.5	N1^{i}—Ni1—N1	83.96 (9)
C13—C14—H14C	109.5	N1^{i}—Ni1—O2	168.49 (6)
H14A—C14—H14C	109.5	N1—Ni1—O2	84.71 (6)
H14B—C14—H14C	109.5	N1^{i}—Ni1—O2^{i}	84.72 (6)
C16—C15—N2	116.49 (15)	N1—Ni1—O2^{i}	168.49 (6)
C16—C15—H15A	108.2	O2—Ni1—O2^{i}	106.67 (8)
N2—C15—H15A	108.2		

O1—C1—C2—O3	−1.0 (3)	C3^{i}—C3—N1—C1	−176.8 (2)
N1—C1—C2—O3	178.08 (18)	C4—C3—N1—Ni1	−176.64 (18)
O1—C1—C2—O2	178.84 (17)	C3^{i}—C3—N1—Ni1	3.0 (3)
N1—C1—C2—O2	−2.0 (2)	C16—C15—N2—C19	−58.4 (2)
N1—C3—C4—C5	−177.5 (2)	C16—C15—N2—C7	−179.28 (16)
C3^{i}—C3—C4—C5	2.9 (4)	C16—C15—N2—C11	59.3 (2)
C3—C4—C5—C5^{i}	0.3 (5)	C20—C19—N2—C15	−62.1 (2)
N2—C7—C8—C9	−174.95 (15)	C20—C19—N2—C7	55.7 (2)
C7—C8—C9—C10	−177.74 (18)	C20—C19—N2—C11	176.60 (16)
N2—C11—C12—C13	177.74 (17)	C8—C7—N2—C15	174.15 (16)
C11—C12—C13—C14	−174.2 (2)	C8—C7—N2—C19	53.2 (2)
N2—C15—C16—C17	−171.21 (16)	C8—C7—N2—C11	−64.3 (2)
C15—C16—C17—C18	−169.98 (18)	C12—C11—N2—C15	50.4 (2)
N2—C19—C20—C21	−175.07 (17)	C12—C11—N2—C19	171.52 (17)
C19—C20—C21—C22	179.53 (19)	C12—C11—N2—C7	−67.8 (2)
O1—C1—N1—C3	−0.8 (3)	O3—C2—O2—Ni1	−177.48 (16)
C2—C1—N1—C3	−179.87 (18)	C1—C2—O2—Ni1	2.7 (2)
O1—C1—N1—Ni1	179.39 (16)	F1—C6—F3—F3^{i}	−131.6 (5)

C2—C1—N1—Ni1	0.4 (2)	F2—C6—F3—F3^{i}	116.8 (5)
C4—C3—N1—C1	3.6 (3)		

Symmetry code: (i) −*x*+2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···O1	0.97	2.42	3.347 (2)	160
C11—H11*B*···O1^{ii}	0.97	2.40	3.368 (2)	172
C15—H15*A*···O2^{iii}	0.97	2.56	3.529 (2)	174
C17—H17*A*···O2^{iv}	0.97	2.41	3.333 (3)	159
C19—H19*A*···O3^{ii}	0.97	2.55	3.441 (2)	152
C21—H21*B*···F1	0.97	2.29	3.208 (4)	156

Symmetry codes: (ii) −*x*+3/2, −*y*+1/2, −*z*+1; (iii) −*x*+3/2, *y*−1/2, −*z*+3/2; (iv) *x*−1/2, *y*−1/2, *z*.