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4,4′-(Propane-1,3-diyl)dipyridinium tetrachloridonickelate(II)

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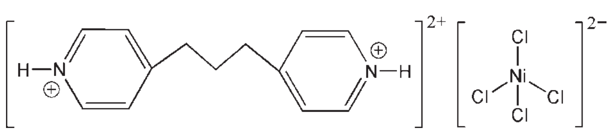
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.045; data-to-parameter ratio = 17.2.

The title compound, $(C_{13}H_{16}N_2)[NiCl_4]$ or $(H_2bpp)\cdot NiCl_4$ [bpp is 1,3-bis(4-pyridyl)propane], is isostructural with its already reported Cu, Zn and Hg analogues. The structure consists of a doubly charged $(H_2bpp)^{2+}$ cation and a tetrahedral $[NiCl_4]^{2-}$ dianion. Both pyridyl N atoms are protonated and form a $(H_2bpp)^{2+}$ cation which adopts an *anti–anti* conformation with a dihedral angle of 6.287 (7)° between the pyridyl rings. The two pyridyl N atoms are both involved in strong N—H⋯Cl hydrogen bonds, which link both units into a dimer.

Related literature

For the isostructural Cu, Zn and Hg analogues, see: Kao & Chen (2004).



Experimental

Crystal data

$(C_{13}H_{16}N_2)[NiCl_4]$
M_r = 400.79
Monoclinic, $P2_1/n$
a = 7.2358 (7) Å
b = 20.773 (2) Å
c = 11.1246 (11) Å
β = 90.627 (1)°
V = 1672.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.79 mm^{-1}
T = 294 K
0.36 × 0.25 × 0.24 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.565, T_{max} = 0.673
12584 measured reflections
3107 independent reflections
2556 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.045
S = 1.90
3107 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯Cl1^{i}	0.86	2.43	3.150 (2)	142
N2—H2A⋯Cl3^{ii}	0.86	2.25	3.114 (2)	178

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2307).

References

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Kao, Y.-C. & Chen, J.-D. (2004). *Struct. Chem.* **15**, 269–276.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2009). E**65**, m1656   [**doi:10.1107/S1600536809048843**]

4,4'-(Propane-1,3-diyl)dipyridinium tetrachloridonickelate(II)

Z.-X. Du and J.-P. Qu

Comment

The title complex (I) is isostructural with its analogues (H_2bpp).$CuCl_4$ and (H_2bpp).MCl_4.H_2O(M = Zn, Hg)(Kao and Chen, 2004), whose structures have been described in detail. The structure consists of a doubly charged $(H_2bpp)^{2+}$ cation and a tetrahedral $NiCl_4^{2-}$ dianion (Figure 1). Both pyridyl N atoms are protonated and form a $(H_2bpp)^{2+}$ cation, which adopts an anti-anti conformations with a dihedral angle of 6.287° between the two pyridyl rings. The two pyridyl N atoms are both involved in strong N—H···Cl hydrogen bonds (Table 1) and link both units into a dimer (Figure 2).

Experimental

$NiCl_2$.6H_2O (1.0 mmol, 0.237 g), bpp (1.0 mmol, 0.198 g) and oxydiacetatic acid (1.0 mmol, 0.134 g) were dissolved in 20 ml of methanol-H_2O (*v*/*v*, 1:3). Then the mixture was sealed in a 25 mL Teflon reactor and kept under autogeneous pressure at 403 K for 5 days. After cooling to room temperature at a rate of 6°C.h^{-1}, blue block shaped crystals suitable for X-ray diffraction were grown from the filtrate by slow evaporation. Yield: 200 mg (50% based on Ni). Anal. Calcd for $C_{13}H_{16}Cl_4N_2Ni$(%): C, 38.92; H, 3.99; N, 6.98. Found: C, 38.85; H, 4.03; N, 6.85. CCDC 752249.

Refinement

H atoms bonded to C and N atoms were positioned geometrically with C—H distance 0.93–0.97Å and N—H distances of 0.8600 Å, and treated as riding atoms, with U_{iso}(H)=1.2U_{eq}(C,N).

Figures

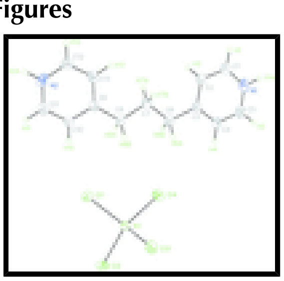

Fig. 1. Molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

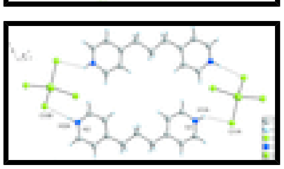

Fig. 2. The dimer of (I) formed by N—H···Cl hydrogen bonds showing as dashed lines.

4,4'-(Propane-1,3-diyl)dipyridinium tetrachloridonickelate(II)

Crystal data

$(C_{13}H_{16}N_2)[NiCl_4]$	F_{000} = 816
M_r = 400.79	D_x = 1.592 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3858 reflections
a = 7.2358 (7) Å	θ = 2.7–25.6º
b = 20.773 (2) Å	μ = 1.79 mm^{-1}
c = 11.1246 (11) Å	T = 294 K
β = 90.6270 (10)º	Block, blue
V = 1672.1 (3) Å^3	0.36 × 0.25 × 0.24 mm
Z = 4	

Data collection

Bruker APEXII CCD area-detector diffractometer	3107 independent reflections
Radiation source: fine-focus sealed tube	2556 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
T = 294 K	θ_{max} = 25.5º
φ and ω scans	θ_{min} = 2.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 8$
T_{min} = 0.565, T_{max} = 0.673	$k = -24 \rightarrow 25$
12584 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.029	H-atom parameters constrained
$wR(F^2)$ = 0.045	$w = 1/[\sigma^2(F_o^2) + (0.P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.90	$(\Delta/\sigma)_{max}$ < 0.001
3107 reflections	$\Delta\rho_{max}$ = 0.36 e Å^{-3}
181 parameters	$\Delta\rho_{min}$ = −0.45 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	0.9569 (3)	0.39288 (13)	0.4993 (2)	0.0604 (7)
H1	0.9940	0.4331	0.5272	0.073*
C2	0.7956 (3)	0.38678 (11)	0.4357 (2)	0.0534 (7)
H2	0.7226	0.4228	0.4204	0.064*
C3	0.7409 (3)	0.32701 (11)	0.3941 (2)	0.0425 (6)
C4	0.8519 (3)	0.27500 (12)	0.4223 (2)	0.0546 (7)
H4	0.8166	0.2340	0.3976	0.066*
C5	1.0119 (4)	0.28295 (13)	0.4855 (2)	0.0611 (7)
H5	1.0862	0.2476	0.5034	0.073*
C6	0.5730 (3)	0.31581 (10)	0.3173 (2)	0.0544 (7)
H6A	0.5005	0.2821	0.3546	0.065*
H6B	0.6135	0.2995	0.2403	0.065*
C7	0.4473 (3)	0.37229 (10)	0.2940 (2)	0.0462 (6)
H7A	0.5157	0.4064	0.2548	0.055*
H7B	0.4019	0.3888	0.3697	0.055*
C8	0.2851 (3)	0.35192 (10)	0.2146 (2)	0.0472 (6)
H8A	0.3340	0.3356	0.1397	0.057*
H8B	0.2227	0.3165	0.2539	0.057*
C9	0.1435 (3)	0.40250 (11)	0.1848 (2)	0.0403 (6)
C10	−0.0087 (3)	0.38588 (11)	0.1149 (2)	0.0497 (7)
H10	−0.0194	0.3442	0.0853	0.060*
C11	−0.1429 (3)	0.42979 (12)	0.0891 (2)	0.0551 (7)
H11	−0.2454	0.4180	0.0429	0.066*
C12	0.0190 (3)	0.50868 (12)	0.1949 (2)	0.0594 (7)
H12	0.0274	0.5511	0.2213	0.071*
C13	0.1564 (3)	0.46566 (11)	0.2227 (2)	0.0519 (7)
H13	0.2589	0.4791	0.2673	0.062*
Cl1	−0.06721 (8)	0.21383 (3)	0.12980 (6)	0.05653 (19)
Cl2	−0.12187 (9)	0.05327 (3)	0.13195 (6)	0.05657 (19)
Cl3	−0.06868 (9)	0.09147 (3)	0.42665 (6)	0.0606 (2)
Cl4	0.29570 (8)	0.16495 (3)	0.30041 (6)	0.0633 (2)

N1	1.0614 (3)	0.34138 (11)	0.52139 (17)	0.0574 (6)
H1A	1.1641	0.3462	0.5601	0.069*
N2	−0.1267 (3)	0.48968 (10)	0.13015 (19)	0.0558 (6)
H2A	−0.2130	0.5170	0.1143	0.067*
Ni1	0.01874 (4)	0.129429 (13)	0.24398 (3)	0.03832 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0602 (19)	0.0548 (17)	0.066 (2)	−0.0016 (15)	−0.0134 (15)	0.0003 (15)
C2	0.0527 (17)	0.0443 (15)	0.0628 (18)	0.0060 (12)	−0.0155 (14)	0.0009 (13)
C3	0.0421 (15)	0.0413 (14)	0.0441 (15)	0.0019 (11)	−0.0031 (12)	0.0036 (12)
C4	0.0569 (17)	0.0454 (15)	0.0613 (18)	0.0086 (13)	−0.0117 (14)	−0.0033 (14)
C5	0.0621 (19)	0.0602 (19)	0.0610 (19)	0.0188 (15)	−0.0051 (15)	0.0026 (16)
C6	0.0508 (17)	0.0451 (15)	0.0669 (19)	0.0019 (12)	−0.0131 (14)	0.0016 (14)
C7	0.0389 (14)	0.0463 (14)	0.0533 (16)	−0.0002 (12)	−0.0049 (12)	0.0005 (13)
C8	0.0482 (16)	0.0417 (14)	0.0515 (16)	0.0019 (12)	−0.0069 (12)	0.0035 (12)
C9	0.0373 (14)	0.0404 (14)	0.0433 (15)	−0.0034 (11)	−0.0009 (11)	0.0003 (12)
C10	0.0482 (16)	0.0410 (15)	0.0597 (18)	−0.0033 (12)	−0.0107 (13)	−0.0013 (13)
C11	0.0455 (16)	0.0567 (17)	0.0627 (19)	−0.0034 (14)	−0.0103 (13)	0.0016 (15)
C12	0.0577 (18)	0.0470 (16)	0.073 (2)	0.0026 (14)	−0.0017 (15)	−0.0097 (15)
C13	0.0435 (15)	0.0476 (15)	0.0643 (18)	−0.0020 (12)	−0.0090 (13)	−0.0059 (14)
Cl1	0.0603 (4)	0.0411 (4)	0.0678 (5)	−0.0059 (3)	−0.0164 (3)	0.0108 (3)
Cl2	0.0692 (5)	0.0397 (4)	0.0607 (4)	−0.0071 (3)	−0.0095 (3)	−0.0059 (3)
Cl3	0.0596 (4)	0.0696 (5)	0.0525 (4)	−0.0197 (3)	−0.0093 (3)	0.0093 (4)
Cl4	0.0462 (4)	0.0542 (4)	0.0892 (5)	−0.0103 (3)	−0.0145 (4)	0.0059 (4)
N1	0.0438 (14)	0.0767 (16)	0.0514 (14)	0.0044 (12)	−0.0110 (11)	0.0038 (13)
N2	0.0462 (14)	0.0540 (14)	0.0671 (16)	0.0139 (11)	0.0006 (11)	0.0067 (12)
Ni1	0.03720 (18)	0.03082 (16)	0.04678 (19)	−0.00285 (13)	−0.00648 (13)	0.00183 (15)

Geometric parameters (Å, °)

C1—N1	1.332 (3)	C8—H8A	0.9700
C1—C2	1.364 (3)	C8—H8B	0.9700
C1—H1	0.9300	C9—C13	1.381 (3)
C2—C3	1.381 (3)	C9—C10	1.385 (3)
C2—H2	0.9300	C10—C11	1.361 (3)
C3—C4	1.380 (3)	C10—H10	0.9300
C3—C6	1.496 (3)	C11—N2	1.330 (3)
C4—C5	1.358 (3)	C11—H11	0.9300
C4—H4	0.9300	C12—N2	1.330 (3)
C5—N1	1.326 (3)	C12—C13	1.370 (3)
C5—H5	0.9300	C12—H12	0.9300
C6—C7	1.505 (3)	C13—H13	0.9300
C6—H6A	0.9700	Cl1—Ni1	2.2487 (6)
C6—H6B	0.9700	Cl2—Ni1	2.2503 (6)
C7—C8	1.521 (3)	Cl3—Ni1	2.2760 (7)
C7—H7A	0.9700	Cl4—Ni1	2.2198 (7)
C7—H7B	0.9700	N1—H1A	0.8600

C8—C9	1.502 (3)	N2—H2A	0.8600
N1—C1—C2	120.2 (2)	C9—C8—H8B	108.1
N1—C1—H1	119.9	C7—C8—H8B	108.1
C2—C1—H1	119.9	H8A—C8—H8B	107.3
C1—C2—C3	119.8 (2)	C13—C9—C10	117.3 (2)
C1—C2—H2	120.1	C13—C9—C8	123.6 (2)
C3—C2—H2	120.1	C10—C9—C8	119.1 (2)
C4—C3—C2	117.6 (2)	C11—C10—C9	120.9 (2)
C4—C3—C6	118.3 (2)	C11—C10—H10	119.6
C2—C3—C6	124.0 (2)	C9—C10—H10	119.6
C5—C4—C3	120.9 (2)	N2—C11—C10	119.6 (2)
C5—C4—H4	119.6	N2—C11—H11	120.2
C3—C4—H4	119.6	C10—C11—H11	120.2
N1—C5—C4	119.6 (2)	N2—C12—C13	119.8 (2)
N1—C5—H5	120.2	N2—C12—H12	120.1
C4—C5—H5	120.2	C13—C12—H12	120.1
C3—C6—C7	117.56 (19)	C12—C13—C9	120.3 (2)
C3—C6—H6A	107.9	C12—C13—H13	119.9
C7—C6—H6A	107.9	C9—C13—H13	119.9
C3—C6—H6B	107.9	C5—N1—C1	121.9 (2)
C7—C6—H6B	107.9	C5—N1—H1A	119.1
H6A—C6—H6B	107.2	C1—N1—H1A	119.1
C6—C7—C8	110.12 (18)	C12—N2—C11	122.0 (2)
C6—C7—H7A	109.6	C12—N2—H2A	119.0
C8—C7—H7A	109.6	C11—N2—H2A	119.0
C6—C7—H7B	109.6	Cl4—Ni1—Cl1	98.27 (2)
C8—C7—H7B	109.6	Cl4—Ni1—Cl2	142.34 (3)
H7A—C7—H7B	108.2	Cl1—Ni1—Cl2	96.59 (3)
C9—C8—C7	116.96 (19)	Cl4—Ni1—Cl3	96.98 (3)
C9—C8—H8A	108.1	Cl1—Ni1—Cl3	134.16 (3)
C7—C8—H8A	108.1	Cl2—Ni1—Cl3	97.04 (3)
N1—C1—C2—C3	−0.1 (4)	C7—C8—C9—C10	−178.6 (2)
C1—C2—C3—C4	1.8 (4)	C13—C9—C10—C11	−2.5 (4)
C1—C2—C3—C6	−176.1 (2)	C8—C9—C10—C11	178.0 (2)
C2—C3—C4—C5	−2.0 (4)	C9—C10—C11—N2	0.8 (4)
C6—C3—C4—C5	176.0 (2)	N2—C12—C13—C9	−0.7 (4)
C3—C4—C5—N1	0.5 (4)	C10—C9—C13—C12	2.4 (4)
C4—C3—C6—C7	176.3 (2)	C8—C9—C13—C12	−178.1 (2)
C2—C3—C6—C7	−5.9 (4)	C4—C5—N1—C1	1.3 (4)
C3—C6—C7—C8	179.9 (2)	C2—C1—N1—C5	−1.5 (4)
C6—C7—C8—C9	178.7 (2)	C13—C12—N2—C11	−1.1 (4)
C7—C8—C9—C13	1.8 (3)	C10—C11—N2—C12	1.0 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···Cl1^{i}	0.86	2.43	3.150 (2)	142
N2—H2A···Cl3^{ii}	0.86	2.25	3.114 (2)	178

Symmetry codes: (i) $x+3/2$, $-y+1/2$, $z+1/2$; (ii) $-x-1/2$, $y+1/2$, $-z+1/2$.

Fig. 1

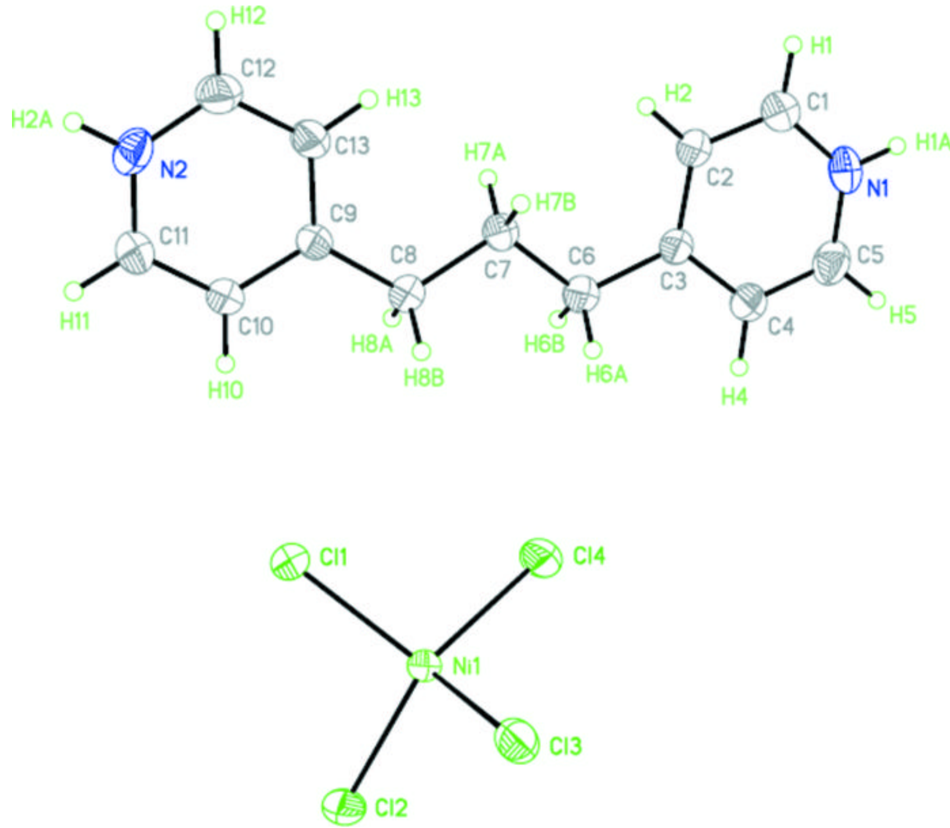

Fig. 2