# Tris(propane-1,3-diamine)nickel(II) bis(tetrafluoroborate)

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The cationic components, $[Ni(C_9H_{30}N_6)]^{2+}$, of the title compound $[Ni(C_9H_{30}N_6)](BF_4)_2$ or $Ni(pn)_3(BF_4)_2$ (pn is 1,3-diaminopropane), are stacked in the crystal *via* hydrogen bonding (N—H···F). The geometry around the $Ni^{II}$ atom is distorted octahedral, with an average Ni—N bond distance of 2.14 (2) Å.

## Comment

The title compound, (I), was obtained in attempt to prepare the analogous compounds $[Ni(en)_2ClO_4](ClO_4)$ and $[Ni(en)_2ClO_4](PF_6)$ (en is ethylenediamine) (Landee *et al.*, 1997). These compounds contain chains of nitrito-bridged nickel(II). These chains of $S = 1$ ions exhibit antiferromagnetic exchange coupling (Meyer *et al.*, 1982). Accordingly, they are ideal candidates for investigating the conjecture by Haldane that integer spin antiferromagnetic chains exhibit an energy gap between the nonmagnetic singlet ground state and the lowest lying excited state (Haldane, 1983).



(I)
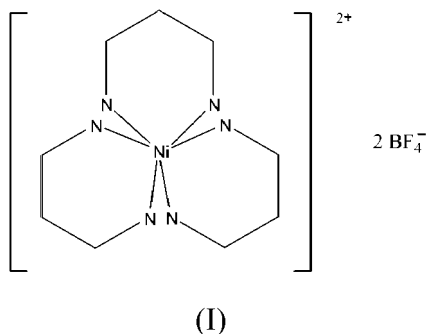
In the title compound, the average Ni—N bond distance is 2.14 (2) Å, with N—Ni—N chelate bite angles ranging from 86.57 (18) to 88.6 (2)°. Furthermore, the *trans* angles are different from the typical octahedral angle 174.2 (2)–179.0 (2)°. The structure is different from the corresponding nitrate structure (Vezzosi *et al.*, 1985). The three six-membered rings are similar to each other, with chair conformations, as shown in Fig. 1. The Ni—N—C and N—C—C angles are close in value, with ranges of 120.5 (4)–123.6 (4) and 112.7 (6)–116.2 (6)°, respectively. The analogous nitrate structure shows a pronounced flattening in one of the six-membered rings, as indicated by wider ranges of the above angles of 114 (1)–128 (1) and 113 (1)–127 (2)°, respectively. N—H···F hydrogen bonds stabilize the packing of the formula units in the crystal. The two tetrafluoroborate anions are disordered, as reflected by the large displacement ellipsoids of the F atoms.

## Experimental

1,3-Diaminopropane (32.2 mmol) was added slowly to a solution of $Ni(BF_4)_2$ (10.6 mmol) in water. To this solution, $Ni(BF_4)_2$ (10.6 mmol) and NaSCN (10.6 mmol) were added. Slow evaporation lead to the formation of purple crystals.

### Crystal data

| | |
|---|---|
| $[Ni(C_9H_{30}N_6)](BF_4)_2$ | Mo $K\alpha$ radiation |
| $M_r = 454.72$ | Cell parameters from 36 |
| Orthorhombic, $Pbca$ | reflections |
| $a = 15.2751 (14)$ Å | $\theta = 2.4–16.0°$ |
| $b = 15.498 (2)$ Å | $\mu = 1.05$ mm$^{-1}$ |
| $c = 16.835 (2)$ Å | $T = 298 (2)$ K |
| $V = 3985.6 (8)$ Å$^3$ | Chunk, purple |
| $Z = 8$ | $0.4 \times 0.3 \times 0.2$ mm |
| $D_x = 1.516$ Mg m$^{-3}$ | |

### Data collection

| | |
|---|---|
| Siemens $P2_1$ diffractometer upgraded to $P4$ | 1657 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.048$ |
| Absorption correction: $\psi$ scan (XPREP in SHELXTL; Bruker, 1996) | $\theta_{max} = 25.3°$ |
| | $h = 0 \rightarrow 18$ |
| | $k = 0 \rightarrow 18$ |
| $T_{min} = 0.654$, $T_{max} = 0.742$ | $l = 0 \rightarrow 20$ |
| 4877 measured reflections | 3 standard reflections |
| 3503 independent reflections | every 100 reflections |
| | intensity decay: none |

### Refinement

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 0.8993P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.059$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.139$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.00$ | $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ |
| 3503 reflections | $\Delta\rho_{min} = -0.22$ e Å$^{-3}$ |
| 311 parameters | |
| H-atom parameters constrained | |

The two tetrafluoroborate anions were found to be disordered and were refined in sets (set 1: F1, F2, F3 and F4; set 2: F1′, F2′, F3′ and F4′; set 3: F11, F12, F13 and F14; set 4: F11′, F12′, F13′ and F14′). They were refined to partial occupancies of 0.571 (18), 0.429 (18), 0.533 (16) and 0.467 (16), respectively. The B—F bond lengths of each tetrafluoroborate anion were restrained to the same value, and the F···F distances were restrained to be 1.633 times the corresponding B—F bond lengths.

Data collection: XSCANS (Bruker, 1996); cell refinement: XSCANS; data reduction: SHELXTL (Bruker, 1996); program(s) used to solve structure: XS in SHELXTL; program(s) used to refine
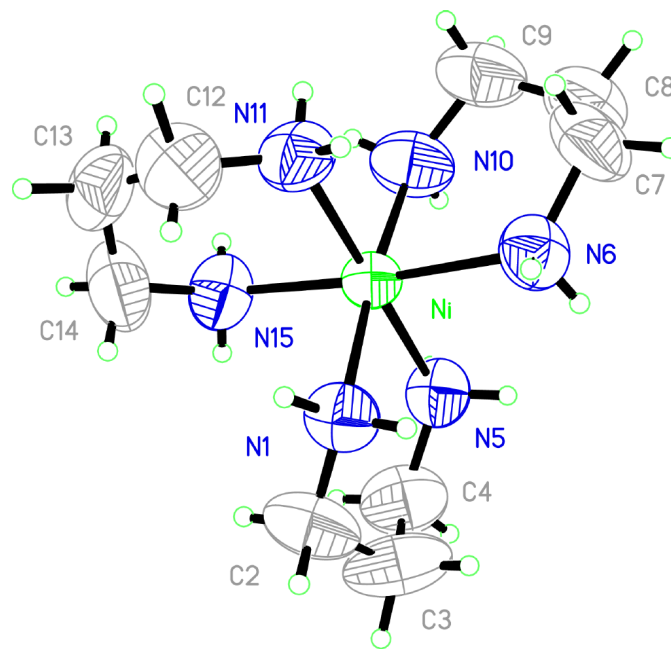


**Figure 1**
Drawing of the cationic component of (I), with 50% displacement ellipsoids.

structure: XL in SHELXTL; molecular graphics: XP in SHELXTL; software used to prepare material for publication: XCIF in SHELXTL.

## References

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