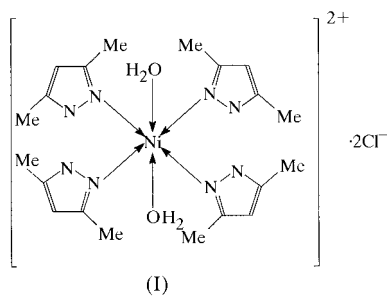


***trans*-Diaquatetrakis(3,5-dimethylpyrazole-*N*²)nickel(II) dichloride**Magdalena Małecka,^{a*} Agnieszka Rybarczyk-Pirek,^a
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The X-ray structure analysis of $[\text{Ni}(\text{C}_5\text{H}_8\text{N}_2)_4(\text{H}_2\text{O})_2]\text{Cl}_2$ was undertaken to elucidate the geometry around the Ni^{2+} ion. The molecule lies on a twofold axis which runs through the O—Ni—O atoms. The geometry around the Ni^{2+} ion is best described as slightly distorted tetragonal bipyramidal.

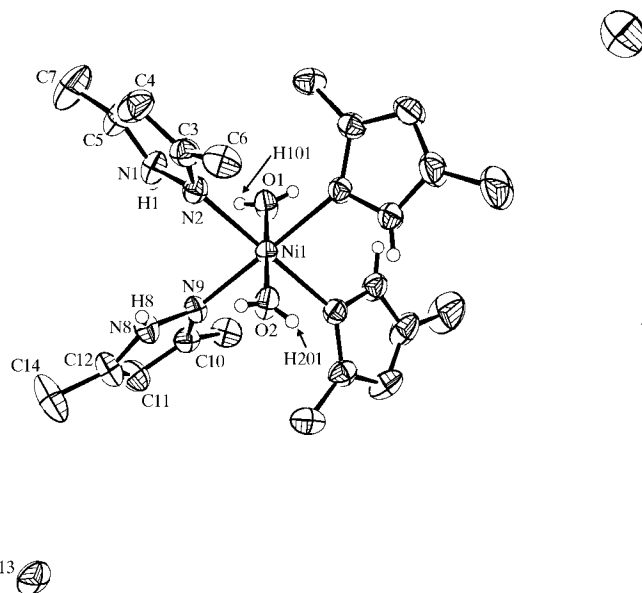
Comment

In recent years, we have investigated the coordination chemistry of metal complexes with N-containing heterocyclic derivatives (Driessen *et al.*, 1988; Małecka *et al.*, 1998; Ochocki *et al.*, 1990, 1992, 1997) because of expected pharmacological activity by analogy to *cis*-platinum complexes (Hollis, 1989; Reedijk, 1996). Previous studies on this kind of complex showed their significant role in biological processes, which is often related to their coordination ability towards transition metal ions (Ochocki *et al.*, 1998). This work extends the study to transition metal complexes with pyrazoles and their derivatives. Nickel is now recognized as an essential element for bacteria, plants, animals and humans, since it plays an important role in the catalytic activity of enzymes.



The X-ray structure analysis of the title compound, (I), was undertaken to elucidate the geometry around the Ni^{2+} ion. The central Ni^{2+} ion is in a special position on the twofold axis running through atoms O1, Ni1 and O2, and is centred in the slightly distorted octahedral environment. The basal plane is formed by N atoms from the 3,5-dimethylpyrazole ligands and

is distorted, with a tetrahedral angle of $178.3(1)^\circ$ (Holm & O'Connor, 1971). The distortion of the local tetrahedral coordination is assigned in a bending angle of $179.4(1)^\circ$ (which shows how far the Ni^{2+} ion is shifted from the geometric centre of the tetrahedron). There is a net of hydrogen bonds (see Table 2). Bond distances and angles in the phenyl and pyrazole rings are in good agreement with expected values (Allen *et al.*, 1987; Orpen *et al.*, 1989).

**Figure 1**

ORTEX (McArdle, 1995) drawing of the title compound. Displacement ellipsoids are drawn at the 40% probability level. H atoms attached to C atoms have been omitted for clarity.

An **ORTEX** (McArdle, 1995) drawing of the title molecule with the atomic numbering scheme is given in Fig. 1.

Experimental

The title compound was obtained by reaction of 3,5-dimethylpyrazole with nickel(II) chloride hexahydrate (4:1 stoichiometric ratio) in ethanol–water (4:1) solution. Light-blue crystals were obtained by slow evaporation from the same solution.

Crystal data

$[\text{Ni}(\text{C}_5\text{H}_8\text{N}_2)_4(\text{H}_2\text{O})_2]\text{Cl}_2$
 $M_r = 550.18$
Monoclinic, $C2/c$
 $a = 10.505(3) \text{ \AA}$
 $b = 14.213(2) \text{ \AA}$
 $c = 18.822(3) \text{ \AA}$
 $\beta = 92.77(2)^\circ$
 $V = 2807.1(10) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.302 \text{ Mg m}^{-3}$
 $D_m = 1.300 \text{ Mg m}^{-3}$

D_m measured by flotation in a mixture of xylene, bromobenzene, toluene and heptane
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
 $\theta = 6.13\text{--}12.76^\circ$
 $\mu = 0.912 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
Block, light blue
 $0.5 \times 0.2 \times 0.2 \text{ mm}$

Data collection

Rigaku AFC-5S diffractometer
 ω scans
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
 $T_{\min} = 0.560$, $T_{\max} = 0.788$
5343 measured reflections
5110 independent reflections
3056 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$
 $\theta_{\text{max}} = 32.57^\circ$
 $h = 0 \rightarrow 15$
 $k = 0 \rightarrow 21$
 $l = -28 \rightarrow 28$
3 standard reflections
frequency: 150 min
intensity decay: $<2\%$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.171$
 $S = 1.050$
 5109 reflections
 185 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0852P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.026$
 $\Delta\rho_{\max} = 0.84 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.85 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Ni1—O1	2.068 (3)	Ni1—N2	2.115 (2)
Ni1—O2	2.070 (3)	Ni1—N9	2.121 (2)
O1—Ni1—N2	90.22 (6)	O2—Ni1—N9	90.83 (6)
O2—Ni1—N2	89.78 (6)	N2—Ni1—N9	91.88 (8)
O1—Ni1—N9	89.17 (6)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H101 \cdots Cl3 ⁱ	0.69 (3)	2.35 (3)	3.044 (2)	175 (3)
O2—H201 \cdots Cl3 ⁱⁱ	0.69 (3)	2.39 (3)	3.065 (2)	166 (4)
N1—H1 \cdots Cl3 ⁱⁱⁱ	0.87 (3)	2.40 (3)	3.242 (3)	164 (3)
N8—H8 \cdots Cl3 ^{iv}	0.75 (4)	2.52 (4)	3.223 (3)	157 (4)

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

H atoms bonded to C atoms were treated as riding ($C-H = 0.96 \text{ \AA}$), while those on N and O atoms were refined (see Table 2 for bond lengths).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989a); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989b); program(s) used to solve

structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *PARST97* (Nardelli, 1996).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1497). Services for accessing these data are described at the back of the journal.

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