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#### Key indicators

Single-crystal X-ray study  
 $T = 301$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.042  
 $wR$  factor = 0.118  
Data-to-parameter ratio = 15.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

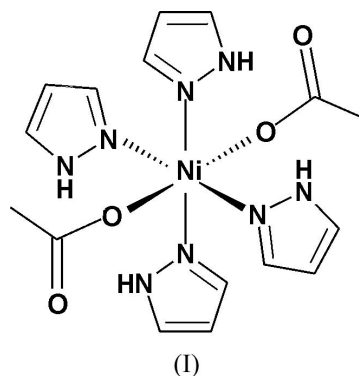
## Bis(acetato- $\kappa\text{O}$ )tetrakis(1*H*-pyrazole- $\kappa\text{N}^1$ )nickel(II)

The title mononuclear complex,  $[\text{Ni}(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_3\text{H}_4\text{N}_2)_4]$ , has been prepared and characterized by X-ray structure analysis. The molecule contains a six-coordinate  $\text{Ni}^{\text{II}}$  atom displaying distorted octahedral coordination geometry defined by the four pyrazole N atoms in the equatorial plane and two O atoms of the carboxylate groups in *trans*-apical positions. The Ni–N(pyrazole) distances range from 2.074 (2) to 2.097 (2) Å and the Ni–O(acetate) distances are 2.060 (2) and 2.072 (2) Å.

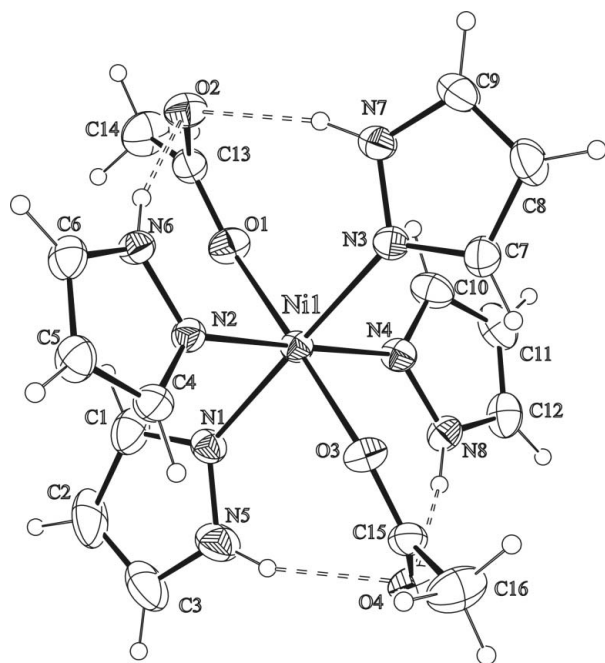
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#### Comment

Complexes containing pyrazole and imidazole ligands present several interesting aspects. In recent years, we have investigated the coordination chemistry of metal complexes with N-containing heterocyclic derivatives (Mašlejová *et al.*, 2001; Boča *et al.*, 2003; Svoboda *et al.*, 2001). Pyrazole (pzH) forms a variety of metal complexes (Otieno *et al.*, 2002; Steel, 1990; Trofimenko, 1972, 1986). There has been one report of the crystal structure of a hydrate of  $[\text{Ni}(\text{Oac})_2(\text{pzH})_4]$  (Doring *et al.*, 1996).



The structure and properties of Ni complexes with N-containing heterocyclic derivatives have attracted much scientific attention due to their potentially useful magnetic properties. In crystal structures, the N-bound H atom in pyrazole complexes is commonly involved in hydrogen bonds, giving rise to supramolecular arrangements which are related to one of the big challenges of chemistry, that is the understanding and control of the organization of molecules (Braga, 2000). The carboxylate ion is a versatile ligand, frequently used for designing complexes with desired magnetic properties. The title compound, (I), was obtained from the reaction of nickel(II) acetate tetrahydrate and an excess of pyrazole; the analogous diacetatotetraimidazolenickel(II) complex (Naumov *et al.*, 2000) is obtained in a different fashion.



**Figure 1**  
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

A series of nickel(II) complexes has been prepared possessing a variety of coordination spheres around the central Ni<sup>II</sup> ion for the study of magnetic anisotropy (Mašlejová *et al.*, 2003). The magnetic properties of (I) were studied down to 2 K (susceptibility and magnetization measurements). It was shown that the complex exhibits an increased magnetic anisotropy (expressed through the axial zero-field splitting parameter  $D$ ); this is determined by the heteroleptic coordination sphere containing ligands of a different crystal-field strength (Papánková *et al.*, 2005).

The molecular structure of  $[\text{Ni}(\text{Oac})_2(\text{pzH})_4]$  consists of discrete monomeric units with the Ni<sup>II</sup> atom in a distorted *trans*-octahedral configuration defined by two acetate anions and four neutral pyrazole ligands. The Ni–O(acetate) distances range from 2.060 (2) to 2.072 (2) Å and the Ni–N(pyrazole) distances are 2.082 (2) and 2.097 (2) Å. All pyrazole NH groups form intramolecular hydrogen bonds, giving rise to seven-membered rings incorporating the Ni atoms. The short intramolecular N–H···O hydrogen bonds [ $\text{H}\cdots\text{O} = 1.91$  and  $2.04$  Å,  $\text{N}\cdots\text{O} = 2.744$  (3)– $2.860$  (4) Å and  $\text{N}–\text{H}\cdots\text{O} = 156$ – $167^\circ$ ] are formed with non-coordinating carboxylate O atoms lying in axial positions and H atoms of the NH groups in the pyrazole rings. As expected, the mean C–O bond distance to the coordinating O atom is 1.251 Å, longer than that to the uncoordinated O atom (mean value = 1.241 Å). Molecules with N–H···O hydrogen bonds form rows connected by van der Waals interactions.

## Experimental

The reaction of  $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$  with a fourfold excess of pyrazole in warm dried ethanol is rapid at room temperature, leading

to the precipitation of  $[\text{Ni}(\text{Oac})_2(\text{pzH})_4]$ . Single blue crystals of (I) were obtained by slow evaporation of an ethanol solution at room temperature after recrystallization.

### Crystal data

$[\text{Ni}(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_3\text{H}_4\text{N}_2)_4]$   
 $M_r = 449.13$   
Monoclinic,  $P2_1/n$   
 $a = 9.925$  (5) Å  
 $b = 14.920$  (7) Å  
 $c = 13.969$  (7) Å  
 $\beta = 98.97$  (5)°  
 $V = 2043.2$  (17) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.460$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 401 reflections  
 $\theta = 2.5$ – $13.7^\circ$   
 $\mu = 0.99$  mm<sup>-1</sup>  
 $T = 301$  (2) K  
Prism, blue  
 $0.54 \times 0.52 \times 0.40$  mm

### Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector  
 $\omega$  scans  
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2002)  
 $T_{\min} = 0.617$ ,  $T_{\max} = 0.693$

12234 measured reflections  
4173 independent reflections  
3171 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.037$   
 $\theta_{\max} = 26.4^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -18 \rightarrow 18$   
 $l = -14 \rightarrow 17$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.118$   
 $S = 1.03$   
4173 reflections  
275 parameters  
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0732P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.003$   
 $\Delta\rho_{\max} = 0.62$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.47$  e Å<sup>-3</sup>  
Extinction correction: *SHELXL97*  
Extinction coefficient: 0.0070 (10)

**Table 1**  
Selected bond lengths (Å).

N1–Ni1	2.074 (2)	N4–Ni1	2.097 (2)
N2–Ni1	2.097 (2)	O1–Ni1	2.072 (2)
N3–Ni1	2.090 (2)	O3–Ni1	2.060 (2)

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

$D–H\cdots A$	$D–H$	$H\cdots A$	$D\cdots A$	$D–H\cdots A$
N5–H5A···O4	0.87 (1)	1.91 (1)	2.764 (3)	167 (3)
N6–H6A···O2	0.86 (1)	1.99 (1)	2.829 (3)	165 (3)
N7–H7A···O2	0.85 (1)	1.92 (1)	2.744 (3)	164 (3)
N8–H8A···O4	0.87 (1)	2.04 (2)	2.860 (4)	156 (3)

H atoms on C atoms were positioned geometrically and treated as riding atoms ( $\text{C}–\text{H} = 0.93$  Å). H atoms on N atoms were located in a difference Fourier map and the N–H distances restrained to 0.86 (1) Å. All  $U_{\text{iso}}(\text{H})$  values were set at  $1.2U_{\text{eq}}$  of the parent atom.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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