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Dinitratotetrapyrazolezinc(II)

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Molecules of the title compound, $[Zn(NO_3)_2(C_3H_4N_2)_4]$, are located on a crystallographic centre of inversion. The Zn atom is in an octahedral environment. The molecular and crystal structures are stabilized by intra- and intermolecular N—H···O hydrogen bonds.

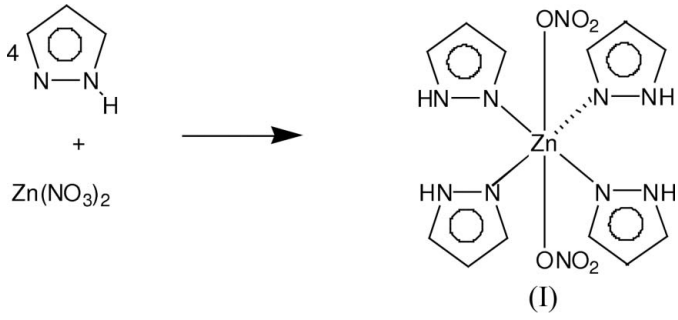
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.024
wR factor = 0.066
Data-to-parameter ratio = 15.1

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

Comment

Tris(1-pyrazolyl)borates ('scorpinates') were invented by Trofimenko (1993) more than 30 years ago and are today well established as ligands in coordination chemistry. Our studies have shown that degradation reactions of scorpionates were preferred in the presence of nucleophilic halides (Bieller *et al.*, 2006). In our aim to reverse deboronation reactions of scorpionates we decided to synthesize pyrazolato complexes with weakly coordinating anions such as nitrates. Therefore we decided to prepare the pyrazolato complex $[Zn(pz)_4(NO_3)_2]$, (I), which is easily accessible from the reaction of one equivalent of $Zn(NO_3)_2$ and four equivalents of pyrazole (pz) in ethanol.



(I)

The title complex has the Zn atom, which is located on a crystallographic centre of inversion, in an octahedral environment. The three N—O bond lengths of the nitrate ligand differ considerably (Table 1). The O atom bonded to Zn shows the longest N—O bond. The O atom involved in NH hydrogen bonding shows a shorter N—O bond. However, the O atom that is only bonded to N displays the shortest N—O bond. The crystal structure reveals intra- and intermolecular hydrogen bonds (Table 2).

Experimental

$Zn(NO_3)_2{\cdot}6H_2O$ (1.487 g) and pyrazole (1.362 g) were combined in ethanol (10 ml) under ambient conditions, forming a clear solution. Cooling of the solution (248 K) led to deposition of crystals of complex (I) (yield 80%).

Crystal data

$[Zn(NO_3)_2(C_3H_4N_2)_4]$
M_r = 461.72
Monoclinic, $P2_1/n$
a = 8.2654 (9) Å
b = 8.7649 (7) Å
c = 13.5367 (14) Å
β = 107.533 (8)°
V = 935.11 (16) Å^3
Z = 2
D_x = 1.640 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.37 mm^{-1}
T = 173 (2) K
Block, colourless
0.42 × 0.39 × 0.36 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.597, T_{max} = 0.639
7448 measured reflections
2145 independent reflections
1982 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.066
S = 1.04
2145 reflections
142 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 0.3842P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.030 (2)

Table 1
Selected bond lengths (Å).

Zn1—N21	2.1261 (12)	N1—O2	1.2396 (16)
Zn1—N11	2.1407 (12)	N1—O3	1.2527 (17)
Zn1—O1	2.1761 (10)	N1—O1	1.2824 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
N12—H12···O3	0.87 (2)	2.15 (2)	2.9922 (17)	162.5 (19)
N22—H22···O3^{i}	0.82 (2)	2.16 (2)	2.9511 (17)	160 (2)

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

H atoms were located in a difference map, but those bonded to C were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C)] using a riding model, with C—H = 0.95 Å. The H atoms bonded to N were refined freely.

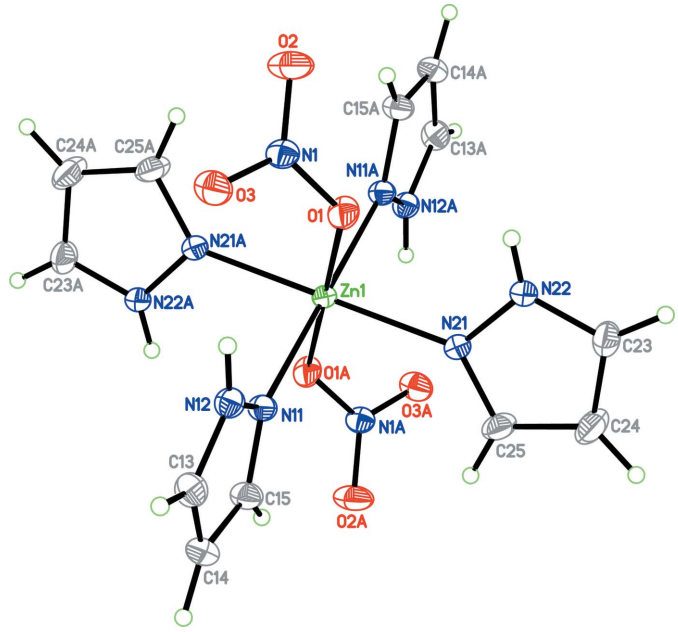


Figure 1
Perspective view of the title compound with the atom-numbering scheme; displacement ellipsoids are at the 50% probability level; H atoms are drawn as small spheres of arbitrary radii. Atoms with suffix A are generated by the symmetry operator $(-x, 1-y, 1-z)$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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