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[Hydridotris(5-methyl-3-phenylpyrazol-1-yl)-borato-$\kappa^3N^2,N^{2'},N^{2''}$](1*H*-imidazole-4-carboxyl-ato-κ^2N,O)zinc(II) methanol solvate

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Key indicators

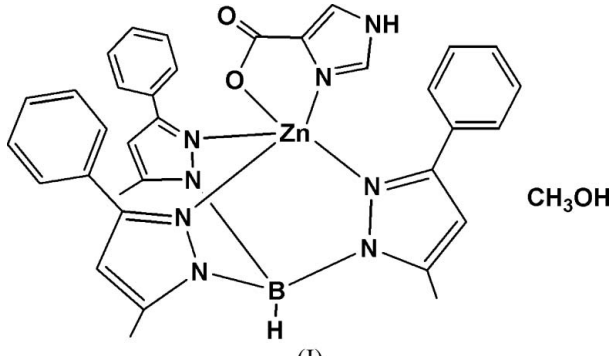
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.053
wR factor = 0.128
Data-to-parameter ratio = 15.2

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

In the title compound, $[Zn(C_{30}H_{28}BN_6)(C_4H_3N_2O_2)]\cdot CH_3OH$, the zinc ion is coordinated by three N atoms of the tridentate pyrazolyl groups, one of the carboxylate O atoms and one N atom of the 1*H*-imidazole-4-carboxylate ligand in a trigonal–bipyramidal geometry.

Comment

One of the most important model complexes of matrix metalloproteinases is the hydridotris(3,5-methylphenyl-pyrazoly)borate zinc hydroxide ($Tp^{Me,Ph}Zn$–OH). This compound exhibits high structural similarity to the catalytic center of the enzymes (Parkin, 2004; Puerta & Cohen, 2003). Structural analysis revealed that the zinc ion in this model compound adopted a tetrahedral geometry with bond distances and bond angles very close to the corresponding parameters in the enzymes. Previously, we (He, 2006*a*,*b*; He *et al.*, 2005) reported the interactions of this compound with a variety of potential synthetic inhibitors of these enzymes and found that their coordination modes to Zn^{2+} ions were highly dependent upon the bite size of the inhibitor. To further examine the preference of monodentate and bidentate coordination of an inhibitor to the Zn^{2+} ion, we have studied the interaction of $[Tp^{Me,Ph}Zn-OH]$ with 1*H*-imidazole-4-carboxylic acid, which can bind to Zn^{2+} through one imidazole N (monodentate), one imidazole N and one carboxylate O (bidentate), or two carboxylate O (bidentate) atoms.



(I)

In the title complex, (I), the tridentate hydridotris(3,5-methylphenylpyrazoly) ligand is coordinated to the zinc *via* pyrazolyl atoms N1, N3 and N5 (Fig. 1). The 1*H*-imidazole-4-carboxylate anion coordinates to Zn^{2+} through the chelation of carboxylate O2 and imidazolyl N7 atoms. The angular structural parameter, τ, is 1.01 (Addison *et al.* 1984). Thus, zinc adopts a trigonal–bipyramidal geometry. Atoms O2 and N3 occupy the axial positions with an angle of 170.15 (12)° about

the Zn atom. Atoms N1, N5 and N7 occupy equatorial positions. The other bond lengths and angles are in normal ranges (Puerta & Cohen, 2002, 2003). These results show that the chelation effect in this molecule is much stronger than that of chelation from two O atoms of the carboxylate anion.

Atom O1 is hydrogen bonded to O3 through H3*A*, whereas O2 is hydrogen-bonded to atom N12 of a second molecule (Table 1).

Experimental

Equimolar quantities of hydridotris(3,5-methylphenylpyrazolyl)-borate zinc hydroxide (69.2 mg, 0.12 mmol) (Puerta & Cohen, 2002) and 1*H*-imidazole-4-carboxylic acid (14.9 mg, 0.12 mmol) were separately dissolved in 10 and 2 ml of methanol, respectively. The solutions were mixed and stirred magnetically for 30 min. The solid formed was collected and dried under vacuum. Yield 48 mg, 69%.

Crystal data

$[Zn(C_{30}H_{28}BN_6)(C_4H_3N_2O_2)]\cdot CH_4O$
M_r = 691.89
Monoclinic, $P2_1/n$
a = 15.371 (3) Å
b = 13.829 (3) Å
c = 16.357 (3) Å
β = 99.46 (3)°
V = 3429.7 (12) Å^3
Z = 4
D_x = 1.340 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.76 mm^{-1}
T = 293 (2) K
Prism, colorless
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART 1K CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.862, T_{max} = 0.928
42524 measured reflections
6734 independent reflections
4349 reflections with $I > 2\sigma(I)$
R_{int} = 0.089
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.128
S = 1.10
6734 reflections
443 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 6.1376P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00088 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*A*···O1	0.82	1.93	2.728 (5)	165
N12—NH12···O2^{i}	0.86	1.89	2.711 (4)	160

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

The H atoms on B1 was refined [B1—BH = 1.10 (4) Å], while the other H atoms were geometrically constrained and refined in riding mode as follows: methyl C—H = 0.96 Å, U_{iso}(H) = 1.5U_{eq}(C); aromatic C—H = 0.93 Å, U_{iso}(H) = 1.2U_{eq}(C); O—H = 0.82 Å, U_{iso}(H) = 1.5U_{eq}(O); N—H = 0.86 Å, U_{iso}(H) = 1.2U_{eq}(N).

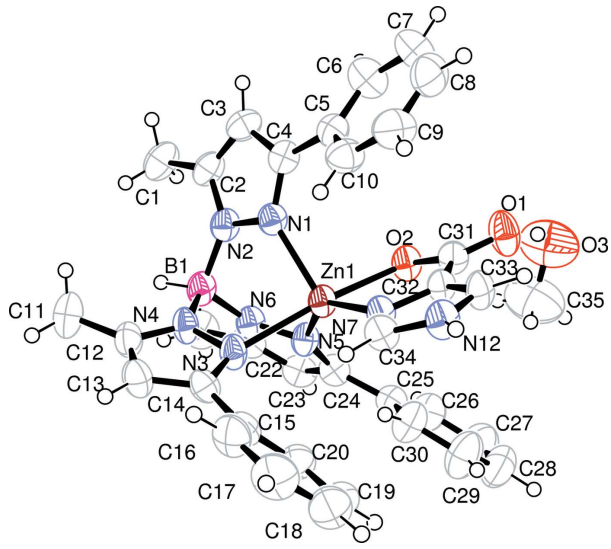


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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