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Key indicators

Single-crystal X-ray study

T = 295 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.028

wR factor = 0.074

Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Diammine[pyridine-2,6-dicarboxylato- $\kappa^3\text{O}^2, \text{N}, \text{O}^6$]zinc(II)

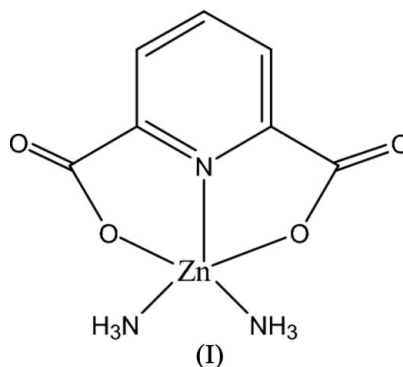
The title complex, $[\text{Zn}(\text{C}_7\text{H}_3\text{NO}_4)(\text{NH}_3)_2]$, was prepared by a hydrothermal reaction at 413 K. The complex has mirror symmetry. The Zn^{II} ion is coordinated by a tridentate pyridinedicarboxylate dianion and two ammonia molecules, in a distorted trigonal-bipyramidal coordination geometry. An intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding network stabilizes the crystal structure.

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Comment

Pyridine-2,6-dicarboxylic acid (H_2PDC) is widely used to construct metal-organic frameworks. We present here the structure of the title complex, (I), in which PDC plays the role of a tridentate ligand.



The molecular structure of (I) is shown in Fig. 1. The Zn^{II} complex has mirror symmetry. The Zn^{II} ion is coordinated by a tridentate PDC dianion and two ammonia molecules, in a distorted trigonal bipyramidal coordination geometry. In the axial direction, the $\text{Zn}-\text{O}1$ bond distance is significantly shorter than the $\text{Zn}-\text{O}3$ distance. The three $\text{Zn}-\text{N}$ bond distances in the equatorial plane are nearly the same (Table 1).

Intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonding between ammonia molecules and carboxylate groups (Table 2) stabilizes the crystal structure of (I).

Experimental

H_2PDC (0.083 g, 0.5 mmol) and concentrated ammonia (1 ml) were added to an aqueous solution (15 ml) of $\text{ZnSO}_4\cdot 7\text{H}_2\text{O}$ (0.143 g, 0.5 mmol). The mixture was placed in a 25 ml Teflon-lined Parr bomb and heated at 413 K for 38 h. The bomb was then cooled to room temperature at 5 K h^{-1} . Crystals were obtained in about 37% yield. Analysis calculated for $\text{C}_7\text{H}_9\text{N}_3\text{O}_4\text{Zn}$: C 31.78, H 3.43, N 15.88%; found: C 31.66, H 3.60, N 15.92%. IR (KBr, cm^{-1}): 3367 (*m*), 3071 (*w*), 1610 (*vs*), 1566 (*m*), 1470 (*s*), 1420 (*s*).

Crystal data

[Zn(C₇H₃NO₄)(NH₃)₂]
M_r = 264.56
 Orthorhombic, *Pbcm*
a = 10.4696 (9) Å
b = 12.6989 (11) Å
c = 7.2309 (6) Å
V = 961.37 (14) Å³
Z = 4
D_x = 1.828 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 121 reflections
 θ = 2.2–26.0°
 μ = 2.55 mm⁻¹
T = 295 (2) K
 Block, white
 0.36 × 0.32 × 0.25 mm

Data collection

Bruker APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.390, *T_{max}* = 0.530
 5637 measured reflections

1244 independent reflections
 1099 reflections with *I* > 2σ(*I*)
R_{int} = 0.020
 θ_{max} = 27.9°
h = -13 → 5
k = -16 → 16
l = -9 → 9

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.028
wR (*F*²) = 0.074
S = 1.16
 1244 reflections
 89 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.2581P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.004$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.79 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

| | | | |
|------------------------|-------------|-----------|-------------|
| Zn1–N1 | 2.0154 (19) | Zn1–O1 | 2.139 (2) |
| Zn1–N2 | 2.0039 (15) | Zn1–O3 | 2.2891 (16) |
| N1–Zn1–O1 | 77.45 (7) | N2–Zn1–O3 | 96.94 (5) |
| N1–Zn1–O3 | 74.76 (7) | O1–Zn1–O3 | 152.21 (6) |
| N1–Zn1–N2 | 127.12 (5) | O1–Zn1–N2 | 99.75 (5) |
| N2–Zn1–N2 ⁱ | 105.58 (9) | | |

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

Table 2

Hydrogen-bond geometry (Å, °).

| <i>D</i> –H... <i>A</i> | <i>D</i> –H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> –H... <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| N2–H2A...O4 ⁱⁱ | 0.89 | 2.23 | 3.050 (2) | 153 |
| N2–H2B...O2 ⁱⁱⁱ | 0.89 | 2.15 | 3.011 (2) | 162 |
| N2–H2C...O3 ^{iv} | 0.89 | 2.32 | 3.123 (2) | 150 |

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

Methyl H atoms were placed in calculated positions, with C–H = 0.89 Å, and refined to fit the electron density, with *U*_{iso}(H) =

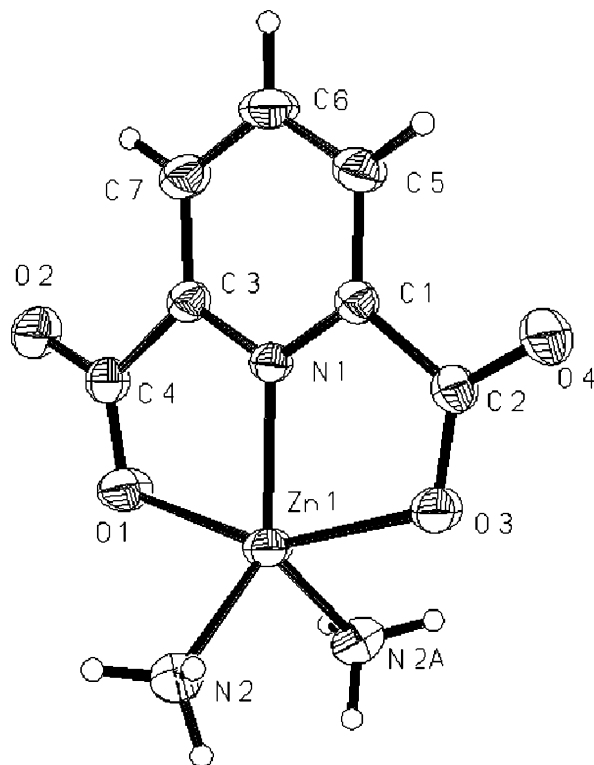


Figure 1

The molecular structure of (I), shown with 50% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry code: (A) *x*, *y*, $\frac{1}{2} - z$].

1.5*U*_{eq}(C). Aromatic H atoms were placed in calculated positions, with C–H = 0.93 Å, and refined as riding, with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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