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Diaquabis(4,4′-bipyridine)zinc bis(2,4,6-trinitrophenolate) dihydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.057
wR factor = 0.152
Data-to-parameter ratio = 13.2

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

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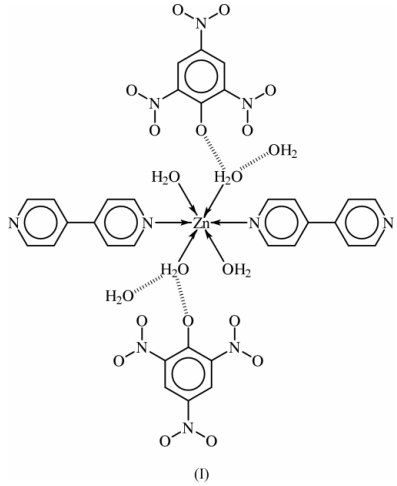
The space group of the title compound, $[Zn(C_{10}H_8N_2)_2(H_2O)_2](C_6H_2N_3O_7)_2\cdot 2H_2O$, reported as *Cc* [Liang *et al.* (2001). *Chin. J. Inorg. Chem.* **17**, 699–703], is revised to *C*2/*c*. In the revised description, the Zn atom lies on a special position of 2 symmetry (Wyckoff 2*e*); one 4,4′-bipyridine entity also lies on a twofold axis that passes through the two N atoms, whereas the other 4,4′-bipyridine entity lies on an inversion center (Wyckoff 4*a*) that lies midway between the 4- and 4′-C atoms.

Comment

The reaction of zinc dipicrate octahydrate with 4,4′-bipyridine gives bis(4,4′-bipyridine)diaquazinc dipicrate (Liang *et al.*, 2000) as a dihydrate (Liang *et al.*, 2001*b*), (I), as well as a monohydrated co-crystal with 4,4′-bipyridine (Liang *et al.*, 2001*a*). The crystal structures of both were refined in the space group *Cc*; that of the dihydrate was refined to 0.087 for reflections with 2584 $I > \sigma(I)$. However, the published packing diagram shows a centrosymmetric arrangement of the contents. In the revised *C*2/*c* setting, the structure refined to 0.057, but some retraints had to be imposed on the model (Fig. 1). In the revised description, the Zn atom lies on a twofold axis as does one of the two 4,4-bipyridine units. This axis passes through the two N atoms. The other spacer unit lies on an inversion center that is mid-way between the 4- and 4′-C atoms. Arising from the revision, the 4,4′-bipyridine–zinc layer motif is better regarded as an almost perfect square. One side is equal in length to the *b*-axial distance, whereas the other is half the *c* axis, as seen from the axial ratio (11.418 Å/22.908 Å) which is nearly 1/2.



(I)

Experimental

The reaction of zinc dipicrate octahydrate with 4,4′-bipyridine gives bis(4,4′-bipyridine)diaquazinc dipicrate (Liang *et al.*, 2000) as a dihydrate (Liang *et al.*, 2001*b*), as well as a monohydrated co-crystal with 4,4′-bipyridine (Liang *et al.*, 2001*a*).

Crystal data

$[Zn(C_{10}H_8N_2)_2(H_2O)_2]$-$(C_6H_2N_3O_7)_2\cdot 2H_2O$
$M_r = 906.01$
Monoclinic, $C2/c$
$a = 14.391\ (2)$ Å
$b = 11.418\ (1)$ Å
$c = 22.908\ (3)$ Å
$\beta = 95.08\ (1)^\circ$
$V = 3749.4\ (8)$ Å^3
$Z = 4$
$D_x = 1.605$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 15.4$–15.9°
$\mu = 0.75$ mm^{-1}
$T = 298\ (2)$ K
Block, yellow
$0.18 \times 0.15 \times 0.12$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.874$, $T_{max} = 0.914$
3829 measured reflections
3679 independent reflections
2020 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.0^\circ$
$h = -17 \rightarrow 0$
$k = -14 \rightarrow 0$
$l = -28 \rightarrow 28$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.152$
$S = 1.01$
3679 reflections
278 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 9.5165P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.61$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}

The aromatic H atoms were positioned geometrically (C—H = 0.93 Å) in a riding-model approximation, and were assigned displacement parameters 1.2 times those of their parent C atoms. The water H atoms were located and refined, subject to O—H = 0.85 (1) Å and H···H = 1.39 (1) Å; the displacement parameters were similarly assigned. The aromatic ring of the anion was refined as a rigid hexagon (C—C = 1.39 Å); the three C—N distances were restrained to be approximately equal within 0.01 Å, as were the six N—O distances.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1988); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

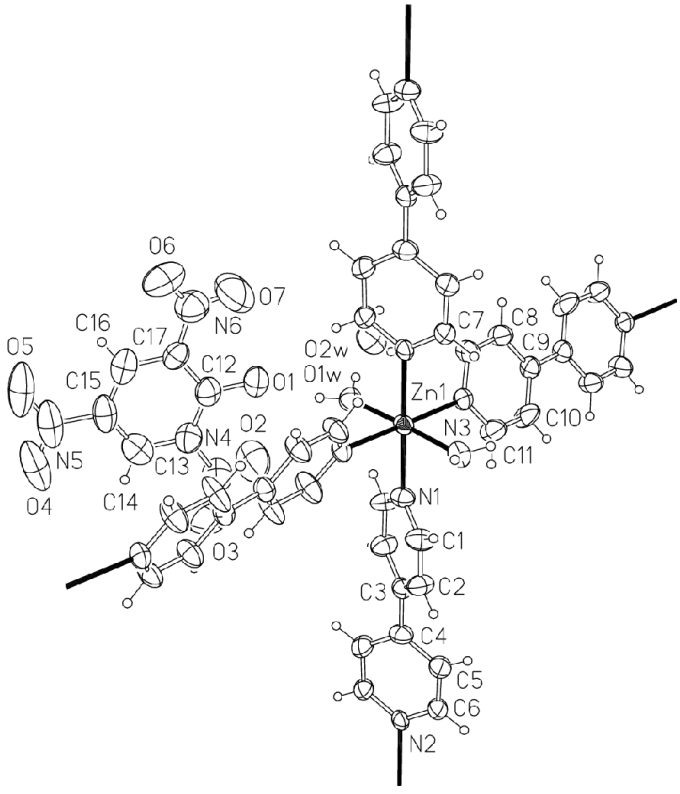


Figure 1
*ORTEP*II (Johnson, 1976) plot of a portion of the crystal structure of (I), with displacement ellipsoids drawn at the 50% probability level.

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