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A reinvestigation of the space group of *catena*-poly[[[triaqua(sulfato-κO)-zinc(II)]-μ-4,4′-bipyridine-κ^2N:N'] dihydrate]

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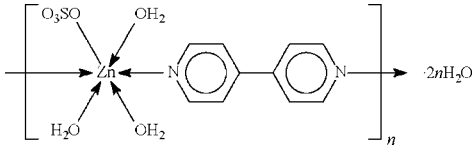
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.010 Å; H-atom completeness 45%; R factor = 0.045; wR factor = 0.124; data-to-parameter ratio = 19.0.

In the title compound, $\{[Zn(SO_4)(C_{10}H_8N_2)(H_2O)_3]\cdot 2H_2O\}_n$, the *N*-heterocycle of (4,4′-bipyridine)triaquasulfatozinc dihydrate links the sulfate- and water-coordinated Zn atom into a helical chain that runs along the *a* axis of the hexagonal $P6_1$ unit cell; adjacent chains are linked by hydrogen bonds into a three-dimensional network. The structure is a twin, the components being nearly 50%.

Related literature

For adducts of zinc sulfate and 4,4′-bipyridine in molar stoichiometries, see Huang *et al.* (1998) and Tong & Chen (2000). For the title compound described in the space group $P6_5$, see Kondo *et al.* (1999) and Ma *et al.* (2000). For the analogous copper compound that is described in the space group $P6_1$, see Lin & Liu (2003), and for the same compound described in the space group $P6_5$, see Hagrman *et al.* (1998) and Yuan *et al.* (2003).

For literature on enantiomorphous space groups, see Ha & Allewell (1997) and for a comment on the rarity of compounds crystallizing in both enantiomorphous space groups, see Brock & Dunitz (1991). For the possible existence of two enantiomorphic forms of ammonium dioxalatotitanate dihydrate, see English & Eve (1993) and Sheu *et al.* (1996).



Experimental

Crystal data

$[Zn(SO_4)(C_{10}H_8N_2)(H_2O)_3]\cdot 2H_2O$
M_r = 407.69
Hexagonal, $P6_1$
a = 11.4275 (3) Å
c = 20.9322 (6) Å
V = 2367.27 (9) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 1.74 mm^{-1}
T = 295 (2) K
0.35 × 0.29 × 0.18 mm

Data collection

Rigaku RAXIS-RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.45, T_{max} = 0.73
22988 measured reflections
3514 independent reflections
3309 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.125
S = 1.11
3514 reflections
185 parameters
41 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Absolute structure: Flack (1983), with 1473 Friedel pairs
Flack parameter: −0.05 (2)

Table 1
Selected bond lengths (Å).

Zn1—O1	2.316 (5)	Zn1—O3*W*	2.108 (5)
Zn1—O1*W*	2.163 (5)	Zn1—N1	2.160 (4)
Zn1—O2*W*	2.034 (6)	Zn1—N2^{i}	2.170 (4)

Symmetry code: (i) $x-1, y, z$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Molecular Structure Corporation & Rigaku, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997) and *PLATON* (Spek, 2003); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG3045).

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supplementary materials

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A reinvestigation of the space group of *catena*-poly[[[triaqua(sulfato-κO)zinc(II)]-μ-4,4'-bipyridine-$\kappa^2 N$:N'] dihydrate\]

S. W. Ng

Comment

The crystal structure of (4,4'-bipyridine)triaquasulfatozinc dihydrate, which is described in the $P6_5$ space group, shows a bipyridine-bridged, helical chain structure having unidentate sulfate groups (Kondo *et al.*, 1999; Ma *et al.*, 2000). The compound with the formulation $2[C_{10}H_{10}N_2]^{2+}$ $[(C_{10}H_8N_2)_3(H_2O)_6(SO_4)_4Zn_3]^{4-}.10H_2O$, isolated at a different pH, has the sulfate groups in μ_2-bridging modes (Tong & Chen, 2000). Under solvothermal conditions, a monoaqua monohydrate is also known but the sulfate group engages in a different bonding mode (Huang *et al.*, 1998).

In the present study, the crystal structure of (4,4'-bipyridine)triaquasulfatozinc dihydrate when refined in the $P6_1$ space group gave a Flack parameter (Flack, 1983) of nearly zero. Interestingly, two previous studies have described the compound in the $P6_5$ space group (Kondo *et al.*, 1999; Ma *et al.*, 2000), so that the present and previous studies hint at the possibility that helices of opposite handedness could be present in a synthesis. Such has not been mentioned in the literature. Unfortunately, the Flack parameter is not given in the earlier reports, and one (Ma *et al.*, 2000) used a large weighting scheme.

The crystal structure of the analogous (4,4'-bipyridine)triaquasulfatocopper dihydrate is known in the $P6_5$ form its Flack parameter refined to nearly zero (Hagrman *et al.*, 1998; Lin & Liu, 2003). A $P6_1$ polymorph has also been reported; however, as its Flack parameter refined to 0.93 (Yuan *et al.*, 2003), the form may be the $P6_5$ form only.

The existence of pairs of enantiomorphic structures is rarely discussed in the literature as finding such compounds is regarded as being unlikely (Brock & Dunitz, 1991). Amonium dioxalatotitanate dihydrate was refined by *SHELX*-76 in $P6_222$; the authors attempted a refinement in $P6_422$ but in view of statistical tests available then, concluded that the space group was $P6_222$ as it led to significantly better agreement (English & Eve, 1993). A later study on the charge density (Sheu *et al.*, 1996) did not comment on the alternative choice of the $P6_422$ setting. The title zinc compound (in $P6_1$) and the isostructural copper compound (in $P6_5$) can be regarded as exemplifying such a pair of achiral metal-organic compounds. In this case, the space groups can be distinguished on the basis of anomalous dispersion (Ha & Allewell, 1997).

Experimental

The compound was the unexpected product from the reaction of zinc sulfate monohydrate (0.09 g, 5 mmol), 3-(3-carboxyphenoxy)propionic acid (0.10 g, 5 mmol), sodium hydroxide (0.04 g, 10 mmol) and 4,4'-bipyridine (0.08 g, 5 mmol) in methanol. Colorless prismatic crystals were obtained from the filtered solution after several days.

Refinement

The structure is twinned; the use of the TwinRotMat routine of the *PLATON* suite (Spek, 2003) gave the TWIN command as (1 1 0 0 − 1 0 0 0 − 1). The twin component refined to 0.54 (1).

For the 4,4'-bipyridine molecule, the C–N bond distances were restrained to 0.005 Å of each other as were the C–C bond distances of the rings. There was some disorder in the carbon atoms as some temperature factors were too small whereas others were too large. As the molecule has approximate twofold rotational symmetry along the N···N vector, the temperature factors of pairs of carbon atoms (*i.e.*, C1/C5, C2/C4, C6/C10 and C7/C9) were restrained to be the same.

The carbon-bound hydrogen atoms were placed at calculated positions (C–H 0.93 Å) and were inlcuded in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C). Although the hydronge atoms of the water molecules could be placed in chemically sensible positions on the basis of hydrogen bonds, more than one such scheme may be envisaged. As such, hydrogen atoms were not included in the refinement.

The final difference Fourier map had a large peak in the neighbourhood of O1W, but was otherwise diffuse.

Figures

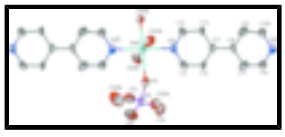


Fig. 1. Thermal ellipsoid plot of a portion of the chain structure of $[(C_{10}H_8N_2)(H_2O)_3(SO_4)Zn]\cdot 2H_2O$. Displacement ellipsoids are drawn at the 70% probability level, and H atoms are not drawn. Translational code (i): $x-1$, y, z.

catena-poly[[[triaqua(sulfato-κ*O*)zinc(II)]-μ- 4,4'-bipyridine-κ^2*N*:*N*'] dihydrate]

Crystal data

$[Zn(SO_4)(C_{10}H_8N_2)(H_2O)_3]\cdot 2H_2O$	$Z = 6$
$M_r = 407.69$	$F_{000} = 1260$
Hexagonal, $P6_1$	$D_x = 1.716$ Mg m^{-3}
Hall symbol: P 61	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.4275$ (3) Å	Cell parameters from 21095 reflections
$b = 11.4275$ Å	$\theta = 3.6$–27.4°
$c = 20.9322$ (6) Å	$\mu = 1.74$ mm^{-1}
$\alpha = 90^\circ$	$T = 295$ (2) K
$\beta = 90^\circ$	Prism, colorless
$\gamma = 120^\circ$	0.35 × 0.29 × 0.18 mm
$V = 2367.27$ (9) Å^3	

Data collection

Rigaku RAXIS-RAPID IP diffractometer	3514 independent reflections
Radiation source: fine-focus sealed tube	3309 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
$T = 295(2)$ K	$\theta_{max} = 27.4^\circ$
ω scan	$\theta_{min} = 3.6^\circ$
Absorption correction: Multi-scan ABSCOR (Higashi, 1995)	$h = -14\rightarrow14$
$T_{min} = 0.45$, $T_{max} = 0.73$	$k = -14\rightarrow14$

22988 measured reflections

$l = -25 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.125$

$S = 1.11$

3514 reflections

185 parameters

41 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0882P)^2 + 0.7556P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 1.46$ e Å^{-3}

$\Delta\rho_{min} = -0.43$ e Å^{-3}

Extinction correction: none

Absolute structure: Flack parameter for 1473 Friedel pairs

Flack parameter: −0.05 (2)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.57094 (6)	1.00177 (10)	0.49998 (4)	0.02328 (15)
S1	0.38825 (14)	0.64977 (15)	0.47291 (7)	0.0273 (3)
O1	0.4543 (5)	0.7708 (5)	0.51774 (19)	0.0286 (10)
O2	0.2571 (5)	0.5504 (5)	0.4983 (3)	0.0487 (13)
O3	0.3648 (5)	0.6913 (5)	0.4104 (2)	0.0472 (14)
O4	0.4787 (7)	0.5936 (6)	0.4647 (3)	0.0566 (16)
O1w	0.6807 (5)	1.2160 (5)	0.4796 (2)	0.0282 (10)
O2w	0.5570 (5)	0.9639 (6)	0.4045 (3)	0.0389 (13)
O3w	0.5760 (6)	1.0252 (6)	0.6000 (3)	0.0412 (15)
O4w	0.1575 (7)	0.6249 (9)	0.6013 (3)	0.074 (2)
O5w	0.5635 (6)	0.6791 (6)	0.3194 (3)	0.0489 (12)
N1	0.7614 (4)	1.0049 (5)	0.5031 (3)	0.0261 (9)
N2	1.3815 (4)	1.0025 (6)	0.4974 (4)	0.0316 (10)
C1	0.7649 (6)	0.8902 (6)	0.5018 (5)	0.0347 (9)
H1	0.6836	0.8088	0.5005	0.042*
C2	0.8846 (5)	0.8873 (6)	0.5022 (4)	0.0318 (8)
H2	0.8827	0.8050	0.5023	0.038*
C3	1.0071 (4)	1.0071 (6)	0.5026 (4)	0.0246 (8)
C4	1.0036 (6)	1.1263 (6)	0.5042 (4)	0.0318 (8)
H4	1.0831	1.2093	0.5054	0.038*
C5	0.8795 (5)	1.1195 (6)	0.5038 (5)	0.0347 (9)
H5	0.8784	1.2004	0.5041	0.042*
C6	1.2611 (5)	0.8924 (7)	0.4970 (7)	0.0646 (18)
H6	1.2595	0.8105	0.4935	0.078*
C7	1.1377 (7)	0.8860 (7)	0.5012 (8)	0.076 (2)
H7	1.0578	0.8038	0.5050	0.092*

C8	1.1372 (5)	1.0062 (6)	0.4997 (4)	0.0263 (9)
C9	1.2614 (6)	1.1228 (7)	0.4997 (8)	0.076 (2)
H9	1.2661	1.2062	0.5029	0.092*
C10	1.3794 (6)	1.1172 (7)	0.4948 (6)	0.0646 (18)
H10	1.4607	1.1975	0.4896	0.078*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0185 (3)	0.0317 (3)	0.0245 (2)	0.0161 (3)	−0.0012 (3)	−0.0016 (2)
S1	0.0231 (6)	0.0313 (7)	0.0282 (6)	0.0142 (6)	−0.0029 (5)	−0.0036 (6)
O1	0.028 (2)	0.038 (3)	0.026 (2)	0.022 (2)	0.0011 (17)	−0.0011 (18)
O2	0.046 (3)	0.044 (3)	0.036 (2)	0.007 (2)	0.008 (2)	0.003 (2)
O3	0.058 (3)	0.040 (3)	0.039 (3)	0.020 (3)	−0.025 (3)	−0.009 (2)
O4	0.062 (3)	0.062 (4)	0.068 (4)	0.048 (3)	−0.021 (3)	−0.026 (3)
O1w	0.023 (2)	0.023 (2)	0.036 (2)	0.0106 (18)	0.0037 (19)	0.0042 (17)
O2w	0.030 (3)	0.044 (3)	0.029 (3)	0.008 (2)	−0.011 (2)	0.000 (2)
O3w	0.069 (4)	0.049 (3)	0.017 (2)	0.038 (3)	−0.002 (2)	−0.002 (2)
O4w	0.047 (3)	0.086 (5)	0.070 (4)	0.018 (3)	0.013 (3)	−0.011 (4)
O5w	0.049 (3)	0.052 (3)	0.054 (3)	0.031 (3)	0.002 (2)	−0.013 (2)
N1	0.019 (2)	0.032 (2)	0.029 (2)	0.013 (2)	0.000 (2)	0.0033 (19)
N2	0.016 (2)	0.035 (2)	0.044 (2)	0.012 (2)	0.006 (3)	−0.0010 (19)
C1	0.017 (2)	0.032 (2)	0.051 (2)	0.009 (2)	0.000 (2)	0.0027 (18)
C2	0.024 (2)	0.0298 (19)	0.050 (2)	0.020 (2)	−0.007 (2)	−0.0013 (17)
C3	0.018 (3)	0.034 (3)	0.0283 (19)	0.018 (3)	0.005 (3)	−0.0024 (19)
C4	0.024 (2)	0.0298 (19)	0.050 (2)	0.020 (2)	−0.007 (2)	−0.0013 (17)
C5	0.017 (2)	0.032 (2)	0.051 (2)	0.009 (2)	0.000 (2)	0.0027 (18)
C6	0.020 (2)	0.032 (2)	0.147 (6)	0.017 (2)	−0.003 (4)	−0.013 (3)
C7	0.022 (2)	0.031 (2)	0.183 (7)	0.019 (2)	0.016 (4)	0.000 (3)
C8	0.010 (2)	0.032 (3)	0.035 (2)	0.009 (2)	0.002 (3)	0.001 (2)
C9	0.022 (2)	0.031 (2)	0.183 (7)	0.019 (2)	0.016 (4)	0.000 (3)
C10	0.020 (2)	0.032 (2)	0.147 (6)	0.017 (2)	−0.003 (4)	−0.013 (3)

Geometric parameters (Å, °)

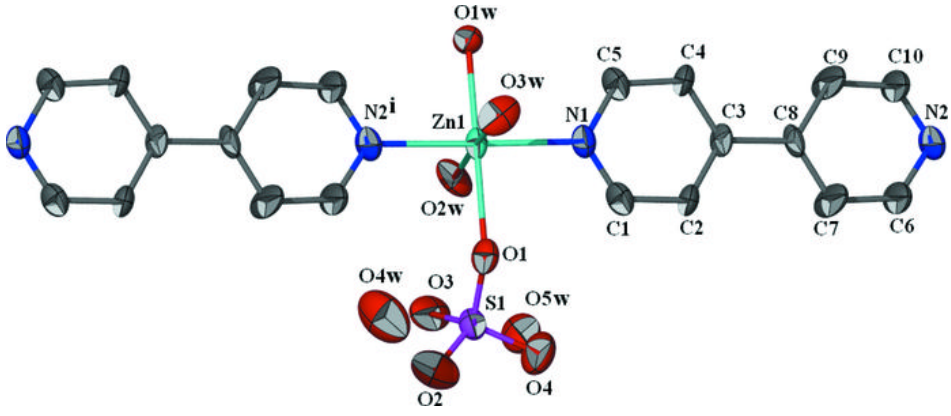
Zn1—O1	2.316 (5)	C1—H1	0.9300
Zn1—O1W	2.163 (5)	C2—C3	1.384 (4)
Zn1—O2W	2.034 (6)	C2—H2	0.9300
Zn1—O3W	2.108 (5)	C3—C4	1.383 (9)
Zn1—N1	2.160 (4)	C3—C8	1.492 (5)
Zn1—N2^{i}	2.170 (4)	C4—C5	1.381 (10)
S1—O2	1.455 (5)	C4—H4	0.9300
S1—O3	1.461 (5)	C5—H5	0.9300
S1—O4	1.474 (5)	C6—C7	1.378 (11)
S1—O1	1.523 (5)	C6—H6	0.9300
N1—C1	1.331 (4)	C7—C8	1.377 (10)
N1—C5	1.330 (4)	C7—H7	0.9300
N2—C6	1.321 (9)	C8—C9	1.378 (10)

N2—C10	1.324 (9)	C9—C10	1.385 (11)
N2—Zn1^{ii}	2.170 (4)	C9—H9	0.9300
C1—C2	1.385 (10)	C10—H10	0.9300
O2w—Zn1—O3w	175.7 (3)	N1—C1—H1	118.7
O2w—Zn1—N1	90.3 (2)	C2—C1—H1	118.7
O3w—Zn1—N1	90.3 (3)	C3—C2—C1	119.9 (5)
O2w—Zn1—O1w	89.2 (2)	C3—C2—H2	120.0
O3w—Zn1—O1w	95.1 (2)	C1—C2—H2	120.0
N1—Zn1—O1w	88.8 (2)	C2—C3—C4	117.4 (4)
O2w—Zn1—N2^{i}	90.2 (3)	C2—C3—C8	120.8 (5)
O3w—Zn1—N2^{i}	89.3 (3)	C4—C3—C8	121.8 (5)
N1—Zn1—N2^{i}	178.9 (2)	C5—C4—C3	118.7 (5)
O1w—Zn1—N2^{i}	90.3 (2)	C5—C4—H4	120.7
O2w—Zn1—O1	88.6 (2)	C3—C4—H4	120.7
O3w—Zn1—O1	87.09 (19)	N1—C5—C4	124.2 (5)
N1—Zn1—O1	90.86 (19)	N1—C5—H5	117.9
O1w—Zn1—O1	177.80 (19)	C4—C5—H5	117.9
N2^{i}—Zn1—O1	90.1 (2)	N2—C6—C7	126.9 (6)
O2—S1—O3	107.2 (3)	N2—C6—H6	116.5
O2—S1—O4	112.1 (4)	C7—C6—H6	116.5
O3—S1—O4	108.6 (4)	C8—C7—C6	117.4 (6)
O2—S1—O1	109.4 (3)	C8—C7—H7	121.3
O3—S1—O1	110.2 (3)	C6—C7—H7	121.3
O4—S1—O1	109.3 (3)	C7—C8—C9	116.6 (4)
S1—O1—Zn1	132.7 (2)	C7—C8—C3	120.4 (5)
C1—N1—C5	117.1 (4)	C9—C8—C3	122.8 (5)
C1—N1—Zn1	120.6 (4)	C8—C9—C10	120.7 (6)
C5—N1—Zn1	122.3 (4)	C8—C9—H9	119.6
C6—N2—C10	114.6 (5)	C10—C9—H9	119.6
C6—N2—Zn1^{ii}	124.2 (4)	N2—C10—C9	122.9 (6)
C10—N2—Zn1^{ii}	121.1 (4)	N2—C10—H10	118.6
N1—C1—C2	122.6 (5)	C9—C10—H10	118.6
O2—S1—O1—Zn1	−136.6 (4)	C2—C3—C4—C5	−1.4 (16)
O3—S1—O1—Zn1	−18.9 (4)	C8—C3—C4—C5	177.0 (8)
O4—S1—O1—Zn1	100.3 (4)	C1—N1—C5—C4	−1.0 (18)
O2w—Zn1—O1—S1	−4.5 (4)	Zn1—N1—C5—C4	−178.3 (7)
O3w—Zn1—O1—S1	175.0 (4)	C3—C4—C5—N1	1.1 (17)
N1—Zn1—O1—S1	−94.8 (4)	C10—N2—C6—C7	−8(2)
N2^{i}—Zn1—O1—S1	85.7 (4)	Zn1^{ii}—N2—C6—C7	173.6 (13)
O2w—Zn1—N1—C1	−76.9 (8)	N2—C6—C7—C8	7(3)
O3w—Zn1—N1—C1	98.8 (8)	C6—C7—C8—C9	−5(2)
O1w—Zn1—N1—C1	−166.1 (8)	C6—C7—C8—C3	178.6 (11)
O1—Zn1—N1—C1	11.7 (8)	C2—C3—C8—C7	−4.7 (16)
O2w—Zn1—N1—C5	100.3 (8)	C4—C3—C8—C7	177.0 (11)
O3w—Zn1—N1—C5	−84.0 (8)	C2—C3—C8—C9	179.7 (11)
O1w—Zn1—N1—C5	11.1 (8)	C4—C3—C8—C9	1.3 (16)
O1—Zn1—N1—C5	−171.1 (8)	C7—C8—C9—C10	6(2)

C5—N1—C1—C2	1.2 (17)	C3—C8—C9—C10	−177.9 (12)
Zn1—N1—C1—C2	178.5 (7)	C6—N2—C10—C9	8(2)
N1—C1—C2—C3	−1.5 (17)	$Zn1^{ii}$—N2—C10—C9	−173.2 (12)
C1—C2—C3—C4	1.6 (16)	C8—C9—C10—N2	−8(2)
C1—C2—C3—C8	−176.8 (8)		

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*+1, *y*, *z*.

Fig. 1


O1w
O3w
N2i
Zn1
N1
C5
C4
C3
C9
C10
C8
N2
O2w
C1
C2
C7
C6
O1
O4w
O3
S1
O5w
O2
O4