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**Key indicators**

Single-crystal X-ray study  
 T = 295 K  
 Mean  $\sigma(N-C) = 0.007 \text{ \AA}$   
 Disorder in solvent or counterion  
 R factor = 0.038  
 wR factor = 0.122  
 Data-to-parameter ratio = 13.6

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

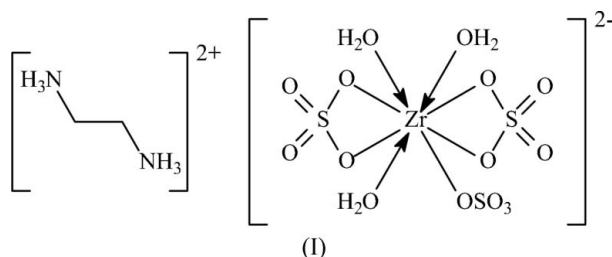
**1,3-Propanediammonium triqua(sulfato- $\kappa O$ )-bis(sulfato- $\kappa^2 O, O'$ )zirconate(IV)**

The eight-coordinate  $Zr^{IV}$  atom in the title compound,  $(C_3H_{12}N_2)[Zr(SO_4)_3(H_2O)_3]$ , has an approximate square-antiprismatic coordination geometry. In the dianion, one of the sulfate groups is unidentate whereas the other two chelate the metal atom. In the crystal structure, cations and anions are linked into a three-dimensional network architecture by hydrogen bonds.

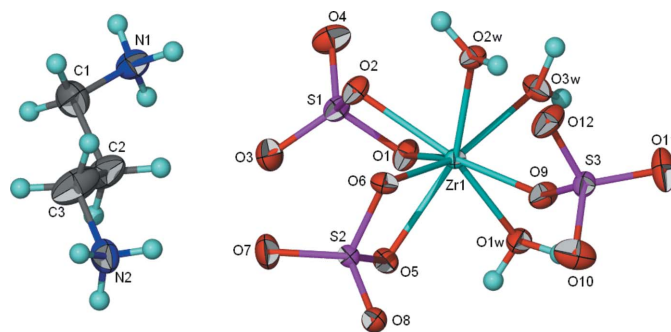
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**Comment**

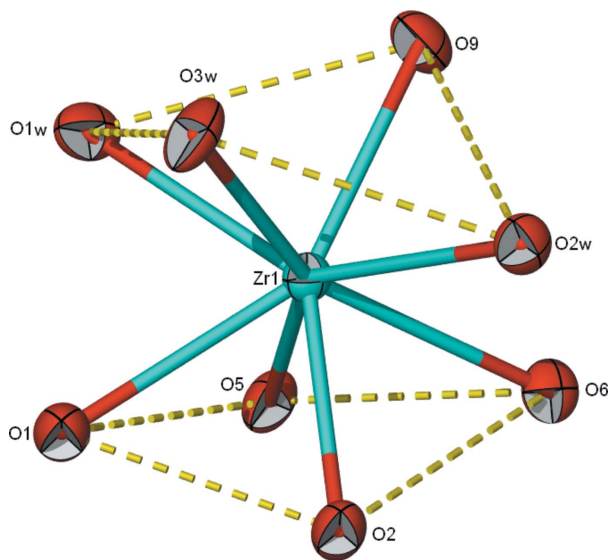
A previous study reported the reaction of zirconyl oxychloride and sulfuric acid in the presence of pyridine, which afforded tetrapyridinium tetrakis(sulfato)zirconate(IV) hydrate. In the anion, the metal atom is chelated by the sulfate groups in a dodecahedral geometry (Fu *et al.*, 2005). Using 1,3-diaminopropanediamine instead of an aromatic amine, the analogous reaction furnishes a tris(sulfate) salt that also has the metal in an eight-coordinate geometry. However, in the title compound, (I), only two sulfate groups engage in chelation (the other is unidentate), and the three other coordination sites are occupied by water molecules (Fig. 1).



Selected bond lengths and angles are given in Table 1. In the dianion the coordination geometry around the  $Zr^{IV}$  approx-



**Figure 1**  
**Fig. 1.** The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level and H atoms as spheres of arbitrary radii. The minor component of disorder is not shown.



**Figure 2**  
**Fig. 2.** A representation of the approximate square antiprismatic coordination geometry of atom Zr1.

imates to a square antiprism; the square planes are, however, only approximately flat (Fig. 2).

The cation and anion are linked by extensive hydrogen bonds into a three-dimensional network (Table 2).

## Experimental

Zirconium oxydichloride octahydrate (0.322 g, 1 mmol) was dissolved in water (10 ml). Sulfuric acid (98%) (0.50 ml, 8.33 mmol) and 1,3-diaminopropane (0.07 ml, 1 mmol) were added. The clear solution was allowed to stand for a week for the formation of colorless block-shaped crystals (in 50% yield based on Zr).

### Crystal data

$(C_3H_{12}N_2)[Zr(SO_4)_3(H_2O)_3]$	$Z = 4$
$M_r = 509.59$	$D_x = 2.160 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 7.9124 (5) \text{ \AA}$	$\mu = 1.18 \text{ mm}^{-1}$
$b = 20.464 (1) \text{ \AA}$	$T = 295 (2) \text{ K}$
$c = 10.4195 (7) \text{ \AA}$	Block, colorless
$\beta = 111.727 (1)^\circ$	$0.16 \times 0.14 \times 0.09 \text{ mm}$
$V = 1567.26 (16) \text{ \AA}^3$	

### Data collection

Bruker APEX area-detector diffractometer	8512 measured reflections
$\varphi$ and $\omega$ scans	3532 independent reflections
Absorption correction: multi-scan SADABS (Sheldrick, 1996)	3079 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.833$ , $T_{\max} = 0.901$	$R_{\text{int}} = 0.034$
	$\theta_{\text{max}} = 27.5^\circ$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 2.5754P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.122$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.14$	$\Delta\rho_{\text{max}} = 0.77 \text{ e \AA}^{-3}$
3532 reflections	$\Delta\rho_{\text{min}} = -0.63 \text{ e \AA}^{-3}$
260 parameters	
H atoms treated by a mixture of independent and constrained refinement	

**Table 1**  
 Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Zr1—O1	2.242 (3)	Zr1—O9	2.097 (3)
Zr1—O2	2.142 (3)	Zr1—O1w	2.207 (3)
Zr1—O5	2.170 (3)	Zr1—O2w	2.202 (3)
Zr1—O6	2.311 (3)	Zr1—O3w	2.171 (3)
O1—Zr1—O2	63.1 (1)	O5—Zr1—O9	98.9 (1)
O1—Zr1—O5	79.4 (1)	O5—Zr1—O1w	75.8 (1)
O1—Zr1—O6	123.6 (1)	O5—Zr1—O2w	137.3 (1)
O1—Zr1—O9	148.2 (1)	O5—Zr1—O3w	148.4 (1)
O1—Zr1—O1w	72.7 (1)	O6—Zr1—O9	81.1 (1)
O1—Zr1—O2w	124.4 (1)	O6—Zr1—O1w	127.7 (1)
O1—Zr1—O3w	78.3 (1)	O6—Zr1—O2w	75.6 (1)
O2—Zr1—O5	90.1 (1)	O6—Zr1—O3w	149.4 (1)
O2—Zr1—O6	76.6 (1)	O9—Zr1—O1w	76.0 (1)
O2—Zr1—O9	148.5 (1)	O9—Zr1—O2w	78.2 (1)
O2—Zr1—O1w	135.5 (1)	O9—Zr1—O3w	88.6 (1)
O2—Zr1—O2w	74.9 (1)	O1w—Zr1—O2w	141.0 (1)
O2—Zr1—O3w	99.5 (1)	O1w—Zr1—O3w	76.3 (1)
O5—Zr1—O6	62.1 (1)	O2w—Zr1—O3w	74.2 (1)

**Table 2**  
 Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1w—H1w1 $\cdots$ O8 <sup>i</sup>	0.85 (3)	2.01 (3)	2.763 (4)	148 (5)
O1w—H1w2 $\cdots$ O10 <sup>i</sup>	0.85 (3)	1.80 (2)	2.624 (5)	164 (5)
O2w—H2w1 $\cdots$ O6 <sup>ii</sup>	0.845 (19)	1.99 (1)	2.834 (4)	178 (4)
O2w—H2w2 $\cdots$ O12	0.85 (3)	1.85 (2)	2.656 (5)	158 (4)
O3w—H3w1 $\cdots$ O8 <sup>iii</sup>	0.85 (3)	1.85 (4)	2.700 (4)	171 (4)
O3w—H3w2 $\cdots$ O11 <sup>iv</sup>	0.85 (3)	1.88 (2)	2.705 (5)	162 (5)
N1—H1n1 $\cdots$ O3 <sup>v</sup>	0.86 (2)	2.19 (3)	2.945 (6)	146 (4)
N1—H1n2 $\cdots$ O10 <sup>vi</sup>	0.86 (2)	2.34 (3)	3.126 (6)	152 (4)
N1—H1n3 $\cdots$ O12 <sup>ii</sup>	0.87 (2)	2.30 (3)	3.007 (6)	139 (4)
N2—H2n1 $\cdots$ O4 <sup>vii</sup>	0.86 (2)	1.99 (2)	2.839 (6)	172 (4)
N2—H2n2 $\cdots$ O6 <sup>viii</sup>	0.84 (2)	2.60 (5)	3.106 (5)	120 (4)
N2—H2n3 $\cdots$ O7	0.86 (2)	2.02 (2)	2.855 (5)	164 (4)

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

The ammonium and water H atoms were refined with distance restraints of  $N-H = O-H = 0.85 (1) \text{ \AA}$  and  $H\cdots H = 1.39 (1) \text{ \AA}$ , and were included in the refinement with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(N,O)$ . The carbon-bound H atoms were positioned geometrically ( $C-H = 0.97 \text{ \AA}$ ) and their displacement factors were similarly tied.

Two of the three carbon atoms of the cation are disordered; the disordered atom occupancies refined to a ratio of 0.66 (1):0.34 (1). The C—N distances were restrained to  $1.45 (1) \text{ \AA}$  and the C—C distances to  $1.50 (1) \text{ \AA}$ ; 1,3-related C $\cdots$ C distances were restrained to  $2.45 (1) \text{ \AA}$ .

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINTE* (Bruker, 2003); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2006).

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