O(20)—Mo(3)—O(22)	74.8 (1)	O(18)—Mo(7)—O(21)	101.2 (2)
O(20)—Mo(3)—O(23)	86.7 (2)	O(18)—Mo(7)—O(22)	101.6 (2)
O(22)—Mo(3)—O(23)	72.0 (1)	O(18)—Mo(7)—O(23)	171.6 (2)
O(4)—Mo(4)—O(8)	104.5 (3)	O(18)—Mo(7)—O(24)	83.9 (2)
O(4)—Mo(4)—O(16)	91.5 (2)	O(21)—Mo(7)—O(22)	141.9 (1)
O(4)—Mo(4)—O(20)	99.5 (2)	O(21)—Mo(7)—O(23)	76.5 (1)
O(4)—Mo(4)—O(22)	87.5 (2)	O(21)—Mo(7)—O(24)	75.6 (2)
O(4)—Mo(4)—O(24)	155.6 (2)	O(22)—Mo(7)—O(23)	76.5 (1)
O(8)—Mo(4)—O(16)	99.9 (2)	O(22)—Mo(7)—O(24)	77.0 (2)
O(8)—Mo(4)—O(20)	98.2 (2)	O(23)—Mo(7)—O(24)	87.7 (1)
O(8)—Mo(4)—O(22)	166.7 (2)		

Intensity data were corrected for Lp effects. The positions of the seven Mo atoms were obtained by direct methods using *MULTAN*80 (Main *et al.*, 1980) and those of O, N and C atoms from subsequent Fourier syntheses. An absorption correction following the *DIFABS* procedure (Walker & Stuart, 1983) was applied at the conclusion of isotropic refinement. A weighting scheme of type $w = w_1.w_2$ with $w_1 = k_1/(a + b|F_o|)^2$ and $w_2 = k_2/(c + d\sin\theta/\lambda + e\sin^2\theta/\lambda)$ was used to obtain flat dependence in $\langle w\Delta F^2\rangle$ *versus* $\langle F_o\rangle$, and *versus* $\langle\sin\theta/\lambda\rangle$ (*PESOS*; Martínez-Ripoll & Cano, 1975); the coefficients used were $k_1 = 0.497$, $k_2 = 1.013$, $a = 4.916$, $b = -0.099$ for $|F_o| \leq 26.20$; $a = 2.513$, $b = 0.000$ for $26.20 < |F_o| \leq 52.81$; $a = 0.791$, $b = 0.028$ for $52.81 < |F_o| \leq 185.61$; $a = -9.380$, $b = 0.079$ for $|F_o| > 185.61$; $c = 23.116$, $d = -129.978$, $e = 185.590$ for $\sin\theta/\lambda \leq 0.37$; $c = 0.885$, $d = -0.553$, $e = 0.000$ for $\sin\theta/\lambda > 0.37$. Non-H atoms were refined anisotropically. The H atoms were positioned from stereochemical criteria and included as fixed contributors. Most calculations were carried out using the *XRAY*76 system (Stewart *et al.*, 1976) running on a MicroVAX 3800 computer. Geometrical calculations were made with *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71830 (59 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1070]

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2,2′-Bipyrimidine (bpm)-Bridged Dinuclear Complexes. 3. Structure of μ-(2,2′-Bipyrimidine-N^1,$N^{1\prime}$:N^3,$N^{3\prime}$)-bis(tetraaquazinc) Disulfate Dihydrate, $[Zn_2(\mu\text{-bpm})(H_2O)_8](SO_4)_2.2H_2O$

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Abstract

The title compound, $[Zn_2(\mu\text{-}C_8H_6N_4)(H_2O)_8]$.$(SO_4)_2.2H_2O$, consists of $[Zn_2(\mu\text{-bpm})(H_2O)_8]^{4+}$ dinuclear units (bpm = 2,2′-bipyrimidine), sulfate counter anions and crystallization water molecules. The bpm ligand acts in a bis-chelating fashion and bridges the two metal ions. Each metal ion is in a distorted octahedral environment created by two N atoms from bpm and four O atoms from the water molecules.

Comment

2,2′-Bipyrimidine is a heterocyclic N-donor ligand which has been the subject of great interest in the last

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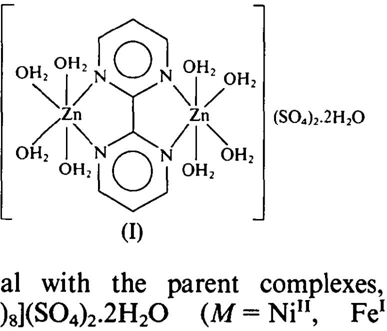
decade because of its versatility as a ligand towards metal ions. It can coordinate to metal centres either as a bis-chelate (De Munno & Bruno, 1984; De Munno, Julve, Nicolò, Lloret, Faus, Ruiz & Sinn, 1993; Brewer & Sinn, 1985; Kaim & Kholmann, 1987; Brewer, Murphy & Petersen, 1987; Julve, De Munno, Bruno & Verdaguer, 1988; Julve, Verdaguer, De Munno, Real & Bruno, 1993) or a chelate (Castro, Julve, De Munno, Bruno, Real, Lloret & Faus, 1992; De Munno, Bruno, Julve & Romeo 1990; De Munno, Julve, Lloret, Faus, Verdaguer & Caneschi, 1993; De Munno, Julve, Verdaguer & Bruno, 1993; De Munno, Nicolò & Julve, 1993; Hiskey & Ruminski, 1986; Ruminski & Petersen, 1985; Matsubayashi, Yamaguchi & Tanaka, 1988).

In recent years, particular attention has been devoted to the synthesis and crystal structure determination of bpm-containing copper(II) complexes. In these compounds, both bidentate (Morgan, Pennington, Petersen, Ruminski, Bennett & Rommel, 1992; De Munno *et al.*, 1990; De Munno, Julve, Verdaguer & Bruno, 1993) and the bis-bidentate (De Munno & Bruno, 1984; Julve *et al.*, 1988; Julve *et al.*, 1993) coordination modes were found. Recently, our attention has focused on dinuclear compounds of bpm with other first-row transition-metal ions to discuss and compare the magnitude of the exchange coupling through the bridging bpm ligand. Along this line, bpm-bridged iron(II) (Andrés, De Munno, Julve, Real & Lloret, 1993) and nickel(II) (De Munno, Julve, Lloret & Derory, 1993) were prepared and characterized.

We report here the synthesis and crystal structure of the bpm-bridged zinc(II) complex $[Zn_2(\mu\text{-bpm})(H_2O)_8](SO_4)_2.2H_2O$, (I). The structure of the compound is composed of $[Zn_2(\mu\text{-bpm})(H_2O)_8]^{4+}$ dinuclear cations, uncoordinated sulfate anions and crystallization water molecules. The compound is isostructural with the parent complexes, $[M_2(\mu\text{-bpm})(H_2O)_8](SO_4)_2.2H_2O$ ($M = Ni^{II}$, Fe^{II}). The molecular geometry and the atom-labelling scheme for the dinuclear unit are depicted in Fig. 1. The sulfate anions and the coordinated and uncoordinated water molecules contribute to the packing by forming an extensive network of hydrogen bonds, as shown in Fig. 2. The dinuclear entity consists of two metal atoms, joined by a bpm molecule, and eight coordinated water molecules. An inversion centre is located halfway through the C(4)—C(4^i) bond of the bpm group. The metal atom is hexacoordinate, being bonded to two N atoms of the bpm ligand and four O atoms of the water molecules. The Zn—N bond lengths are very similar, with a mean value of 2.198 (2) Å. This value is larger than the corresponding distance in the nickel(II) complex [2.115 (2) Å], but smaller than that of iron(II) [2.221 (2) Å]. The four Zn—O distances exhibit significant differences: Zn—O(1) and Zn—O(2) are 2.105 (2) and 2.104 (2) Å, respectively, whereas Zn—O(4) assumes the largest value [2.143 (2) Å] found in compounds of this family. The best equational plane is defined by the atoms N(1), N(2^i), O(1) and O(2) [largest deviation from the mean plane is 0.031 (2) Å for O(2)]. The Zn atom is 0.013 (1) Å out of this plane. Significant deviations from the idealized orthogonal geometry were found at the Zn atom in the five-membered Zn(1)—N(1)—C(4)—C(4^i)—N(2^i) chelate ring [bite angle 75.3 (1)°]. The Zn(1)···Zn(1^i) separation through bpm is 5.848 (1) Å. These values compare well with the those reported for the iron(II) complex [74.9 (1)° and 5.836 (1) Å], but they differ from those of the nickel(II) compound [78.5 (1)° and 5.660 (1) Å].



(I)

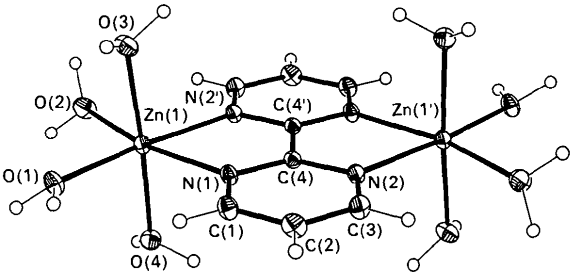


Fig. 1. View of the $[Zn_2(\mu\text{-bpm})(H_2O)_8](SO_4)_2.2H_2O$ complex, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. All H atoms are drawn with uniform isotropic displacement parameters.

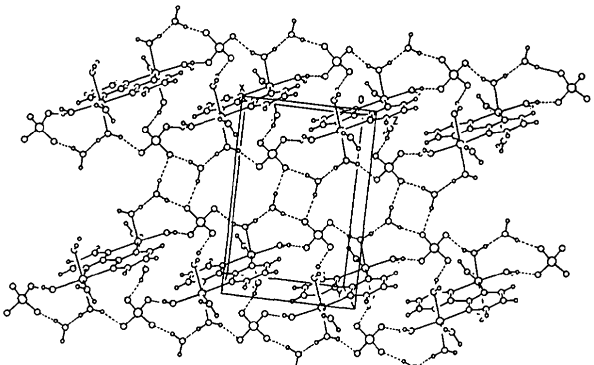


Fig. 2. View of the crystal packing of the complex along the *c* axis, showing some hydrogen bonds.

The C—C and C—N bonds are in the same range as those observed for uncoordinated (Fernholt, Romming & Samdal, 1981), chelating (De Munno *et al.*, 1990; Morgan *et al.*, 1992; De Munno, Julve, Verdaguer & Bruno, 1993) and bis-chelating bpm (De Munno *et al.*, 1984; Julve *et al.*, 1988; Julve *et al.*, 1993). The pyrimidine rings of bpm are planar [maximum deviation from the mean plane of 0.013 (2) Å at C(4)] and the bpm ligand as a whole is also planar. The Zn(1) atom is 0.042 (1) Å out of the bpm plane towards O(3). The dihedral angle between bpm and the equatorial plane of N(1), N(2^i), O(1) and O(2) is 4.8 (1)°.

The dinuclear entities are held together by means of hydrogen bonds involving water molecules and sulfate anions (see Table 3). Some of these hydrogen bonds (corresponding to the first and last three of Table 3) are depicted in Fig. 2, where it may be seen that each sulfate group contributes with all four of its O atoms.

Experimental

Colourless crystals were obtained by evaporation of an aqueous solution of a mixture of bpm and zinc(II) sulfate heptahydrate in a 2:1 molar ratio.

Crystal data

$[Zn_2(C_8H_6N_4)(H_2O)_8]$-
$(SO_4)_2.2H_2O$
$M_r = 661.2$
Monoclinic
$P2_1/c$
$a = 8.128$ (1) Å
$b = 11.818$ (2) Å
$c = 11.719$ (2) Å
$\beta = 91.89$ (1)°
$V = 1125.1$ (3) Å^3
$Z = 2$

$D_x = 1.952$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5$-$15°$
$\mu = 2.45$ mm^{-1}
$T = 298$ K
Polyhedral
0.39 × 0.28 × 0.22 mm
Colourless

Data collection

Siemens *R*/3*mV* four-circle diffractometer
ω-2θ scans
Absorption correction: empirical (ψ scan)
$T_{min} = 0.337$, $T_{max} = 0.384$
2942 measured reflections
2578 independent reflections
2217 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.008$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = -15 \rightarrow 15$
2 standard reflections monitored every 150 reflections
intensity variation: ±2%

Refinement

Refinement on F
$R = 0.0280$
$wR = 0.0296$
$S = 1.059$
2217 reflections
184 parameters
$w = 1/[\sigma^2(F) + 0.0010F^2]$

$(\Delta/\sigma)_{max} = 0.033$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.92$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Zn(1)	0.18638 (3)	0.10734 (2)	0.18822 (2)	0.020 (1)
S(1)	0.72870 (6)	0.28374 (4)	−0.02002 (4)	0.020 (1)
O(1)	0.4229 (2)	0.1778 (2)	0.2122 (1)	0.029 (1)
O(2)	0.1145 (2)	0.1405 (2)	0.3502 (1)	0.036 (1)
O(3)	0.1101 (2)	0.2724 (2)	0.1450 (2)	0.032 (1)
O(4)	0.2741 (2)	−0.0563 (1)	0.2405 (1)	0.027 (1)
O(5)	0.8392 (2)	0.2286 (1)	−0.1005 (2)	0.032 (1)
O(6)	0.6051 (2)	0.3554 (2)	−0.0827 (2)	0.039 (1)
O(7)	0.8275 (2)	0.3614 (2)	0.0546 (2)	0.032 (1)
O(8)	0.6475 (2)	0.1963 (2)	0.0467 (2)	0.036 (1)
O(9)	0.3231 (2)	0.4086 (2)	0.0308 (2)	0.035 (1)
N(1)	0.2033 (2)	0.0613 (2)	0.0073 (1)	0.020 (1)
C(1)	0.3269 (3)	0.0806 (2)	−0.0628 (2)	0.026 (1)
C(2)	0.3176 (3)	0.0472 (2)	−0.1749 (2)	0.027 (1)
C(3)	0.1767 (3)	−0.0089 (2)	−0.2121 (2)	0.026 (1)
N(2)	0.0519 (2)	−0.0283 (2)	−0.1431 (1)	0.020 (1)
C(4)	0.0706 (2)	0.0092 (2)	−0.0374 (2)	0.018 (1)

Table 2. *Selected geometric parameters* (Å, °)

Zn(1)—O(1)	2.105 (2)	Zn(1)—O(2)	2.042 (2)
Zn(1)—O(3)	2.104 (2)	Zn(1)—O(4)	2.143 (2)
Zn(1)—N(1)	2.197 (2)	Zn(1)—N(2^i)	2.199 (2)
N(1)—C(1)	1.338 (3)	N(1)—C(4)	1.335 (3)
C(1)—C(2)	1.371 (3)	C(2)—C(3)	1.382 (3)
C(3)—N(2)	1.337 (3)	N(2)—C(4)	1.320 (2)
C(4)—C(4^i)	1.482 (4)	S(1)—O(5)	1.475 (2)
S(1)—O(6)	1.489 (2)	S(1)—O(7)	1.485 (2)
S(1)—O(8)	1.466 (2)		
O(1)—Zn(1)—O(2)	95.0 (1)	O(1)—Zn(1)—N(2^i)	173.3 (1)
O(1)—Zn(1)—N(1)	98.1 (1)	O(1)—Zn(1)—O(3)	85.7 (1)
O(1)—Zn(1)—O(4)	91.5 (1)	O(2)—Zn(1)—N(2^i)	91.6 (1)
O(2)—Zn(1)—N(1)	166.7 (1)	O(2)—Zn(1)—O(3)	87.4 (1)
O(2)—Zn(1)—O(4)	90.6 (1)	N(1)—Zn(1)—N(2^i)	75.3 (1)
O(3)—Zn(1)—N(2^i)	94.9 (1)	O(4)—Zn(1)—N(2^i)	88.1 (1)
O(3)—Zn(1)—N(1)	91.4 (1)	O(4)—Zn(1)—N(1)	91.2 (1)
O(3)—Zn(1)—O(4)	176.4 (1)	Zn(1)—N(1)—C(1)	128.5 (1)
Zn(1)—N(1)—C(4)	114.7 (1)	C(1)—N(1)—C(4)	116.8 (2)
N(1)—C(1)—C(2)	121.4 (2)	C(1)—C(2)—C(3)	117.3 (2)
C(2)—C(3)—N(2)	121.9 (2)	C(3)—N(2)—C(4)	116.4 (2)
C(3)—N(2)—Zn(1^i)	127.4 (1)	C(4)—N(2)—Zn(1^i)	116.2 (1)
N(1)—C(4)—N(2)	126.1 (2)	N(1)—C(4)—C(4^i)	117.8 (2)
N(2)—C(4)—C(4^i)	116.0 (2)	O(5)—S(1)—O(6)	110.5 (1)
O(5)—S(1)—O(7)	108.7 (1)	O(6)—S(1)—O(7)	106.4 (1)
O(5)—S(1)—O(8)	108.9 (1)	O(6)—S(1)—O(8)	110.8 (1)
O(7)—S(1)—O(8)	111.5 (1)		

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

Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	A	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O(1)	H(1*w*)	O(8)	0.95 (2)	1.78 (2)	2.71 (1)	173 (3)
O(3)	H(5*w*)	O(9)	0.95 (2)	1.81 (2)	2.74 (1)	172 (2)
O(9)	H(9*w*)	O(6)	0.95 (2)	1.82 (2)	2.76 (1)	171 (2)
O(2)	H(4*w*)	O(9^i)	0.95 (2)	1.79 (2)	2.73 (1)	175 (2)
O(1)	H(2*w*)	O(6^i)	0.95 (2)	1.87 (2)	2.81 (1)	175 (2)
O(2)	H(3*w*)	O(5^{ii})	0.94 (2)	1.89 (2)	2.80 (1)	160 (2)
O(4)	H(7*w*)	O(7^{iii})	0.95 (2)	1.80 (2)	2.74 (1)	173 (3)
O(3)	H(6*w*)	O(7^{iv})	0.94 (2)	1.82 (2)	2.71 (1)	156 (2)
O(4)	H(8*w*)	O(5^v)	0.95 (2)	1.82 (2)	2.75 (1)	166 (3)
O(9)	H(10*w*)	O(6^{vi})	0.93 (2)	2.00 (2)	2.91 (1)	164 (2)

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

Lorentz and polarization corrections were applied to the intensity data. The structure was solved by standard Patterson methods and subsequently completed by Fourier syntheses. All non-H atoms were refined anisotropically. The H atoms of the water molecules were located on a ΔF map and refined with constraints, while the H atoms of bpm were set in calculated positions and refined as riding atoms. A common fixed isotropic parameter was assigned to all H atoms. Solution and refinement were performed with the *SHELXTL-Plus* system (Sheldrick, 1990). The final geometrical calculations were carried out with the *PARST* program (Nardelli, 1983). The graphical manipulations were performed using the *XP* utility of the *SHELXTL-Plus* system.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71836 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1055]

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Yttrium(III) and Lanthanum(III) Complexes of a 15-Membered Macrocycle with Three Pendant Carboxymethyl Groups

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Abstract

The yttrium(III) and lanthanum(III) complexes of 9,14-dioxo-1,4,7,10,13-pentaaza-1,4,7-cyclopentadecanetriacetic acid, namely, diaquabis(9,14-dioxo-O^{14}:O^9-1,4,7,10,13-pentaaza-N^1,N^4:N^7-1,4,7-cyclopentadecanetriacetato-O^1,O^4:O^7)diyttrium(III) tetradecahydrate $[Y_2(C_{16}H_{24}N_5O_8)_2(H_2O)_2].14H_2O$, and diaquabis(9,14-dioxo-O^{14}:O^9-1,4,7,10,13-pentaaza-N^1,N^4:N^7-1,4,7-cyclopentadecanetriacetato-O^1,O^4:-O^7)dilanthanum(III) tetradecahydrate, $[La_2(C_{16}H_{24}N_5O_8)_2(H_2O)_2].14H_2O$, have a binuclear structure in which two metal ions are located between two macrocycles. The coordination geometry around each metal ion is distorted tricapped trigonal prismatic. The geometrical properties are defined by the inflexible conformation of the macrocycle and the strong coordination of the O atoms.

Comment

The 15-membered macrocycle with three pendant carboxymethyl groups 9,14-dioxo-1,4,7,10,13-pentaaza-1,4,7-cyclopentadecanetriacetic acid, abbreviated as (15-dten)H_3, forms a novel binuclear gadolinium(III) complex, $[Gd_2(15\text{-dten})_2(H_2O)_2]$, in which two Gd^{III} ions are located between two macrocycles (Inoue, Inoue, Muñoz, Bruck & Fernando, 1993). This finding prompted us to study the structures of the complexes (I) of other rare earth elements with a

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