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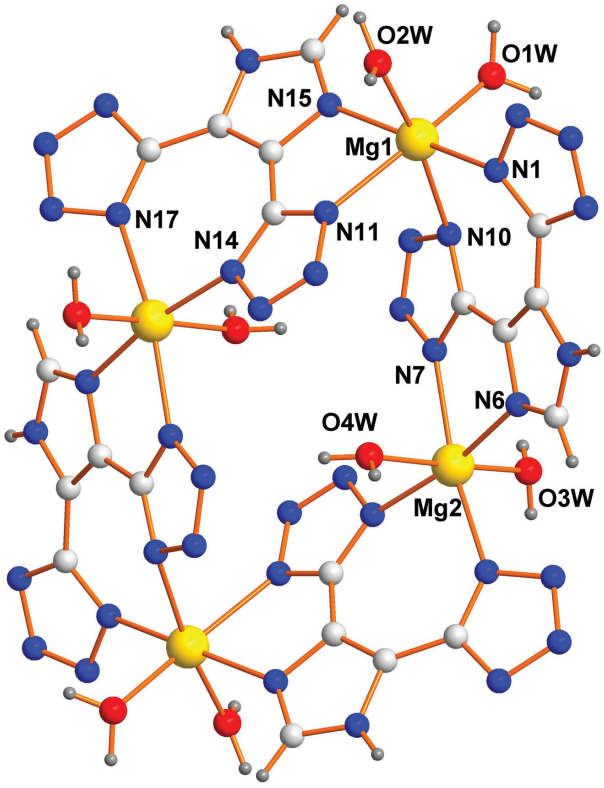
Crystal structure of *cyclo*-[octaaqua-tetrakis(μ$_2$-5,5′-(1*H*-imidazole-4,5-diyl)bis(tetrazol-2-ido)-κ^4*N*,*N*′,*N*″,*N*‴)tetramagnesium(II)], $C_{20}H_{24}N_{40}O_8Mg_4$



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Abstract

$C_{10}H_{12}N_{20}O_4Mg_2$, monoclinic, $P2_1/n$ (no. 14), $a = 7.4934(3)$ Å, $b = 16.1409(5)$ Å, $c = 16.1522(5)$ Å, $\beta = 101.827(3)°$, $Z = 2$, $V = 1912.14(12)$ Å^3, R_{gt} $(F) = 0.0577$, wR_{ref} $(F^2) = 0.1726$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.19 × 0.18 × 0.17 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.20 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, *φ* and *ω*-scans
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	19435, 4702, 0.054
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3974
$N(param)_{refined}$:	328
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

Source of material

A mixture of 5,5′-(1*H*-imidazole-4,5-diyl)bis(2*H*-tetrazole) (1 mmol), magnesium chloride hexahydrate (1 mmol), and *N*,*N*-dimethylformamide (2 mL) were added to deionized water (0.4 mL) in a glass tube. The mixture was carefully stirred, and then frozen the tube, finally sealed with a gas burner. The tube was placed in the oven at 170 °C under the hydro-thermal reaction conditions for 3 days to get good quality white block crystal appeared in the tube wall.

Experimental details

All hydrogen atoms were positioned in accord with geometric considerations.

Discussion

Coordination polymers and metal organic frameworks based on tetrazole linkers have attracted more interests, which reveal that tetrazole groups can possess similar coordination characteristics to those of carboxylate groups [5–8]. These coordination polymers can be applied potentially in gas storage, separation, luminescence and so on. Due to the complexity of reaction, it is difficult to control topologies [9–12].

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Mg1	0.51480(9)	0.17074(4)	−0.01807(4)	0.02125(19)
Mg2	0.31265(9)	0.48915(4)	0.15205(4)	0.01863(18)
O1W	0.3050(2)	0.08815(9)	−0.04491(9)	0.0279(3)
H1WA	0.2595	0.0787	−0.0004	0.042*
H1WB	0.3446	0.0398	−0.0565	0.042*
O2W	0.7266(2)	0.09335(11)	−0.03597(11)	0.0360(4)
H2WA	0.8297	0.1173	−0.0185	0.054*
H2WB	0.7214	0.0864	−0.0891	0.054*
O3W	0.0387(2)	0.48209(9)	0.14629(10)	0.0265(3)
H3WA	−0.0176	0.4720	0.0953	0.040*
H3WB	−0.0025	0.5298	0.1572	0.040*
O4W	0.5868(2)	0.49939(11)	0.14678(11)	0.0331(4)
H4WA	0.5968	0.5082	0.0958	0.050*
H4WB	0.6399	0.4529	0.1593	0.050*
N1	0.5752(2)	0.13363(10)	0.11326(10)	0.0223(4)
N2	0.6398(3)	0.05565(11)	0.12736(11)	0.0282(4)
N3	0.6529(3)	0.03700(12)	0.20668(11)	0.0290(4)
N4	0.5986(3)	0.10101(11)	0.24763(11)	0.0252(4)
N5	0.5246(2)	0.26505(11)	0.29299(10)	0.0247(4)
H5	0.5737	0.2357	0.3361	0.030*
N6	0.3943(2)	0.37097(10)	0.21838(10)	0.0231(4)
N7	0.2827(2)	0.39647(10)	0.04957(10)	0.0220(4)
N8	0.2225(3)	0.39035(12)	−0.03444(11)	0.0300(4)
N9	0.2454(3)	0.31444(12)	−0.05797(11)	0.0286(4)
N10	0.3247(2)	0.26828(11)	0.01014(10)	0.0227(4)
N11	0.6958(2)	0.27948(11)	−0.01469(10)	0.0239(4)
N12	0.7901(3)	0.33071(12)	0.04346(11)	0.0289(4)
N13	0.8097(3)	0.40180(11)	0.00805(11)	0.0275(4)
N14	0.7283(2)	0.39891(10)	−0.07515(10)	0.0222(4)
N15	0.4899(2)	0.20526(10)	−0.14969(10)	0.0227(4)
N16	0.4454(2)	0.22913(11)	−0.28626(10)	0.0234(4)
H16	0.4110	0.2216	−0.3399	0.028*
N17	0.6457(2)	0.44000(10)	−0.27148(10)	0.0221(3)
N18	0.6701(3)	0.47881(12)	−0.34256(11)	0.0285(4)
N19	0.6194(3)	0.42919(12)	−0.40711(12)	0.0309(4)
N20	0.5610(3)	0.35693(12)	−0.38209(11)	0.0275(4)
C1	0.5513(3)	0.15895(12)	0.18877(11)	0.0194(4)
C2	0.4932(3)	0.24014(12)	0.20986(11)	0.0198(4)
C3	0.4112(3)	0.30636(12)	0.16421(11)	0.0193(4)
C4	0.3418(3)	0.32083(11)	0.07479(11)	0.0185(4)
C5	0.4651(3)	0.34321(13)	0.29498(13)	0.0271(4)
H5A	0.4728	0.3741	0.3442	0.032*
C6	0.6616(3)	0.32261(12)	−0.08661(11)	0.0192(4)
C7	0.5618(3)	0.28197(12)	−0.16262(11)	0.0194(4)
C8	0.5338(3)	0.29813(12)	−0.24763(11)	0.0191(4)
C9	0.5800(3)	0.36546(12)	−0.29839(12)	0.0195(4)
C10	0.4224(3)	0.17577(12)	−0.22555(12)	0.0247(4)
H10	0.3659	0.1244	−0.2358	0.030*

Single crystal X-ray diffraction reveals that cyclic title complex contains four magnesium atoms, four ligands and eight water ligands (see the figure). The central of both crystallographically independent magnesium are coordinated with two oxygen atoms from two water molecules, and four nitrogen atoms from two deprotonated organic ligands. The magnesium atom (Mg1) is coordinated by four nitrogen atoms, two (N11, N15) from one imidazole ring and one tetrazole ring, two (N1, N10) from two another tetrazole ring. Similarly, the coordination environment of all central atoms is the same as the Mg1. It is also interesting to note that the complex is centrally. The distant of magnesium atoms and oxygen atoms are from 2.039 Å ~ 2.178 Å, and the bond length between magnesium atoms and nitrogen atoms from 2.161 Å ~ 2.232 Å.

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