Crystal structure of hexaaquamagnesium bis(*p*-nitrophenolate) dihydrate, $Mg(H_2O)_6(O_2NC_6H_4O)_2 \cdot 2H_2O$

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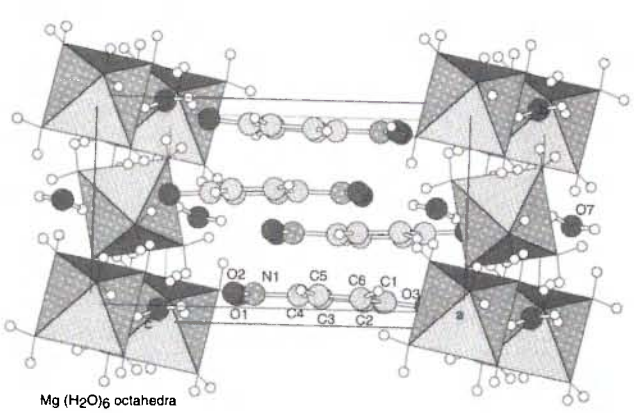

Abstract

$C_{12}H_{24}MgN_2O_{14}$, monoclinic, $P12_1/a1$ (No. 14), $a = 11.833(1)$ Å, $b = 6.681(1)$ Å, $c = 12.396(1)$ Å, $\beta = 93.68(1)°$, $V = 978.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.029$, $wR(F) = 0.044$, $T = 296$ K.

Source of material

2×10^{-3} mole of 4-nitrophenol was dissolved in 20 cm^3 of water containing 1.5×10^{-3} mole $MgCO_3$. The mixture was maintained a few minutes at 333 K and then cooled. The excess $MgCO_3$ was separated and well faceted orange crystals up to 5 mm × 3 mm × 3 mm in size formed after a day. The chemical formula was deduced from the crystal structure determination.

Discussion

The inorganic network is formed by a staggered arrangement of $Mg(H_2O)_6$ octahedra bridged by water molecules through hydrogen bonds. This inorganic layer is located in (001) plane. The organic network contains 4-nitrophenolate anions sandwiched between inorganic layers. The NO_2 and phenoxy groups act as hydrogen acceptors of water molecules. No oxygen atoms of the anion participate to the coordination sphere of Mg atom in contrast to the crystal structure of magnesium bis(4-nitrophenolate) dihydrate in which polymeric chains of edge shared MgO_6 octaedra are built both with water molecules and oxygen atoms of NO_2 group, so inducing herringbone motifs of 4-nitrophenolate anions [1].

Table 1. Data collection and handling.

Crystal:	orange prism, size 0.28 × 0.37 × 0.44 mm
Wavelength:	Ag K_α radiation (0.5608 Å)
μ:	0.95 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius Kappa-CCD, 180 exposures, $\Delta\varphi = 1°$
$2\theta_{max}$:	35.46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1326, 1362
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 1128
$N(param)_{refined}$:	169
Programs:	SIR92 [2], teXsan [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.592(1)	0.087(2)	0.498(1)	0.051
H(2)	4*e*	0.550(1)	0.063(2)	0.315(1)	0.051
H(3)	4*e*	0.876(1)	0.070(2)	0.264(1)	0.051
H(4)	4*e*	0.925(1)	0.079(2)	0.4474(9)	0.051
H(5)	4*e*	0.0858(9)	–0.118(2)	0.1949(9)	0.051
H(6)	4*e*	0.1001(9)	–0.288(2)	0.1332(9)	0.051
H(7)	4*e*	–0.211(1)	–0.029(2)	0.093(1)	0.051
H(8)	4*e*	–0.161(1)	–0.208(2)	0.0625(9)	0.051
H(9)	4*e*	–0.048(1)	0.326(2)	0.0992(9)	0.051
H(10)	4*e*	0.063(1)	0.301(2)	0.1162(9)	0.051
H(11)	4*e*	0.733(1)	–0.035(2)	0.923(1)	0.051
H(12)	4*e*	0.693(1)	0.015(2)	0.820(1)	0.051

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mg(1)	2*a*	0	0	0	0.0194(2)	0.0269(3)	0.0222(2)	–0.0001(2)	0.0002(2)	–0.0025(2)
O(1)	4*e*	0.70788(9)	0.0815(1)	0.66544(7)	0.1005(7)	0.0720(7)	0.0302(5)	0.0027(5)	0.0093(5)	0.0012(4)
O(2)	4*e*	0.88724(8)	0.0778(1)	0.64101(7)	0.0870(6)	0.0687(6)	0.0378(5)	0.0119(5)	–0.0245(5)	–0.0065(4)
O(3)	4*e*	0.68506(6)	0.0473(1)	0.16448(5)	0.0297(4)	0.0457(4)	0.0250(4)	0.0063(3)	–0.0008(3)	–0.0006(3)
O(4)	4*e*	0.07584(6)	–0.1664(1)	0.13019(5)	0.0379(4)	0.0369(5)	0.0267(4)	0.0053(3)	–0.0052(3)	0.0015(3)
O(5)	4*e*	–0.15945(5)	–0.0903(1)	0.04484(5)	0.0253(3)	0.0299(4)	0.0296(4)	–0.0003(3)	0.0057(3)	0.0007(3)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(6)	4*e*	0.00192(5)	0.2460(1)	0.09487(5)	0.0243(3)	0.0360(4)	0.0406(4)	–0.0002(3)	–0.0003(3)	–0.0137(3)
O(7)	4*e*	0.67206(6)	0.0083(1)	0.88330(6)	0.0264(4)	0.0456(5)	0.0317(4)	–0.0025(3)	0.0009(3)	0.0030(3)
N(1)	4*e*	0.78678(9)	0.0810(1)	0.60561(7)	0.0859(7)	0.0277(6)	0.0298(6)	0.0046(5)	–0.0031(5)	–0.0014(4)
C(1)	4*e*	0.76112(9)	0.0823(2)	0.49068(8)	0.0555(7)	0.0233(6)	0.0271(6)	0.0008(5)	–0.0055(5)	–0.0001(4)
C(2)	4*e*	0.6488(1)	0.0828(2)	0.45154(9)	0.0537(7)	0.0394(7)	0.0317(7)	0.0006(5)	0.0121(5)	–0.0003(5)
C(3)	4*e*	0.62332(9)	0.0740(2)	0.34221(9)	0.0324(6)	0.0471(7)	0.0351(6)	0.0020(5)	0.0018(5)	0.0008(5)
C(4)	4*e*	0.70885(8)	0.0638(1)	0.26881(8)	0.0330(5)	0.0238(6)	0.0268(6)	0.0026(4)	–0.0008(4)	0.0011(4)
C(5)	4*e*	0.82213(9)	0.0707(2)	0.31216(9)	0.0313(6)	0.0465(7)	0.0334(6)	0.0030(5)	0.0013(4)	–0.0004(5)
C(6)	4*e*	0.84715(9)	0.0788(2)	0.42125(9)	0.0401(6)	0.0410(7)	0.0365(7)	0.0032(5)	–0.0087(5)	–0.0006(5)

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