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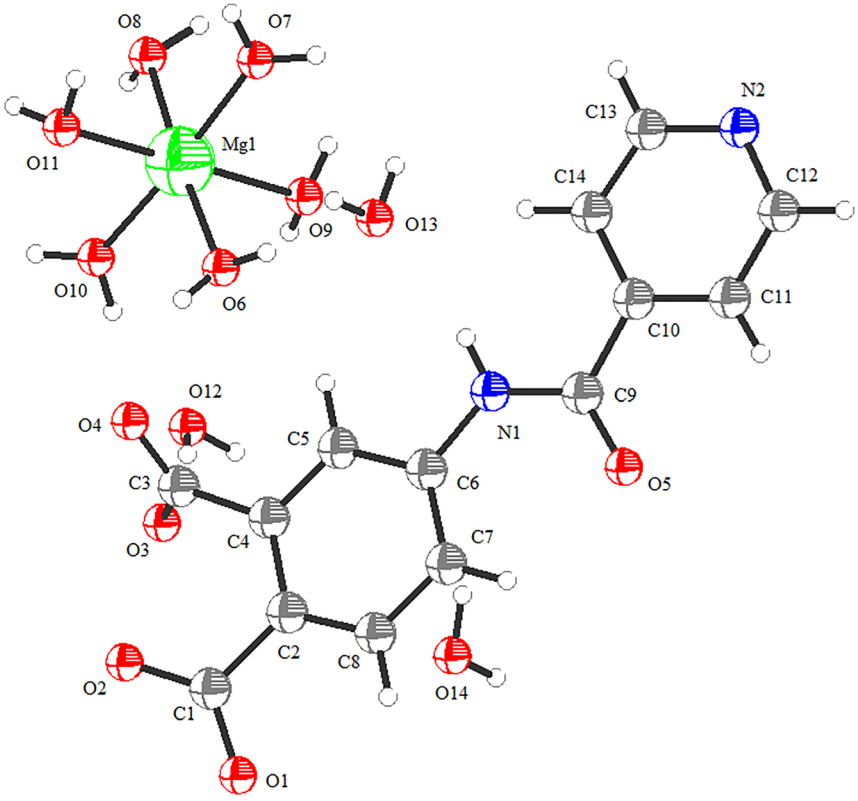
The crystal structure of [hexaaquamagnesium(II)] 4-[(pyridine-4-carbonyl)-amino]-phthalate trihydrate, $C_{14}H_{26}N_2O_{14}Mg$



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Abstract

$C_{14}H_{26}N_2O_{14}Mg$, triclinic, $P\bar{1}$ (no. 2), a = 7.0646(2) Å, b = 8.5008(2) Å, c = 17.2402(2) Å, α = 86.230(2)°, β = 81.415(2)°, γ = 82.768(2)°, V = 1014.45(4) Å^3, Z = 2, $R_{gt}(F)$ = 0.0340, $wR_{ref}(F^2)$ = 0.0904, T = 216(40) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.15 × 0.13 × 0.12 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	1.48 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max}, completeness:	68.2°, 98 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9832, 3636, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3474
$N(param)_{refined}$:	314
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Mg1	1.20332 (7)	1.21782 (5)	0.10440 (3)	0.01783 (15)
O1	0.58862 (17)	0.31442 (12)	0.23873 (7)	0.0277 (3)
O2	0.58280 (18)	0.51095 (13)	0.14656 (6)	0.0305 (3)
O3	0.93963 (15)	0.67067 (13)	0.12554 (6)	0.0238 (2)
O4	0.71985 (15)	0.88297 (12)	0.13287 (6)	0.0234 (2)
O5	0.65949 (17)	0.77752 (13)	0.55220 (6)	0.0277 (3)
O6	1.00959 (17)	1.07575 (14)	0.16622 (7)	0.0283 (3)
H6A	0.915405	1.042562	0.148250	0.042*
O7	1.11861 (17)	1.39461 (13)	0.18091 (6)	0.0263 (3)
H7A	1.055881	1.485366	0.170946	0.039*
H7B	1.126829	1.393866	0.230196	0.039*
O8	1.40785 (17)	1.35563 (14)	0.04790 (6)	0.0299 (3)
H8A	1.467673	1.390538	0.082029	0.045*
H8B	1.495806	1.296926	0.019270	0.045*
O9	1.39710 (15)	1.11249 (12)	0.17829 (6)	0.0233 (2)
H9A	1.469374	1.181182	0.187938	0.035*
H9B	1.475887	1.040203	0.153878	0.035*
O10	1.27367 (18)	1.05305 (13)	0.02313 (6)	0.0297 (3)
H10A	1.274262	1.063944	−0.026855	0.045*
H10B	1.311512	0.954107	0.031540	0.045*
O11	0.98885 (18)	1.32415 (15)	0.03864 (7)	0.0343 (3)
H11A	1.041571	1.352681	−0.007753	0.051*
H11B	0.935965	1.411892	0.058978	0.051*
O12	1.33507 (17)	0.75170 (14)	0.08851 (8)	0.0340 (3)
H12A	1.407191	0.692673	0.117436	0.051*
H12B	1.220836	0.727182	0.104583	0.051*
O13	0.86067 (16)	1.17220 (13)	0.31765 (6)	0.0242 (2)
H13A	0.932456	1.243105	0.324530	0.036*
O14	1.11796 (17)	0.39367 (14)	0.33967 (7)	0.0294 (3)
H14A	1.119108	0.492084	0.348228	0.044*
H14B	1.202882	0.336585	0.363587	0.044*
N1	0.72972 (17)	0.89988 (15)	0.43162 (7)	0.0179 (3)
N2	0.83244 (18)	1.29712 (16)	0.62544 (8)	0.0250 (3)
C1	0.6063 (2)	0.45577 (17)	0.21403 (9)	0.0201 (3)
C2	0.65489 (19)	0.56631 (16)	0.27123 (8)	0.0173 (3)
C3	0.7987 (2)	0.75587 (16)	0.16081 (8)	0.0170 (3)
C4	0.72923 (19)	0.70964 (16)	0.24585 (8)	0.0161 (3)
C5	0.7500 (2)	0.81516 (17)	0.30112 (8)	0.0179 (3)
H5	0.796382	0.913511	0.283764	0.022*
C6	0.70475 (19)	0.78088 (16)	0.38166 (8)	0.0165 (3)
C7	0.6382 (2)	0.63627 (17)	0.40722 (8)	0.0187 (3)
H7	0.610673	0.608965	0.461716	0.022*
C8	0.6126 (2)	0.53256 (17)	0.35160 (8)	0.0189 (3)
H8	0.564467	0.434961	0.369103	0.023*
C9	0.70918 (19)	0.89309 (17)	0.51120 (8)	0.0181 (3)
C10	0.75101 (19)	1.03823 (17)	0.54865 (8)	0.0187 (3)
C11	0.7387 (2)	1.03465 (18)	0.63000 (9)	0.0223 (3)
H11	0.702426	0.943630	0.660865	0.027*
C12	0.7800 (2)	1.16563 (19)	0.66554 (9)	0.0256 (3)
H12	0.770652	1.161752	0.721161	0.031*
C13	0.8415 (2)	1.29985 (19)	0.54737 (9)	0.0253 (3)
H13	0.877167	1.392684	0.517978	0.030*
C14	0.8020 (2)	1.17557 (18)	0.50686 (9)	0.0232 (3)
H14	0.809525	1.183976	0.451284	0.028*
H1	0.763 (3)	0.983 (2)	0.4075 (11)	0.026 (5)*
H6B	0.963 (3)	1.105 (3)	0.2114 (15)	0.046 (6)*
H13B	0.763 (3)	1.222 (3)	0.2981 (13)	0.041 (6)*

1 Source of materials

The title compound has been synthesized as following: 0.286 g (1.0 mmol) 4-[(pyridine-4-carbonyl)-amino]-phthalic acid, 0.080 g (2.0 mmol) NaOH, and 0.2033 g (1.0 mmol) magnesium chloride hexahydrate were together added to a solution of 15 ml water-ethanol (v:v = 1:2) under stirring. The solution was heated to boiling, then cooled to 75 °C and further stirred for 4 h. After cooling to room temperature, further stirring for another 3 h. Filtered and transfered the filtrate to a small beaker, colourless block crystals were received after 10 days.

2 Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.95 Å, O–H = 0.84–0.87 Å). Their U_{iso} values were set to 1.2 U_{eq} or 1.5 U_{eq} of the parent atoms.

3 Comment

In the last few decades, scientists have studied the structures and properties of many magnesium complexes. For example, Hu et al. designed multifunctional Mg^{2+}-based luminescent sensors to detect dangerous explosives, especially the TNT [5]. Chen et al. found magnesium pyrazolyl-indolyl complexes could efficiently catalyze the ring-opening polymerization of L-lactide in the presence of alcohol [6]. We have also synthesized and structurally characterized some Mg(II) complexes [7–10]. In order to enrich the coordination behavior of the magnesium ions. Hence, we present herein the synthetic conditions and single-crystal structural analysis of a new Mg(II) complex using 4-[(pyridine-4-carbonyl)-amino]-phthalic acid, NaOH, and magnesium chloride hexahydrate as materials. The coordination environment of the Mg(II) is shown in the Figure. The Mg(II) complex crystallizes in a triclinic space group $P\bar{1}$ (no. 2). The asymmetric unit of Mg(II) complex contains one $[Mg(H_2O)_6]^{2+}$ cation, one 4-[(pyridine-4-carbonyl)-amino]-phthalate anion, and three lattice H_2O molecules. The Mg atom is coordinated to six O atoms (O6, O7, O8, O9, O10, O11) of six coordinated H_2O molecules, forming a distorted octahedral coordination geometry (Figure). And the O8 and O6 atoms are at the axial positions where the bond angle of O8–Mg1–O6 is 176.44(5)°. The 4-[(pyridine-4-carbonyl)-amino]-

phthalate anion does not coordinate with Mg(II) ion. The Mg–O bond lengths range from 2.0116(12) to 2.1051(12) Å, which agree well with those of other Mg(II) complexes [7–10]. The angles around the Mg(II) ion range from 87.54(5) to 95.12(5)° in the basal plane. The $[Mg(H_2O)_6]^{2+}$ unit is linked with 4-[(pyridine-4-carbonyl)-amino]-phthalate by O–H···O hydrogen bonds. The Mg(II) complex molecules form 2D layered structure by various hydrogen bonds including oxygen atoms of $[Mg(H_2O)_6]^{2+}$ cation unit, lattice water molecules, and 4-[(pyridine-4-carbonyl)-amino]-phthalate anion.

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