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NOTE

Synthesis and Crystal Structure of $Dy(H_2O)_9(1,4\text{-dimethylbenzene})_2Cl_4{\cdot}H_3O$

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One-pot solvothermal reaction of $DyCl_3{\cdot}6H_2O$ with 1,4-dimethylbenzene and water generates $Dy(H_2O)_9(1,4\text{-dimethylbenzene})_2Cl_4{\cdot}H_3O$ that is characterized by single-crystal X-ray diffraction. The compound crystallizes in monoclinic, space group $P2_1/m$ with a = 7.8378(10), b = 25.103(3), c = 7.9068(10) Å, β = 119.3850(10), V = 1355.5(3) Å^3, $C_{16}H_{41}Cl_4DyO_{10}$, Mr = 697.79, Dc = 1.710 g/cm^3, μ(MoK_α) = 3.194 mm^{-1}, F(000) = 702, Z = 2, the final R = 0.0503 and wR = 0.1323 for 2157 observed reflections (I > 2σ(I)). The 2-D supramolecular layer is built up from the compound by hydrogen bonds.

Keywords: Chloride, Crystal, Dimethylbenzene, Dysprosium, Solvothermal.

1,4-Dimethylbenzene (namely, *p*-xylene) is a derivative of benzene and an important raw material for chemical industries[1-3]. As one of the basic chemical products, the *p*-xylene has become the essential element in people's life. Hydrogen bonds have recently been applied in the crystal engineering of metal-organic coordination supramolecular compounds[4,5]. Solvothermal reactions have been employed to prepare single crystals due to the obvious advantages[6,7]. In this paper, we reported the preparation and crystal structure of a dimethylbenzene compound $Dy(H_2O)_9(1,4\text{-dimethylbenzene})_2Cl_4{\cdot}H_3O$. The molecules are linked by hydrogen bonds to form 2-D supramolecular layer.

Synthesis of $Dy(H_2O)_9(1,4\text{-dimethylbenzene})_2Cl_4{\cdot}H_3O$: A mixture of $DyCl_3{\cdot}6H_2O$ (1 mmol, 375 mg), 1,4-dimethylbenzene 3 mL and H_2O_9 mL was sealed in a 25 mL Teflon-lined stainless steel reactor and heated to 175 °C for 5 days and then cooled to room temperature spontaneously. The crystals are washed with H_2O to give the title compound in about 25 % yield (based on $DyCl_3{\cdot}6H_2O$). The crystallographic data is given in Table-1.

X-ray structural determination: The X-ray diffraction data was collected at 296(2) K on a CCD area detector equipped with a graphite-monochromatized MoK_α radiation. Data processing was conducted with a SAINT program. The

TABLE-1
CRYSTALLOGRAPHIC DATA AND STRUCTURE REFINEMENT SUMMARY

Empirical formula	$C_{16}H_{41}O_{10}DyCl_4$	Temperature (K)	296(2)
Formula weight	697.79	Radiation (MoK_α), λ/Å	0.71073
Crystal size/mm^3	0.11 × 0.09 × 0.06	μ/mm^{-1}	3.194
Crystal system	Monoclinic	Dc/(g/cm^3)	1.710
Space group	P2(1)/m	F(000)	702
a/Å	7.8378(10)	Index ranges (h, k, l)	-9/8, -29/29, -9/9
b/Å	25.103(3)	Reflections collected/unique	7498/2234 (R_{int} = 0.0216)
c/Å	7.9068(10)	Observed reflections I > 2σ(I)	2157
β/°	119.3850(10)	Data/restraints/parameters	2157/49/151
V/Å^3	1355.5(3)	Goodness-of-fit on F^2	1.092
Z	2	R, wR (I > 2σ(I))	R1 = 0.0503, wR2 = 0.1323
θ range/°	2.98/25.02	R, wR (all data)	R1 = 0.0517, wR2 = 0.1335
(Δ/σ)max	0	Large diff. peak and hole/(e/Å^3)	1.297 and -1.365

single-crystal structure was solved by direct methods with SHELXS-97 program and refined on F^2 using SHELXL-97 program by full-matrix least-squares methods with equivalent isotropic displacement parameters for all non-hydrogen atoms. The hydrogen atoms were placed according to theoretical models. CCDC: 998313.

X-ray single-crystal diffraction study reveals that the title compound crystallizes in the P2(1)/m space group and is composed of $Dy(H_2O)_9(1,4\text{-dimethylbenzene})_2Cl_4{\cdot}H_3O$ (Fig. 1). Asymmetric unit contains one crystallographically independent Dy center, one 1,4-dimethylbenzene ligand, two Cl^- ions, one H_3O and six coordinated water molecules. The Dy^{3+} center adopts nine-coordinated mode with nine O atoms from nine coordinated water molecules to construct a mono-capped square antiprism. The Dy-O bond lengths are in the normal range of 2.438(6)-2.536(7) Å (Table-2), similar to those observed in the reported Dy-compounds[8-10]. An interesting structure feature of the title compound is hydrogen bond interactions. As illustrated in Fig. 2, because of hydrogen bond interactions among $Dy(H_2O)_9$ groups, Cl^- ions and H_3O molecules, the title compound presents a two-dimensional (2-D) supramolecular layer structure.

TABLE-2
SELECTED BOND LENGTHS (Å)

Bond	(Å)	Bond	(Å)
Dy(1)-OW2	2.438(6)	Dy(1)-OW4	2.468(5)
Dy(1)-OW2#1	2.438(6)	Dy(1)-OW5	2.530(7)
Dy(1)-OW3	2.462(6)	Dy(1)-OW6	2.535(7)
Dy(1)-OW3#1	2.462(5)	Dy(1)-OW7	2.536(7)
Dy(1)-OW4#1	2.468(5)		
Symmetry code: #1 x, -y + 3/2, z			

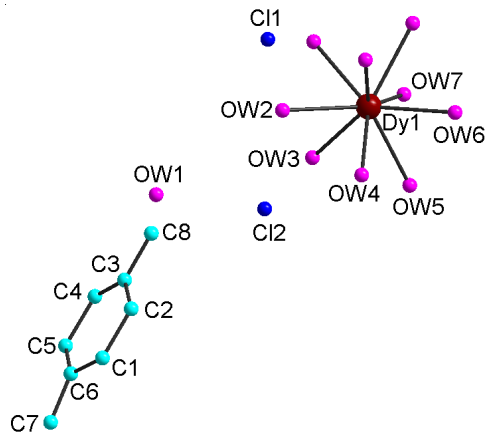

Fig. 1. Coordination environment of Dy1 and the crystal structure of the title compound

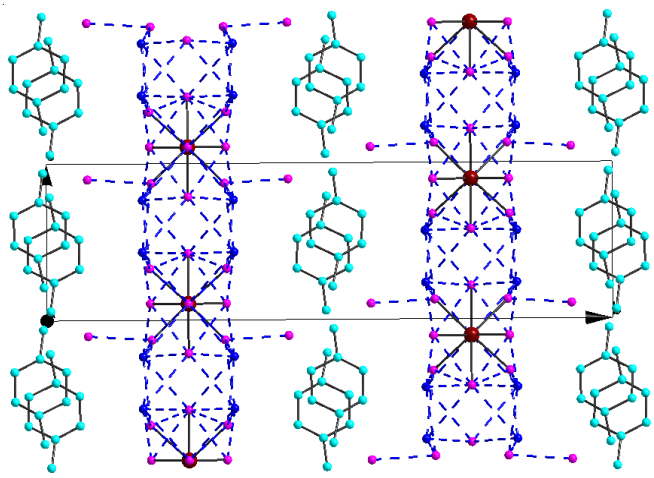

Fig. 2. A packing drawing of the title compound. The dashed lines are hydrogen bonds

Conclusion

One-pot solvothermal reaction gives $Dy(H_2O)_9(1,4\text{-dimethylbenzene})_2Cl_4{\cdot}H_3O$ is characterized by single-crystal X-ray diffraction. The 2-D supramolecular layer is built up from the compound by hydrogen bonds.

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