Asian Journal of Chemistry; Vol. 27, No. 9 (2015), 3519-3520

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2015.18565

NOTE

Synthesis and Crystal Structures of Two Inorganic Compounds

YI ZHU* and HUA-ZE DONG

College of Chemistry and Chemical Engineering, Hefei Normal University, Hefei 230601, P.R. China

*Corresponding author: E-mail: dapdong@163.com

Received: 6 August 2014; *Accepted*: 2 February 2015; *Published online*: 26 May 2015; AJC-17289

Two novel compounds $[Zn(H_2O)_6K_2(SO_4)_2]$ and $[Ni(H_2O)_6K_2(SO_4)_2]$ were synthesized and characterized by single-crystal X-ray. The two crystals are isostructure with monoclinic, space group $P2_1/c$. The oxygen atoms of sulfate in two compounds not only chelate four metal ions but also are all involved in hydrogen bonding as the acceptor, while the coordination water molecules also act as hydrogen donors to form intermolecular hydrogen bonds.

Keywords: Isostructure, Crystal structure.

Electron donor-acceptor molecules play an important role in the understanding of charge-transfer processes. The structural and physical characteristics of these donor and acceptor groups strongly influence the electron-transfer mechanism, which modulate the electron charge-transfer processes[1-4]. Recently, in our laboratory, a series of transition metal compounds have been synthesized and studied[5-7]. In this paper, we will report the synthesis and crystal structure of two compounds, $[Zn(H_2O)_6K_2(SO_4)_2]$ (**1**) and $[Ni(H_2O)_6K_2(SO_4)_2]$ (**2**).

1,2,4-Benzenetricarboxylic acid (H_3btc) were purchased from Aldrich and used without further purification. All the other reagents were commercially available and used as received.

Synthesis: 1,2,4-Benzenetricarboxylic acid (105 mg, 0.5 mmol), KOH (20 mg, 0.5 mmol) and $ZnSO_4$ (0.966 g, 6 mmol) or $NiSO_4$ (1.578 g, 6 mmol) were stirred for 0.5 h in 20 mL of ethanol and water (1:1) mixed solvent then at 160 °C for 4 days in a sealed 30 mL Teflon-lined stainless steel vessel under autogenous pressure. After the reaction mixture was slowly cooled to room temperature, the colorless block crystals of compound **1** (yield 48 %) and the pale green block crystals of compound **2** (yield 32 %) were produced.

Crystal structure determination: Data collection for compounds **1** and **2** were performed on a Bruker SMART 1K CCD diffractometer at 291 K employing graphite-monochromatized MoK_α radiation (λ = 0.71073 Å). The data were collected using SMART and reduced by the program SAINT[8]. The structure was solved by the direct method and refined by full-matrix least squares fitting on 'F^2_{obs}' by SHELXTL-PC[9]. All non-hydrogen atoms were refined with anisotropic thermal parameters. The O-H hydrogen atoms were localized by difference Fourier maps and refined by fixing the bond lengths to 0.840(1) Å. The crystallographic data for compounds **1** and **2** are listed in Table-1.

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINEMENT PARAMETERS FOR COMPOUNDS **1** AND **2**

	Compound **1**	Compound **2**
Formula	$[Zn(H_2O)_6K_2(SO_4)_2]$	$[Ni(H_2O)_6K_2(SO_4)_2]$
M_r	443.83	437.13
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a (Å)	6.114(2)	6.130(2)
b (Å)	12.148(4)	12.188(5)
c (Å)	8.990(3)	9.009(4)
α (°)	90	90.00
β (°)	104.814(4)	105.052(5)
γ (°)	90	90.00
V (Å^3)	645.5(4)	650.0(4)
Z	2	2
ρ_{cald} (g cm^{-3})	2.283	2.234
$\mu(MoK_\alpha)$ (mm^{-1})	2.939	2.522
Temp. (K)	296(2)	296(2)
F(000)	448	444
Data collected	3149	3191
Unique data (R_{int})	1134(0.030)	1147 (0.018)
Observed data [I>2σ(I)]	1101	1082
Final R[I>2σ(I)]	0.0353	0.0242
wR	0.0873	0.0654
S	1.17	1.07
$\Delta\rho_{min}$, $\Delta\rho_{max}$ [e Å^{-3}]	-1.68, 0.67	-0.53, 0.37

TABLE-2
SELECTED BOND DISTANCES (Å) AND ANGLES (°) OF COMPOUND **1**

Bond	Length	Angle	(°)	Angle	(°)
Zn(1)-O(1)W	2.114	O(1)W-Zn(1)-O(2)W	90.11	O(1)-K(1)-O(2)	48.27
S(1)-O(1)	1.454	O(1)W-Zn(1)-O(3)W	91.13	K(1)-O(1)-S(1)	98.18
K(1)-O(1)	3.249	O(3)W-K(1)-O(3)	72.61	K(1)-O(3)W-Zn(1)	137.62
K(1)-O(2)	2.882	O(1)W-K(1)-O(1)	131.96	O(1)-S(1)-O(2)	109.74
K(1)-O(1)W	3.080	O(3)W-K(1)-O(1)W	77.76	O(2)-S(1)-O(3)	108.05

TABLE-3
SELECTED BOND DISTANCES (Å) AND ANGLES (°) OF COMPOUND **2**

Bond	Length	Angle	(°)	Angle	(°)
Ni(1)-O(1)W	2.078	O(1)W-Ni(1)-O(2)W	89.81	O(1)-K(1)-O(2)	46.00
S(1)-O(1)	1.466	O(1)W-Ni(1)-O(3)W	91.50	K(1)-O(1)-S(1)	97.30
K(1)-O(1)	2.965	O(3)W-K(1)-O(3)	79.22	K(1)-O(3)W-Ni(1)	138.36
K(1)-O(2)	2.725	O(1)W-K(1)-O(1)	132.08	O(1)-S(1)-O(2)	108.58
K(1)-O(1)W	3.112	O(3)W-K(1)-O(1)W	78.46	O(2)-S(1)-O(3)	108.25

Compounds **1** and **2** have the same structure in formula of $[M(H_2O)_6K_2(SO_4)_2]$ (M = Zn, Ni) and the major difference lies in the nature of metal ions. All the compounds are heteronuclear polymers consisting of two sulfate ligands, six water molecules, one zinc ion or nickel ion and two potassium ions. As the representative one, the structure of complex **1** is shown in Fig. 1 with the atom-numbering scheme. All com-pounds crystallize in the monoclinic system, $P2_1/c$ space group. The Zn(II) ion lies in an octahedral coordinated geometry, which is completed by six coordination water molecules, while the potassium ions locate in distorted pentagonal bipyramidal configuration, which is completed by five oxygen atoms from four different sulfates and two water molecules (Fig. 2). The axial O2-k-O4 bond angle is 139.2º in **1** while the axial O3-k-O4 bond angle is in the range, about 138.9º in **2** (Tables 1 and 2). Moreover, there are classicly intermolecular hydrogen bonds O–H···O, which reinforces the structural stability of compounds.

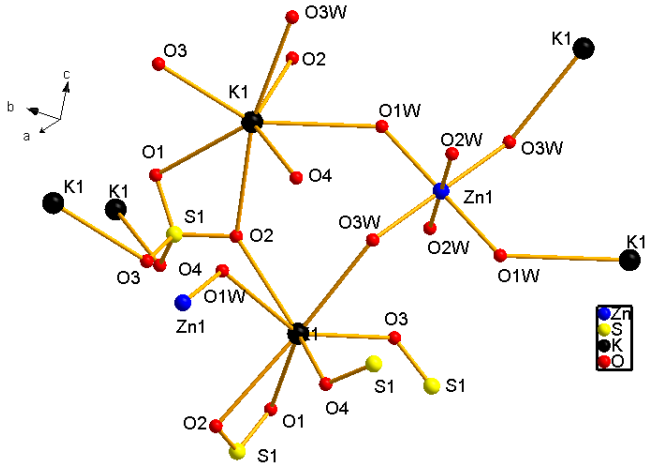

Fig. 1. Atom labelling scheme for $[Zn(H_2O)_6K_2(SO_4)_2]$

Supplementary materials: Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Center with CCDC deposition numbers 1016713-1016714 for compounds **1** and **2**, respectively. Copies of this information may be obtained free of charge *via* www.ccdc.cam.ac.uk/data request.cif.

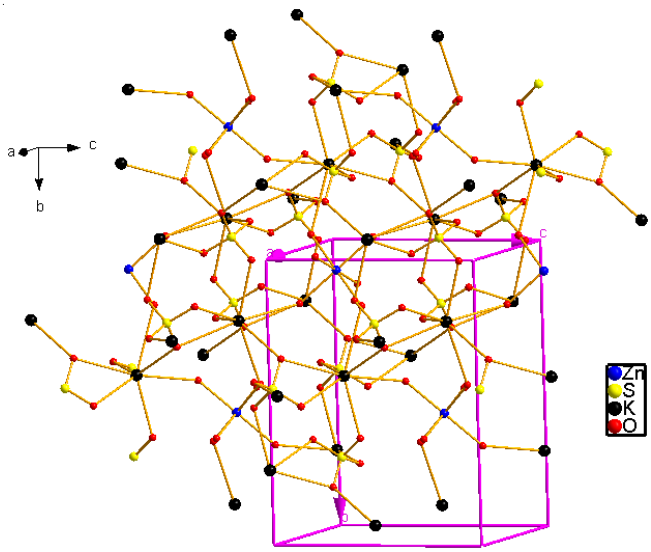

Fig. 2. Packing diagram of compound **1**

ACKNOWLEDGEMENTS

The authors are indebted to Natural Science Foundation of Anhui Province Science Research Projects (1408085MB30), Foundation for scholar of Hefei Normal University (2014136kjc06).

REFERENCES

1. M.J. Hannon and L.J. Childs, *Supramol. Chem.*, **16**, 7 (2004).
2. M. Greenwald, D. Wessely, E. Katz, I. Willner and Y.J. Cohen, *J. Org. Chem.*, **65**, 1050 (2000).
3. M. Greenwald, D. Wessely, I. Goldberg and Y. Cohen, *New J. Chem.*, **23**, 337 (1999).
4. C. Dietrich-Buchecker and J.-P. Sauvage, *Chem. Commun.*, 615 (1999).
5. J.H. Bi, F.X. Xie, X.D. Zhao, Q. Chen and J.D. Xu, *Asian J. Chem.*, **16**, 137 (2004).
6. J.H. Bi, J.M. Song, Z.X. Huang, Y.H. Wang and L.T. Kong, *Asian J. Chem.*, **18**, 2365 (2006).
7. N.L. Hu, H.Z. Dong, Z.X. Huang and F.X. Xie, *Asian J. Chem.*, **17**, 1276 (2005).
8. Siemens, SAINT Version 4 Software Reference Manual (Siemens Analytical X-ray Systems, Inc.), Madison: WI (1996).
9. Siemens, SAINT Version 5 Reference Manual (Siemens Analytical X-ray Systems, Inc.), Madison: WI (1996).