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NOTE

Hydrothermal Synthesis and Crystal Structure of A Special Sodium Sulfate Decahydrate ($Na_2SO_4 \cdot 10H_2O$)

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One new sodium compound sodium sulfate decahydrate is formed by $MnSO_4$, thiophene-2,5-dicarboxylic acid and sodium glutamate has been successfully synthesized. The compound has been characterized by X-ray single-crystal diffraction, compound shows a one-dimensional framework. The 3D supramolecular structure is formed *via* hydrogen bonding connection.

Keywords: Coordination polymer, Crystal structure, Sodium sulfate.

Metal organic frameworks (MOFs) have received much attention in the field of crystal engineering and supramolecular chemistry because of their diverse structures and promising applications in functional materials such as luminescent materials, gas adsorption and magnetism[1-4]. Hydrogen bonds are well suited for the design of polymeric arrangement and crystal engineering because of their important directional interactions and because they can interlink 1-D, or 3-D structures into higher-dimensionality systems[5,6].

All reagent and solvents employed were commercially available and used as received without further purification.

A mixture of $MnSO_4$ (0.5 mmol), thiophene-2,5-dicarboxylic acid (0.45 mmol) and sodium glutamate (0.5 mmol) and distilled water (10 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner 433 K for 24 h, followed by slow cooling to room temperature. Colourless crystals of the compound formed.

Detection method: Diffraction intensity data of the single crystal of the five compounds were collected on a Bruker SMART APEX-II CCD diffractometer equipped with a graphite monochromated MoK_α radiation ($\lambda = 0.71073$ Å) by using a ω-scan mode. All the structures were solved by direct methods and refined by full-matrix least-squares methods on F^2 using the program SHEXL 97[7]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by geometrically calculations and their positions and thermal parameters were fixed during the structure refinement. The crystallographic data and experimental details of structural analyses for coordination polymers are summarized in Table-1. Selected bond and angle parameters are listed in Table-2.

The molecular structure of $Na_2SO_4 \cdot 10H_2O$ is given in Fig. 1. X-ray diffraction analysis revealed that the fundamental building unit consists of metal sodium and sulfate radical as bridging ligands to construct a new coordination polymer. The sodium atom is six-coordinated [NaO_6] in a distorted octahedral manner. The Na(1)-O(3) and Na(1)-O(5) bond lengths are 2.397(3) Å and 2.401(3) Å, respectively. and four oxygen atoms (O11, O12, O13 and O14) from sulfate radical ligands.

TABLE-1
CRYSTALLOGRAPHIC DATA AND STRUCTURE REFINEMENT SUMMARY

Empirical formula	$H_{20}O_{14}SNa_2$
Formula weight	322.20
Crystal system space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 11.517(4) Å; b = 10.386(4) Å c = 12.851(5) Å
Volume (Å^3)	1464.1(9)
θ range for data collection	2.57-25.49
Final R indices [I>2σ(I)]	$R_1 = 0.0577$; $wR_2 = 0.1523$
Z, calculated density (mg/m^3)	4, 1.462
Absorption coefficient (mm^{-1})	0.336
F(000)	680
Limiting indices	$-13 \le h \le 13$; $-12 \le k \le 12$; $-15 \le l \le 15$
Largest diff. peak and hole (e/Å^3)	0.964 and -0.651
Goodness-of-fit on F^2	1.079
R indices (all data)	$R_1 = 0.0675$, $wR_2 = 0.1620$

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (°) FOR $Na_2SO_4 \cdot 10H_2O$

Na(1)-O(4)	2.391(3)	Na(1)-O(5)	2.401(3)
Na(1)-O(3)	2.397(3)	Na(1)-O(10)	2.432(3)
Na(1)-O(9)	2.446(3)	Na(1)-O(8)#1	2.477(3)
O(4)-Na(1)-O(3)	92.52(10)	O(4)-Na(1)-O(5)	92.82(9)
O(4)-Na(1)-O(10)	175.77(9)	O(3)-Na(1)-O(5)	173.12(9)
O(3)-Na(1)-O(8)#1	89.96(9)	O(3)-Na(1)-O(9)	86.94(9)
O(5)-Na(1)-O(10)	83.95(9)	O(4)-Na(1)-O(9)	92.13(9)

Symmetry codes: #1 x, -y+1/2, z+1/2

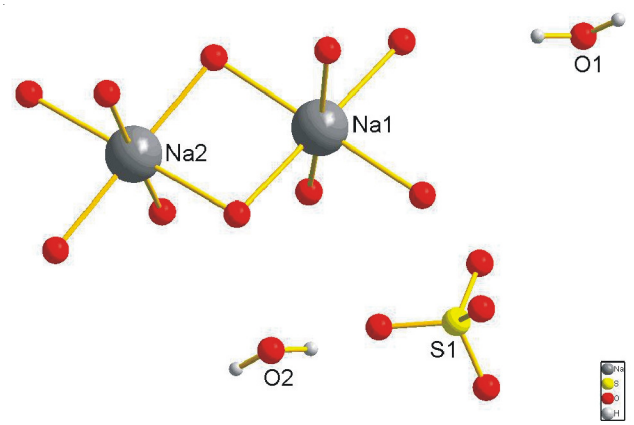

Fig. 1. Molecular structure of sodium sulfate decahydrate at 30 % probability displacement ellipsoids

The S-O bond lengths are 1.452(3) Å, 1.447(3) Å, 1.471(2)Å and 1.446(3) Å, respectively. The hydrogen bonds as follows: O(3)-H(6W)···O(7)#6, [O···O = 2.813(3)Å, O–H···O = 165.6°]; O(4)-H(7W)···O(6)#1, [O···O = 2.796(4)Å, O–H···O = 161.8°]; O(5)-H(10W)···O(2)#9, [O···O = 2.801(3)Å, O–H···O = 170.4°]; O(6)-H(12W)···O(4)#9, [O···O = 2.835(4)Å, O–H···O = 165.0°]; O(7)-H(14W)···O(3)#6, [O···O = 2.813(3)Å, O–H···O = 165.8°]; O(9)-H(17W)···O(2), [O···O = 2.882(4)Å, O–H···O = 175.3°]. Symmetry codes: #1 x, -y + 1/2, z + 1/2; #6 -x, -y + 1, -z; #9 -x + 1, y-1/2, -z + 1/2. The chains are further assembled by the intermolecular hydrogen bonding interaction leading to the formation of a 3D framework (Fig. 2).

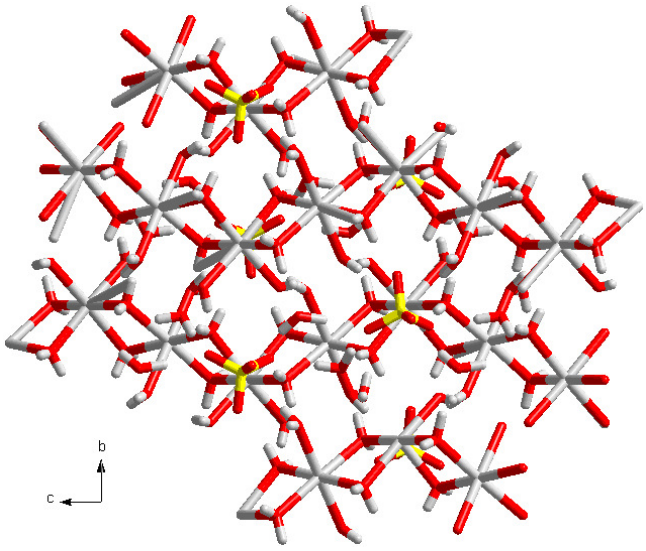

Fig. 2. 3D structure formed *via* hydrogen bonding interactions

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