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NOTE

Synthesis and Crystal Structure of 3D Network Barium(II) Complex with Succinic Acid

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A new dimensional alkaline-earth metal-organic framework complex, [Ba(L)]n (L = succinic acid) (**1**), has been synthesized by hydrothermal method and characterized by single crystal X-ray diffraction studies. Structural determination reveals that the coordinated geometry of Ba is a capped distorted pentagonal bipyramidal geometry. Each metal ion is coordinated by eight O from six different carboxylate groups. While each succinate acid ligand coordinates six alkaline earth metal centers to form 3D network.

Key Words: Succinic acid, Supramolecule.

Recent studies have witnessed great interesting in hybrid inorganic-organic compounds probably due to their fascinating architectures and potential applications such as catalysis[1,2] materials[3-5] and biochemistry[6]. In such materials, metal dicarboxylates are particularly interesting because they exhibit wide application in open-framework structures, as they act as building blocks for porous net works[7].

Alkaline-earth metal complexes is an emerging area of interest as they have some advantages for the application in materials science compared with transition metal ions. They mostly are nontoxic, cheap and generally soluble in aqueous preparation[8]. And alkaline-earth metal dicarboxylates, as new type functional materials, occur emerging interest not only because a wealth of topology and the accumulation variety of ways, but also tremendously potential applications such as optical properties, magnetic properties, porous materials and so on[9]. Among the alkaline-earth metal ions, barium(II) ion has larger ionic radius and therefore could adopt a higher coordination number and may lead to interesting topological arrangement.

This paper focuses on the preparation and structural characterization of an alkaline-earth-metal-based complex that affords a new 3D structure involving barium and Succinic acid.

All chemicals purchased were of analytical grade and used without further purification. Elemental analysis was measured on a Perkin-Elmer 1400C analyzer. Infrared spectra were recorded on a Nicolet Magna 750 FI-IR spectrometer using KBr pellets in the range of 4000-400 cm^{-1}.

Synthesis of 3D network: A mixture of $BaNO_3$ (0.522 g, 2 mmol), succinic acid (0.236 g, 2 mmol) and 15 mL H_2O was placed in a 23 mL Parr Teflon-lined autoclave at 160 ºC for 3 days. After being slowly cooled to the room temperature, colourless block crystals of **1** were isolated (yield: 46.7 %). Anal. calcd. (%) for $C_4H_4O_4Ba$: C, 18.96; H,1.59. Found (%): C, 18.93; H, 1.61. IR (KBr, cm^{-1}): 1553 (s), 1415 (s), 1330 (s), 1290(m), 1036 (m), 929 (m), 803 (w).

Crystal structure determination: A single crystal of compound with dimensions of 0.21 mm × 0.32 mm × 0.38 mm was selected for the crystallographic data collection at 291(2)K and structure determination on a Bruker SMART CCD-4K diffractometer employing graphite-monochromated MoK_α radiation (λ = 0.71073 Å). A total of 1327 reflections were collected in the range of $3.30^\circ \le \theta \le 25.0^\circ$, of which 156 reflections were unique with R_{int} = 0.032. The data were collected using SMART and reduced by the program SAINT[10]. All the structures were solved by direct methods and refined by full-matrix least squares method on F^2_{obs} by using SHELXTL-PC[11] software package. Non-hydrogen atoms were placed in geometrically calculated positions. Hydrogen atoms were added according to theoretical model. The final full-matrix least-squares refinement including 25 variable parameters for 156 reflections with $I > 2\sigma(I)$ and converged with unweighted and weighted agreement factors of

$$R_1 = \frac{\Sigma(||F_0| - |F_c||)}{\Sigma|F_0|} = 0.021 \quad (1)$$

TABLE-1
SELECTED BOND DISTANCES (Å) AND ANGLES (°)

Ba-Ba1#	4.5875(4)	Ba-O1	2.865(5)	Ba1-O1#c	2.702(5)
Ba1-O1#d	2.702(5)	Ba1-O1#g	2.865(5)	Ba1-O1#j	2.865(5)
Ba1-O1#o	2.702(5)	Ba1-O1#t	2.865(5)	Ba1-O1#z	2.702(5)
C1-C2	1.51(8)	C1- C3	1.52(8)	C2-C3	1.53(11)
O1-Ba1-O1#c	88.53(10)	O1#j-Ba1-O1#t	148.56(10)	O1-Ba1-O1#g	148.56(10)
O1-Ba1-O1#j	148.56(10)	O1-Ba1-O1#o	88.53(10)	O1#d-Ba1-O1#z	176.82(15)
O1-Ba1-O1#z	114.12(15)	O1#c-Ba1-O1#d	90.04(1)	O1#g-Ba1-O1#t	148.56(10)
O1#c-Ba1-O1#j	114.12(15)	O1#c-Ba1-O1#o	176.82(15)	O1#d-Ba1-O1#t	114.12(15)

Symmetry codes: c = 5/4+y, 5/4-x,-1/4 + z; d = x,-y, -z; g = 5/4-y, 5/4-x, -1/4-z; j = 3/4 + y, 5/4-x, - 1/4 - z; o = 3/4-y, 5/4-x, -1/4 + z; t =2-x, 1/2-y, z; z = 2-x, 1/2 + y, -z.

and $$wR_2 = \left\{\frac{\Sigma[w(F_0^2 - F_C^2)^2]}{\Sigma w(f_0^2)^2}\right\}^{1/2} = 0.0479 \quad (2)$$

where $w = 1/[\sigma^2(F_0^2) + (0.0068P)^2 + 7.9697P]$ and $P = (F_0^2 + 2F_C^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.62 and -0.54 e/Å^3, respectively.

The selected bonds are shown in Table-1 and a perspective view of the present complex with the atom-numbering scheme is shown in Fig. 1. The packing diagram of the present complex is shown in Fig. 2.

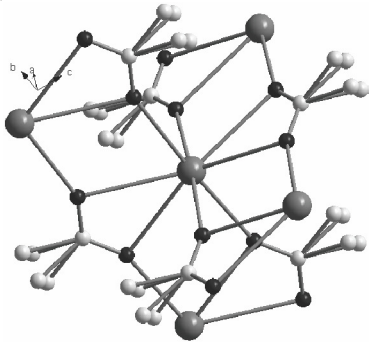

Fig. 1. Coordination environment of Ba ion

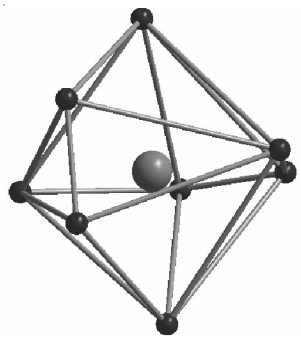

Fig. 2. View of polyhedron around the Ba center

Complex **1** crystallizes in the $I4_1/amd$ space group. As depicted in Fig. 1, in each symmetric unit of 1 there are one Ba^{2+} ion and one succinic acid and the barium atom located in eight-coordinated circumstance with the capped distorted pentagonal bipyramidal geometry. The center of barium ion is coordinated by eight O atoms from different six ligands. (Fig. 2). The Ba-O bond distances range from 2.702(5) to 2.865(5) Å, which are in agreement with the Ba-O bond lengths observed in other alkaline earth metal carboxylate complexes[12]. All the carboxylate oxygen atoms are doubly bridged. Each sucinnic acid coordinates to six Ba metal centers through the carboxylato oxygen atoms, adopting fascinating μ_6-η^4 coordinated model to afford 3D network complex (Fig. 3).

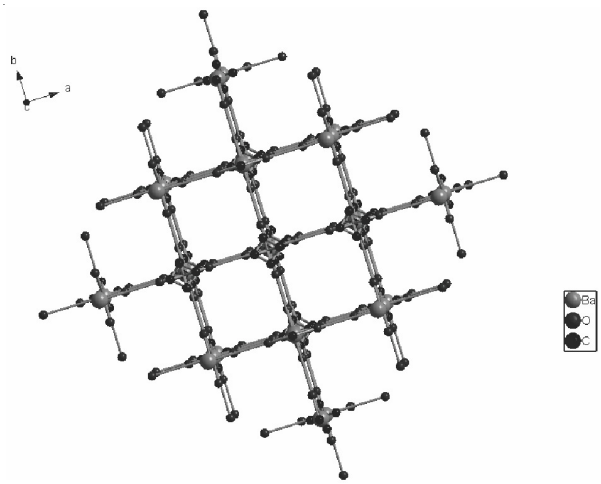

Fig. 3. 3D supromolecular network down the c-axes

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