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Preparation, Structure Characterizations and Thermal Analysis of $[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2] \cdot (CH_3CH_2OH) \cdot 2.5H_2O$ Complex

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Abstract: The $Ba_2(H_2TNP)_2(OH)_2(H_2O)_2] \cdot (CH_3CH_2OH) \cdot 2.5H_2O$ (H_2TNP: 3,5-dihydroxy-2,4,6-trinitrophenolate) complex was prepared by mixing barium nitrate solution and sodium H_2TNP solution, which was obtained through the reaction of sodium bicarbonate and trinitrophloroglucinol. The molecular structure was characterized by using element analysis, IR analysis and X-ray diffraction. The complex 1 crystallizes in tetragonal system, space group I4(1)/amd, with crystal parameters $a = 19.911(3)$ nm, $b = 19.911(3)$ nm, $c = 13.265(3)$ nm, $V = 5259.0(15)$ nm^3, $Z = 8$, $R = 0.0335$ with 1092 reflections. The structure consists of a barium dimmer unit held together by two oxygens bridging. Each Ba atom is 10-coordinated with eight oxygen atoms from four H_2TNP, an oxygen atom from water molecule and a coordinated hydroxy ion. In complex the metal atoms are linked via oxygen bridges to infinite networks structure. Uncoordinating water molecules and ethanol molecules are present in the cavities of the crystal structure. The thermal decomposition of the complex is studied using differential scanning calorimetry (DSC), thermogravimetry-derivative thermogravimetry (TG-DTG) and IR techniques. The final residue is $BaCO_3$ at 550 ℃.

Key words: physical chemistry; trinitrophloroglucinate; barium complex; crystal structure; thermal analysis

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1 Introduction

2,4,6-Trinitro-1,3,5-benzenetriol (trinitrophloroglucinol, TNPG) has been used in chemical industry as an ingredient for making dyes and explosive industry as an ingredient for priming composition, percussion caps and detonator formulations[1]. Some metal salts of the TNPG are efficient primary explosives and are also important uses in the military and commercial industry[2]. The crystallographic structures of potassium salts of TNPG have been reported[3]. Gorshev et al.[4] have reported the synthesis and the combustion behavior of salts of TNPG, and results obtained for the bivalent alkali-earth metal salts are that the burning rate grows with increasing metal atomic weight and radius: Mg < Ca < Sr < Ba. Besides, the burning rate of the salts is found to be highly dependent on the position of the metal atom in the molecule. The research of the structure-properties relationship for the energetic materials is an object of permanent interest.

The barium complexes have also been given considerable attention because of their structural, novel electronic and properties. More recently, a very large variety of barium *b*-diketonate compounds have been reported. This may, in part, be due to the requirement for a volatile barium source for utilization in metal organic chemical vapor deposition techniques[5-7]. Melanie A. and Pierce-Butler have reported the structure of the barium salt of 2,4,6-trinitro-1,3-benzenediol (TNR) monohydrate[8]. Hu Rongzu et al. have reported kinetic parameters and mechanism of thermal decomposition reaction behavior of $Ba(TNR) \cdot H_2O$ and $Pb_{0.25}Ba_{0.75}(TNR) \cdot H_2O$ complex[9,10]. Unlike many salts of nitro derivatives of mono- and dihydroxybenzenes, which structure, explosive and burning properties are fairly known[11-13], metallic salts of TNPG still remain poorly explored. Considering their importance remarkably, little is known about the nature of Ba-TNPG compound. Structure determinations have been carried out on the title complex as part of a program of research into the structural properties of primary explosives.

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At the same time, it is of particular interest to investigate any correlations between molecular geometry and sensitivity. In this paper, the authors report the structure characterizations and the thermal decomposition mechanism on the mono-substituted barium complex of TNPG, $[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2]\cdot(CH_3CH_2OH)\cdot 2.5H_2O$.

2 Experimental

2.1 Preparation of Materials

The synthesis of the TNPG was carried out as described in the literature[14].

TNPG was dissolved in distilled water with vigorously stirring and heated to about 60 ℃. Sodium bicarbonate was added to the solution, and carbon dioxide released during the reaction. Sodium H_2TNP (3,5-dihydroxy-2,4,6-trinitrophenolate) solution ($0.5\ mol\cdot L^{-1}$) was obtained at the end of the reaction. An aqueous solution of barium nitrate ($0.5\ mol\cdot L^{-1}$) was added very slowly to the sodium H_2TNP at 60 - 70 ℃ with continuous stirring. Then, the reacting mixture was kept at 65 ℃ for 40 min to complete the reaction. Finally, cooled the solution to ambient temperature and filtered to obtain yellow crystal product barium H_2TNP. Single crystals suitable for X-ray measurement were obtained from the mother liquid and ethanol solution with pH = 4 - 5 by slow evaporation at room temperature for 7 days.

Anal. calcd. (found) for $C_7H_{10.5}BaN_3O_{12.75}$: C, 17.57% (17.51%), H, 2.20% (2.18%), N, 8.79% (8.72%). IR (ν, KBr pellets, cm^{-1}): 3446 (m, ν O—H), 1538 (s, ν Ph—OH), 1460 (w, δ C=C), 1252 (s, ν_s NO_2), 936w, 762w, 713w (δ ring bone).

2.2 Physical measurements

Elemental analyses for H, C and N were performed at a Flash EA 1112 full-automatic trace element analyzer. Infrared spectrum (400 - 4000 cm^{-1}) was measured on KBr pellet with a Bruker Equinox 55 FT-IR spectrophotometer. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were carried out on a model Pyris-1 differential scanning calorimeter and a model Pyris-1 thermogravimeter analyzer respectively using dry oxygen-free nitrogen as atmosphere with flowing speed of 20 $mL\cdot min^{-1}$. The sample mass was about 1.0 mg sealed in aluminum pans using α-Al_2O_3 as reference sample for DSC with heating rate of 10 ℃ $\cdot min^{-1}$ from 50 ℃ to 500 ℃, held in platinum pans for TG with heating rate of 10 ℃ $\cdot min^{-1}$.

The diffraction data collection were performed on a Rigaku RAXIS-RAPID X-ray single-crystal diffractometer using Mo Kα radiation (λ = 0.071073 nm) with a graphite monochromator. The corrections for Lorentz Polarization factors and empirical absorption were applied to the original data. The structure was solved by direct methods and refined by least-squares on F^2 using SHELX-98 program[15]. Full-matrix least-squares anisotropic refinements for all non-hydrogen atoms were completed. All of the hydrogen atoms were identified but not included in the refinement process. Crystallographic data and experimental details for structure analyses are listed in Table 1.

Table 1 Crystal data and structure refinement for the title complex

items	values
empirical formula	$C_{14}H_{21}Ba_2N_6O_{25.5}$
formula weight	956.04
crystal system	tetragonal
unit cell dimensions /nm	a = 19.911(3), b = 19.911(3), c = 13.256(3)
volume /nm^3	5259.0(15)
Z	8
temperature /K	293(2)
space group	I4(1)/amd
wavelength /nm	0.071073
density (calculated)/$g\cdot cm^{-3}$	2.402
absorption coefficient /mm^{-1}	3.106
$F(000)$	3704
crystal size /mm	0.40 × 0.30 × 0.20
theta range for data collection	2.76 to 25.03
limiting indices	$-23\leq h\leq 23$, $-16\leq k\leq 16$, $-15\leq l\leq 15$
reflections collected	15456
independent reflections	1244[R(int) = 0.0478]
data / restraints / parameters	1244 / 0 / 117
goodness-of-fit on F^2	1.037
final R indices [$I>2\sigma(I)$]	$R1$ = 0.0335, $wR2$ = 0.0945
rindices (all data)	$R1$ = 0.0387, $wR2$ = 0.0970
largest diff. peak and hole (e/nm^3)	1.208 and -0.747

3 Results and discussion

3.1 The crystal structure of the title complex

The single crystal X-ray diffraction performed on the compound reveals that the title complex consists of the 2D network built from centrosymmetric dimeric

$[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2]$ units, and one uncoordinating CH_3CH_2OH and two point five uncoordinating H_2O molecules are also present in the complex, so the chemical formula of title complex should be written as $[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2]\cdot(CH_3CH_2OH)\cdot 2.5H_2O$. The barium coordination mode in the crystal is shown in Fig. 1, together with the numbering scheme (The H atoms are omitted for clarity). Selected bond lengths and angles around barium ions are listed in Tables 2 and 3.

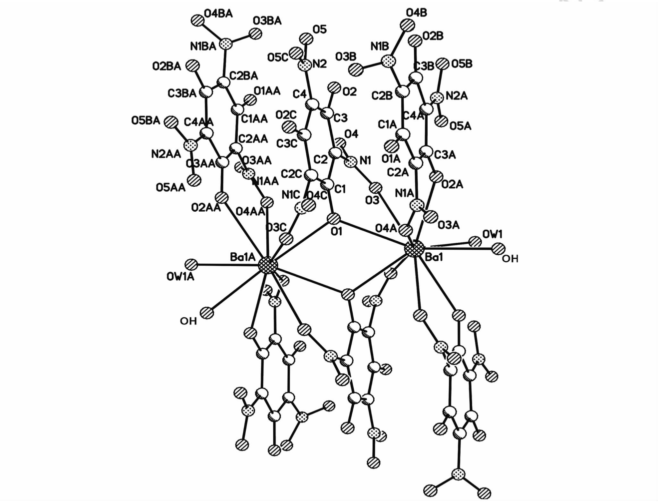

Fig. 1 Complex cation coordination modes of the title complex and numbering scheme

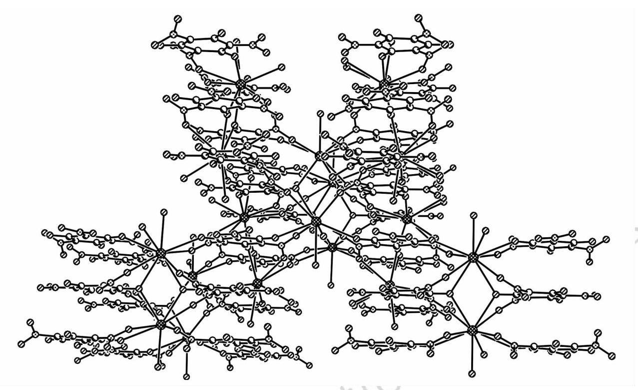

Fig. 2 Extended coordination scheme of barium ion in the title complex

In complex with large atomic radii metal of barium, a new coordination mode has been observed for H_2TNP ligand. The barium atoms are ten coordinated with eight oxygen atoms from four H_2TNP ligand anions, one oxygen atom (OW1) from one water molecule and one O atom (OH) from one hydroxy ion. It is noted that two oxo bridges are formed between the adjacent Ba^{2+} ions by the two phenolic hydroxyl oxygen atoms from two H_2TNP anions forming a binuclear structural unit. Hydroxy ions acting as anionic supporting ligands link with the Ba^{2+} ions. The coordination atoms form four six-memberd chelate rings around each Ba^{2+} ion, and every two neighboring Ba^{2+} ions and their oxo atoms form a four-memberd ring. All of the six-memberd rings are not planar. The bond distances of Ba—O lie in the expected region as observed for the distances 2.710 - 2.989 nm (Table 2), which are similar to the sums of the appropriate ionic radii: $Ba^{2+} + O^{2-} = 2.87$ nm[8], well within the ranges reported for comparable barium complexes[5,16,17]. Furthermore, in complex, the bond distances of Ba—O(1) and Ba—OH are rather short, demonstrating the predominantly electrostatic nature of Ba—O bonds in the mono-substituted barium complex of TNPG. And short Ba—Ba distance is found (4.5969 nm).

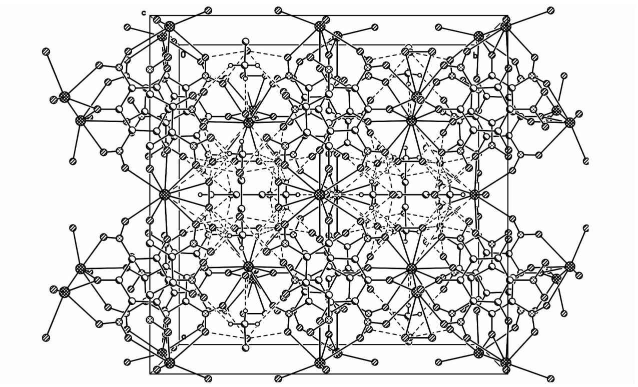

Fig. 3 Packing arrangement of the title complex

Table 2 Selected bond distances (nm)

Ba(1)—O(1)	2.710(2)	Ba(1)—OW1	2.846(10)
Ba(1)—O(1)#1	2.710(2)	Ba(1)—O(4)#3	2.879(4)
Ba(1)—OH	2.753(6)	Ba(1)—O(4)#2	2.879(4)
Ba(1)—O(2)#2	2.805(4)	Ba(1)—O(3)	2.989(4)
Ba(1)—O(2)#3	2.805(4)	Ba(1)—O(3)#4	2.989(4)

Note: symmetry transformations used to generate equivalent atoms: #1: $-x+1/2$, $-y+1/2$, $-z+1/2$; #2: $-x+1/4$, $-y+3/4$, $z-1/4$; #3: $x+1/4$, $-y+3/4$, $z-1/4$; #4: $x+1/4$, $-y+1/2$, z. Same as Table 3

Table 3 Selected bond angles (°)

O(1)—Ba(1)—O(1)#1	63.98(15)	O(2)#2—Ba(1)—O(3)	135.26(9)
O(1)—Ba(1)—OH	142.37(12)	O(3)#2—Ba(1)—O(3)	71.24(11)
O(1)—Ba(1)—O(2)#2	143.42(11)	O(4)#3—Ba(1)—O(3)	109.64(11)
O(1)#1—Ba(1)—O(2)#2	79.79(11)	O(4)#2—Ba(1)—O(3)	159.12(11)
OH—Ba(1)—O(2)#2	67.91(8)	O(2)#2—Ba(1)—O(3)#4	67.78(8)
O(1)—Ba(1)—O(3)	53.91(8)	N(1)—O(3)—Ba(1)	135.4(3)
O(2)#3—Ba(1)—O(4)#3	55.29(9)	C(1)—O(1)—Ba(1)	121.99(8)
O(2)#2—Ba(1)—OW1	85.66(11)	Ba(1)—O(1)—Ba(1)#1	116.02(15)

Acting as a multidentate ligand, H_2TNP anion is coordinated with Ba^{2+} ion via four oxygens of two nitro groups and three phenolic hydroxyl oxygen atoms, and two oxygen atoms on N(2) is not coordinated with Ba^{2+} ion. TNPG is an acidic compound when it reacts with $NaHCO_3$, O(1)H on the benzene ring of TNPG gives its H^+ ion out and forms a univalent anion H_2TNP. Ba^{2+} ions combine directly with O(1) on the benzene ring of TNPG to form a special coordination bond resulted in the title complex more stability. The phenolic hydroxyl oxygen atom O(1) is coordinated with two different Ba^{2+} ion, which is served as oxygen bridge atom. Three nitro groups are not coplane with the benzene ring, one of which is not coordinated with the Ba^{2+} ion. One hydroxy ion is also coordinated with the Ba^{2+} ion in order to ensure the electrical neutrality of the system.

The metals are linked via oxygen bridges to infinite structure. The H_2TNP anions are aligned in parallel planes. The extended coordination scheme of barium ion in the complex is illustrated in Fig. 2. The crystal packing of the complex shows the dinuclear clusters are aligned in Fig. 3. Uncoordinating water molecules and ethanol molecules are present in the cavities of the crystal structure. At the same time, ethanol molecules exist as out-of-order state. The coordination Ba^{2+} cations, H_2TNP and hydroxy anions are linked by electrostatic interaction, weak *van der Waals* forces.

3.2 Thermal analysis

The typical DSC and TG-DTG curves of the thermal decomposition of the title complex under the condition of flowing N_2 gas are shown in Fig. 4 and Fig. 5. The thermal decomposition processes of $[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2]\cdot(CH_3CH_2OH)\cdot 2.5H_2O$ are comprised of four major stages from 50 ℃ to 500 ℃ in the DSC curve. The first and second stages are two endothermic processes, and the third and four stages are two exothermic processes. The TG curve consists of four-stage mass loss process. Due to the complicated coordination sphere, the decomposition of title complex is also intricate.

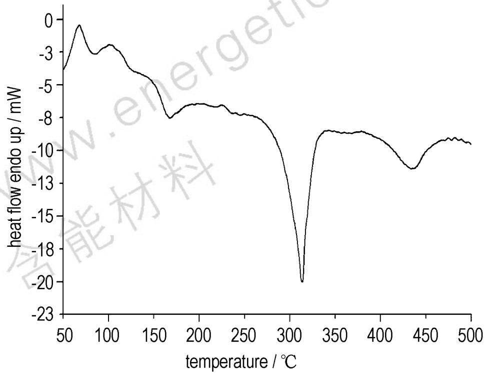

Fig.4 DSC curve of the title complex

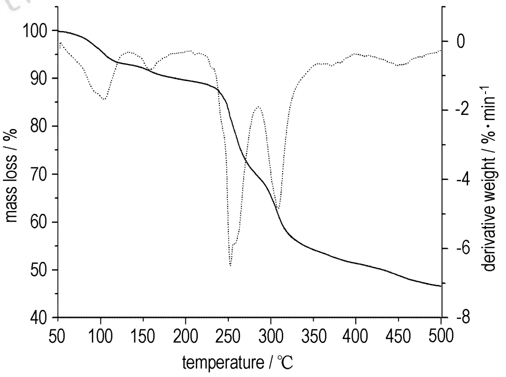

Fig. 5 TG-DTG curves of the title complex

The DSC curve shows some endothermic peaks from 50 ℃ to 200 ℃. Accordingly, its TG-DTG curves confirm a mass loss process of about 9.48 wt-% in the range 68 - 118 ℃, and other mass loss process of about 3.82 wt-% in the range 120 - 200 ℃. The results show that the endothermic processes attribute to the losses of one ethanol and two point five crystal water molecules (expected 9.52 wt-%) and two coordination water molecules (expected 3.77 wt-%) from the title complex. Then, the DSC curve indicates two exothermal decomposition processes with two peaks at 317 ℃ and 435 ℃. The first exothermic process occurs from 247 ℃ to 352 ℃, and the corresponding mass loss process also appears on TG-DTG curves continuously with a mass loss of 33.5 wt-%. The enthalpy of the first exothermal decomposition reaction is 428.0 kJ·mol^{-1}. The characteristic absorption peaks of the residue at the temperature of 370 ℃ shows that some new absorption bands appear at 1460 cm^{-1} and 827 cm^{-1} for $BaCO_3$, at 3350, 1620 cm^{-1} for[—CO—NH—], which conforms that metal carbonate and polyamide are formed during decomposition processes. Because the thermal decomposition residue is a mixture, it cannot be calculated quantitatively. The second exothermic decomposition process occurs in 400 - 500 ℃. The final residue formed after second exothermic process at 550 ℃ is $BaCO_3$ as verified by IR absorption peaks at 1460, 1056, 845, 687 cm^{-1}. Those peaks at 3350, 1620 cm^{-1} for [—CO—NH—] disappeared already. It is obvious that the exothermic process is caused by the decomposition of the polymer. The formation of $BaCO_3$ from the original sample should be accomplished with a theoretical overall mass loss of 58.7 wt-%. This value is in agreement with the experimental value of 57.9 wt-%.

The thermal decomposition mechanism of the title complex might be concluded as following:

$$[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2]\cdot(CH_3CH_2OH)\cdot 2.5H_2O$$

$$\xrightarrow[68-118\,^{\circ}C]{-EtoH,\,2.5H_2O}[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2]$$

$$\xrightarrow[120-200\,^{\circ}C]{-2H_2O}Ba_2(H_2TNP)_2(OH)_2$$

$$\xrightarrow[247-352\,^{\circ}C]{}\text{noncrystalline inorganic compound}$$

$$\xrightarrow[247-352\,^{\circ}C]{}BaCO_3$$

4 Conclusions

The crystal structure of the title complex presents the 2D network structure, which is connected by Ba—O—Ba and H_2TNP ligands links, and two H_2TNP anions act as bridging to connect the adjacent metal Ba(II) center. The thermal decomposition mechanism of the title complex comprises four major stages from 50 ℃ to 500 ℃, which also approves the existence of water molecules and ethanol molecules in the complex, and enthalpy of the major exothermic decomposition reaction is 428.0 kJ · mol^{-1}. The final residue is $BaCO_3$ at 550 ℃.

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$[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2]\cdot(CH_3CH_2OH)\cdot2.5H_2O$ 配合物的合成、结构表征和热分析

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摘要: 将 $NaHCO_3$ 与三硝基间苯三酚分散于蒸馏水中,加热搅拌制备出3,5-二羟基-2,4,6-三硝基苯酚钠溶液,再与硝酸钡水溶液反应合成了3,5-二羟基-2,4,6-三硝基苯酚钡盐。在乙醇的水溶液中培养得到了配合物（$[Ba_2(H_2TNP)_2(OH)_2(H_2O)_2]\cdot(CH_3CH_2OH)\cdot2.5H_2O$（$H_2TNP$: 3,5-二羟基-2,4,6-三硝基苯酚盐）,其化学式为 $C_{14}H_{21}Ba_2N_6O_{25.5}$。对其进行了元素分析和红外表征,利用单晶分析测定了晶体结构,晶体属于四角形晶系,空间群为 I4(1)/amd,晶体学数据为: $a = 19.911(3)$ nm, $b = 19.911(3)$ nm, $c = 13.265(3)$ nm, $V = 5259.0(15)$ nm^3, $Z = 8$,对 1092 个衍射点 $R = 0.0335$。分子结构由二个氧桥联钡的二聚体单元组成。每个 Ba 原子是 10 配位,配位的氧原子分别为: 8 个来自 4 分子 H_2TNP,一个来自一分子 H_2O,一个来自氢氧根离子。金属原子由氧桥连成网状结构,非配位的水分子和乙醇分子位于晶体的空穴中。用 DSC,TG-DTG 和 IR 技术研究了标题物的热分解机理,在 550 ℃,分解残渣主要为 $BaCO_3$。

关键词: 物理化学; 三硝基间苯三酚盐; 钡配合物; 晶体结构; 热分析

中图分类号: TJ55; O614.2 **文献标识码:** A

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