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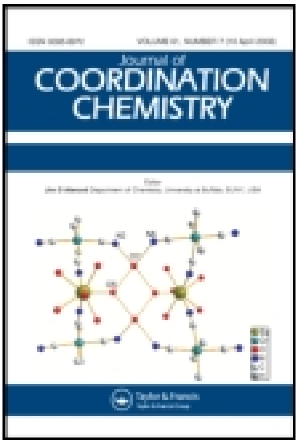

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gcoo20

Synthesis, crystal structure, thermal decomposition, and explosive properties of $[Bi(tza)_3]_n$ (tza=tetrazole acetic acid)

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Published online: 09 Aug 2011.

To cite this article: Shiwei Wang , Li Yang , Tonglai Zhang , Guotao Zhang , Jianguo Zhang & Zunning Zhou (2011) Synthesis, crystal structure, thermal decomposition, and explosive properties of $[Bi(tza)_3]_n$ (tza=tetrazole acetic acid), Journal of Coordination Chemistry, 64:15, 2583-2591, DOI: 10.1080/00958972.2011.601812

To link to this article: http://dx.doi.org/10.1080/00958972.2011.601812

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Journal of Coordination Chemistry
Vol. 64, No. 15, 10 August 2011, 2583–2591

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Synthesis, crystal structure, thermal decomposition, and explosive properties of $[Bi(tza)_3]_n$ (tza = tetrazole acetic acid)

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(*Received 7 April 2011; in final form 6 June 2011*)

A coordination compound based on tetrazole acetic acid (Htza) and bismuth(III), $[Bi(tza)_3]_n$, was synthesized and characterized by single crystal X-ray diffraction analysis, elemental analysis, FT-IR, and ^{1}H NMR spectroscopy. The crystallographic data show that the crystal belongs to monoclinic, $P2_1/n$ space group, $a = 0.91968(19)$ nm, $b = 0.94869(19)$ nm, $c = 1.7824(4)$ nm, $\beta = 101.488(3)^\circ$, and $Z = 4$. The central bismuth(III) is nine-coordinate by three nitrogens from three tetrazole rings and six oxygens of the carboxylate of another three tza^- ions, with each tza^- tridentate, chelating, bridging coordination. The coordination bonds and the intramolecular hydrogen bonds make the complex pack into a layered structure in polymer form. The thermal decomposition mechanism of the title complex was investigated by DSC and TG-DTG techniques. Under nitrogen at a heating rate of $10^\circ C\,min^{-1}$, thermal decomposition of the complex contains two intense exothermic processes between 217.4°C and 530.3°C in the DSC curve; the final decomposed residue at 570°C was Bi_2O_3. Sensitivity tests showed that $[Bi(tza)_3]_n$ was sensitive to impact and flame stimulus.

Keywords: Bismuth; Tetrazole acetic acid; Crystal structure; Thermal decomposition; Sensitivity

1. Introduction

Energetic, environmental-friendly, low-pressure index, and low signature propellants have become an important developmental direction of solid propellants. Since the 1990s, much research on burning rate catalysts are based on traditional Pb and Cu catalysts, which are harmful to both people and environment. So, lead-free or some less toxic energetic catalysts will be the focus of future research. Bismuth is a heavy metal which is less toxic and bismuth ions mainly exist as trivalent and pentavalent ions; trivalent has larger radius, stronger deformation, and polarization properties, able to form stable complexes, with coordination number up to 10. Because of the stereochemical activity of the outer lone pair the complexes have structural diversity. A number of complexes of bismuth have been synthesized and widely used in medicine,

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Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online
DOI: 10.1080/00958972.2011.601812

pharmaceuticals, chemical catalysts, and many other fields [1–8]. Most bismuth complexes are seven or nine coordinate, which is very different from other metals [9–13].

Tetrazole-based compounds are a kind of energetic material which are insensitive and non-toxic. As a catalyst, when added into propellants, they may reduce the characteristic signal of propellants and are used as components to propellants [14]. Scientists have made progress in making tetrazole-based energetic compounds as catalysts of solid propellants. Tetrazole acetic acid (Htza) is an important pharmaceutical intermediate and an important raw material to prepare varieties of antibiotic drugs; tetrazole acetic acid is also widely used to prepare polishing agents. In recent years, researchers found that tetrazole acetic acid has excellent coordination properties, with the complexes containing tetrazole acetic acid having novel structures, optical and magnetic properties [15–18]. Dong *et al.* [19] reported that the crystal structure of $[Ag(tza)]_n$ exhibits a helical chain and $[Cu(tza)_2]_n$ features a wave-like layered structure. They also synthesized $[Cd(tza)_2]_n$, $[Mn(tza)_2]_n$, $[Co(tza)_2]_n$, α-$[Zn(tza)_2]_n$, and β-$[Zn(tza)_2]_n$ that also have special structures [20].

In this article, we report the synthesis, crystal structure, thermal decomposition, and explosive properties of a nine-coordinate complex of tetrazole acetic acid and bismuth, ($[Bi(tza)_3]_n$).

2. Experimental

2.1. *Materials and methods*

All reagents and solvents were of analytical grade and used as commercially obtained.

2.2. *Physical techniques*

Elemental analyses (carbon, hydrogen, and nitrogen) were performed on an Elementar Vario MICROCUBE (Germany) elemental analyzer. FT-IR spectra were recorded on a Bruker Equinox 55 infrared spectrometer (KBr pellets) from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1}. ^1H NMR spectra were obtained using a Bruker AVANCE DRX 500 nuclear magnetic resonance spectrometer with DMSO-d_6 as solvent. DSC and TG measurements were carried out using a Pyris-1 differential scanning calorimeter and a Pyris-1 thermogravimetric analyzer (Perkin Elmer, USA), respectively, with dry nitrogen flowing at 20 mL min^{-1}. The conditions for thermal analysis were as follows: for Pyris-1 DSC, the crystal sample was powdered and sealed in aluminum pans with a linear heating rate of 10°C min^{-1} from 50°C to 600°C; for Pyris-1 TGA, the crystal sample was powdered and put in platinum open pans with heating rate of 10°C min^{-1} from 50°C to 600°C.

2.3. *Synthesis of $[Bi(tza)_3]_n$*

Tetrazole acetic acid (23 g, 0.18 mol) was dissolved into 120 mL distilled water, and then $Bi(NO_3)_3 \cdot 5H_2O$ (19.4 g, 0.04 mol) was added in portions with heating and stirring. $Bi(NO_3)_3 \cdot 5H_2O$ dissolves in the solution and then $[Bi(tza)_3]_n$ precipitates gradually.

The resultant mixture was filtered and kept undisturbed at room temperature, and single crystals suitable for X-ray analysis were obtained after 3 days.

2.4. *Characterization*

Elemental analysis for $BiC_9H_9N_{12}O_6$ (formula weight: $590.26\,g\,mol^{-1}$): Calcd (%): C, 18.30; H, 1.52; N, 28.46. Found (%): C, 18.14; H, 1.62; N, 28.24. FT-IR (cm^{-1}, KBr): 3417, 3145, 3125, 3099, 3000, 2959 (C–H), 1642, 1636, 1616, 1400 (C=O, C=N, N=N), basically consistent with the literature values [21]. ^1H NMR (DMSO-d_6, δ, ppm): 9.33 (s), 5.26 (s).

2.5. *X-ray data collection and structure refinement*

A colorless single crystal with dimensions of 0.30 mm × 0.27 mm × 0.23 mm was selected for X-ray diffraction analysis. The data collection was performed on a Rigaku AFC-10/Saturn 724^+ CCD detector diffractometer with graphite monochromated Mo-Kα radiation ($\lambda = 0.\ 071073$ nm) at 103 (2) K with *phi* and *omega* scan techniques. A total of 13,878 reflections (3465 unique, $R_{int} = 0.0443$) were measured from $3.12^\circ \leq \theta \leq 27.48^\circ$, of which 3210 were observed with $I \geq 2\sigma(I)$. The scaled maximum and minimum transmission factors are 0.1724 and 0.1280, respectively. A semi-empirical absorption correction (SADABS) was applied to the raw intensities [22]. The structure was solved by direct methods using SHELXS-97 [23] and refined by full-matrix least-squares methods on F^2 with SHELXL-97 [24]. All non-hydrogen atoms were obtained from the difference Fourier map and refined anisotropically. Hydrogens were obtained geometrically and treated by a constrained refinement. The detailed crystallographic data are listed in table 1; selected bond lengths and angles are listed in tables 2 and 3, respectively.

3. Results and discussion

3.1. *Crystal structure*

The molecular structure and packing of $[Bi(tza)_3]_n$ are shown in figures 1 and 2, respectively. There are six tetrazole acetates coordinated with one bismuth, three monodentates, and the other three bidentates. In other words, the central bismuth coordinates with three nitrogens from three tetrazole rings and six oxygens of the carboxylate of another three tetrazole acetates to form a nine-coordinate geometry. As a tridentate-bridging ligand, two tza^- molecules connect two neighboring Bi ions, Bi1 and Bi1A. The two neighboring Bi ions link with one nitrogen and two oxygens of the same tza^- into a circle that makes the entire structure extend infinitely to form a network structure. In this circle (Bi1–tza–Bi1A–tza), the two tetrazole rings and the two Bi1–O1–C3–O2 rings are parallel, leading to minimum steric hindrance.

The bond lengths between Bi1 and O1–O6 are 0.2640(3), 0.2287(3), 0.2594(3), 0.2266(3), 0.2389(3), and 0.2550(3), respectively. Distances between the central bismuth and the three nitrogens are not exactly the same, 0.2689 for Bi1–N9A, 0.2868 for Bi1–N5A, and 0.2907 for Bi1–N2A, all longer than the Bi–O bonds. Bond lengths of Bi1–N2A and Bi1–N5A are longer than that of Bi1–N9A, showing that bismuth is weakly coordinated with N2A and N5A.

Table 1. Crystal data and structure refinement parameters of $[Bi(tza)_3]_n$.

Compound	$[Bi(tza)_3]_n$
Empirical formula	$BiC_9H_9N_{12}O_6$
Formula weight	590.26
Temperature (K)	103(2)
Crystal system	Monoclinic
Space group	$P2_1/n$
Crystal size (mm^3)	$0.30 \times 0.27 \times 0.23$
Unit cell dimensions (nm, °)	
a	0.91968 (19)
b	0.94869 (19)
c	1.7824 (4)
β	101.488 (3)
Z	4
Limiting indices	$-11 \leq h \leq 12$; $-12 \leq k \leq 12$; $-23 \leq l \leq 22$
Calculated density ($Mg \cdot m^{-3}$)	2.573
λ (nm)	0.071073
Absorption coefficient (mm^{-1})	11.638
$F(000)$	1112
θ range for data collection (°)	$3.12 \leq \theta \leq 27.48$
Reflections collected	13,878
Independent reflection	3465 [R(int) = 0.0443]
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0278$, $wR_2 = 0.0552$
R indices (all data)	$R_1 = 0.0319$, $wR_2 = 0.0575$
Goodness-of-fit on F^2	0.999
Largest difference peak and hole ($e\ nm^{-3}$)	2707 and −1394

Table 2. Selected bond lengths of $[Bi(tza)_3]_n$.

Bonds	Length (nm)	Bonds	Length (nm)
Bi(1)–O(4)	0.2266(3)	N(4)–C(2)	0.1456(6)
Bi(1)–O(2)	0.2287(3)	N(5)–C(4)	0.1319(6)
Bi(1)–O(5)	0.2389(3)	N(5)–N(6)	0.1365(6)
Bi(1)–O(6)	0.2550(3)	N(6)–N(7)	0.1300(6)
Bi(1)–O(3)	0.2594(3)	N(7)–N(8)	0.1342(5)
Bi(1)–O(1)	0.2640(3)	N(8)–C(4)	0.1323(6)
Bi(1)–N(9)#1	0.2689(4)	N(8)–C(5)	0.1456(5)
O(1)–C(3)	0.1234(5)	N(9)–C(7)	0.1311(6)
O(2)–C(3)	0.1284(5)	N(9)–N(10)	0.1365(5)
O(3)–C(6)	0.1236(5)	N(9)–Bi(1)#2	0.2689(4)
O(4)–C(6)	0.1288(6)	N(10)–N(11)	0.1288(5)
O(5)–C(9)	0.1263(6)	N(11)–N(12)	0.1351(5)
O(6)–C(9)	0.1243(5)	N(12)–C(7)	0.1337(6)
N(1)–C(1)	0.1316(6)	N(12)–C(8)	0.1447(6)
N(1)–N(2)	0.1360(6)	C(2)–C(3)	0.1515(7)
N(2)–N(3)	0.1293(6)	C(5)–C(6)	0.1518(6)
N(3)–N(4)	0.1345(6)	C(8)–C(9)	0.1527(6)
N(4)–C(1)	0.1335(6)		

Symmetry transformations used to generate equivalent atoms: #1: $x-1/2$, $-y+1/2$, $z-1/2$; #2: $x+1/2$, $-y+1/2$, $z+1/2$.

Table 3. Selected bond angles of $[Bi(tza)_3]_n$.

Bonds	Angles (°)	Bonds	Angles (°)
O(4)–Bi(1)–O(2)	81.03(11)	C(1)–N(4)–C(2)	131.0(4)
O(4)–Bi(1)–O(5)	82.32(11)	N(3)–N(4)–C(2)	120.3(4)
O(2)–Bi(1)–O(5)	78.74(11)	C(4)–N(5)–N(6)	105.3(4)
O(4)–Bi(1)–O(6)	78.71(11)	N(7)–N(6)–N(5)	110.6(4)
O(2)–Bi(1)–O(6)	129.32(11)	N(6)–N(7)–N(8)	106.1(4)
O(5)–Bi(1)–O(6)	52.88(10)	C(4)–N(8)–N(7)	109.0(4)
O(4)–Bi(1)–O(3)	53.91(11)	C(4)–N(8)–C(5)	129.1(4)
O(2)–Bi(1)–O(3)	128.32(11)	N(7)–N(8)–C(5)	121.7(4)
O(5)–Bi(1)–O(3)	113.90(11)	C(7)–N(9)–N(10)	106.7(4)
O(6)–Bi(1)–O(3)	70.19(10)	C(7)–N(9)–Bi(1)#2	131.5(3)
O(4)–Bi(1)–O(1)	128.42(11)	N(10)–N(9)–Bi(1)#2	121.7(3)
O(2)–Bi(1)–O(1)	52.87(11)	N(11)–N(10)–N(9)	109.8(4)
O(5)–Bi(1)–O(1)	106.42(11)	N(10)–N(11)–N(12)	107.0(4)
O(6)–Bi(1)–O(1)	146.75(10)	C(7)–N(12)–N(11)	108.3(4)
O(3)–Bi(1)–O(1)	139.05(10)	C(7)–N(12)–C(8)	130.2(4)
O(4)–Bi(1)–N(9)#1	75.70(11)	N(11)–N(12)–C(8)	121.3(4)
O(2)–Bi(1)–N(9)#1	73.59(12)	N(1)–C(1)–N(4)	109.1(4)
O(5)–Bi(1)–N(9)#1	146.83(11)	N(4)–C(2)–C(3)	110.7(4)
O(6)–Bi(1)–N(9)#1	141.87(11)	O(1)–C(3)–O(2)	123.3(4)
O(3)–Bi(1)–N(9)#1	71.94(11)	O(1)–C(3)–C(2)	121.3(4)
O(1)–Bi(1)–N(9)#1	70.50(11)	O(2)–C(3)–C(2)	115.3(4)
C(3)–O(1)–Bi(1)	84.3(3)	N(5)–C(4)–N(8)	109.0(4)
C(3)–O(2)–Bi(1)	99.5(3)	N(8)–C(5)–C(6)	112.8(4)
C(6)–O(3)–Bi(1)	84.4(3)	O(3)–C(6)–O(4)	123.4(4)
C(6)–O(4)–Bi(1)	98.2(3)	O(3)–C(6)–C(5)	118.4(4)
C(9)–O(5)–Bi(1)	95.3(3)	O(4)–C(6)–C(5)	118.2(4)
C(9)–O(6)–Bi(1)	88.3(3)	N(9)–C(7)–N(12)	108.2(4)
C(1)–N(1)–N(2)	105.1(4)	N(12)–C(8)–C(9)	115.5(4)
N(3)–N(2)–N(1)	111.4(4)	O(6)–C(9)–O(5)	123.2(4)
N(2)–N(3)–N(4)	106.0(4)	O(6)–C(9)–C(8)	115.1(4)
C(1)–N(4)–N(3)	108.5(4)	O(5)–C(9)–C(8)	121.7(4)

Symmetry transformations used to generate equivalent atoms: #1: $x-1/2$, $-y+1/2$, $z-1/2$; #2: $x+1/2$, $-y+1/2$, $z+1/2$.

The bismuth and three carboxyls form three-, four-membered rings (Bi–O–C–O), and the plane equations can be expressed as

Bi1–O1–C3–O2 (plane 1): $5.475x-0.464y+11.895z=12.3588$, $R=0.0084$;
Bi1–O3–C6–O4 (plane 2): $2.712x-6.006y+11.455z=8.3898$, $R=0.0077$;
Bi1–O5–C9–O6 (plane 3): $9.134x+0.825y-4.893z=5.1384$, $R=0.0270$.

The angles between the three planes are 141.3° for 1 and 2, 58.4° for 1 and 3, and 100.5° for 2 and 3.

From the packing plot of $[Bi(tza)_3]_n$, there are many intramolecular hydrogen bonds because of the existence of N–H and O–H; all the hydrogen bonds exist in different layers of tza^- making the molecule pack into a layered structure. The coordination bonds and the intramolecular hydrogen bonds make the complex a polymer.

3.2. *Thermal decomposition*

In order to investigate the thermal behavior of the title compound, DSC and TG-DTG experiments were carried out. The DSC curves at a heating rate of $10°C\,min^{-1}$ are

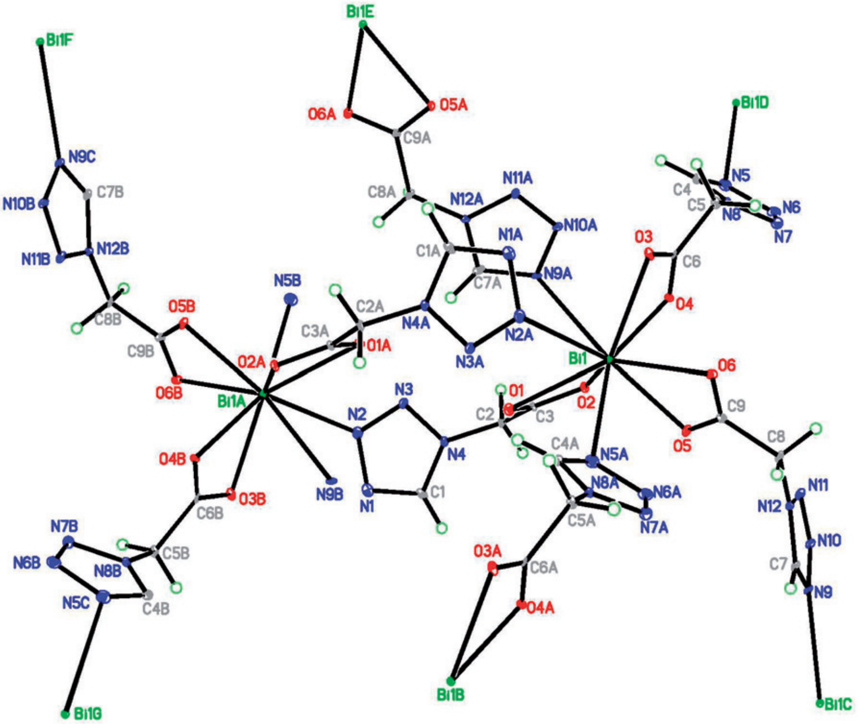

Figure 1. Molecular structure of $[Bi(tza)_3]_n$.

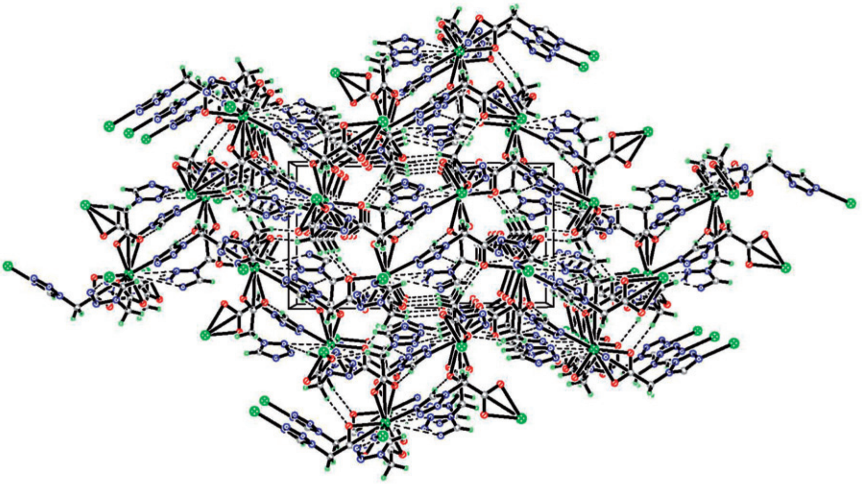

Figure 2. Packing plot of $[Bi(tza)_3]_n$ viewed along the *a*-axis of the unit cell.

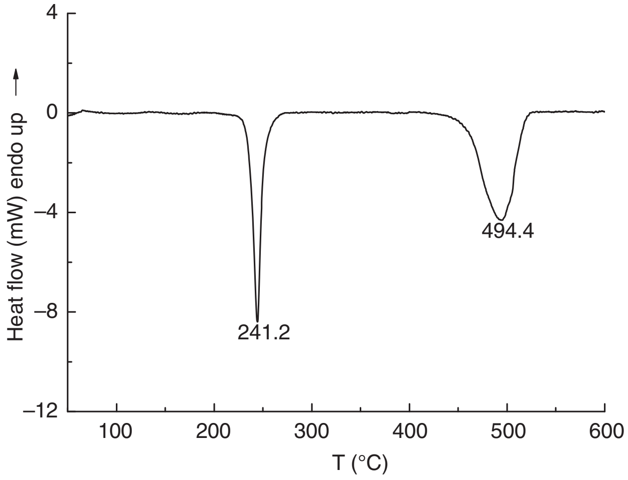

Figure 3. DSC curve of the title complex with heating rate of 10°C min^{-1}.

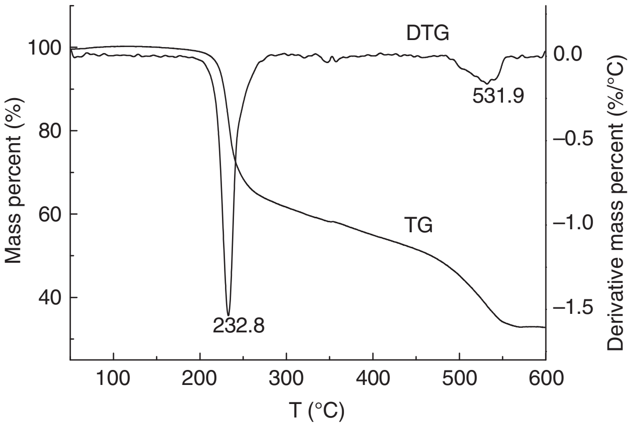

Figure 4. TG-DTG curve of the title complex with heating rate of 10°C min^{-1}.

shown in figure 3; TG-DTG curves at 10°C·min^{-1} in flowing nitrogen at 20 mL min^{-1} are illustrated in figure 4.

In the DSC curve, there are two intense exothermic processes. The first occurs from 217.4°C to 276.4°C and this peak is very sharp with the peak temperature and the onset temperature 244.1°C and 236.3°C, respectively; the exothermic enthalpy of this exothermic process is 713.9 kJ mol^{-1}. Corresponding to this exothermic process, there is a mass loss of 33% of the initial mass in the TG-DTG curves, caused by the violent decomposition of $[Bi(tza)_3]_n$.

With the temperature increasing, the second exothermic decomposition process, which is also an intense exothermic process, appears between 438.1°C and 530.3°C, with the peak temperature and the onset temperature 494.0°C and 460.6°C, respectively; the exothermic enthalpy of this exothermic process is 2240.0 kJ mol^{-1}. In the

TG-DTG curves, another mass loss of 28% is observed. The decomposition of $[Bi(tza)_3]_n$ stopped at 570°C, and the mass of the final residue is 38% of the initial mass, coincident with the calculated value of Bi_2O_3, 39%. The absorption band at 2850–2920 cm^{-1} in the FT-IR spectrum of the residue proves the final residue is Bi_2O_3.

3.3. *Sensitivity test*

One of the most important indicators to evaluate an energetic material is the sensitivity, a measure of the response of the composition to external stimulus such as impact, friction, heat, etc. The impact sensitivity, friction sensitivity, and flame sensitivity were measured according to the "Gunpower test method" of GJB 770 A-97.

Impact sensitivity was determined by a Fall Hammer Apparatus. 30 mg of $[Bi(tza)_3]_n$ was added into the mold, and hit by a 10 kg drop hammer. The test results showed that the 50% firing height (h_{50}) was 16.0 cm.

Friction sensitivity was determined by a Pendulum apparatus. When 20 mg of $[Bi(tza)_3]$ was compressed between two steel poles with mirror surfaces at 2.45 MPa and hit horizontally with a 1.5 kg hammer from a 66° angle, the title compound did not fire.

According to the flame sensitivity test, 20 mg of $[Bi(tza)_3]_n$ was compacted to a copper cap under a pressure of 58.8 MPa and was ignited by black powder pellet. The 50% firing height (h_{50}) was 10.8 cm.

4. Conclusions

We have synthesized and characterized a new coordination compound, $[Bi(tza)_3]_n$. The central bismuth(III) is nine-coordinate by three nitrogens of three tetrazole rings and six oxygens of carboxylate of another three tza^- ions; each tza^- is tridentate chelating bridging. Molecules of the title complex are linked into a layered-network structure by both coordination bonds and intramolecular hydrogen bonds. Thermal analysis indicates there are two intense exothermic processes, and the final decomposed residue is Bi_2O_3. The sensitivity tests show that $[Bi(tza)_3]_n$ was sensitive to external stimulus, especially impact and flame stimulus. All the studies indicate that $[Bi(tza)_3]_n$ has potential applications in energetic materials and may be used as a catalyst to solid propellants.

Supplementary material

Crystallographic data for the structures reported in this article have been deposited with the Cambridge Crystallographic Data Centre (CCDC). Copies of the data can be obtained free of charge on quoting the numbers CCDC-807863 for the title compound from CCDC (12 Union Road, Cambridge, CB2 1EZ, UK [Fax +44(0)1223/336033; Email: deposit@ccdc.cam.ac.uk].

Acknowledgments

This project was financially supported by Science and Technology fund on Applied Physical Chemistry Laboratory (No. 9140C3703051105), fund of the State Key Laboratory of Explosion Science and Technology (No. YBKT10–05 and ZDKT10–01b), and Program for New Century Excellent Talents in University (No. NCET–10–0051 and No. CNET–09–0051).

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