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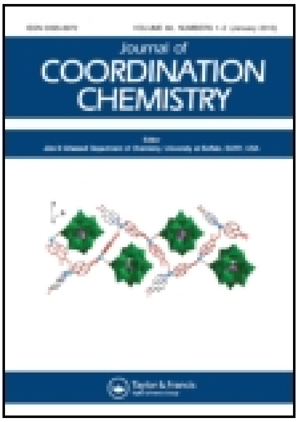

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Synthesis, structural characterization, and thermal properties of a new energetic zinc-FOX-7 complex

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Synthesis, structural characterization, and thermal properties of a new energetic zinc-FOX-7 complex

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A new energetic zinc-FOX-7 complex (FOX-7 = 1,1-diamino-2,2-dinitroethylene), $Zn(NH_3)_2(FOX\text{-}7)_2$, was synthesized and structurally characterized by single crystal X-ray diffraction. Zn^{2+} was coordinated by four nitrogens from two ammonias and two $FOX\text{-}7^-$ anions, forming a distorted tetrahedron. Thermal decomposition of $Zn(NH_3)_2(FOX\text{-}7)_2$ was studied by differential scanning calorimetry and thermogravimetry/differential thermogravimetry. The apparent activation energy and pre-exponential constant of the first decomposition process are 144.8 kJ M^{-1} and $10^{13.99}$ s^{-1}, respectively. The self-accelerating decomposition temperature and critical temperature of thermal explosion of $Zn(NH_3)_2(FOX\text{-}7)_2$ are 183.2 and 195.8 °C, respectively. The thermal stability of $Zn(NH_3)_2(FOX\text{-}7)_2$ is good, but $Zn(NH_3)_2(FOX\text{-}7)_2$ is still sensitive.

Keywords: 1,1-Diamino-2,2-dinitroethylene (FOX-7); Zinc complex; Crystal structure; Thermal behavior

1. Introduction

Scientists have long sought high-energy materials with low sensitivity to impact and friction. 1,1-Diamino-2,2-dinitroethylene (FOX-7) is a high-energy material with high thermal stability and low sensitivity to impact and friction, with a density of 1.885 g cm^{-3}, a heat formation of 32 kcal M^{-1}, the same insensitive to TATB (1,3,5-triamino-2,4,6-trinitrobenzene) and similar energy density with RDX (1,3,5-trinitroperhydro-1,3,5-triazine) and HMX (cyclotetramethylene tetranitramine) [1]. Since first synthesized in 1999 [1], FOX-7 has received attention and is considered as the main component to be used in insensitive ammunitions and solid propellants. Many studies have reported synthesis, mechanism, structure, thermal behavior, explosive performance, and applications of FOX-7 [1–10]. FOX-7 is a representative "push-pull" nitroenamine compound, which possesses a highly polarized carbon–carbon double bond with positive and negative charges stabilized by the amino and nitro groups, respectively, and presents certain acidic properties [11–13]. So, FOX-7 can react with strong alkalis to prepare energetic salts, such as potassium salt, rubidium salt, cesium salt, and guanidine salt [14–16]. Other salts and metal complexes of FOX-7 also can be synthesized through replacement reactions. Garg and He *et al.* have reported Ag(amine)(FOX-7) [amine: ammonia, methylamine, and

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*Corresponding author. Email: xukz@nwu.edu.cn propylamine], Cu(amine)$_2$(FOX-7)$_2$ [amine: ammonia, methylamine, propylamine and dimethylamine, ethylenediamine, and 1,3-propane diamine], and other copper (nickel) bipyridyl (phenanthroline) FOX-7 complexes [17–20]. Despite those studies, the reactivity of FOX-7 is still an unresolved issue. In this article, we report a new zinc-FOX-7 complex, study its crystal structure and thermal decomposition behavior, and hope to research the structure–property relationships of these FOX-7 complexes.

2. Experimental

2.1. *Materials and physical measurements*

All chemicals were analytical-grade commercial products. FOX-7 came from Xi'an Modern Chemistry Research Institute (purity > 99%). K(FOX-7)·H$_2$O was prepared according to the reference [15].

Elemental analyzes were performed on a VarioEL Ш elemental analyzer (Elementar Co., Germany). IR spectra were determined on a EQUINX55 with KBr pellets. Differential scanning calorimetry (DSC) experiments were performed using a DSC200 F3 apparatus (NETZSCH, Germany) under nitrogen at a flow rate of 80 mL min^{-1}. The heating rates were 5.0, 7.5, 10.0, 12.5, and 15.0 °C min^{-1} from ambient temperature to 350 °C. The thermogravimetry/differential thermogravimetry (TG/DTG) experiment was performed using a SDT-Q600 apparatus (TA, USA) under nitrogen at a flow rate of 100 mL min^{-1}. The heating rate was 10.0 °C min^{-1} from ambient temperature to 500 °C. The impact and friction sensitivities were determined by using a ZBL-B impact sensitivity instrument and a MGY-2 friction sensitivity instrument (Nachen Co., China), respectively. The mass of fall-weight is 2.0 kg. The swing angle and gage pressure are 50° and 0.6 MPa. The sample weight for each test is 30 mg.

2.2. *Synthesis*

K(FOX-7)·H$_2$O (0.01 M) was put into excess ammonia water (15 mL) to form a clear solution, and then zinc nitrate (0.005 M) was added. After reaction at room temperature for 20 min, the resulting mixture was stored at room temperature. Yellow crystals of Zn(NH$_3$)$_2$(FOX-7)$_2$ formed, were filtered, washed with water and dried under vacuum, yielding 0.83 g (42%). Anal. Calcd (%) for $C_4H_{12}N_{10}O_8Zn$: C, 12.20; H, 3.073; N, 35.58. Found: C, 12.11; H, 3.11; N, 35.36. IR (KBr): 3407, 3326, 2737, 2505, 1645, 1613, 1508, 1467, 1357, 1232, 1128, 802, 758 cm^{-1}.

2.3. *Determination of the single crystal structure*

Single crystals suitable for X-ray measurement were obtained by slow evaporation of solution. A bright yellow crystal with dimensions of 0.36 × 0.25 × 0.14 mm was chosen for X-ray determination. The data were collected on a Bruker SMART APEX CCD X-ray diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.071073 nm). The structure was solved by direct methods (SHELXTL-97) and refined by full-matrix least-squares on F^2 with anisotropic thermal parameters for all non-hydrogen atoms [21, 22]. Hydrogens were added according to theoretical models.

Crystal data and refinement results are summarized in table 1. Selected bond lengths and angles are listed in table 2.

3. Results and discussion

3.1. *Crystal structure*

$Zn(NH_3)_2(FOX\text{-}7)_2$ crystallizes in the monoclinic crystal system with space group *C*2/*c* containing four molecules per unit cell. As shown in figure 1, $Zn(NH_3)_2(FOX\text{-}7)_2$ is made of one Zn^{2+}, two FOX-7^- and two ammonias. Zn^{2+} is coordinated by four nitrogens from two FOX-7^- (N1 and N1A), and two ammonias [N5 and N5A]. The whole complex exhibits a cross-symmetrical structure around Zn^{2+}. Four nitrogens form a distorted tetrahedron with bond lengths [Zn1–N1 (1.9934(18) Å) and Zn1–N5 (2.0277(19) Å)] and bond angles [N1A–Zn1–N1 (114.19(11)°), N1A–Zn1–N5A (107.99(8)°), N1–Zn1–N5A (108.61(8)°), N1A–Zn1–N5 (108.61(8)°), N1–Zn1–N5 (107.99(8)°) and N5A–Zn1–N5 (109.38(12)°)]. The coordination structure of $Zn(NH_3)_2(FOX\text{-}7)_2$ is similar with that of $Cu(NH_3)_2(FOX\text{-}7)_2$, but they have different symmetry and spatial configuration. Four nitrogens around Cu^{2+} form a planar cross-shaped structure in $Cu(NH_3)_2(FOX\text{-}7)_2$ [18]. In addition, there are six kinds of N–H···O and one kind of N–H···N hydrogen bonds in the crystal packing of $Zn(NH_3)_2(FOX\text{-}7)_2$ (table 3). The coordination interactions, electrostatic attractions, and hydrogen bonds form the crystal packing of $Zn(NH_3)_2(FOX\text{-}7)_2$ (figure 2).

An ammonia, Zn^{2+} ion, and two amino groups (non-hydrogens) of one FOX-7^- anion form a plane structure supported by the torsion angles N5–Zn1–N1–C1 (−176.3°), Zn1–N1–C1–N2 (−1.5°), and Zn1–N1–C1–C2 (179.6°). Moreover, the space configuration of FOX-7^- changes from original one plane to two approximate orthogonal planes (non-hydrogens) [2], according to the torsion angles N1–C1–C2–N4 (89.2°) and N2–C1–C2–N4 (89.8°), and the intersection of two approximate orthogonal planes is the C1–C2 bond.

Table 1. Crystal data and structure refinement details of $Zn(NH_3)_2(FOX\text{-}7)_2$.

Chemical formula	$C_4H_{12}N_{10}O_8Zn$
Formula weight (g M^{-1})	393.61
Temperature (K)	296(2)
Crystal system	Monoclinic
Space group	*C*2/*c*
a (Å)	18.468(2)
b (Å)	6.454(1)
c (Å)	12.979(2)
β (°)	117.11
Volume ($Å^3$)	1377.1(3)
Z	4
D_{Calcd} (g cm^{-3})	1.898
Absorption coefficient (mm^{-1})	1.850
F (0 0 0)	800
θ range (°)	2.48–25.10
Index ranges	$-16 \le h \le 22$, $-7 \le k \le 7$, $-15 \le l \le 15$
Reflections collected	3340
Reflections unique	1227
Goodness-of-fit on F^2	1.077
Final *R* indices [$l > 2\sigma(l)$]	$R_1 = 0.0252$, $wR_2 = 0.0594$
R indices (all data)	$R_1 = 0.0279$, $wR_2 = 0.0607$
Largest diff. peak and hole (e $Å^{-3}$)	0.272 and −0.250

Table 2. Selected bond lengths (Å) and angles (°).

	Exp.	Calcd		Exp.	Calcd
Zn1–N1#1	1.993(2)	2.003	N1A–Zn1–N1#1	114.19(11)	111.28
Zn1–N1A	1.993(2)	2.003	N1A–Zn1–N5A	107.99(8)	101.72
Zn1–N5#1	2.028(2)	2.100	N1#1–Zn1–N5A	108.61(8)	115.35
Zn1–N5A	2.028(2)	2.100	N1A–Zn1–N5#1	108.61(8)	115.35
N1#1–C1	1.290(3)	1.309	N1#1–Zn1–N5#1	107.99(8)	101.72
N2–C1	1.328(3)	1.330	N5A–Zn1–N5#1	109.38(12)	112.42
N3–O2	1.233(2)	1.225	C1–N1#1–Zn1	131.70(15)	120.80
N3–O1	1.249(2)	1.288	O2–N3–O1	121.86(19)	120.84
N3–C2	1.385(3)	1.356	O2–N3–C2	123.02(19)	123.93
N4–O3	1.248(2)	1.226	O1–N3–C2	115.10(19)	115.23
N4–O4	1.263(2)	1.264	O3–N4–O4	120.21(19)	122.80
N4–C2	1.363(3)	1.375	O3–N4–C2	123.52(18)	122.76
C1–C2	1.500(3)	1.480	O4–N4–C2	116.26(18)	114.44
			N1#1–C1–N2	123.50(20)	120.37
			N1#1–C1–C2	121.27(19)	121.68
			N2–C1–C2	115.27(19)	117.78
			N4–C2–N3	122.49(19)	125.81
			N4–C2–C1	118.89(18)	117.41
			N3–C2–C1	118.04(18)	116.38

Symmetry transformation: #1 −*x*, *y*, −*z* + 1/2.

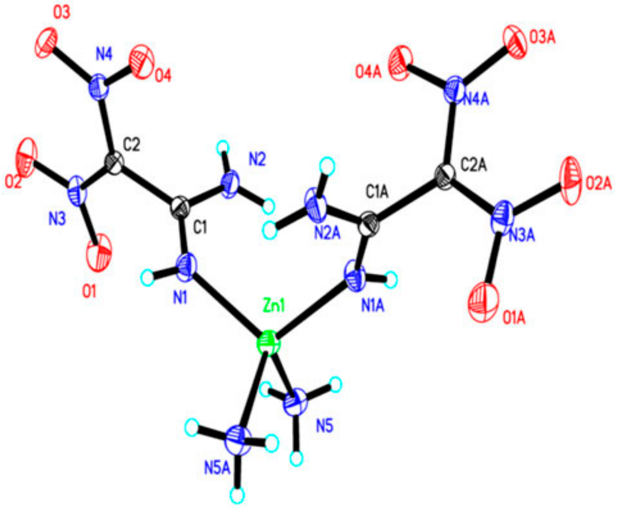

Figure 1. Molecular structure of $Zn(NH_3)_2(FOX\text{-}7)_2$ with 30% probability displacement ellipsoids.

Structural change can be found in other salts of FOX-7. The bond lengths and bond angles also change. The symmetrical and approximately equal bond lengths and bond angles in FOX-7 are no longer consistent with each other through different interactions. Theoretical C(1)–C(2) double bond (1.500(3) Å) in FOX-7$^-$ is much closer to C–C single bond (1.53 Å) than that in FOX-7 molecule. Equilong C(1)–N(1) and C(1)–N(2) bonds present big deviations (1.290(3) and 1.328(3) Å). C(1)–N(1) in FOX-7$^-$ is a typical C–N double bond. FOX-7 has changed to one of its tautomer.

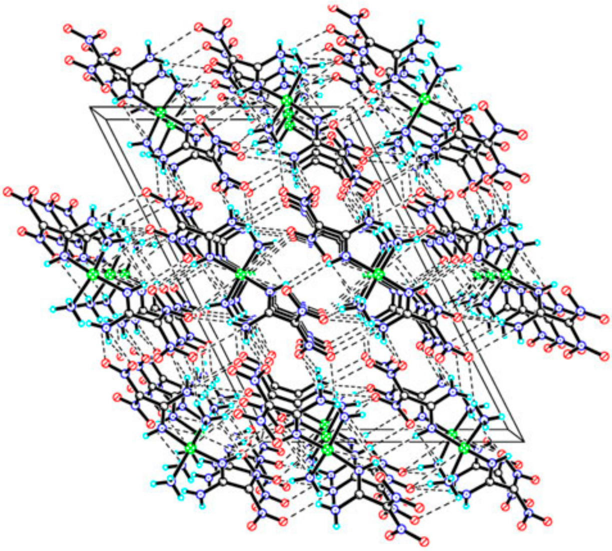

Figure 2. Crystal packing of $Zn(NH_3)_2(FOX\text{-}7)_2$.

3.2. *Theoretical calculation*

According to the crystal structure of $Zn(NH_3)_2(FOX\text{-}7)_2$, a crystal unit was selected as the initial structure, while DFT-B3LYP/6-311G method in Gaussian 03 package [23] was used to optimize the structure and compute the frequencies. Vibration analysis showed that the optimized structure was in accord with the minimum points on the potential energy planes, which means no imaginary frequencies, proving that the obtained optimized structure was stable. All the convergent precisions are the system default values, and the calculation was carried out on a *Lenovo* T260 server. The optimized bond lengths and bond angles are listed in table 2. Mulliken net charges of non-hydrogen atoms are listed in table 4. A careful comparison of the molecular conformation predicted by Density Functional Theory (DFT) calculations and X-ray studies show close agreement between the two. The symmetry of coordinated bonds is increased (Zn1–N1 2.003 Å and Zn1–N5 2.100 Å). Conjugation effects of C1–C2 (1.480 Å), N1–C1 (1.309 Å), and N2–C1 (1.330 Å) are further enhanced. But the calculated values of bond angles and dihedral angles are significantly different from the experimental values. The reason may be that only a single gas-phase molecule was used in theoretical calculation process, which is clearly different from the actual solid-phase compound. The calculation of Mulliken net charges of atoms (table 4) indicates C(1) has positive charge (0.6620 e), but C(2) has some negative charge (−0.0054 e); C1–C2 is the polarized C–C double bond, which is a typical characteristic of "push-pull" nitro-enamine.

3.3. *Thermal analysis*

Typical DSC and TG-DTG curves (figures 3 and 4) indicate that the thermal decomposition of $Zn(NH_3)_2(FOX\text{-}7)_2$ can be divided into two continuous exothermic decomposition processes. The first is an intense exothermic decomposition process, which occurs at

Table 3. Hydrogen bonds for $Zn(NH_3)_2(FOX\text{-}7)_2$ (Å and °).

D−H···A	d(D−H)	d(H···A)	d(D···A)	∠(DHA)
N1–H···O4#1	0.860	2.395	3.211	158.61
N1–H···O3#1	0.860	2.581	3.098	119.66
N2–H···O3#2	0.860	2.180	3.028	168.49
N2–H···O1#3	0.860	2.140	2.939	154.38
N5–H···O2#3	0.890	2.218	3.045	154.49
N5–H···O3#2	0.890	2.356	2.984	127.60
N1–H···N4#1	0.858	2.690	3.360	135.67

Symmetry transformations: #1:$-x$, $-y + 2$, $-z$; #2: x, $-y + 2$, $z + 1/2$; #3: $-x + 1/2$, $y + 1/2$, $-z + 1/2$.

Table 4. Mulliken net charges of non-hydrogen atoms (e).

Atom		Atom	
Zn(1)	1.7476	O(1)	−0.6571
N(1)	−1.073	O(2)	−0.3973
N(2)	−0.7873	O(3)	−0.4039
N(3)	0.5394	O(4)	−0.5458
N(4)	0.5401	C(1)	0.6620
N(5)	−1.1726	C(2)	−0.0054

180–235 °C with a mass loss of about 37.5%. Extrapolated onset temperature and peak temperature are 198.1 and 204.4 °C, at a heating rate of 10.0 °C min^{-1}, respectively. The second is a slow exothermic decomposition process with a mass loss of about 28.1% at 235–330 °C, and the peak temperature is 279.3 °C at a heating rate of 10.0 °C min^{-1}. The decomposition heat of the two continuous processes is 2070 ± 70 J g^{-1}. The final residue percentage at 500 °C is about 28%. Comparing with the thermal decomposition of $Cu(NH_3)_2(FOX\text{-}7)_2$, they present similar thermal decomposition processes, but $Zn(NH_3)_2(FOX\text{-}7)_2$ has much higher thermal stability than $Cu(NH_3)_2(FOX\text{-}7)_2$ [18]. Energetic copper complexes are often used as combustion catalysts in solid propellant [24–27]. Cu^{2+} has good catalytic action to energetic components, so the thermal stability of compound will descend with the introduction of Cu^{2+} to the stable FOX-7. However, Zn^{2+} has no catalytic action, which should be the reason for $Zn(NH_3)_2(FOX\text{-}7)_2$ exhibiting higher thermal stability than $Cu(NH_3)_2(FOX\text{-}7)_2$. The thermal stability of FOX-7 declines when it becomes salts or complexes, and the decomposition process also becomes severe [14–20].

In order to obtain the kinetic parameters (the apparent activation energy (E) and the pre-exponential constant (A)) of the first exothermic decomposition process, multiple heating methods (Kissinger method [28] and Ozawa method [29]) were employed. The determined values of the extrapolated onset temperature (T_e) and peak temperature (T_p) at different heating rates are listed in table 5. The calculated values of kinetic parameters (E and A) are also listed in table 5. The apparent activation energy obtained by Kissinger method is consistent with that by Ozawa method. All the linear correlation coefficients (r) are close to 1. So the results are credible. Moreover, the apparent activation energy of the exothermic decomposition process is low, indicating that $Zn(NH_3)_2(FOX\text{-}7)_2$ easily decomposes above 180 °C.

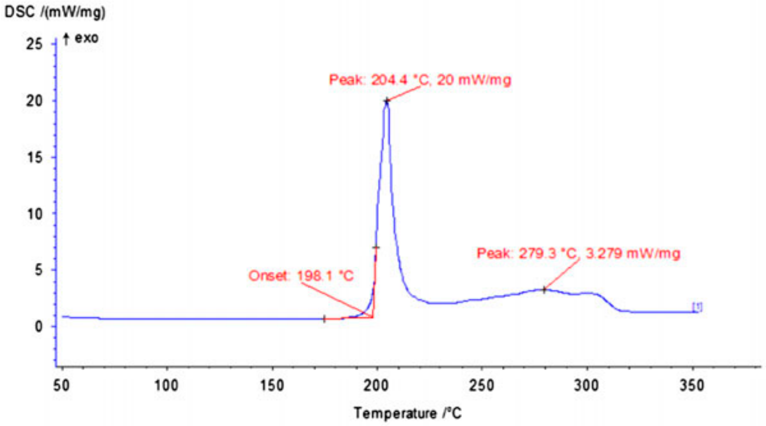

Figure 3. DSC curve of $Zn(NH_3)_2(FOX\text{-}7)_2$ at a heating rate of 10.0 °C min^{-1}.

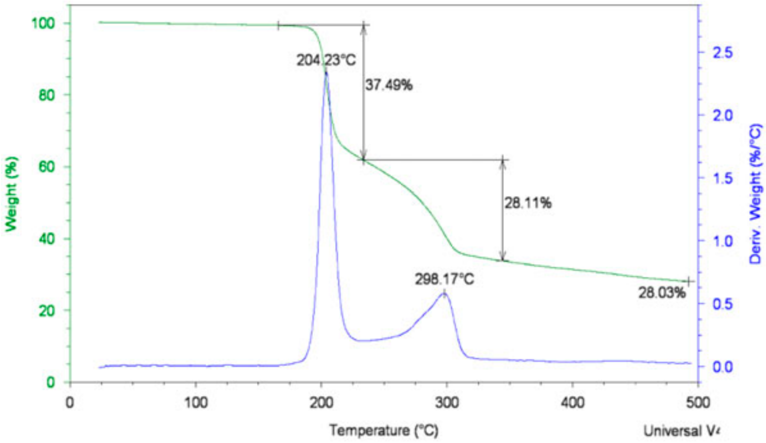

Figure 4. TG/DTG curves of $Zn(NH_3)_2(FOX\text{-}7)_2$ at a heating rate of 10.0 °C min^{-1}.

3.4. *Self-accelerating decomposition temperature and critical explosion temperature*

The self-accelerating decomposition temperature (T_{SADT}) and critical temperature of thermal explosion (T_b) are two important parameters required to ensure safe storage and process operations for energetic materials and then to evaluate the thermal stability. T_{SADT} and T_b can be obtained by equations (1) and (2) [30–32], respectively,

$$T_{SADT} = T_{e0} = T_{ei} - n\beta_i - m\beta_i^2 \quad i = 1-5 \tag{1}$$

$$T_b = \frac{E_O - \sqrt{E_O^2 - 4E_O R T_{e0}}}{2R} \tag{2}$$

where E_O is the apparent activation energy obtained by Ozawa method.

Table 5. The values of T_0, T_e, T_p, T_{00}, T_{e0} and kinetic parameters of the first exothermic decomposition process for $Zn(NH_3)_2(FOX\text{-}7)_2$ determined from the DSC curves at various heating rates (β).

β (K min^{-1})	T_e (°C)	T_p (°C)	E_k (kJ M^{-1})	log (A/s^{-1})	r_k	E_O (kJ M^{-1})	r_O
5.0	189.7	195.7	144.8	13.99	0.9795	145.2	0.9815
7.5	195.6	198.4					
10.0	198.1	204.4					
12.5	201.4	206.3					
15.0	203.5	207.8					

Subscript k, data obtained by Kissinger method; subscript O, data obtained by Ozawa method.

T_{SADT} and T_b for $Zn(NH_3)_2(FOX\text{-}7)_2$ are 183.2 and 195.8 °C, respectively, which all are much higher than those of analogous $Cu(NH_3)_2(FOX\text{-}7)_2$ as 142.10 and 152.86 °C [33], also indicating $Zn(NH_3)_2(FOX\text{-}7)_2$ has better thermal stability than $Cu(NH_3)_2(FOX\text{-}7)_2$.

3.5. *Sensitivity*

The experimental results indicate that the characteristic fall-height (H_{50}) of $Zn(NH_3)_2(FOX\text{-}7)_2$ is 45 cm (about 9 J). Explosion probability for friction sensitivity is 48% (25 time experiments). So $Zn(NH_3)_2(FOX\text{-}7)_2$ is sensitive. However, $Zn(NH_3)_2(FOX\text{-}7)_2$ still exhibits lower impact sensitivity than analogous $Cu(NH_3)_2(FOX\text{-}7)_2$ (5 J) [18].

4. Conclusions

$Zn(NH_3)_2(FOX\text{-}7)_2$ was synthesized and structurally characterized. $Zn(NH_3)_2(FOX\text{-}7)_2$ crystallizes in the monoclinic crystal system with space group $C2/c$. Zn^{2+} is coordinated by four nitrogens from two ammonias and two FOX-7$^-$ anions to form a distorted tetrahedron.

The thermal behavior of $Zn(NH_3)_2(FOX\text{-}7)_2$ presents two continuous exothermic decomposition processes. The self-accelerating decomposition temperature and critical temperature of thermal explosion of $Zn(NH_3)_2(FOX\text{-}7)_2$ are 183.2 and 195.8 °C, respectively. $Zn(NH_3)_2(FOX\text{-}7)_2$ has better thermal stability and less sensitivity than analogous $Cu(NH_3)_2(FOX\text{-}7)_2$.

Supplementary material

CCDC-873109 contains the supplementary crystallographic data for this article. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Center (CCDC), 12 Union Road, Cambridge, CB2, 1EZ, UK; Fax:+44 1223 336033; E-mail: deposit@ccdc.cam.ac.uk.

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References

[1] N.V. Latypov, J. Bergman, A. Langlet, U. Wellmar, U. Bemm. *Tetrahedron*, **54**, 11525 (1998).
[2] U. Bemm, H. Östmark. *Acta Crystallogr. C*, **54**, 1997 (1998).
[3] W.A. Trzciński, S. Cudzilo, Z. Chylek, L. Szymańczyk. *J. Hazard. Mater.*, **157**, 605 (2006).
[4] H.X. Gao, F.Q. Zhao, R.Z. Hu, Q. Pan, B.Z. Wang, X.W. Yang, Y. Gao, S.L. Gao, Q.Z. Shi. *Chin. J. Chem.*, **24**, 177 (2006).
[5] X.Z. Fan, J.Z. Li, Z.R. Liu. *J. Phys. Chem. A*, **111**, 13291 (2007).
[6] G. Majano, S. Mintova, T. Bein, T.M. Klapötke. *J. Phys. Chem. C*, **111**, 6694 (2007).
[7] J.J. Zhao, H. Liu. *Comp. Mater. Sci.*, **42**, 698 (2008).
[8] J.H. Ahn, J.K. Kim, H.S. Kim, E.J. Kim, K.K. Koo. *J. Chem. Eng. Data*, **54**, 3259 (2009).
[9] B. Huang, Z.Q. Qiao, F.D. Nie, M.H. Cao, J. Su, H. Huang, C.W. Hu. *J. Hazard. Mater.*, **184**, 561 (2010).
[10] V. Venkatesan, B.G. Polke, A.K. Sikder. *Comput. Theor. Chem.*, **995**, 49 (2012).
[11] S. Rajappa. *Tetrahedron*, **37**, 1453 (1981).
[12] G. Hervé, G. Jacob, N. Latypov. *Tetrahedron*, **61**, 6743 (2005).
[13] G. Hervé, G. Jacob, N. Latypov. *Tetrahedron*, **63**, 953 (2007).
[14] K.Z. Xu, C.R. Chang, J.R. Song, F.Q. Zhao, H.X. Ma, X.Q. Lv, R.Z. Hu. *Chin. J. Chem.*, **26**, 495 (2008).
[15] K.Z. Xu, X.G. Zuo, J.R. Song, F. Wang, J. Huang, C.R. Chang. *Chem. J. Chin. Univ.*, **31**, 638 (2010).
[16] J.A. Luo, K.Z. Xu, M. Wang, J.R. Song, X.L. Ren, Y.S. Chen, F.Q. Zhao. *Bull. Korean Chem. Soc.*, **31**, 2867 (2010).
[17] S. Garg, H.X. Gao, Y.H. Joo, D.A. Parrish, Y. Huang, J.M. Shreeve. *J. Am. Chem. Soc.*, **132**, 8888 (2010).
[18] S. Garg, H.X. Gao, D.A. Parrish, J.M. Shreeve. *Inorg. Chem.*, **50**, 390 (2011).
[19] T.T. Vo, D.A. Parrish, J.M. Shreeve. *Inorg. Chem.*, **51**, 1963 (2012).
[20] F. He, K.Z. Xu, H. Zhang, Q.Q. Qiu, J.R. Song, F.Q. Zhao. *J. Coord. Chem.*, **66**, 845 (2013).
[21] G.M. Sheldrick. *SHELXS*, University of Göttingen, Germany (1997).
[22] G.M. Sheldrick. *SHELXL, Program for X-ray Crystal Structure Refinement*, University of Göttingen, Germany (1997).
[23] M.J. Frisch, G.W. Trucks, H.B. Schlegel, *et al. Gaussian 03, Revision B.01*, Gaussian Inc., Pittsburgh, PA (2003).
[24] G. Fan, Y.L. Zhang, J.J. Sun, M.Y. Zheng. *J. Coord. Chem.*, **64**, 3711 (2011).
[25] Q. Yang, S.P. Chen, G. Xie, S.L. Gao. *J. Coord. Chem.*, **65**, 2584 (2012).
[26] Q. Yang, S.P. Chen, G. Xie, S.L. Gao. *J. Hazard. Mater.*, **197**, 199 (2011).
[27] J. Stierstorfer, K.R. Tarantik, T.M. Klapötke. *Chem. Eur. J.*, **15**, 5775 (2009).
[28] H.E. Kissinger. *Anal. Chem.*, **29**, 1702 (1957).
[29] T. Ozawa. *Bull. Chem. Soc. Jpn.*, **38**, 1881 (1965).
[30] R.Z. Hu, S.L. Gao, F.Q. Zhao, Q.Z. Shi, T.L. Zhang, J.J. Zhang. *Thermal Analysis Kinetics*, 2nd Edn, Science Press, Beijing (2008) (in Chinese).
[31] T.L. Zhang, R.Z. Hu, Y. Xie, F.P. Li. *Thermochim. Acta*, **244**, 171 (1994).
[32] S.W. Wang, L. Yang, T.L. Zhang, G.T. Zhang, J.G. Zhang, Z.N. Zhou. *J. Coord. Chem.*, **64**, 2583 (2011).
[33] Y.S. Chen, K.Z. Xu, M. Wang, H.X. Ma, F.Q. Zhao. *Chin. J. Explos. Propellants*, **34**, 5 (2011) (in Chinese).