Crystal Structure and Enthalpy of Combustion of AEFOX-7

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Abstract：The single crystal of 1-amino-1-ethylamino-2,2-dinitroethylene（AEFOX-7）was obtained. The crystal structure of AEFOX-7 · H_2O is orthorhombic, space group $Pna2(1)/m$ with crystal parameters of $a=1.3692(3)$ nm, $b=0.71240(16)$ nm, $c=0.9024(2)$ nm, $\beta=90°$, $V=0.8802(4)$ nm^3, $Z=4$, $\mu=0.133$ mm^{-1}, $F(000)=408$, $D_c=1.465$ g·cm^{-3}, $R_1=0.0306$ and $wR_2=0.0855$. Enthalpies of combustion of AEFOX-7, AMFOX-7 (1-amino-1-methylamino-2,2-dinitroethylene) and FOX-7 at 298.15 K were determined, and the corresponding enthalpies of combustion were −(2347.83±4.84), −(1819.96±5.94) kJ·mol^{-1} and −(1159.77±1.30) kJ·mol^{-1}, respectively.

Key words：1,1-diamino-2,2-dinitroethylene（FOX-7）; 1-amino-1-ethylamino-2,2-dinitroethylene（AEFOX-7）; crystal structure; enthalpy of combustion

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1 Introduction

1,1-Diamino-2,2-dinitroethylene（FOX-7）is a novel high-energy material with high thermal stability and low sensitivity, which has a same insensitivity to TATB and a similar energy density with RDX and HMX[1]. When first synthesized in 1998[1], FOX-7 received much attention and was considered as the main component potentially in insensitive ammunitions and solid propellants. Many researches have been carried out about the synthesis, mechanism, molecule structure, thermal behavior, detonation performance and application of FOX-7[2-4].

FOX-7 can react with some nucleophiles to synthesize new high energetic derivatives due to its "push-pull" nitroenamine structure, which possesses a highly polarized carbon-carbon double bond with positive and negative charges stabilized by the amino group and nitro group respectively[5-7]. The positive end of the carbon-carbon double bond can be attacked by nucleophilic reagents to synthesize new single substituted derivatives or disubstituted closed-loop derivatives. We have synthesized many derivatives of FOX-7, such as 1-amino-1-hydrazino-2,2-dinitroethene（AHDNE）, 1-amino-1-(2,4-dinitrophenylhydrazinyl)-2,2-dinitroethylene（APHDNE）, 1-amino-1-methylamino-2,2-dinitroethylene（AMFOX-7）, 2-(dinitromethylene)-1,3-diazacyclopentane（DNDZ）, 5-(dinitromethylene)-tetrazole（DNMT）, series of potassium and other salts or complexes[8-13], some of which will be used as energetic catalysts or energetic flame suppressor in propellant.

1-Amino-1-ethylamine-2,2-dinitroethene（AEFOX-7）was a derivative synthesized by our group in 2010, whose synthesis , theoretical calculation and thermal behavior have

been reported[13]. But the crystal structure of AEFOX-7 was not got by many methods. However, we obtain it with an unexpected method. This paper mainly reports the crystal structure and enthalpy of combustion of AEFOX-7.

2 Experiments

2.1 Sample

FOX-7 was obtained from Xi'an Modern Chemistry Research Institute. AEFOX-7 and AMFOX-7 were synthesized according to References [13-14]. The purities of the three compounds are all over 99%.

2.2 Determination of the single crystal structure

We tried to prepare Cd(ethamine)$_2$(FOX-7)$_2$ by putting Cd(NO$_3$)$_2$ and K(FOX-7)·H_2O into ethylamine aqueous solution（50%）. Although there was no success of Cd(ethamine)$_2$(FOX-7)$_2$, we finally got many crystals of AEFOX-7·H_2O. In the system, substitution reaction of FOX-7 happened at the catalytic action of Cd^{2+} at room temperature for 3 months.

The crystal with dimensions of 0.39 nm × 0.21 nm × 0.19 mm was chosen for X-ray determination. The data were collected on a Bruker SMART APEX CCD X-ray diffractometer using graphite-monochromated Mo K$_\alpha$ radiation（$\lambda=0.071073$ nm）. The structure was solved by the direct methods（SHELXTL-97）and refined by the full-matrix-block least-squares method on F^2 with anisotropic thermal parameters for all non-hydrogen atoms[15-16]. The hydrogen atoms were added according to the theoretical models. Crystal data and refinement results of AEFOX-7·H_2O are summarized in Table 1. CCDC No.：1042545.

2.3 Measurement of enthalpy of combustion

Enthalpy of combustion was determined by IKA C5000 oxygen-bomb calorimeter（German）adiabatically. The calorimeter was calibrated with the standard substance benzoic acid having a purity of 99.99%, and each sample was tested with 6 times. The calibration result of −(26503±73.30) J·g^{-1}

for the standard substance benzoic acid is very close to the standard value as $-(26434\pm5.8)$ J·g^{-1} at 298.15 K, indicating that the measuring system is accurate and reliable [17].

Table 1   Crystal data and structures refinement details

chemical formula	$C_4H_{10}N_4O_5$
formula mass/g·mol^{-1}	194.16
temperature /K	296(2)
wavelength/nm	0.071073
crystal system	orthorhombic
space group	$Pna2(1)$
a/nm	1.3692(3)
b/nm	0.71240(16)
c/nm	0.9024(2)
volume/nm^3	0.8802(4)
Z	4
D_c/g·cm^{-3}	1.465
absorption coefficient/mm^{-1}	0.133
$F(000)$	408
θ/(°)	2.98−25.10
index ranges	$-9\leqslant h\leqslant16$, $-8\leqslant k\leqslant8$, $-10\leqslant l\leqslant10$
reflections collected	1451
reflections unique	1279
refinement method	full-matrix least-squares on F^2
goodness-of-fit on F^2	1.060
final R indices [$I>2\sigma(I)$]	$R_1=0.0306$, $wR_2=0.0855$
R indices (all data)	$R_1=0.0352$, $wR_2=0.0904$
largest diff. peak and hole/(e·Å^{-3})	0.13 and −0.201

3   Results and Discussion

3.1   Crystal Structure

Molecular structure and crystal packing of AEFOX-7·H$_2$O are illustrated in Fig.1 and Fig.2. Selected bond lengths and bond angles of AEFOX-7·H$_2$O are listed in Table 2.

Table 2   Selected bond lengths and bond angles of AEFOX-7·H$_2$O

bond	length/nm	bond	angle/(°)
N(1)—O(1)	0.1186(6)	O(1)—N(1)—O(2)	122.0(4)
N(1)—O(2)	0.1267(4)	O(1)—N(1)—C(1)	117.3(3)
N(1)—C(1)	0.1396(6)	O(2)—N(1)—C(1)	120.6(3)
N(2)—O(3)	0.1204(4)	O(3)—N(2)—O(4)	121.5(3)
N(2)—O(4)	0.1323(5)	O(3)—N(2)—C(1)	126.7(3)
N(2)—C(1)	0.1352(5)	O(4)—N(2)—C(1)	111.8(3)
N(3)—C(2)	0.13077(19)	C(2)—N(4)—C(3)	123.43(13)
N(4)—C(2)	0.13004(18)	N(2)—C(1)—N(1)	123.25(13)
N(4)—C(3)	0.14674(18)	N(2)—C(1)—C(2)	120.6(4)
C(1)—C(2)	0.14877(19)	N(1)—C(1)—C(2)	116.0(4)
C(3)—C(4)	0.1502(2)	N(4)—C(2)—N(3)	123.19(13)
		N(4)—C(2)—C(1)	118.80(12)
		N(3)—C(2)—C(1)	118.00(12)
		N(4)—C(3)—C(4)	109.74(14)

The analytical results indicate that the molecule of AEFOX-7·H$_2$O is made up of an amino group, an ethyl-ami-

no group, two nitryls and a crystal water as shown in Fig.1. There are three orthogonal planes in the molecule of AEFOX-7: the nitro-nitro groups plane, the amino-imino groups plane and the ethyl group plane, the intersection of the first two planes is C(1)—C(2), while N(4)—C(3) combines the last two planes, and the plane to which the crystal water belongs parallels with the nitro-nitro groups plane. Comparing the results with that of the parent compound FOX-7 and other derivatives, such as 1-amino-1-methylamino-2, 2-dinitroethylene (AMFOX-7), one amino group is substituted by the nucleophile of methylamine. Consequently, the molecular symmetry is broken in a certain extent, the molecular configuration changes and the molecular structure almost transforms into own two orthogonal planes (the nitro-nitro groups plane and the amino-methylamino groups plane).
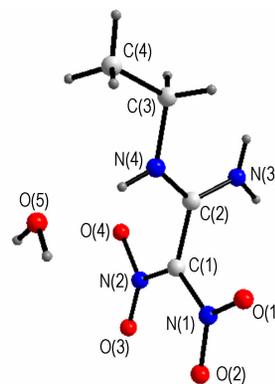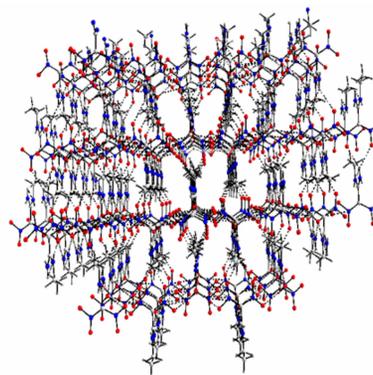

Fig.1   Molecular structure of AEFOX-7·H$_2$O

Fig.2   Crystal packing of AEFOX-7·H$_2$O

In accordance with the structural analysis, there is a hydrogen bond in the molecule. Each molecule of AEFOX-7 forms four hydrogen bonds, which are formed by the hydrogen atoms of the crystal water and connecting two adjacent nitryls of two adjoining molecules, with neighboring molecules to form an infinite 1D chain. The adjacent two 1D chains are attached by two kinds of hydrogen bonds to form a 2D monolayer network. These two sorts of hydrogen bonds appear between the amino groups of one 1D chain and the nitryls of the neighboring chain. The crystal packing (Fig.2) is formed by weak N—H···O interactions between adjacent 2D layers. The details of the above hydrogen bonds are shown in Table 3.

From Table 2, we can see that the bond length of

C(1)—C(2) (0.14877 nm) is longer than the average length of C—C double bond (0.132 nm) and close to the length of C—C single bond (0.153 nm). The bond lengths of C(2)—N(3) and C(2)—N(4) (all equal to 0.130 nm approximately) are shorter than the average length of C—N single bond (0.147–0.150 nm), and close to the length of conjugated C—N double bond (0.128 nm). All these prove that the obvious conjugative effect occurs between bond C(1)—C(2), C(2)—N(3), and C(2)—N(4).

Comparing related bond lengths of EAFOX-7 with that of FOX-7, it can be seen that the change is great[5]. The bond length

of C(1)—C(2) (0.14877 nm) is longer than that of FOX-7 as 1.454 nm. Bond C(1)—N(1) and C(1)—N(2) change greatly from 0.1426, 0.1387 nm in FOX-7 to 0.1396, 0.1352 nm in EAFOX-7. Corresponding C(2)—N(3) and C(2)—N(4) bond shorten form 0.1317, 0.1313 nm to 0.13077, 1.3004 nm. The introduction of ethyl group (electron-donating group) has an obvious effect on the configuration of molecule. The whole molecule presents a shift trend from amino groups to nitro groups. The introduction of ethyl group makes the "push-pull" effect enhance, also making EAFOX-7 exhibit many different physicochemical properties with FOX-7[13].

Table 3　Hydrogen bonds of AEFOX-7 · H$_2$O

D—H···A	d(D—H)/nm	d(H···A)/nm	d(D···A)/nm	∠DHA/(°)
	hydrogen bond in the molecule			
N(4)—H(4)···O(5)	0.086	0.2011	0.2863	171.12
	hydrogen bonds between adjacent molecules			
O(5)—H(5)A···O(4)#1	0.0823	0.1975	0.279	170.38
O(5)—H(5)A···N(2)#1	0.0823	0.2686	0.3433	151.66
O(5)—H(5)B···O(1)#2	0.082	0.1963	0.2773	169.17
O(5)—H(5)B···N(1)#2	0.082	0.2678	0.3386	145.54
	hydrogen bonds between 1D chains			
N(3)—H(3)A···O(3)#3	0.086	0.2365	0.3113	145.6
N(3)—H(3)A···O(2)#3	0.086	0.2386	0.3117	143.13
	hydrogen bond between 2D layers			
N(3)—H(3)B···O(5)#4	0.086	0.2031	0.2883	170.94

Note：(#1) -x+1/2, y-1/2, z+1/2; (#2) -x+1/2, y-1/2, z-1/2; (#3) -x-1/2, -y+3/2, z; (#4) x, y+1, z.

3.2　Constant-volume Combustion Enthalpy

From the determination results in Table 4, it can be seen that theconstant-volume combustion enthalpies for AEFOX-7, AMFOX-7

Table 4　Determination results of enthalpy of combustion

sample	no.	m/g	ΔT/K	−Δ$_c$U /J·g^{-1}
AEFOX-7	1	0.17203	0.2217	13328
	2	0.17208	0.2203	13235
	3	0.17237	0.2217	13301
	4	0.17224	0.2227	13375
	5	0.17224	0.2215	13299
	6	0.17316	0.2247	13429
	mean			13327.83±27.46
AMFOX-7	1	0.13845	0.1542	11254
	2	0.13897	0.1536	11359
	3	0.13840	0.1529	11156
	4	0.13898	0.1526	11278
	5	0.13838	0.1523	11111
	6	0.13842	0.1543	11194
	mean			11225.33±36.65
FOX-7	1	0.15387	0.1216	7821
	2	0.15396	0.1220	7844
	3	0.15412	0.1222	7850
	4	0.15325	0.1214	7838
	5	0.15471	0.1218	7792
	6	0.15265	0.1196	7741
	mean			7831.00±8.75

Note：m is mass of sample；ΔT is temperature rise；Δ$_c$U is energy of combustion.

and FOX-7 are −(13327.83±27.46), −(11225.33±36.65) J·g^{-1} and −(7831.00±8.75) J·g^{-1}, respectively. The sequence of enthalpy of combustion for these compounds is AEFOX-7 > AMFOX-7>FOX-7. So, the enthalpy of combustion rises with the increase of the number of C atom in molecule.

3.3　Enthalpy of Formation

The standard molar enthalpy of combustion ($\Delta_c H_m^\theta$) was referred to the combustion enthalpy change of the following i-dealized reaction formula (1) at T = 298.15 K and p^θ = 101.325 kPa[18].

$$M(s)+aO_2(g)=bCO_2(g)+cH_2O(l)+dN_2(g) \quad (1)$$

M = AEFOX-7: $C_4H_8O_4N_4$, a=4, b=4, c=4, d=2;
AMFOX-7: $C_3H_6O_4N_4$, a=5/2, b=3, c=3, d=2;
FOX-7: $C_2H_4O_4N_4$, a=1, b=2, c=2, d=2.

$\Delta_c H_m^\theta$ can be derived from the standard molar enthalpy of combustion in accordance with equations (2) and (3). The calculated results are listed in Table 5.

$$\Delta_c H_m^\theta = \Delta_c U_m^\theta + \Delta nRT \quad (2)$$

$$\Delta n = \sum n_i(\text{products, g}) - \sum n_i(\text{reactants, g}) \quad (3)$$

where $\sum n_i$ is the total molar amount of gases in products or reactants.

The standard molar enthalpy of formation of the compound($\Delta_f H_m^\theta$) can be calculated by Hess's law [18], according to the above thermochemical equation. Taking AEFOX-7 for instance, the standard molar enthalpy of formation is：

$$\Delta_f H_m^\theta(\text{AEFOX-7, s}) = 4\Delta_f H_m^\theta(CO_2, g) + $$
$$4\Delta_f H_m^\theta(H_2O, l) - \Delta_c H_m^\theta(\text{AEFOX-7, s})$$

The standard molar enthalpies of formation for CO_2 (g) and H_2O (l) recommended by CODATA, $\Delta_f H_m^\theta$ (CO_2, g) = $-(393.51\pm0.13)$ kJ $\cdot$ mol^{-1} and $\Delta_f H_m^\theta$ (H_2O, l) = $-(285.83\pm0.04)$ kJ $\cdot$ mol^{-1}, are employed in the calculation of $\Delta_f H_m^\theta$ (compound, s) values[17-18]. The standard molar enthalpies of formation for AEFOX-7, AMFOX-7 and FOX-7 can be calculated and listed in Table 5, and the results also indicate that the standard molar enthalpy of formation rises with the number of C atom in molecule. Moreover, the standard molar enthalpy of formation of FOX-7 [$-(206.35\pm1.33)$ kJ $\cdot$ mol^{-1}] is close to the literature value obtained by theoretical calculation -133.7 kJ $\cdot$ mol^{-1}[19].

Table 5 Standard molar enthalpies of constant-volume combustion, standard molar enthalpies of combustion and standard molar enthalpies of formation of the three compounds at 298.15 K

sample	M /g $\cdot$ mol^{-1}	$-\Delta_c U_m$ /kJ $\cdot$ mol^{-1}	$-\Delta_c H_m^\theta$ /kJ $\cdot$ mol^{-1}	$-\Delta_f H_m^\theta$ /kJ $\cdot$ mol^{-1}
AEFOX-7	176.16	2347.83±4.84	2342.87±4.84	374.49±4.87
AMFOX-7	162.13	1819.96±5.94	1813.77±5.94	224.25±5.95
FOX-7	148.10	1159.77±1.30	1152.33±1.30	206.35±1.33

Note: M is molecular mass; $\Delta_c U_m$ is standard molar enthalpy of constant-volume combustion; $\Delta_c H_m^\theta$ is standard molar enthalpy of combustion; $\Delta_f H_m^\theta$ is standard molar enthalpy of formation.

4　Conclusions

The single crystal of AEFOX-7 $\cdot$ H_2O is obtained unexpectedly. The crystal is orthorhombic with space group of $Pna2(1)/m$. The enthalpies of combustion of AEFOX-7, AMFOX-7 and FOX-7 are measured to be $-(2347.83\pm4.84)$, $-(1819.96\pm5.94)$, $-(1159.77\pm1.30)$ kJ $\cdot$ mol^{-1} respectively. Their corresponding standard molar enthalpies of formation are $-(374.49\pm4.87)$, $-(224.25\pm5.95)$ and $-(206.35\pm1.33)$ kJ $\cdot$ mol^{-1}, respectively. Both enthalpies of combustion and standard molar enthalpies of formation rise with the increase of the number of C atom in molecule.

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AEFOX-7 的晶体结构和燃烧焓

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摘　要：制备了1-氨基-1-乙氨基-2,2-二硝基乙烯(AEFOX-7)水合物的单晶并测定了其结构，该晶体属于立方晶系，空间群是 $Pna2(1)/m$，晶体参数为：$a=1.3692(3)$ nm，$b=0.71240(16)$ nm，$c=0.9024(2)$ nm，$\beta=90°$，$V=0.8802(4)$ nm^3，$Z=4$，$\mu=0.133$ mm^{-1}，$F(000)=408$，$D_c=1.465$ g·cm^{-3}，$R_1=0.0306$ and $wR_2=0.0855$。测定了 AEFOX-7、1-氨基-1-甲氨基-2,2-二硝基乙烯(AMFOX-7)和1,1-二氨基-2,2-二硝基乙烯(FOX-7)的燃烧焓，相应的标准摩尔燃烧焓分别是：−(2347.83±4.84)，−(1819.96±5.94) kJ·mol^{-1} 和−(1159.77±1.30) kJ·mol^{-1}。

关键词：1,1-二氨基-2,2-二硝基乙烯(FOX-7)；1-氨基-1-乙氨基-2,2-二硝基乙烯(AEFOX-7)；晶体结构；燃烧焓

中图分类号：TJ55；O614.1　　　　　　　　**文献标志码**：A　　　　　　　　**DOI**：10.11943/j.issn.1006-9941.2015.12.017

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2015 国际推进剂、炸药、烟火技术秋季研讨会在青岛成功举行

　　"2015国际推进剂、炸药、烟火技术秋季研讨会"于9月16−18日在青岛成功举行。出席会议的国内领导及知名专家有全国人大常委、中国科协副主席、北京理工大学冯长根教授，中国兵工学会副秘书长许毅达研究员，中北大学副校长肖忠良教授，西安近代化学研究所书记、副所长刘晓东研究员，湖北航天化学技术研究所副所长庞爱民研究员，爆炸科学与技术国家重点实验室主任张庆明教授，西安航天动力技术研究所科技委主任史宏斌研究员，以及胡双启教授、庞思平教授、冯昊研究员、张庆华研究员、李明研究员、吕龙研究员、史良伟研究员、彭汝芳教授、邵颖惠研究员等；国际知名学者有国际弹道学会主席 Clive R. Woodley 博士、美国航空航天学会终身成就奖获得者 Hugh J. McSPA DDEN 先生、美国 Karl Rink 教授、韩国 Chang Ha Lee 教授、意大利 Luigi De Luca 教授、俄罗斯 Valery P. Sinditskii 教授、荷兰 Antoine van der Heijden 研究员、土耳其 Burhan Lemi Türker 教授、捷克 Svatopluk Zeman 教授、以色列 Michael Gozin 教授、俄罗斯 Valeriy Yu. Filimonov 教授、俄罗斯 A. S. Smirnov 教授、俄罗斯 Alexander Lukin 研究员、俄罗斯 Vladimir A. Petrov 教授、英国 M Eamon Colclough 研究员、白俄罗斯 M. S. ASSAD 教授、韩国 Jin Seuk Kim 教授、韩国 Ju Young Kim 教授、韩国 Cheol Ho Choi 教授、韩国 Woo-Jae Kim 教授、韩国 Young Gyu Kim 教授。

　　参加此次会议代表共202人(境外72人)，分别来自中国、美国、英国、俄罗斯、白俄罗斯、加拿大、荷兰、以色列、意大利、印度、捷克、韩国、土耳其、新加坡、巴基斯坦、伊朗等。会议期间，Karl Rink 教授等11位国际知名学者作了大会报告，Young Gyu KIM 教授等6位教授作了分会场特邀报告，50篇论文在分会场进行了口头交流，20篇论文进行了海报展示。

　　"2015国际推进剂、炸药、烟火技术秋季研讨会"由中国兵工学会、北京理工大学、中国工程物理研究院化工材料研究所、应用物理化学重点实验室(陕西应用物理化学研究所)、中北大学、南京理工大学、湖北航天化学技术研究所、西安航天动力技术研究所、西安近代化学研究所和西南科技大学和青岛市科协联合主办；由北京理工大学火炸药研究院和中国兵工学会火炸药专业委员会协办；由爆炸科学与技术国家重点实验室(北京理工大学)和安全与防护协同创新中心共同承办。

　　"国际推进剂、炸药、烟火技术秋季研讨会"是一个系列会议，每两年举办一次。前10届会议分别在北京、深圳、成都、绍兴、桂林、北京、西安、昆明、南京和成都举行，得到了国内外与会学者的一致好评。这次国际会议的召开将进一步促进相关技术在我国的发展和繁荣，增进国内外学者之间的学术交流。