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Research paper

Synthesis and Characterization of Metal Derivatives of 1,1,2,2-Tetranitraminoethane (TNAE)

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Abstract: The introduction of anions and cations into energetic materials can significantly improve their physicochemical and energetic properties. The K, Zn, Ba, Cu and Pb salts of 1,1,2,2-tetranitraminoethane (TNAE) were synthesized using TNAE as the starting material, and were characterized by IR spectroscopy, NMR spectroscopy and elemental analysis. All of the energetic salts exhibited relatively high thermal stabilities (decomposition temperatures ranging from 195.1 °C to 279.3 °C) and high densities (ranging from 2.1 g·cm⁻³ to 3.7 g·cm⁻³). It was demonstrated that the introduction of metal ions conveys better thermal stability than covalent TNAE itself. More importantly, the Zn, Ba and Cu salts exhibited good thermal stability, high density, low sensitivity, and therefore have the potential to be a new class of insensitive, highly energetic explosives for practical applications.

Keywords: Ionic compounds, 1,1,2,2-tetranitraminoethane, thermal property, safety performance, thermal decomposition kinetics

Supporting information (SI) available at:

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1 Introduction

1,1,2,2-Tetranitraminoethane (TNAE) is a high-energy, oxygen-rich explosive that can be prepared by hydrolysis of tetranitroglycoluril (TNGU) or

1,3,4,5,7,8-hexanitrooctahydrodiimidazo-[4,5-b:4',5'-e]-pyrazine-2,6(1*H*,3*H*)-dione (HHTDD) in the presence of alkali [1, 2]. The four primary nitramino groups in TNAE possess good reactivity, and can be further functionalized to produce heterocyclic nitramines [3] and cage-like energetic materials such as CL-20 [2]. Although a lot of energetic materials with great detonation properties have been reported, TNAE being one such, high energy is always accompanied by a major disadvantage, high sensitivity. Therefore, the modification of these energetic compounds to reduce the sensitivity while maintaining good detonation performance has become an important research direction. There are many reports that have shown good results can be achieved by combining energetic materials, such as furazans, azoles, nitriles, nitrobenzenes and non-aromatics, with anions and cations [4-12]. Therefore, preparation of energetic salts or complexes has provided a novel strategy for searching for insensitive, highly energetic materials. It has been successfully applied in the fields of primary explosives, energetic catalysts, rocket propellants, *etc.* [13-15].

With poor thermal stability, TNAE slowly undergoes hydrolysis in mildly acidic and neutral conditions. Due to the acidity of the nitramino groups, TNAE can form stable salts with counter ions, such as metal ions, carbamylguanidinium, ammonium, guanidinium, hydrazinium, hydroxylamine, triaminoguanidine, which were found to be interesting as new energetic ingredients [2, 16]. The sodium salt of TNAE has significantly improved detonation performance and is by far one of the highest energy compounds [17, 18]. Recently, Born *et al.* [19] reported the crystal structures, sensitivities, and aquatic toxicity of the K and Na salts of TNAE, in which the results showed that both the K and Na salts were sensitive to impact. In order to search for more insensitive and higher energy TNAE salts, we used TNAE as the parent compound to synthesize the K, Ba, Cu, Zn and Pb salts of TNAE by deprotonation. Their structural characterization and related performances were tested. The thermal stability of all of the salts obtained were greatly improved and the crystal density had increased significantly. Among these salts, the Zn salt, Ba salt and Cu salt were very insensitive to impact and friction.

2 Experimental

Caution: TNAE and its salts are energetic materials and are sensitive to impact and friction. In the process of these experiments, the necessary personal protective equipment is strongly recommended, *e.g.* explosion-proof shield, leather gloves, and small scale are strongly encouraged!

2.1 Materials

Tetranitroglycoluril and TNAE were prepared in the laboratory [17, 18, 20], and all others reagents were purchased from local commercial suppliers and used without any further purification.

Tetra-potassium 1,1,2,2-tetranitraminoethane (TNAE-K): TNAE (5.40 g, 20 mmol) was dissolved in MeOH (100 mL) and K_2CO_3 (5.52 g, 40 mmol) was added in portions. After mechanical stirring at room temperature for 2 h, the reaction product was filtered off and the filter cake was washed with methanol (50 mL) and dried *in vacuo* to give the target product (7.82 g, 92.7%). 1H NMR (600 MHz, D_2O): 5.91 (s, 1H, CH) ppm; ^{13}C NMR (150 MHz, D_2O): 77.5 ppm; IR (KBr, cm^{-1}) ν : 2961 (C-H), 1674 (N-N), 1404 (N-O), 1380 (N-O), 1319 (N-N), 1247 (C-C), 1105 (C-N), 959 (C-C); Analysis Calcd. for $K_4C_2H_2N_8O_8 \cdot H_2O$: C 5.45, H 0.92, N 25.44%; Found: C 6.18; H 0.88; N 25.44%.

Di-barium 1,1,2,2-tetranitraminoethane (TNAE-Ba): $Ba(CH_3COO)_2$ (10.24 g, 40 mmol) was slowly added to a solution of TNAE (5.40 g, 20 mmol) in distilled water (100 mL). The reaction mixture was stirred at room temperature for 1 h to give a white suspension. The precipitated product was filtered off, and washed with methanol and water until the filtrate was neutral. The product obtained was dried *in vacuo* to give Ba_2TNAE (10.65 g, 98.3%). IR (KBr, cm^{-1}) ν : 2942 (C-H), 1646 (N-N), 1429 (N-O), 1311 (N-N), 1274 (C-N), 1237 (C-C), 1118 (C-N), 973 (C-C); Analysis Calcd. for $Ba_2C_2H_2N_8O_8 \cdot 3H_2O$: C 4.04, H 1.36, N 18.84%; Found: C 4.18; H 1.60; N 18.51%.

Di-copper 1,1,2,2-tetranitraminoethane (TNAE-Cu): The compound $Cu(NO_3)_2$ (2.24 g, 12 mmol) was added to a solution of K_4TNAE (2.11 g, 5 mmol) in water (60 mL); the reaction solution immediately turned blue and a blue solid precipitated. After mechanical stirring at room temperature for 1 h, the precipitated product was filtered off, and was washed with methanol and water until the filtrate was neutral. The product obtained was dried *in vacuo* to give Cu_2TNAE (1.42 g, 72.3%). IR (KBr, cm^{-1}) ν : 1622 (N-N), 1419 (N-O), 1342 (N-N), 1281 (C-N), 1142 (C-N), 1026 (C-C); Analysis Calcd. for $Cu_2C_2H_2N_8O_8 \cdot H_2O$: C 5.84, H 0.98, N 27.25%; Found: C 4.14; H 1.52; N 19.11%.

Di-zinc 1,1,2,2-tetranitraminoethane (TNAE-Zn): The compound $Zn(NO_3)_2 \cdot 6H_2O$ (3.35 g, 12 mmol) was added to a solution of K_4TNAE (2.11 g, 5 mmol) in water (60 mL). A white solid precipitated immediately from the reaction solution. After mechanical stirring at room temperature for 1 h, the precipitated product was filtered off, and was washed with methanol and water until the filtrate was neutral. The product obtained was dried *in vacuo* to give Zn_2TNAE (0.88 g, 44.7%). IR (KBr, cm^{-1}) ν : 2973 (C-H), 1633 (N-N), 1367 (N-O), 1285 (C-N), 1130 (C-N), 1024 (C-C); Analysis Calcd. for $Zn_2C_2H_2N_8O_8 \cdot 2H_2O$: C 5.55, H 1.4,

N 25.89%; Found: C 5.54; H 1.20; N 24.73%.

Di-lead 1,1,2,2-tetranitraminoethane (TNAE-Pb): The compound $\text{Pb}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (3.57 g, 12 mmol) was added to a solution of K_4TNAE (2.11 g, 5 mmol) in water (60 mL). A white solid precipitated immediately from the reaction solution. After mechanical stirring at room temperature for 1 h, the precipitated product was filtered off, and was washed with methanol and water until the filtrate was neutral. The product obtained was dried *in vacuo* to give Pb_2TNAE (2.51 g, 73.5%). IR (KBr, cm^{-1}): 1627 (N-N), 1415 (N-O), 1347 (N-N), 1278 (C-N), 1146 (C-N), 1027 (C-C); Analysis Calcd. for $\text{Pb}_2\text{C}_2\text{H}_2\text{N}_8\text{O}_8 \cdot \text{H}_2\text{O}$: C 3.44, H 0.58, N 16.04%; Found: C 4.37; H 0.58; N 14.99%.

2.2 Instrumental methods

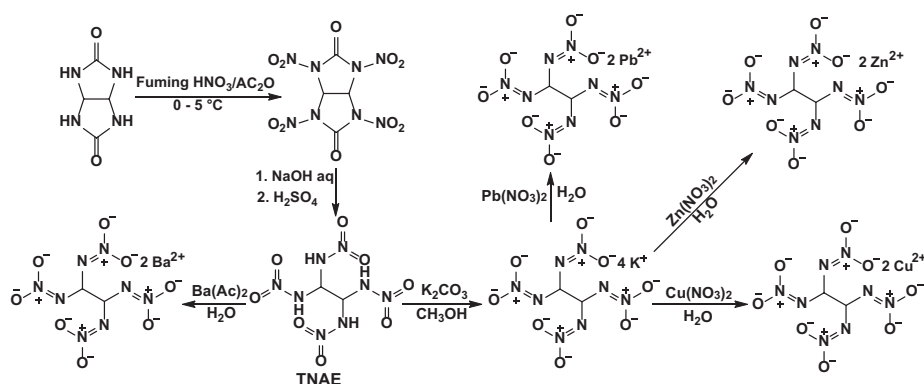
IR spectra were recorded on a PerkinElmer 1600 Fourier transform infrared spectrometer (Boston, MA, USA). ^1H NMR (600 MHz) and ^{13}C NMR (150 MHz) spectra were recorded with a Bruker Avance II 600 spectrometer (Munich, Germany). Elemental analyses were obtained on an Elementar Vario MICRO CUBE (Germany) elemental analyzer. Thermal analysis was carried out using a Shimadzu DSC-60 (Japan) instrument under a nitrogen atmosphere at a flow rate of $50 \text{ mL} \cdot \text{min}^{-1}$; sample weight was about 0.7 mg; heating rates $5 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$, $10 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$, $15 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$, $20 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$ and $25 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$, from ambient temperature to $450 \text{ }^\circ\text{C}$. Density data were determined using a Micromeritics Accupyc II 1340 gas pycnometer at $25 \text{ }^\circ\text{C}$.

The sensitivity to impact and friction stimuli were tested according to China's national military standard (GJB 772A-97) [21]. The sensitivity to impact was tested using a Fall Hammer WB-IIB (China), and the method in detail was as follow: a 10 kg drop hammer was allowed to fall freely from a height of 25 cm onto the test sample ($50 \pm 1 \text{ mg}$), and the results were expressed as a percentage of the impact sensitivity (the ratio of the number of explosions to the number of experiments). Sensitivity to friction was measured on a Friction Apparatus WI-IIB (China), and a sample weight of $20 \pm 1 \text{ mg}$ was measured under the following conditions: drop hammer: 1.5 kg, tilt angle: $90 \pm 1^\circ$, positive pressure: $3.92 \pm 0.07 \text{ MPa}$, and the sensitivity to friction was expressed as a percentage of the friction sensitivity (the ratio of the number of explosions to the number of experiments). During the sensitivity to impact and friction tests, the experiments were repeated 25 times for each group, a total of 2 parallel experiments for each kind of sample, and the final results were taken as the average.

3 Results and Discussion

3.1 Synthesis

The synthetic method for TNAE was based on previous literature [17, 18] (Scheme 1). Basically, the TNAE salts were mainly prepared by the solubility differences of the salts in water and alcohol. K salt was insoluble in CH_3OH and soluble in H_2O , hence the reaction was carried out in CH_3OH , and the target product was obtained by direct filtration after the reaction was complete. It may be further purified by recrystallization from water. Analogously, TNAE reacted with $\text{Ba}(\text{CH}_3\text{COO})_2$ in water to afford the Ba salt. The Zn salt, Cu salt and Pb salt were prepared by a substitution reaction between the K salt and the corresponding nitrates. All of the salts obtained were characterized by FTIR and elemental analysis. In all of the IR spectra of the TNAE salts, the strong IR absorption around 3200 cm^{-1} (N-H) of TNAE was absent, while other characteristic absorption peaks of TNAE were retained. Since the K salt could be dissolved in water, but the other salts were insoluble in both organic solvents and water, only the K salt could be identified by NMR spectroscopy.



Scheme 1. Synthetic route to TNAE and its salts

3.2 Thermal properties

The DSC curves, at a heating rate of $5\text{ }^\circ\text{C}/\text{min}$, for TNAE-Pb, TNAE-K, TNAE-Zn, TNAE-Cu and TNAE-Ba are shown in Figure 1. The decomposition temperature (T_d) of the K salt was the highest ($279.3\text{ }^\circ\text{C}$), and possessed the best thermal stability, followed by the Zn salt ($244.3\text{ }^\circ\text{C}$), the Ba salt ($234.4\text{ }^\circ\text{C}$), the Pb salt ($199.7\text{ }^\circ\text{C}$) and the Cu salt ($195.1\text{ }^\circ\text{C}$). Obviously, the thermal stability is greatly improved by salt formation (T_d (TNAE) = $158.7\text{ }^\circ\text{C}$ [1]). Of these salts, the thermal stabilities of the K, Ba, and Zn salts were better than that of RDX

($T_d = 230\text{ }^\circ\text{C}$) [22], and slightly lower than HMX ($T_d = 287\text{ }^\circ\text{C}$) [22], which can be classified as a heat-resistant energetic material. There were two exothermic peaks in the curve for the K salt, one corresponding to the decomposition of the K salt and the other likely to be due to the decomposition of K_2O [23]. In addition, it is important to note that the data we obtained for TNAE-K was very close to that reported by Lee *et al.* ($284\text{ }^\circ\text{C}$) [2], and considerably different from that of Born ($225\text{ }^\circ\text{C}$) [19], which is probably related to the presence of crystal water. In the curve for the Zn salt, there was a small exothermic peak after the main exothermic peak, which is presumed to be due to the secondary decomposition of the decomposition residue.

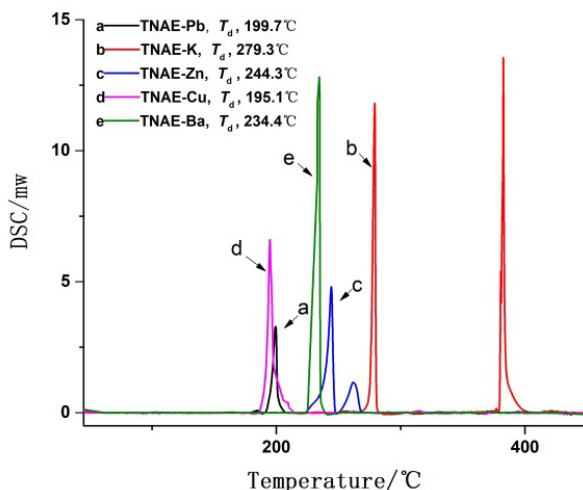


Figure 1. DSC curves of the Pb, K, Zn, Cu and Ba salts of TNAE at a heating rate of $5\text{ }^\circ\text{C}\cdot\text{min}^{-1}$

3.3 Safety tests

The sensitivity of the salts to impact and friction are listed in Table 1. The Pb salt was the most sensitive to impact (100%) and friction (100%). Although the K salt was very insensitive to friction (8%), it was very sensitive to impact (100%). The Ba, Cu and Zn salts all had a low degree of friction and impact sensitivity (percentage), with the Ba salt being the most insensitive. In order to compare the performance of these salts with commonly used explosives, we measured the sensitivities of HMX and RDX under the same experimental conditions. The sensitivities of the Ba, Cu and Zn salts were obviously lower than those of HMX, RDX and TNAE. Moreover, the density data was also obtained using the standard container method (Table 1). Compared with TNAE ($1.836\text{ g}\cdot\text{cm}^{-3}$) [1],

the density was significantly improved after the introduction of the metal ions. In particular, the density of the Pb salt was up to $3.7 \text{ g}\cdot\text{cm}^{-3}$, but its high sensitivity limits its application. Since TNAE has 4 nitramino groups and only 2 carbon atoms, it possesses a positive oxygen balance (5.92%). Upon formation of the salts, the oxygen balance becomes closer to zero, which suggested that these energetic salts only release a very limited amount of harmful gas (e.g. N_xO_y).

Table 1. Results of the sensitivity tests and other selected parameters of the five TNAE salts and commonly used explosives

Compound	IS ^a [%]	FS ^b [%]	T_d^c [°C]	ρ^d [g·cm ⁻³]	Ω^e [%]	N ^f [%]
TNAE-K	100	8	279.3	2.1032	+3.79	26.55
TNAE-Ba	0	12	234.4	2.3935	+2.95	20.67
TNAE-Cu	28	16	195.1	2.4752	+4.08	28.58
TNAE-Zn	40	4	244.3	2.8662	+4.06	28.44
TNAE-Pb	100	100	199.7	3.7059	+2.35	16.42
HMX	100	96	287	1.91	-21.6	37.84
RDX	80	76	230	1.816	-21.6	37.84
TNAE	98	90	158.7	1.836	+5.92	41.48

^a Impact sensitivity; ^b Friction sensitivity; ^c Decomposition temperature (DSC/5 °C·min⁻¹);

^d Measured density; ^e Oxygen balance $\Omega = 1600 \cdot [w\text{O} - 2\text{C} - 0.5\text{yH} - 2]/M_w$; ^f Nitrogen content

3.4 Thermolysis

Our data so far had shown that the K salt had the best stability, that the Zn salt had moderate thermal stability and sensitivity, and that Pb salt was a commonly used addition component in explosives. Therefore, we studied further the thermal decomposition kinetics of these salts using different DSC heating rates (5 °C·min⁻¹, 10 °C·min⁻¹, 15 °C·min⁻¹, 20 °C·min⁻¹ and 25 °C·min⁻¹). The data was processed using the Kissinger method [24], and gave the thermal decomposition kinetic parameters for the exothermic decomposition peaks (Table 2). As shown in Table 2, the decomposition temperature increased as the heating rate was increased. The apparent activation energy of the Zn salt was 213.75 kJ·mol⁻¹, which was higher than the K salt (181.91 kJ·mol⁻¹), the Pb salt (156.39 kJ·mol⁻¹) and TNAE (151.32 kJ·mol⁻¹) [25]. This indicated that the Zn salt had theoretically a better stability. The K salt had the highest decomposition temperature in the actual test, but the theoretically calculated thermal stability was not as good as that of the Zn salt, which may be related to the crystal structure and particle morphology of the salts. In addition, all linear correlation coefficients were greater than 0.98, which indicated that the

correlations were good. Simultaneously, the corresponding Arrhenius equation was also calculated and is listed in the Table 2.

Table 2. Kinetic parameters for the K, Zn and Pb salts

Compound	β^a [°C·min ⁻¹]	T_p^b [K]	E_k^c [kJ·mol ⁻¹]	$\ln(A)^d$ [s ⁻¹]	r^e	Arrhenius equation, $\ln k =$
TNAE-K	5	551.15	181.91	38.70	0.988	$38.70-181.91 \times 10^3/RT$
	10	560.59				
	15	566.16				
	20	568.39				
	25	574.03				
TNAE-Zn	5	517.48	213.75	48.99	0.982	$48.99-213.75 \times 10^3/RT$
	10	524.77				
	15	527.12				
	20	529.75				
	25	534.98				
TNAE-Pb	5	469.33	156.39	39.30	0.987	$39.30-156.39 \times 10^3/RT$
	10	475.73				
	15	480.09				
	20	484.68				
	25	487.82				

^a DSC heating rates; ^b Decomposition temperature; ^c Calculated activation energies; ^d Calculated preexponential factor; ^e Linear correlation

4 Conclusions

In summary, five metal salts of TNAE have been synthesized and characterized. After the introduction of the metal ions, the TNAE-based salts showed much higher density and better thermodynamic stability compared to the undeprotonated TNAE. The Zn salt and the Ba salt were only outperformed by HMX, RDX and TNAE in the thermodynamic and sensitivity tests, and these results make them promising candidates as insensitive, highly energetic ingredients for practical applications. Furthermore, the thermal decomposition kinetics of the K, Zn and Pb salts were studied, and the apparent activation energies were 181.91 kJ·mol⁻¹, 213.75 kJ·mol⁻¹ and 156.39 kJ·mol⁻¹, respectively. This can provide an important reference for the further study of the synthesis and applications of TNAE and its salts.

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