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Thermal behavior of 3,4,5-triamino-1,2,4-triazole nitrate

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ABSTRACT

The thermal decomposition behaviors of 3,4,5-triamino-1,2,4-triazole nitrate were studied using a C-500 type Calvet Micro-calorimeter at four different heating rates. Its apparent activation energy and pre-exponential factor of exothermic decomposition reaction are 137.16 kJ mol^{-1} and $10^{10.03}$ s^{-1}. The critical temperature of thermal explosion is 441.37 K. The entropy of activation ($\Delta S^{\neq}$), enthalpy of activation ($\Delta H^{\neq}$), and free energy of activation ($\Delta G^{\neq}$) of the decomposition reaction are −65.65 J mol^{-1} K^{-1}, 132.97 kJ mol^{-1} and 166.35 kJ mol^{-1}, respectively. The self-accelerating decomposition temperature (T_{SADT}) is 429.56 K. The specific heat capacity was determined with a Micro-DSC method and a theoretical calculation method. Specific heat capacity (J g^{-1} K^{-1}) equation is $C_p = 0.356 + 2.957 \times 10^{-3}T$ (283.1 K < T < 353.2 K). The adiabatic time-to-explosion is calculated to be a certain value between 702.22 s and 712.22 s. The critical temperature of Hot-Spot initiation is 600.325 °C, and the characteristic drop height of impact sensitivity (H_{50}) is 27 cm.

1. Introduction

The most accepted definition of an ionic liquid is a salt with melting point below 373.15 K [1–3]. Ionic liquids attract increasing attention among academic and industrial chemists for their unique properties. The chemical and thermodynamic stabilities of these compounds, combined with their low vapor pressures and low melting points, have put them forth as potential replacements of many noxious organic solvents used in the chemical industry.

Recently, many studies in developing energetic salts and energetic ionic liquids based on 1,2,3-triazole and 1,2,4-triazole as cations and nitrates, perchlorates, and dinitramides as anions have been made [4–6]. On the thermal aspect, kinetic and thermodynamic of 3,4,5-triamino-1,2,4-triazole nitrate is a basic task in the content of thermal process. The thermal behavior of materials may be determined in a short time using thermal analysis techniques such as DSC, TG-DTG, but more intensive and precise thermal analysis can be carried out by using Micro-calorimeter.

In this work, a C-500 Calvet Micro-calorimeter was applied for researching the thermal decomposition behavior of 3,4,5-triamino-1,2,4-triazole nitrate at four different heating rates, and the data were analyzed to obtain the self acceleration decomposition temperature (T_{SADT}) and critical temperature of thermal explosion (T_b), adiabatic time-to-explosion and critical temperature of Hot-Spot initiation were obtained at the same time.

2. Experimental

2.1. Materials

3,4,5-Triamino-1,2,4-triazole nitrate in this research was prepared by Beijing Institute of Technology and Xi'an Modern Chemistry Research Institute. The structure was characterized by means of organic elemental analyzer, ^{15}N NMR, ^{13}C NMR and HPLC–MS. The sample's purity was >99.5 mass%. The structure for 3,4,5-triamino-1,2,4-triazole nitrate are shown in Scheme 1. The 3,4,5-triamino-1,2,4-triazole nitrate was further purified by subjecting the sample to a very low pressure of about 5×10^{-3} Pa at temperature about 70 °C for approximately 5 h. This procedure removed any volatile chemicals and water from the triazole ionic salts.

2.2. Equipment and conditions

The heat flow curves of the exothermic decomposition process of 3,4,5-triamino-1,2,4-triazole nitrate was measured using a Calvet Micro-calorimeter, type C-500 (Setaram, France), which had a sensitivity of 0.10 μV mW^{-1} and was equipped with two 10 mL ves-

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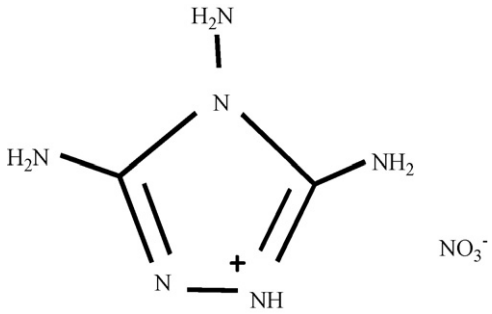

Scheme 1. The structure of 3,4,5-triamino-1,2,4-triazole nitrate.

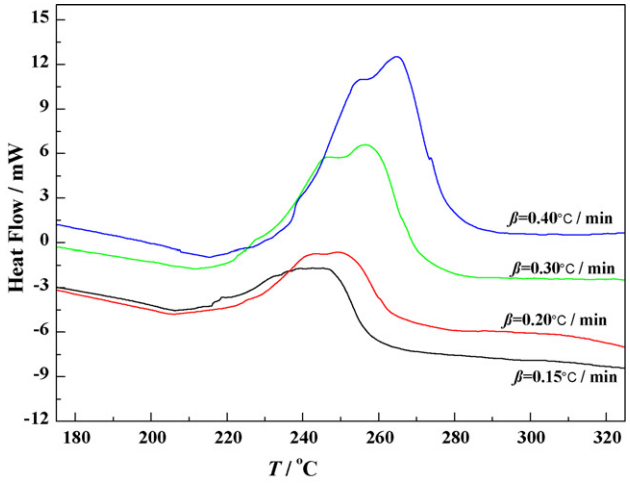

Fig. 1. Heat flow curves of 3,4,5-triamino-1,2,4-triazole nitrate at different heating rates.

sels. The precision of enthalpy measurement was less than 0.5% after adjusting. The four different heating rates used were 9 K h^{-1} (0.15 K min^{-1}), 12 K h^{-1} (0.20 K min^{-1}), 18 K h^{-1} (0.30 K min^{-1}) and 24 K h^{-1} (0.40 K min^{-1}), respectively. Sample used was about 10 mg, and the reference sample was α-Al_2O_3. Each process was repeated three times, and the heat flow curves under same conditions overlap with each other, indicating that the reproducibility of test is satisfactory.

The measurement of specific heat capacity (C_p) was performed using a Micro-DSC III apparatus (Setaram, France). The amount of used sample was 479.29 mg. The heating rate was 9 K h^{-1} from temperatures 283.1 K to 353.2 K.

3. Results and discussion

3.1. Thermal decomposition behavior

The heat flow curves of 3,4,5-triamino-1,2,4-triazole nitrate at different heating rates are show in Fig. 1, which show only one exothermic peak. The characteristic temperatures obtained by heat flow curves at different heating rates are listed in Table 1. In Table 1,

Table 1
Original data of the thermal decomposition reaction of 3,4,5-triamino-1,2,4-triazole nitrate at different heating rates.

β (K min^{-1})	T_e (K)	T_p (K)	$Q = -\Delta H$ (kJ mol^{-1})
0.15	482.23	518.36	365.40
0.20	487.67	522.06	367.80
0.30	490.99	527.74	369.34
0.40	496.01	533.91	361.56

Table 2
Kinetic parameters obtained from the data in Table 1.

Kissinger method			Ozawa method		T_{p0} (K)
E_k (kJ mol^{-1})	lg(A_k) (s^{-1})	r_k	E_{op} (kJ mol^{-1})	r_{op}	
137.16	10.03	0.9938	138.75	0.9945	508.41

β is the heating rate, T_p the peak temperature, T_e the onset temperature, and ΔH the enthalpy of decomposition reaction.

In order to obtain the apparent activation energy (E) and the pre-exponential constant (A) of the decomposition reaction for 3,4,5-triamino-1,2,4-triazole nitrate, multiple heating methods (Kissinger method [7] and Ozawa method [8]) were employed. Kissinger and Ozawa equations are as follows [9–14]

$$\ln\left(\frac{\beta_i}{T_{pi}^2}\right) = \ln\frac{AR}{E} - \frac{E}{R}\frac{1}{T_{pi}} \quad [i = 1, 2, \ldots, 4] \tag{1}$$

$$lg\beta_i = lg\left(\frac{AE}{RG(\alpha)}\right) - 2.315 - 0.4567\frac{E}{RT_i} \quad [i = 1, 2, \ldots, 4] \tag{2}$$

where β_i is the linear heating rate, T_{pi} represents the peak temperature of the decomposition process, R is the gas constant.

From the original data in Table 1, the values of E and A obtained by Kissinger method (with a subscript of k) and Ozawa method (with a subscript of o) are listed in Table 2.

With the data listed in Table 2, entropy of activation ($\Delta S^{\neq}$), enthalpy of activation ($\Delta H^{\neq}$), and free energy of activation ($\Delta G^{\neq}$) of the decomposition reaction corresponding to $T = T_p$, $E = E_k$ and $A = A_k$ obtained by Eqs. (3)–(5) are −65.65 J mol^{-1} K^{-1}, 132.97 kJ mol^{-1} and 166.35 kJ mol^{-1}, respectively.

$$A_k = \left(\frac{k_B T_{p0}}{h}\right) e^{\Delta S^{\neq}/R} \tag{3}$$

$$\Delta H^{\neq} = E_k - RT_{p0} \tag{4}$$

$$\Delta G^{\neq} = \Delta H^{\neq} - T_{p0}\,\Delta S^{\neq} \tag{5}$$

where k_B the Boltzmann constant (1.3807 × 10^{-23} J K^{-1}), and h the Plank constant (6.626 × 10^{-34} J s^{-1}).

The positive value of $\Delta G^{\neq}$, indicates that the exothermic decomposition reaction for 3,4,5-triamino-1,2,4-triazole nitrate must proceed under the heating condition.

3.2. Self-accelerating decomposition temperature (T_{SADT}) and critical temperature of thermal explosion (T_b)

The value T_{e0} (T_{SADT}) of T_e corresponding to $\beta = 0$ can be obtained by substituting the T_e and β from Table 1 into Eq. (6) [13–18]. The value of T_{e0} (T_{SADT}) is obtained is 429.56 K.

$$T_{(e,p)i} = T_{(e,p)0} + b\beta_i + c\beta_i^2 + d\beta_i^3 \quad [i = 1, 2, \ldots, 4] \tag{6}$$

where b, c and d are coefficients.

The critical temperature of thermal explosion T_b obtained from Eq. (7) taken from Refs. [13–18] is 441.37 K, where $E_o = E_{op}$.

$$T_b = \frac{E_o - \sqrt{E_o^2 - 4E_o RT_{SADT}}}{2R} \tag{7}$$

3.3. Specific heat capacity (C_p)

Fig. 2 shows the determination results of 3,4,5-triamino-1,2,4-triazole nitrate using a continuous specific heat capacity mode of a Micro-DSC III apparatus. In determining temperature range, specific heat capacity of 3,4,5-triamino-1,2,4-triazole nitrate presents

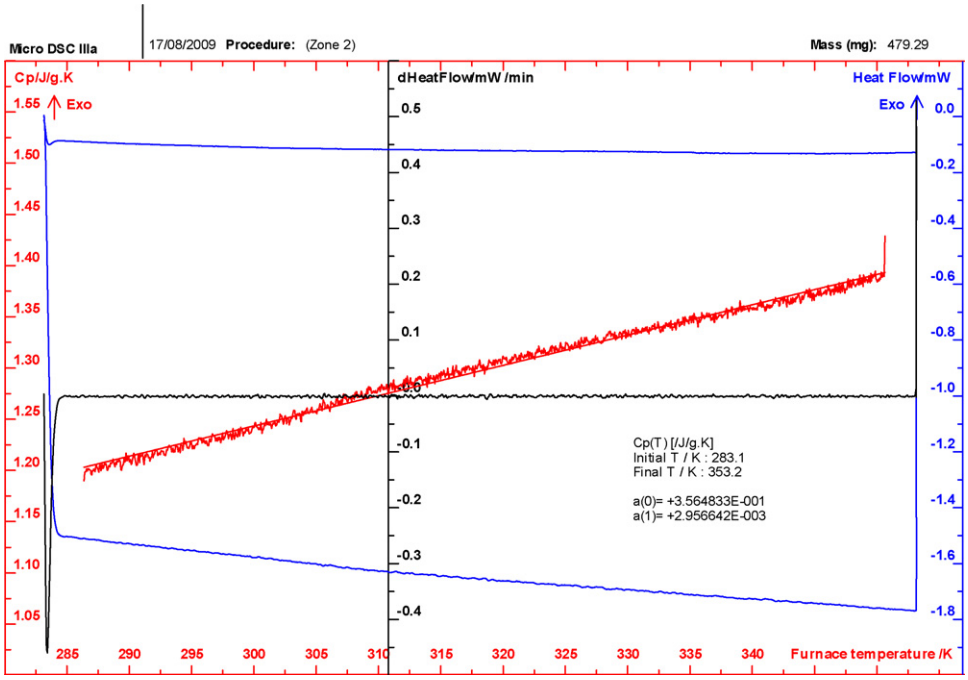

Fig. 2. Determination results of the continuous specific heat capacity.

a good linear relationship with temperature. Specific heat capacity ($J\,g^{-1}\,K^{-1}$) equation is shown as:

$$C_p = 0.356 + 2.957 \times 10^{-3}T \quad (283.1\,K < T < 353.2\,K) \tag{8}$$

The molar heat capacity of 3,4,5-triamino-1,2,4-triazole nitrate is 221.81 $J\,mol^{-1}\,K^{-1}$ at 298.15 K. Although only a 70.0 K range was taken in the determination process, the specific heat capacity equation obtained was a stable and continuous equation, which can provide a reference and some help for wide temperature applications.

3.4. Adiabatic time-to-explosion

Energetic materials need a time from the beginning thermal decomposition to thermal explosion in the adiabatic conditions. The time is named the adiabatic time-to-explosion [16–20]. Ordinarily, the heating rate (dT/dt) and critical heating rate $(dT/dt)_{T_b}$ in a thermal decomposition reaction are used to estimate the thermostability of energetic materials. However, the adiabatic time-to-explosion (t) can be calculated by the following Eqs. (9)–(12) [20–22] if a series of experimental data are obtained. Thereby, as an important parameter, it is easier to estimate the thermostability of energetic materials according to the length of the adiabatic time-to-explosion.

$$C_p\frac{dT}{dt} = QA\exp\left(\frac{-E}{RT}\right)f(\alpha) \tag{9}$$

$$f(\alpha) = (1-\alpha)^n \tag{10}$$

$$\alpha = \int\frac{C_p}{Q}dT \tag{11}$$

$$C_p = a + bT \tag{12}$$

where C_p is the specific heat capacity ($J\,mol^{-1}\,K^{-1}$), T is the absolute temperature (K), t is the adiabatic decomposition time (s), Q is the exothermic energy ($J\,mol^{-1}$), A is the pre-exponential factor (s^{-1}), E is the apparent activation energy of the thermal decomposition reaction ($J\,mol^{-1}$), R is the gas constant ($J\,mol^{-1}\,K^{-1}$), $f(\alpha)$ is the most probable kinetic model function and α is the conversion degree, a and b are coefficients.

The combination of Eqs. (9)–(12) can give the following equation:

$$t = \int_0^t dt = \int_{T_{e0}}^{T}\frac{C_p\exp(E/RT)}{QAf(\alpha)}dT = \frac{1}{QA}\int_{T_{e0}}^{T}\frac{(a+bT)\exp(E/RT)}{(1-\alpha)^n}dT$$
$$= \frac{1}{QA}\int_{T_{e0}}^{T}\frac{(a+bT)\exp(E/RT)}{\left[1-(1/Q)\int_{T_0}^{T}(a+bT)dT\right]^n}dT \tag{13}$$

The limit of the temperature integral in Eq. (13) is from T_{e0} to T_b.

(1) When $n = 0$

$$t_0 = \frac{1}{QA}\int_{T_{e0}}^{T_b}(a+bT)\exp\left(\frac{E}{RT}\right)dT \tag{14}$$

(2) When $n = 1$

$$t_1 = \frac{1}{QA\{1-(1/Q)[a(T_{e0}-T_b)+(b/2)(T_{e0}^2-T_b^2)]\}} \times \int_{T_{e0}}^{T_b}(a+bT)\exp\left(\frac{E}{RT}\right)dT \tag{15}$$

(3) When $n = 2$

$$t_2 = \frac{1}{QA\{1-(1/Q)[a(T_{e0}-T_b)+(b/2)(T_{e0}^2-T_b^2)]\}^2} \times \int_{T_{e0}}^{T_b}(a+bT)\exp\left(\frac{E}{RT}\right)dT \tag{16}$$

We can directly get $t_2 = 712.20$ s, $t_1 = 707.22$ s and $t_0 = 702.28$ s from Eqs. (14)–(16), so the adiabatic time-to-explosion of 3,4,5-triamino-1,2,4-triazole nitrate is a certain value between 702.22 s and 712.22 s.

Table 3
Explosive parameters and comparison of experimental and predicted 50% drop heights (H_{50}).

Acronym	$10^4\lambda^{(1)}$ ($J\,cm^{-1}\,s^{-1}\,K^{-1}$)	ρ ($g\,cm^{-3}$)	$\lg A_k$ (s^{-1})	Q_d ($J\,g^{-1}$)	$E^{(2)}$ ($J\,mol^{-1}$)	H_{50} (cm)		n	D_2	D_3
						Exp.(1)	Pre.			
HMX	34.43	1.79	33.80	2764	373700	32	33.4	0.564623	33.8765	−0.34717
RDX	10.58	1.66	12.50	2810	140000	26	20.1			
TNT	21.30	1.57	11.10	1506	155017	59	56.4			
BTF	20.92	22.81	22.81	2949	255000	28	28			
3,4,5-Triamino-1,2,4-triazole nitrate	32.40	1.80	11.80	1972	137160	–	27			

Note: (1) cited from Ref. [23]; (2) cited from Ref. [24]. T_{room} = 300 K.

3.5. Critical temperature of Hot-Spot initiation $T_{cr,hot\text{-}spot}$

In order to obtain the critical temperature of Hot-Spot initiation ($T_{cr,hot\text{-}spot}$) of 3,4,5-triamino-1,2,4-triazole nitrate, assuming that $T_{cr,hot\text{-}spot}$ is a function of the size and duration of Hot-Spot and of the physical and chemical properties of the explosive, the equation for calculating the value of $T_{cr,hot\text{-}spot}$ may be expressed as [19–21]

$$\left(\frac{4}{3}\pi a^3\right)\rho Q_d\{1-\exp[-(t-t_0)Ae^{-E/RT_{cr}}]\} = \int_a^\infty 4\pi r^2\rho C_p\left[\frac{a\theta_0}{r}erfc\left[\frac{r-a}{2\sqrt{Bt}}\right]\right]dr = \int_a^\infty 4\pi r^2\rho c_p\left\{\frac{a(T_{cr,hot\text{-}spot}-T_{room})}{r}erfc\left[\frac{r-a}{2\sqrt{\lambda t/\rho c_p}}\right]\right\}dr \quad (17)$$

where a is the radius of the Hot-Spot in cm; ρ is the density in $g\,cm^{-3}$; Q_d is the heat of reaction in $J\,g^{-1}$; $t-t_0$ is the time interval in s; A is the frequency factor in s^{-1}; E is the activation energy in $J\,mol^{-1}$; R is the gas constant in $J\,mol^{-1}\,K^{-1}$; $T_{cr,hot\text{-}spot}$ is the critical temperature of Hot-Spot initiation in K; C_p is the specific heat in $J\,g^{-1}\,K^{-1}$; T_{room} is the ambient temperature in K; λ is the thermal conductivity in $J\,cm^{-1}\,s^{-1}\,K^{-1}$.

By substituting the following data of 3,4,5-triamino-1,2,4-triazole nitrate: $a = 10^{-3}$ cm, $\rho = 1.80\,g\,cm^{-3}$ [23], $Q_d = 1971.8\,J\,g^{-1}$, $t-t_0 = 10^{-4}$ s, $A = A_k = 10^{10.03}\,s^{-1}$, $E = E_k = 137.16\,kJ\,mol^{-1}$, $R = 8.3145\,J\,mol^{-1}\,K^{-1}$, $C_p = 1.25\,J\,g^{-1}\,K^{-1}$, $T_{room} = 300$ K, $\lambda = 32.40\times10^{-4}\,J\,cm^{-1}\,s^{-1}\,K^{-1}$ into Eq. (17), the value of $T_{cr,hot\text{-}spot}$ of 600.325 °C is obtained.

3.6. Characteristic drop height of impact sensitivity H_{50}

To obtain the characteristic drop height of impact sensitivity (H_{50}) of 3,4,5-triamino-1,2,4-triazole nitrate, by substituting the values of λ, ρ, A, Q_d and E of 3,4,5-triamino-1,2,4-triazole nitrate in Table 3 and the values of n, D_2 and D_3 into Eq. (18) [19–21]. The corresponding value of H_{50} of 3,4,5-triamino-1,2,4-triazole nitrate is 27 cm, showing that it has impact sensitivity level approaching those of HMX, RDX and BTF.

$$\frac{1}{2}n\lg H_{50} + \lg\sqrt{\frac{\lambda}{A\rho Q_d}} + D_3 + \frac{0.02612E}{T_1 + D_2H_{50}^n} = 0 \quad (18)$$

where n, D_2 and D_3 are parameters of the correlation.

4. Conclusions

The thermal decomposition process of the exothermic decomposition reaction of 3,4,5-triamino-1,2,4-triazole nitrate at four different heating rates under atmospheric pressure could be described by Fig. 1 show in the text. The apparent activation energy and pre-exponential factor of the above-mentioned reaction are 137.16 kJ mol^{-1} and $10^{10.03}$ s^{-1}, respectively. The critical temperature of thermal explosion is 441.37 K. The entropy of activation ($\Delta S^{\neq}$), enthalpy of activation ($\Delta H^{\neq}$), and free energy of activation ($\Delta G^{\neq}$) of the reaction are −65.65 J mol^{-1} K^{-1}, 132.97 kJ mol^{-1} and 166.35 kJ mol^{-1}, respectively. The self-accelerating decomposition temperature (T_{SADT}) is 429.56 K. The critical temperature of Hot-Spot initiation ($T_{cr,hot\text{-}spot}$) is 600.325 °C and the characteristic drop height of impact sensitivity (H_{50}) is 27 cm. The molar heat capacity of 3,4,5-triamino-1,2,4-triazole nitrate is 221.81 J mol^{-1} K^{-1} at 298.15 K. The adiabatic time-to-explosion of 3,4,5-triamino-1,2,4-triazole nitrate was calculated to be a certain value between 702.22 s and 712.20 s.

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