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Thermal Behaviors of Bis(nitroguanidine)methane (BNGM)

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Abstract: To further evaluate the thermal stability of bis(nitroguanidine)methane (BNGM) and explore its potential application as energetic material, the thermal behavior, specific heat capacity, adiabatic time-to-explosion and impact sensitivity of BNGM were studied by differential scanning calorimetry(DSC), micro-DSC, thermogravimetry /differential thermogravimetry(TG/DTG) and impact experiment. Results show that the thermal behavior of BNGM can be divided into two exothermic decomposition processes. The peak temperatures of the two decomposition processes at a heating rate of 10 ℃·min^{-1} are 208.1 ℃ and 292.5 ℃, respectively. Its self-accelerating decomposition temperature (T_{SADT}) and critical temperature of thermal explosion (T_b) are 189.6 ℃ and 190.9 ℃, respectively. Its molar heat capacity at 298.15 K is 251.9 J·mol^{-1}·K^{-1}. Adiabatic time-to-explosion is estimated to be about 280 s. BNGM has a low impact sensitivity, which is higher than 23.5 J. The thermal stability of BNGM is good.

Key words: energetic materials; bis(nitroguanidine)methane (BNGM); thermal behavior; impact sensitivity

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1 Introduction

Compounds containing nitroxyl group (N—NO$_2$) are of interest for practical use as energetic materials owing to their high energy density and nitrogen content [1-4]. Nitroguanidine (NG) is a simple nitroxyl compound. The effect of electron withdrawing of nitro group makes the lower electron density in the single carbon atom, so NG can behave as an active starting material in the reactions with nucleophilic reagents to get a series of NG-based energetic materials [5-9]. Because of the excellent property and low cost, NG has become an important moiety to design and synthesize other new high-nitrogen energetic materials [6-8,10-12].

Bis(nitroguanidine)methane (BNGM) was reported firstly by Yu et al[13] through the reaction of NG and formaldehyde. BNGM was considered as an important intermediate to synthesize other heterocyclic compounds because of the existence of $>$C═NNO$_2$ and —NH$_2$ groups. It is remarkable to note that when BNGM was treated with nitric acid/acetic anhydride, 2,4-dinitramino-1,5-dinitro-1,3,5-triazine was obtained [13-16]. Namely, the deammoniation cyclization and the nitration of cyclized product occurred simultaneously. 2,4-Dinitramino-1,5-dinitro-1,3,5-triazine is a nitrogen-rich explosive with slight positive oxygen balance. Unfortunately, its stability decreased obviously in comparison to that of BNGM. Containing two explosive groups ($>$C═N—NO$_2$) and possessing good thermal stability will make BNGM serve as an appropriate candidate of energetic materials.

In this paper, we mainly report the thermal behavior, specific heat capacity and adiabatic time-to-explosion of BNGM, further estimating its thermal stability and exploring essential application values as an energetic material. The difference of thermal

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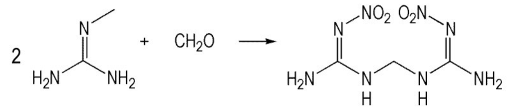
behavior between the parent material and BNGM is also discussed.

2 Experimental

2.1 Synthesis

The parent materials were purchased from the trade, and formaldehyde was AR grade product. The purity of nitroguanidine was 98% and the concentration of hydrochloric acid was 38%. BNGM was prepared according to Ref [15] and the synthetic route of BNGM was showed in Scheme 1. ^{1}H NMR (DMSO-d_6, 500 MHz): 8.157, 4.597, 3.345. ^{13}C NMR (DMSO-d_6, 500 MHz): 159.532, 73.329. IR (KBr, ν/cm^{-1}): 3319, 3180, 1586, 1542, 1367, 1252, 983, 719. Anal. calcd for $C_3H_8N_8O_4$ (%): C 16.36, H 3.64, N 50.91; Found: C 16.45, H 3.74, N 51.17.



Scheme 1 Synthetic route of BNGM

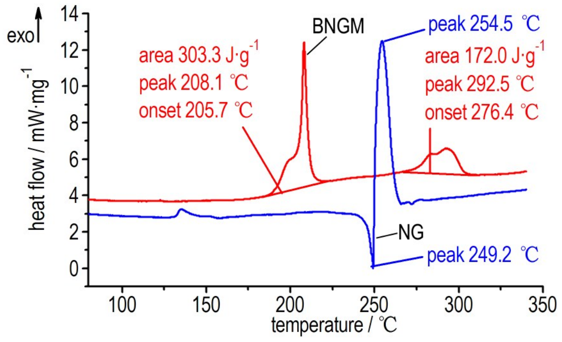
2.2 Experimental Measurements

The differential scanning calorimetry (DSC) experiments were performed using a DSC200 F3 (NETZSCH, Germany) apparatus under a nitrogen atmosphere with a flow rate of 80 mL · min^{-1}. The heating rates were 5.0, 7.5, 10.0, 12.5 ℃ · min^{-1} and 15.0 ℃ · min^{-1} from ambient temperature to 350 ℃, respectively. The thermogravimetry/differential thermogravimetry (TG/DTG) experiment was determined using a SDT-Q600 apparatus (TA, USA) under a nitrogen atmosphere at the flow rate of 100 mL·min^{-1}. The heating rate used was 10.0 ℃·min^{-1} from ambient temperature to 500 ℃. The specific heat capacity was determined using a Micro-DSC Ⅲ apparatus (SETARAM, France), and the sample mass was 196.9 mg. The heating rate used was 0.15 ℃ · min^{-1} from 10 ℃ to 80 ℃. Energy 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 the sample was tested with 6 times. The impact sensitivity was determined by using a ZBL-B impact sensitivity instrument (Nachcn, China). The mass of drop hammer was 2.0 kg, and the sample mass for test was 30 mg.

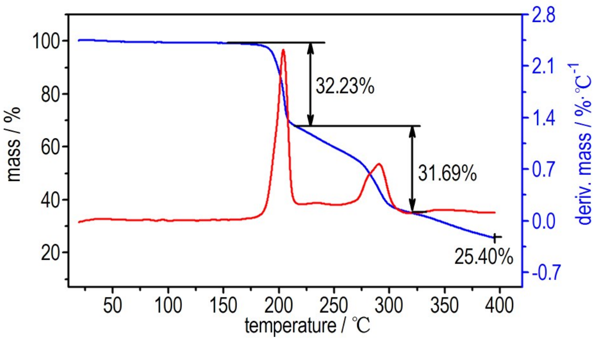
3 Results and Discussion

3.1 Thermal Decomposition Behavior

Typical DSC and TG-DTG curves (Fig. 1) indicate that the thermal behavior of BNGM can be divided into two exothermal decomposition processes. The onset temperature, peak temperature and decomposition enthalpy at a heating rate of 10 ℃ · min^{-1} are 205.7 ℃, 208.1 ℃ and 303.3 J · g^{-1} with a mass loss of about 32.2% for the first decomposition process, and 276.4 ℃, 292.5 ℃, 172.0 J · g^{-1} with a mass loss of about 31.7% for the second process, respectively. The final residue is about 25.4% at 400 ℃. The DSC curve of NG was also obtained, and there is an endothermic peak as a melting peak at 249.2 ℃. Subsequently, an intense exothermic process occurred at 254.5 ℃ for NG. It can be seen that the DSC



a. DSC



b. TG/DTG

Fig. 1 Typical DSC curves of BNGM and NG and TG/DTG curves of BNGM at a heating rate of 10 ℃·min^{-1}

curves of NG and BNGM are obviously different. Although BNGM possesses a good thermodynamic stability, it is lower than that of NG and the reason should be the new unstable —NH—CH_2—NH— bond of BNGM molecule.

In order to obtain the kinetic parameters (the apparent activation energy (E) and pre-exponential constant (A)) of the first exothermic decomposition process, a multiple heating method (Kissinger's method[17] and Ozawa's method[18]) was employed according to the data in Fig. 2. The determined values of the beginning temperature (T_0), extrapolated onset temperature (T_e) and peak temperature (T_p) at different heating rates are listed in Table 1. The values of T_{00} and T_{e0} corresponding to heating rate $\beta \rightarrow 0$ obtained by Eq.(1) are 164.1 ℃ and 189.6 ℃[19].

$$T_{(0\,\mathrm{or}\,e)i} = T_{(00\,\mathrm{or}\,e0)} + n\beta_i + m\beta_i^2 + p\beta_i^3,\ i = 1-5 \quad (1)$$

where n, m and p are coefficients.

The calculated values of kinetic parameters (E and A) are listed in Table 1. E obtained by Kissinger's method is consistent with that by Ozawa's method. The linear correlation coefficients(r) are all close to 1. So the results are credible.

Table 1 The values of T_0, T_e, T_p and kinetic parameters of the first decomposition process for BNGM

β/ ℃·min^{-1}	T_0 /℃	$T_{e/}$℃	T_p / ℃	-ΔH_d/J·g^{-1}	E_K / kJ·mol^{-1}	log(A /s^{-1})	r_K	E_O/kJ·mol^{-1}	r_O
5.0	165.3	200.1	202.6	225	230.40	23.30	0.9999	226.69	0.9999
7.5	167.2	203.4	205.9	251					
10.0	169.8	205.8	208.1	294					
12.5	171.5	207.6	209.9	250					
15.0	172.8	209.1	211.5	243					

Notes: E is the apparent activation energy. A is the pre-exponential constant. r is the linear correlation coefficient. Subscript K is data obtained by Kissinger's method. Subscript O is data obtained by Ozawa's method.

The self-accelerating decomposition temperature (T_{SADT}) and critical temperature of thermal explosion (T_b) are two important parameters required in evaluation of their safe storage and process operations for energetic materials and then to evaluate the thermal stability. T_{SADT} and T_b for BNGM are calculated as 189.6 ℃ and 190.9 ℃, respectively[19-20], indicating that the thermal stability of BNGM is good.

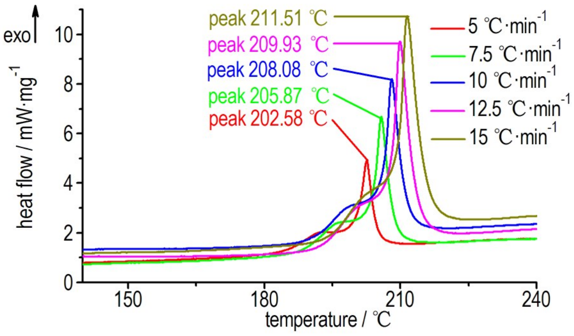


Fig. 2 DSC curves of BNGM at different heat rates

3.2 Specific Heat Capacity

Figure 3 shows the determination result of specific heat capacity (c_p) for BNGM, using a continuous specific heat capacity mode of apparatus. It can be seen that c_p presents a good linear relationship with temperature in determined temperature range. Specific heat capacity equation is shown as:

$$c_p=0.1763+3.2461\times10^{-3}T(285.0\,\mathrm{K}<T<350.0\,\mathrm{K}) \quad (2)$$

The specific heat capacity and molar heat capacity of BNGM are 1.15 J·g^{-1}·K^{-1} and 251.9 J·mol^{-1}·K^{-1} at 298.15 K, respectively.

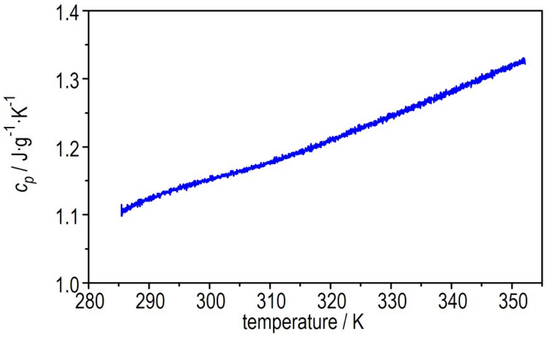


Fig. 3 Determination results of the continuous specific heat capacity for BNGM

3.3 Adiabatic Time-to-explosion

The adiabatic time-to-explosion is also an important parameter for evaluating the thermal stability of energetic materials and can be calculated by Eqs. (3) and (4) [19,21-25].

$$c_p \frac{\mathrm{d}T}{\mathrm{d}t} = QA\exp(-E/RT)f(\alpha) \tag{3}$$

$$\alpha = \int_{T_0}^{T} \frac{c_p}{Q}\mathrm{d}T \tag{4}$$

where c_p is the specific heat capacity, J·mol^{-1}·K^{-1}; T is the absolute temperature, K; Q is the exothermic values, 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.

After integrating of Eq. (3), the adiabatic time-to-explosion equation can be obtained as:

$$t = \int_0^t \mathrm{d}t = \int_{T_0}^{T} \frac{c_p\exp(E/RT)}{QAf(\alpha)}\mathrm{d}T \tag{5}$$

where the limit of temperature integration is from T_{00} to T_b.

In fact, the conversion degree (α) of energetic materials from the beginning thermal decomposition to thermal explosion in the adiabatic conditions is very small, and it is very difficult to obtain the most probable kinetic model function $f(\alpha)$ at the process. So, we separately used Power-low model, Reaction-order model, Avrami-Erofeev model and the above obtained kinetic model function to estimate the adiabatic time-to-explosion and supposed different rate orders (n) [19,26]. The calculation results are listed in Table 2.

From Table 2, we can see that the calculation results exhibit some deviations and the decomposition model has big influence on the estimate of adiabatic time-to-explosion. From the whole results, we believe the adiabatic time-to-explosion of BNGM should be about 280 s. It is a relatively long time, and the result indicates that BNGM has a good thermal stability.

Table 2 The calculation results of adiabatic time-to-explosion

model	rate order	equation	time/s
power-low model	1	$f(\alpha) = 1$	272.2
	2	$f(\alpha) = 2\alpha^{1/2}$	326.3
	3	$f(\alpha) = 3\alpha^{2/3}$	291.5
	4	$f(\alpha) = 4\alpha^{3/4}$	252.6
reaction-order model	1	$f(\alpha) = 1$	272.2
	2	$f(\alpha) = 1-\alpha$	329.5
	3	$f(\alpha) = (1-\alpha)^2$	398.9
avrami-erofeev model	1	$f(\alpha) = 1-\alpha$	329.5
	2	$f(\alpha) = 2(1-\alpha)[-\ln(1-\alpha)]^{1/2}$	376.8
	3	$f(\alpha) = 3(1-\alpha)[-\ln(1-\alpha)]^{2/3}$	331.1
	4	$f(\alpha) = 4(1-\alpha)[-\ln(1-\alpha)]^{3/4}$	285.0

Notes: In the calculation of adiabatic time-to-explosion, the following data, T_{00} =437.25 K, T_b =464.05 K, E_K =226.69 kJ·mol^{-1}, A_K =10$^{23.30}$ s^{-1}, c_p =1.15 J·g^{-1}·K^{-1}, Q = 252.5 J·g^{-1} and R = 8.314 J·mol^{-1}·K^{-1}, are used.

3.4 Enthalpy of Combustion

The determination results of the energy of constant volume combustion are shown in Table 3, so the constant-volume combustion enthalpy for BNGM is (−10171.4±27.5) J·g^{-1}.

Table 3 Determination results of enthalpy of combustion at 298.15 K

sample	No.	m / g	ΔT / K	$-\Delta_c U$ / J·g^{-1}
BNGM	1	0.20747	0.2064	10292
	2	0.20932	0.2058	10170
	3	0.21118	0.2072	10153
	4	0.19880	0.1950	10115
	5	0.20040	0.1972	10154
	6	0.21051	0.2064	10144
	mean			10171.4±27.5

The standard molar enthalpy of combustion ($\Delta_c H_m^\theta$) was referred to the energy of combustion change of the following idealized reaction formulas (a) and (b) at T = 298.15 K and p^θ = 101.325 kPa. Under the ideal condition of sufficient oxygen, we believe that the oxidation product of nitro group is NO_2 (g) and other N element would be oxidized to N_2 (g).

$$C_3H_8N_8O_4(s) + 5O_2(g) = 3CO_2(g) + 4H_2O(l) + 3N_2(g) + 2NO_2(g) \tag{a}$$

$$2NO_2(g) + \frac{2}{3}H_2O(l) = \frac{4}{3}HNO_3(l) + \frac{2}{3}NO(g) \tag{b}$$

$\Delta_c H_m^\theta$ can be derived from the standard molar enthalpy of combustion in accordance with equations (6) and (7).

$$\Delta_c H_m^\theta = \Delta_c U_m^\theta + \Delta n_1 RT + \Delta n_2 RT \tag{6}$$

$$\Delta n = \sum n_i(\text{products},g) - \sum n_i(\text{reactants},g) \tag{7}$$

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

The standard molar enthalpy of formation ($\Delta_f H_m^\theta$) can be calculated by Hess's law [27], according to the above thermochemical equations. For BNGM, the standard molar enthalpy of formation is:

$$\begin{aligned}\Delta_f H_m^\theta(\text{BNGM},s) = {} & 3\Delta_f H_m^\theta(\text{CO}_2,g) + \frac{10}{3}\Delta_f H_m^\theta(\text{H}_2\text{O},l) \\ & + \frac{2}{3}\Delta_f H_m^\theta(\text{NO},g) + \frac{4}{3}\Delta_f H_m^\theta(\text{HNO}_3,l) \\ & - \Delta_c H_m^\theta(\text{BNGM},s)\end{aligned} \tag{8}$$

The standard molar enthalpies of formation for CO_2 (g), H_2O (l), HNO_3 (l) and NO (g) are as follows: $\Delta_f H_m^\theta(CO_2, g) = -(393.51 \pm 0.13)$ kJ·mol^{-1}, $\Delta_f H_m^\theta(H_2O, l) = -(285.83 \pm 0.04)$ kJ·mol^{-1}, $\Delta_f H_m^\theta(HNO_3, l) = -(174.10 \pm 0.8)$ kJ·mol^{-1}, $\Delta_f H_m^\theta(NO, g) = (90.25 \pm 0.75)$ kJ·mol^{-1}[28].

The standard molar enthalpy of combustion ($\Delta_c H_m^\theta$) and the standard molar enthalpy of formation ($\Delta_f H_m^\theta$) were calculated as (-2238.53 ± 6.04) kJ·mol^{-1} and (-66.73 ± 6.05) kJ·mol^{-1} (T = 298.15 K and p^θ = 101.325 kPa).

3.5 Sensitivity

The test result indicates that impact sensitivity of BNGM is higher than 23.5 J. So, BNGM is relatively less sensitive. The sensitivity is close to that of parent material NG (>30 J), but much lower than that of RDX (7.4 J) [29].

4 Conclusions

(1) The thermal decomposition behavior of BNGM presents two exothermic decomposition processes. The self-accelerating decomposition temperature (T_{SADT}) and critical temperature of thermal explosion (T_b) are 189.6 ℃ and 190.9 ℃, respectively.

(2) Specific heat capacity equation of BNGM is $c_p = 0.1763 + 3.2461 \times 10^{-3} T$ (285.0 K< T <350.0 K), and the molar heat capacity is 251.9 J·mol^{-1}·K^{-1} at 298.15 K. Adiabatic time-to-explosion of BNGM is estimated to be about 280 s. The standard molar enthalpy of formation of BNGM is (-66.73 ± 6.05) kJ·mol^{-1}, and the impact sensitivity of BNGM is higher than 23.5 J. The thermal stability of BNGM is good.

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亚甲基二硝基胍(BNGM)的热行为

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摘　要: 为进一步评估亚甲基二硝基胍(BNGM)的热稳定性,采用差示扫描量热法(DSC),微量热仪,热重-微分热重分析(TG/DTG)和撞击实验,研究了BNGM的热分解行为、比热容、绝热至爆时间,并测试了其撞击感度。结果表明:BNGM的热行为分为两个放热分解过程,10 ℃·min^{-1}下两个分解过程的峰温分别为208.1 ℃和292.5 ℃,其自加速分解温度和热爆炸临界温度分别为189.6 ℃和190.9 ℃,298.15 K时摩尔热容为251.9 J·mol^{-1}·K^{-1},估算绝热至爆时间约为280 s,撞击感度大于23.5 J,表明BNGM热稳定性良好。

关键词: 含能材料;亚甲基二硝基胍(BNGM);热行为;撞击感度

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