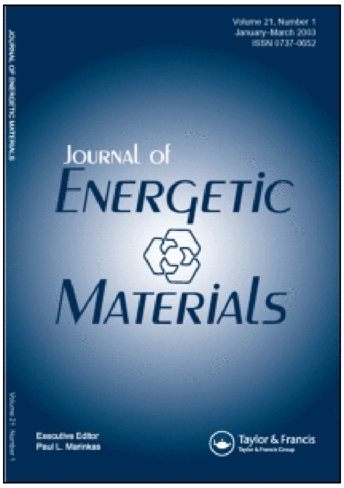

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Thermal Safety Study on TEGDN/NG/NC Gun Propellant

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The self-accelerating decomposition temperature (T_{SADT}), critical temperature of thermal explosion (T_b), adiabatic time to explosion (t_{TIad}), 50% drop height of impact sensitivity (H_{50}), critical temperature of hot spot initiation caused by impact ($T_{cr,hotspot}$), safety degree (S_d), critical thermal explosion ambient temperature (T_{acr}), and thermal explosion probability (P_{TE}) of the gun propellant composed of triethyleneglycol dinitrate (TEGDN), nitroglycerin (NG), and nitrocellulose (NC) were studied. The results of thermal safety evaluation on the gun propellant were obtained: (1) $T_{00} = 433.4\,K$, $T_{SADT} = T_{e0} = 441.1\,K$, $T_b = T_{bp0} = 468.4\,K$; (2) when $E_k = 205.3 \times 10^3\,J\,mol^{-1}$ and $A_k = 10^{20.62}\,s^{-1}$, $t_{TIad} = 48.3\,s$, $H_{50} = 17.93\,cm$, $T_{cr,\ hotspot} = 634.9\,K$; for a sphere sample, $T_{S(T)max} = 369.0K$, $T_{acr} = 364.2\,K$, $S_d = 65.03\%$, $P_{TE} = 34.96\%$.

Keywords: gun propellant, nitroglycerin (NG), thermal decomposition, thermal safety, triethyleneglycol dinitrate (TEGDN)

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Introduction

Triethyleneglycol dinitrate (TEGDN) is extensively used as an important plasticizer in gun propellants, and it can improve some performance characteristics of gun propellant, such as low-temperature mechanical properties. In this article, a thermal safety study on gun propellant composed of triethyleneglycol dinitrate (TEGDN), nitroglycerin (NG), and nitrocellulose (NC) was carried out. This is quite useful to evaluate the heat-resistance ability for gun propellants under nonisothermal conditions and to explore the phenomenon, mechanism, and process from thermal decomposition to explosion.

Sample

The formulation of the TEGDN/NG/NC gun propellant containing the mixed nitric ester was designed as follows: the mass ratio of the mixed ester of TEGDN to NG was 9.5:28 [1]; the mass fraction of NC and some additives were 60 and 2.5%, respectively. The gun propellant sample used in the experiment was prepared by a solventless propellant extrusion technique.

Equipment and Conditions

Thermogravimetric–derivative thermogravimetric (TG-DTG) and differential scanning calorimetry (DSC) curves under the condition of flowing nitrogen gas (purity, 99.999%; atmospheric pressure) were obtained by using a TA2950 thermal analyzer (TA Co., New Castle, DE) and a 204HP differential scanning calorimeter (Netzsch Co., Selb, Bavaria, Germany). The conditions of TG-DTG were: sample mass, approximately 1 mg; N_2 flow rate, $40\,cm^3\,min^{-1}$; heating rate (β), $10\,K\,min^{-1}$. The conditions of DSC analyses were: N_2 flow rate, $50\,cm^3\,min^{-1}$; heating rate, 5, 10, 15, 20, 25, and $30\,K\,min^{-1}$; sample mass, approximately 1 mg; furnace pressure, 0.1 MPa; reference sample, α-Al_2O_3; type of crucible, aluminum pan with a pierced lid.

The specific heat capacity (C_p, J g^{-1} K^{-1}) was determined using continuous C_p mode on a Micro-DSC III microcalorimeter

(Setaram Co., Caluire, France). Heating rate, $0.15\,K\,min^{-1}$; sample mass, approximately 100 mg; atmosphere, nitrogen; reference sample, calcined α-Al_2O_3.

Thermal Behavior Analysis

Typical TG-DTG and DSC curves of the gun propellants are shown in Figs. 1 and 2. The TG curve shows two mass loss stages (stages I and II), corresponding to the two peaks in the DTG curve. Stage I stops at about 450 K, accompanied by about 36.1% mass loss, which is close to the total mass (37.5%) of the mixed ester of TEGDN and NG, and is attributable to the volatilization and decomposition of the mixed ester. Stage II stops at 531 K, accompanied by about 43% mass loss, which is likely caused by the thermal decomposition of NC and centralite II (C_2). The two mass loss processes occur in succession, and the temperature ranges of the two processes are neighbouring. Because the two mass loss temperatures in the TG-DTG are so near to each other, they appear as a single exotherm in the DSC.

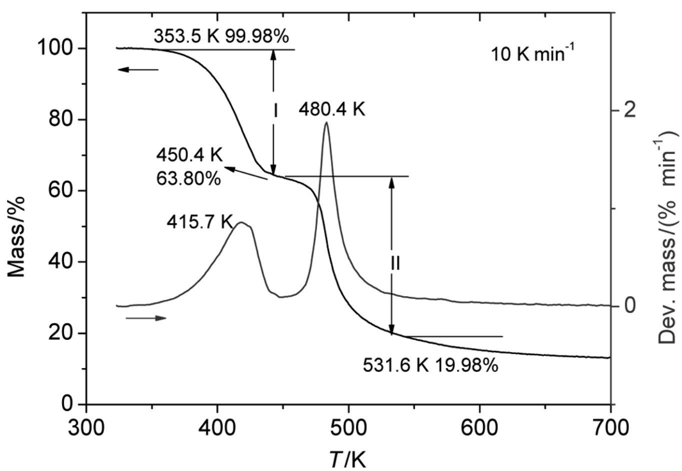

Figure 1. TG-DTG curve for the gun propellant at the heating rate of $10\,K\,min^{-1}$.

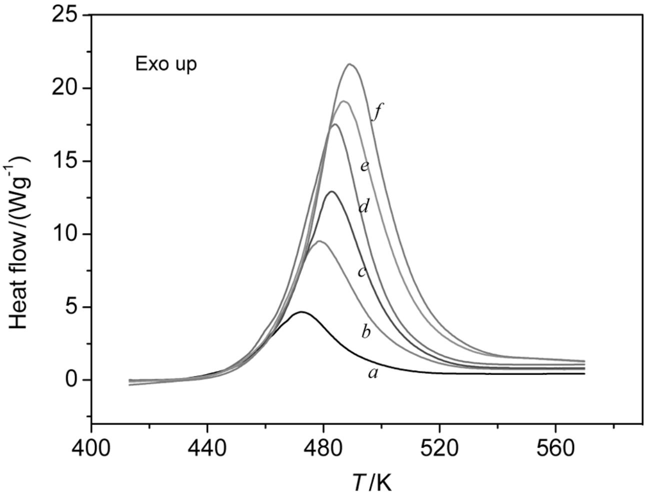

Figure 2. DSC curves for the gun propellant. Heating rate ($K\,min^{-1}$): (a) 5; (b) 10; (c) 15; (d) 20; (e) 25; (f) 30.

Kinetic Parameters Based on Onset Temperature (T_e) and Peak Temperature (T_p) at Six Heating Rates

In order to obtain the kinetic parameters (the apparent activation energy E_a and pre-exponential constant A) of the exothermic decomposition reaction of the gun propellant, the values of E_k and A_k are obtained by Kissinger's multiple heating method [2] (Eq. (1)), and the values of E_{Oe} and E_{Op} are obtained by Ozawa's method [3] (Eq. (2)) as shown in Table 1.

$$\ln\left(\frac{\beta_i}{T_{pi}^2}\right) = \ln\frac{A_k R}{E_k} - \frac{E_k}{R}\frac{1}{T_{pi}} \tag{1}$$

$$\lg\beta_i = \lg\left[\frac{AE_{Oe(or\ Op)}}{RG(\alpha)}\right] - 2.315 - \frac{0.4567E_{Oe(or\ Op)}}{RT_{e(or\ p)i}} \tag{2}$$

Thermodynamic Parameters of Activation Reaction

The entropy of activation ($\Delta S^{\neq}$), enthalpy of activation ($\Delta H^{\neq}$), and Gibbs free energy of activation ($\Delta G^{\neq}$) corresponding to

Table 1
Original data and kinetic parameters for the gun propellant

	Initial value			Calculated value							
β (K min^{-1})	T_0 (K)	T_e (K)	T_p (K)	T_{00} (K)	T_{e0} (K)	T_{p0} (K)	E_K (10^3 J mol^{-1})	lg (A_K/s^{-1})	r_K	E_{Op} (10^3 J mol^{-1})	r_{Op}
5	421.0	451.8	473.3	433.4	441.1	459.4	205.3	20.62	0.9959	202.8	0.9962
10	421.2	455.1	479.3								
15	428.1	459.6	483.5								
20	428.8	463.4	484.7								
25	431.9	466.6	488.2								
30	433.4	468.8	489.3								

$T = T_{p0} = 459.4\,K$, $E = E_k = 205.3 \times 10^3 J\,mol^{-1}$, and $A = A_k = 10^{20.62} s^{-1}$ obtained by Eqs. (3)–(5) [4–6] are 137.9 $J\,mol^{-1}K^{-1}$, 201.5 $kJ\,mol^{-1}$, and 138.1 $kJ\,mol^{-1}$, respectively. The positive value of $\Delta G^{\neq}$ indicates that the exothermic decomposition reaction for the gun propellant must proceed under the heating condition.

$$Ae^{-E/RT} = \frac{k_B T}{h} e^{\frac{-\Delta G^{\neq}}{RT}} \tag{3}$$

$$\Delta H^{\neq} = E - RT \tag{4}$$

$$\Delta G^{\neq} = \Delta H^{\neq} - T\Delta S^{\neq} \tag{5}$$

where k_B is the Boltzmann constant ($1.381 \times 10^{-23} J\,K^{-1}$) and h is Planck's constant ($6.626 \times 10^{-34} J\,s$).

Self-accelerating Decomposition Temperature (T_{SADT})

Setting T_0 as the initial decomposition temperature at which the DSC curve deviates from the baseline, T_e as the onset temperature, and T_p as the peak temperature and defining $T_{00(\mathrm{or\,e0\,or\,p0})}$ as the value of $T_{0(\mathrm{or\,e\,or\,p})i}$ corresponding to $\beta \to 0$ and T_{e0} as the self-accelerating decomposition temperature (T_{SADT}), we have

$$T_{0\mathrm{or}\;e\;\mathrm{orp}} = T_{00\,\mathrm{or\,e0\,or\,p0}} + b\beta_i + c\beta_i^2 + d\beta_i^3 + e\beta_i^4, \quad i = 1, 2, \ldots, 6 \tag{6}$$

and

$$T_{e0} = T_{SADT} \tag{7}$$

The values of T_{00}, T_{e0}, and T_{p0} obtained by using linear regression of T_{0i}, T_{ei}, and T_{pi} against β_i as described in Eq. (6) [4–6] are all listed in Table 1. The value of T_{SADT} of 441.1 K is obtained by the value of T_{e0} in Table 1 and Eq. (7).

Critical Temperature of Thermal Explosion (T_b)

The critical temperature of thermal explosion (T_{be0} and T_{bp0}) is an important parameter for evaluating the safety and elucidating the transition tendency from thermal decomposition to thermal explosion for small-scale energetic materials (EMs).

For the gun propellant, the values of T_b obtained by the Zhang-Hu-Xie-Li equation (Eq. (8)) [4–8] using the values of T_{e0} and T_{p0} and the value of E_{Op} listed in Table 1 are 449.3 and 468.4 K, respectively.

$$T_{be0(\text{or bp0})} = \frac{E_{Op} - \sqrt{E_{Op}^2 - 4E_{Op}RT_{e0(\text{or p0})}}}{2R} \tag{8}$$

The high value of T_b for the gun propellant shows that the transition from thermal decomposition to thermal explosion does not easily take place.

Adiabatic Time to Explosion (t_{TIad})

The adiabatic time to explosion (t_{TIad}) of EMs is the time of EM decomposition transiting to explosion under the adiabatic conditions and is an important parameter for assessing the thermal stability and safety of EMs. In order to acquire the value of t_{TIad} of the gun propellant, substituting the following data—C_p $(\mathrm{J\,g^{-1}\,K^{-1}}) = -1.15 + 1.29 \times 10^{-2}\quad T - 1.43 \times 10^{-5}\quad T^2$, $Q_d = 1.50 \times 10^3\ \mathrm{Jg^{-1}}$, $A = 10^{20.62}\ \mathrm{s^{-1}}$, $E = E_k = 205.3 \times 10^3\ \mathrm{J\,mol^{-1}}$, $R = 8.314\ \mathrm{J\,mol^{-1}\,K^{-1}}$, $f(\alpha) = (1-\alpha)^2$ [1], $T_1 = T_{e0} = 441.1\ \mathrm{K}$, $T_2 = T_{b\,p0} = 468.4\ \mathrm{K}$—into Eqs. (9)–(11) [9–11] results in the value of t_{TIad} of 48.3 s being obtained.

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

$$t_{TIad} = \int_0^t dt = \frac{1}{Q_d A} \int_{T_1}^{T_2} \frac{C_p \exp(E/RT)}{f(\alpha)} dT \tag{10}$$

$$\alpha = \int_{T_1}^{T_2} \frac{C_p}{Q_d} dT \tag{11}$$

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 the gun propellant, assuming that $T_{cr,hot\text{-}spot}$ is a function of the size and duration of the hot spot and of the physical and chemical properties of the explosive, the equation for calculating the value of $T_{cr,hot\text{-}spot}$ can be expressed as Eq. (12) [12,13].

$$\left(\frac{4}{3}\pi a^3\right)\rho Q_d\{1-\exp[-(t-t_0)A\exp(-E/RT_{cr,hot\text{-}spot})]\}$$
$$=\int_a^\infty 4\pi r^2\rho C_p\left[\frac{a\theta_0}{r}\mathrm{erfc}\left[\frac{r-a}{2\sqrt{Bt}}\right]\right]\mathrm{d}r$$
$$=\int_a^\infty 4\pi r^2\rho C_p\left[\frac{a(T_{cr,hot\text{-}spot}-T_{room})}{r}\right.$$
$$\left.\times\,\mathrm{erfc}\left[\frac{r-a}{2\sqrt{\frac{\lambda}{\rho C_p}t}}\right]\right]\mathrm{d}r \tag{12}$$

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

By substituting the following data of the gun propellant—$a=10^{-3}$ cm, $\rho=1.612\,g\,cm^{-3}$, $Q_d=1.50\times10^3\,J\,g^{-1}$, $t-t_0=10^{-4}$ s, $A=A_k=10^{20.62}\,s^{-1}$, $E=E_k=205.3\times10^3\,J\,mol^{-1}$, $R=8.314\,J\,mol^{-1}\,K^{-1}$, $C_p=1.425\,J\,g^{-1}\,K^{-1}$, $T_{room}=293.2$ K, $\lambda=20.50\times10^{-4}\,J\,cm^{-1}\,s^{-1}\,K^{-1}$—into Eq. (12), the value of $T_{cr,hot\text{-}spot}$ of 634.9 K is obtained.

Characteristic Drop Height of Impact Sensitivity (H_{50})

To obtain the characteristic drop height of impact sensitivity (H_{50}) of the gun propellant, by substituting the values of λ, ρ,

A, Q_d, and E of eight explosives with known 50% drop height listed in Table 2 into Eq. (13) [14–17], the corresponding values of n of 0.564623, D_2 of 33.8765, and D_3 of -0.347174 are obtained. By substituting the values of λ, ρ, A, Q_d, and E of the gun propellant listed in Table 2 and the values of n, D_2, and D_3 into Eq. (13), the corresponding the value of H_{50} of the gun propellant is obtained as 17.93 cm.

$$\frac{1}{2} n \lg H_{50} + \lg \sqrt{\frac{\lambda}{A\rho Q_d}} + D_3 + \frac{0.02612E}{T_1 + D_2 H_{50}^n} = 0 \qquad (13)$$

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

Critical Thermal Explosion Ambient Temperature (t_{acr}), Thermal Sensitivity Probability Density Function ($S(T)$), Safety Degree (S_d), and Thermal Explosion Probability (P_{TE})

In order to explore the heat-resistance ability of the gun propellant, the values of T_{acr}, $S(T)$ vs. T relation, S_d, and P_{TE} are calculated by the Frank-Kamenetskii formula (14) [18] and Wang-Du's formulas (15)–(20) [19–21] in Fig. 3. In formulas (14)–(20), T_{acr} is the critical thermal explosion ambient temperature (K); E is the activation energy ($\mathrm{J\,mol^{-1}}$); A is the pre-exponential constant ($\mathrm{s^{-1}}$); R is the gas constant ($8.314\,\mathrm{J\,K^{-1}\,mol^{-1}}$); λ is the thermal conductivity ($\mathrm{W\,m^{-1}\,K^{-1}}$); δ is the Frank-Kamenetskii (FK) parameter; δ_{cr} is the criticality of thermal explosion of exothermic system; r is characteristic measurement of reactant (m); Q_d is decomposition heat ($\mathrm{J\ kg^{-1}}$); ρ is density ($\mathrm{kg\,m^{-3}}$); μ_T is the average value of temperature (K); σ_δ is the standard deviation of the FK parameter; σ_T is the standard deviation of ambient temperature; T is surrounding temperature; $S(T)$ is the thermal sensitivity probability density function; S_d is safety degree; and P_{TE} is thermal explosion probability.

Substituting E with $205.3 \times 10^3\,\mathrm{J\,mol^{-1}}$, A with $10^{20.62}\,\mathrm{s^{-1}}$, ρ with $1.61 \times 10^3\,\mathrm{kg\,m^{-3}}$, λ with $0.205\,\mathrm{W\,m^{-1}\,K^{-1}}$, Q_d

Table 2
Explosive parameters and comparison of experimental and predicted 50% drop height (H_{50})

							H_{50} (cm)				
No.	Sample	λ^a (10^{-4}J cm^{-1}s^{-1} K^{-1})	ρ (gcm^{-3})	lg $(A/s^{-1})^b$	$Q_d{}^c$ (10^3 J g^{-1})	E^b (10^3J mol^{-1})	Exp.[a]	Predicted	n	D_2	D_3
1	HMX	34.43	1.79	33.80	2.76	373.7	32	33.4	0.564623	33.8765	−0.347174
2	RDX	10.58	1.66	12.50	2.81	140.0	26	20.1			
3	TNT	21.30	1.57	11.10	1.51	155.0	59	56.4			
4	PETN	25.10	1.68	10.40	3.26	112.3	16	15.60			
5	BTF	20.92	1.81	22.81	2.95	255.0	28	30.0			
6	HNS	8.53	1.65	22.63	1.39	289.0	54	50.1			
7	Tetryl	18.74	1.67	16.90	1.90	172.5	17[(d)]	17.6			
8	NG	12.55	1.60	16.09	2.09	150.1	7[(d)]	9.4			
9	the gun propellant	20.50	1.61	20.62	1.50	205.3	—	17.93			

[a]Cited from Dong and Zhou [14].
[b]Cited from Dong et al. [15].
[c]Cited from Hu et al. [16].
[d]Cited from Friedman [17].
$T_{room} = 350\,K$.

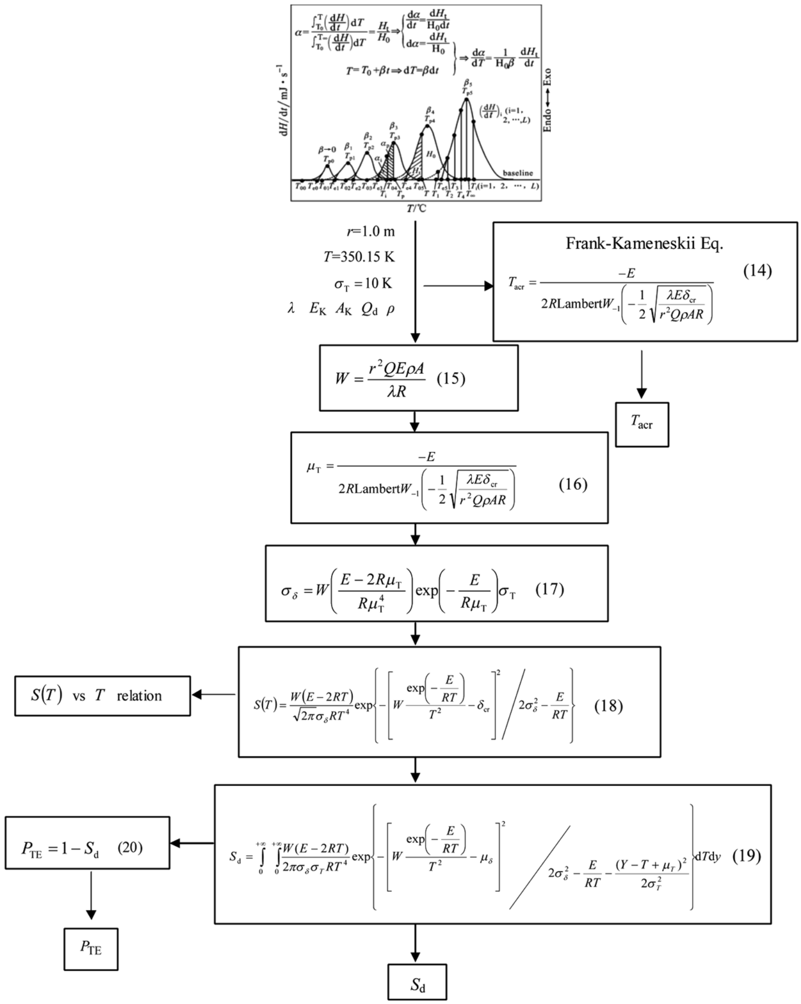

Figure 3. Block diagram of the process of computing the values of T_{acr}, $S(T)$ vs. T relation, S_d, and P_{TE}.

with $1.50 \times 10^6 \mathrm{J\,kg^{-1}}$, r with 1.0 m, T with 350 K, and σ_T with 10 K in Eqs. (14)–(20) in Fig. 3, the maximum value of $S(T)$ vs. T relation curve ($T_{S(T)\max}$), S_d, T_{acr}, and P_{TE} of the gun propellant are obtained as 369.0 K, 65.03%, 364.2 K, and

34.96%, showing the thermal safety and the accelerating tendency from adiabatic decomposition to explosion of the gun propellant.

Conclusion

The thermal safety evaluation on the gun propellant was obtained and the results show that the gun propellant has a high safety degree and low thermal explosion probability, such as high self-accelerating decomposition temperature and critical temperature of thermal explosion, long adiabatic time to explosion, and low impact sensitivity.

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