

Article

Safety Risk Assessment of HMX Synthesis Using Acetic Anhydride Method

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Abstract

To comprehensively evaluate the thermal risk parameters of the HMX synthesis process via the acetic anhydride method, we systematically investigated the safety of raw materials, ingredient mixing, nitration, and crystal transformation processes using DSC, ARC, and reaction calorimetry, which enabled the optimization of feeding strategies based on the exothermic characteristics observed during both ingredient mixing and nitration. Results indicate that the decomposition temperatures of raw materials and products are all above 200 °C, showing excellent thermal stability. Thus, multi-batch feeding is preferred for reaction material preparation. For the nitration process, continuous and stable feeding must be guaranteed during the feeding stage. During nitration, the temperature relationship satisfies $T_p < \text{MTSR} < \text{MTT} < T_{D24}$, wherein the risk of secondary decomposition and overflow is low. Additionally, both the nitration filtrate and crystal transformation filtrate exhibit low thermal hazards. These collective findings indicate that the acetic anhydride-based HMX synthesis process maintains relatively safe operational characteristics under standard processing conditions.

Keywords: octogen; acetic anhydride method; thermal safety; reaction safety risk assessment

1. Introduction

Energetic materials (EMs) serve as the energy source for weapon systems, with a high energy density and rapid energy release rate being the perpetual goals of their development [1,2]. However, high energy and high safety are often mutually exclusive [3]. While pursuing high energy, it is imperative to strengthen the management and control of safety risks during the synthesis process of energetic materials. By enhancing the safety of the technological process, theoretical guidance can be provided for the production, use, and storage of EMs, thereby avoiding substantial costs arising from missing safety data or unclear safety mechanisms, materials, and experiments [4–6]. Nitrification, a key reaction in energetic material synthesis, is one of the core supervised processes in the chemical industry due to its high heat release and significant thermal risk [7,8]. Thus, obtaining full-process safety assessment data for nitrification is crucial for guiding safe production and promoting the process of intrinsic safety.



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Octogen (1,3,5,7-tetranitro-1,3,5,7-tetrazocane, $C_4H_8N_8O_8$, HMX) is currently the highest-performance explosive widely used in military applications due to its advantages of high energy output, detonation velocity, and stability [9–11]. It is widely employed in warheads for artillery shells and missiles, as well as in high-power explosive charges for naval mines and landmines [12]. Additionally, HMX serves as a key component in aerospace propellants. Bachmann and Sheehan [13] first prepared HMX via the acetic anhydride method, which later became the primary industrial synthetic process (Figure 1). Leach and Staples [14] were the first to conduct numerous destructive experiments on HMX synthesis via the acetic anhydride method, exploring the boundary explosion temperature for its feeding process. Tang et al. [15] obtained the thermal decomposition characteristic parameters of HMX using TG-DSC analysis and conducted an in-depth investigation into the thermal stability of this high-energy explosive via the Malek method. Zhang et al. [16] conducted a study on the thermal sensitivity characteristics of HMX. Peng [17] studied the thermal hazards associated with the acetic anhydride-based HMX synthesis process, with a particular focus on the safety aspects of hexamine nitration. Based on the evaluation results, the nitration process was optimized to enhance the safety of HMX production. In summary, existing studies lack systematic research on the safety of additional raw materials, nitration filtrate, and crystal transfer filtrate involved in acetic anhydride-based HMX synthesis, as well as the processes of feed solution preparation and crude product crystal transfer. The incompleteness of safety data highlights the necessity of obtaining safety assessment data for the whole process of nitrification.

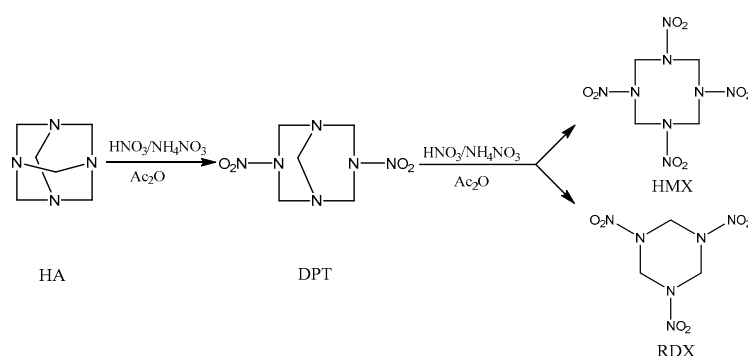


Figure 1. Reaction process of HMX synthesis via acetic anhydride method.

This study conducts a comprehensive safety risk assessment of the entire production process of HMX. Differential Scanning Calorimetry (DSC) was used to evaluate the thermal stability of key materials involved in the acetic anhydride-based HMX synthesis, including the raw materials hexamine (HA), ammonium nitrate (AN), acetic acid (HAc), and acetic anhydride (Ac_2O), as well as the crude product and nitration liquor. The reaction calorimeters (EasyMax and RC1) were utilized to investigate the exothermic behavior during three critical stages of the process: (1) preparation of ammonium nitrate-nitric acid (AN-NA) solution, (2) preparation of hexamine-acetic acid (HA-HAc) solution, and (3) implementation of the nitration reaction process. Adiabatic Accelerating Rate Calorimetry (ARC) was employed to analyze the thermal stability of six process-related solutions, namely AN-NA solution, HA-HAc solution, nitration solution, nitration filtrate, nitric acid transfer solution, and transcrystallization filtrate. Based on the integrated thermal parameters obtained from these assays, a thermal risk assessment was performed for the acetic anhydride-mediated HMX synthesis process, with corresponding precautionary measures proposed [18–20]. This study aims to provide fundamental safety data and control strategies to ensure the safe production of HMX.

2. Experimental Materials and Conditions

2.1. Materials

The information about the materials used in the experiment is shown in Table 1.

Table 1. Detailed information about the materials used in the experiment.

Chemical Name	Source	CAS No.	Boiling Point	Mass Fraction Purity ^a
HA	Sinopharm Group Chemical Reagent Co. Ltd. (Shanghai, China)	100-97-0	-	≥98.0%
AN	Sinopharm Group Chemical Reagent Co., Ltd. (Shanghai, China)	229-347-8	-	≥98.0%
HNO ₃	Sinopharm Group Chemical Reagent Co., Ltd. (Shanghai, China)	7697-37-2	86–88	≥98.0%
HAc	Sinopharm Group Chemical Reagent Co., Ltd. (Shanghai, China)	64-19-7	117.9	≥95.0%
Ac ₂ O	Sinopharm Group Chemical Reagent Co., Ltd. (Shanghai, China)	108-24-7	139.5	≥98.0%

^a No specific purification steps were applied, and the purity information was provided directly by the supplier.

2.2. Experimental Equipment

The experimental equipment include the following: DSC-25 differential scanning calorimeter, TA Company, New Castle, DE, USA; EasyMax 102 HFCal and RC1mx HFCal automatic reaction calorimeters, Mettler-Toledo, Greifensee, Switzerland; Phi-TEC I adiabatic accelerated calorimeter, HEL Company, London, UK. The calibration details of the equipment are provided in the Supplementary File.

2.3. Experimental Conditions

The thermal decomposition properties of the raw materials, products, and nitrification liquor were investigated via DSC. The DSC test used a stainless-steel, high-pressure crucible with a maximum tolerance of 15 MPa. The DSC test conditions were as follows: sample mass: 1.0 ± 0.2 mg; temperature range: 50–400 °C; nitrogen flow: 50 mL·min^{−1}; heating rate: 10 °C min^{−1}.

EasyMax and RC1 were used to investigate the heat release during the preparation of AN-NA solution, the preparation of HA-HAc solution, and the nitration process.

The self-decomposition exothermic behaviors of the AN-NA solution, HA-HAc solution, nitrification liquor, filtrate, crystal transformation solution, and its filtrate were investigated via ARC. The nitrification liquor refers to the solution after the second-stage feeding, and the filtrate is the liquid obtained after the first filtration. The specific test conditions were as follows: sample mass: 1.0 ± 0.1 g; mass of Hastelloy sample cell: 14.4127 g; Heat-Wait-Search (H-W-S) mode with a temperature step of 5 °C; temperature range: 50–400 °C; wait time: 15 min; temperature rate sensitivity: 0.02 °C·min^{−1}.

3. Experimental Results and Analysis

3.1. DSC Thermal Decomposition Experimental Results of Raw Materials, Products, and Nitrification Liquor

As demonstrated by the DSC experimental results in Figure 2a,b and Table 2, HAc exhibits no distinct decomposition point, indicating excellent thermal stability. The de-

composition peak temperatures of the other three raw materials (HA, AN, and Ac_2O) are 296.39 °C, 341.87 °C, and 226.68 °C, respectively. Two decomposition peaks are observed for the product HMX, with corresponding peak temperatures of 251.2 °C and 282.1 °C. All four substances have decomposition temperatures exceeding 200 °C, thus exhibiting a low risk of thermal hazards during routine storage and handling. Their exothermic enthalpies are determined to be 549.65, 1339.3, 226.68, and 2159.1 $\text{J}\cdot\text{g}^{-1}$, respectively. Notably, HMX and AN possess considerably higher decomposition enthalpies than Ac_2O and HAc, posing a higher risk of severe consequences in the event of thermal runaway. Based on the combined analysis of decomposition temperature and enthalpy data, it is imperative to strictly control the temperature of the ambient environment and reaction system during the storage and handling of raw materials and HMX, to prevent thermal decomposition of the materials.

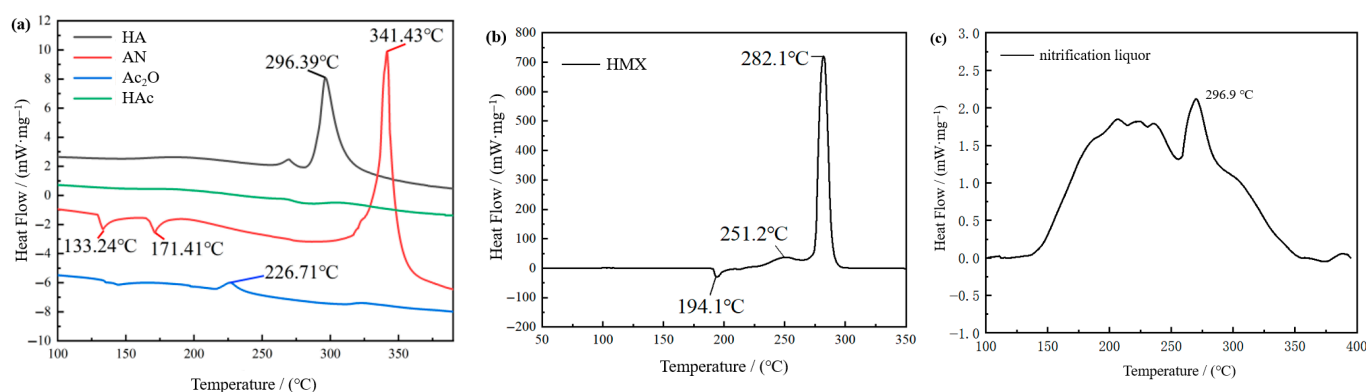


Figure 2. DSC curves of HMX raw materials and product synthesized using acetic anhydride method ((a): raw materials, (b): product, (c): nitrification liquor).

Table 2. DSC test results of raw materials, product, and nitrification liquor of HMX synthesized with acetic anhydride method.

Sample	Peak Exothermic Temperature (°C)	Enthalpy Value ($\text{J}\cdot\text{g}^{-1}$)
HMX	251.2; 282.1	2159.1
HA	296.4	549.6
AN	341.4	1339.3
Ac_2O	226.7	36.9
HAc	-	-
Nitrification liquor	296.9	1349.0

Figure 2c shows the DSC curve of the nitrification liquor. Two overlapping exothermic peaks were observed, with an exothermic temperature range of 132.79 °C to 338.84 °C. The maximum peak appears at 296.9 °C, with a corresponding exothermic enthalpy of 1349.0 $\text{J}\cdot\text{g}^{-1}$. Compared with the raw materials and the product, the nitrification liquor exhibits a lower decomposition temperature and a relatively high exothermic enthalpy. Therefore, it is crucial to strictly control the temperature of the reaction system during the process, and corresponding technical measures must be adopted to ensure that the temperature is below its decomposition temperature.

3.2. Calorimetric Studies of Solution Preparation and Nitration Reactions

EasyMax or RC1 was used to investigate the heat release during the preparation of AN-NA solution and HA-HAc solution, as well as during the nitration process. The thermal

cumulation of the system caused by the accumulation degree of the reaction material can be computed via the following equation:

$$X_{ac} = X_{fd} - X = \frac{\eta \cdot m_t}{m_{fd}} - X \quad (1)$$

where X_{ac} is the heat accumulation degree, %; X_{fd} is the dosing ratio, %; X is the thermal conversion rate, %; η is the excess ratio; m_t is the instantaneous mass of dosing, g; m_{fd} is the total mass of dosing, g.

The maximum temperature that the reaction process can reach (MTSR) is the highest temperature that the reaction system can reach in the case of cooling failure. It can be calculated using Equation (2) [21]:

$$MTSR = (T_{cf})_{max} = \left(T_p + X_{ac} \cdot \Delta T_{ad} \cdot \frac{m_{rf}}{M_{r(t)}} \right)_{max} \quad (2)$$

where T_p is the process temperature, °C. X_{ac} is the heat accumulation degree, %. m_{rf} is the total mass of the reaction mixture, g. $M_{r(t)}$ is the instantaneous total mass of the reaction mixture at any time, g.

The adiabatic temperature rise (ΔT_{ad}) of the system caused by heat accumulation can be calculated with Equation (3) [22]:

$$\Delta T_{ad,r} = \frac{C}{M_t \cdot C_p} \quad (3)$$

where M_t is the total mass of the reactive system at time t , kg. C_p is the SPC (specific heat capacity) of the reactive system at time t , $\text{kJ} \cdot ^\circ\text{C}^{-1} \cdot \text{kg}^{-1}$.

The exothermic characteristics of different feeding methods during the preparation of AN-NA solution were investigated using EasyMax reaction calorimetry, with the results shown in Figure 3. As shown in Figure 3a, the one-time feeding method exhibited concentrated heat release during the preparation of the AN-NA solution. The heat release of AN-NA solution was determined to be $75.99 \text{ J} \cdot \text{g}^{-1}$, $\Delta T_{ad} = 18.79 \text{ }^\circ\text{C}$, and the maximum heat release rate was 158.31 W . If heat dissipation was inadequate, significant bubble generation and overflow would occur, leading to an elevated spillage risk at higher feeding amounts. In contrast, Figure 3b shows experimental results of a five-batch experiment conducted at equal time intervals (total feeding amount unchanged). The first batch showed the maximum exothermic peak, corresponding to a maximum heat release rate of 62.42 W , and heat release gradually decreased in subsequent batches. The total heat release of batch feeding was $75.21 \text{ J} \cdot \text{g}^{-1}$, $\Delta T_{ad} = 18.43 \text{ }^\circ\text{C}$. Overall, the heat release and adiabatic temperature rise in two different feeding methods (batch feeding vs. one-time feeding) during the preparation process of AN-NA solution are nearly identical. However, the one-time feeding method generates a substantially higher instantaneous heat release rate, which constitutes the key safety concern for this operation.

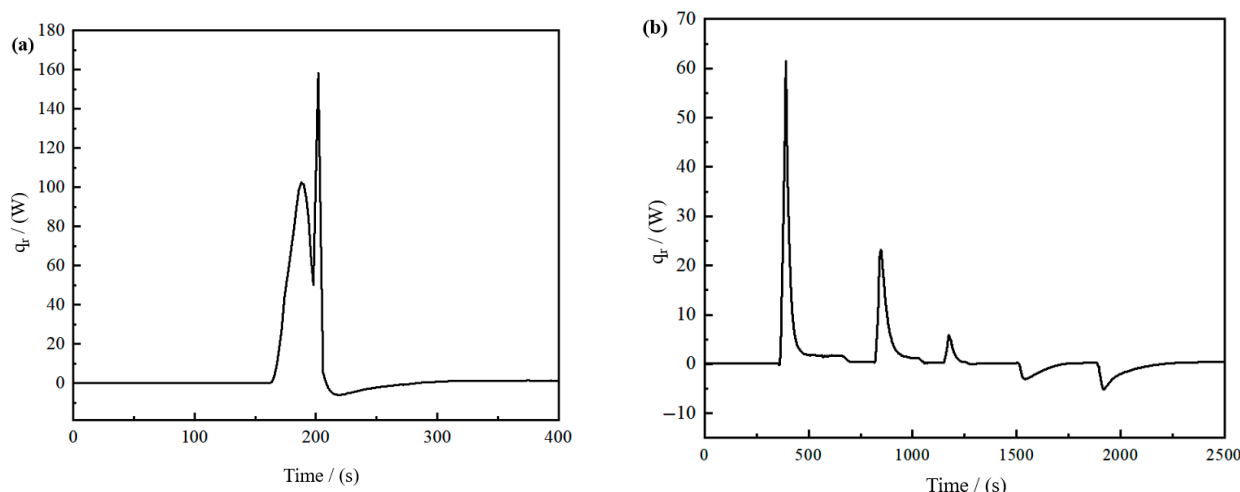


Figure 3. The calorimetric curves of different feeding methods in the preparation of AN-NA solution ((a): one-time feeding test; (b): batch-feeding test).

The exothermic characteristics of different feeding methods during the preparation of HA-HAc solution are presented in Figure 4. Similarly to the AN-NA solution preparation process, the one-time feeding method exhibited concentrated heat release, whereas batch feeding yielded smaller, more consistent heat release per batch. The total heat release of one-time feeding was calculated to be $74.97 \text{ J} \cdot \text{g}^{-1}$, $\Delta T_{ad} = 27.01 \text{ }^{\circ}\text{C}$, and the maximum heat release rate was 113.92 W . The total heat release of batch feeding was $71.54 \text{ J} \cdot \text{g}^{-1}$, $\Delta T_{ad} = 29.87 \text{ }^{\circ}\text{C}$. Overall, batch feeding is a safer approach than one-time feeding for the preparation of these two solutions.

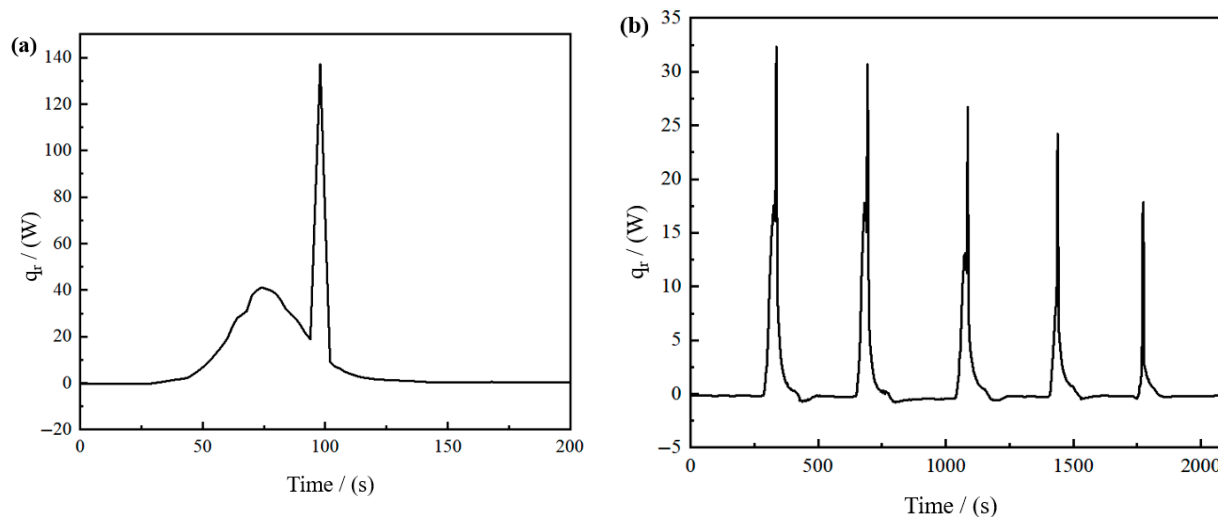


Figure 4. The calorimetric curves of different feeding methods in the preparation of HA-HAc solution ((a): one-time feeding test; (b): batch-feeding test).

The reaction calorimetry results of the nitration process are shown in Figure 5 and Table 3. The study investigated the heat release patterns during the nitration synthesis of HMX using two different feeding methods: constant-pressure funnel feeding and constant-flow pump feeding.

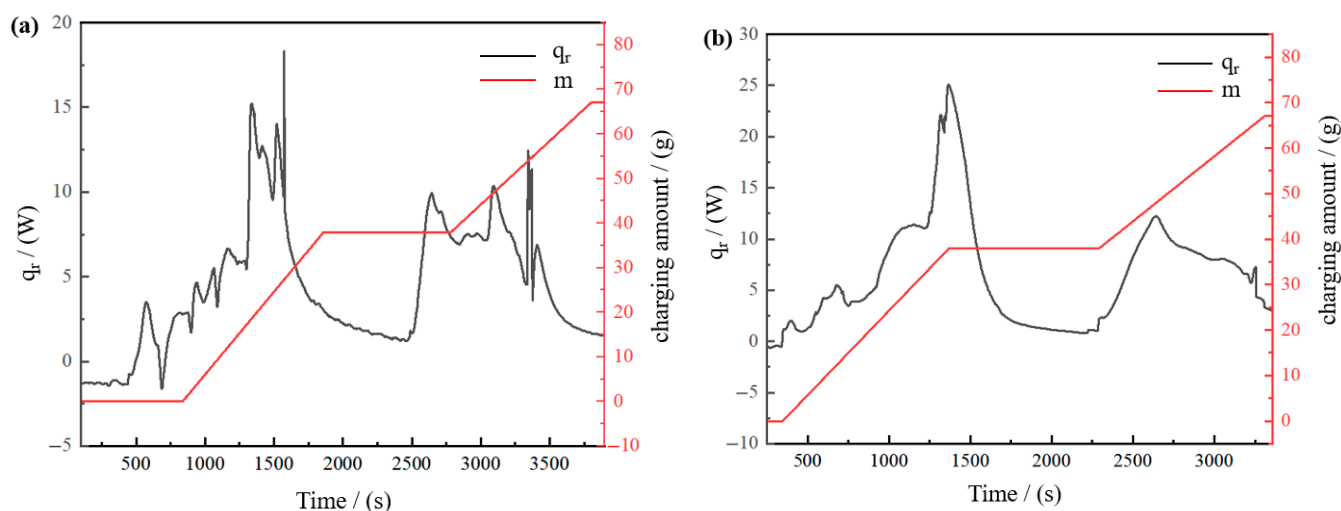


Figure 5. Thermal curves of HMX synthesis by nitrification using two feeding methods ((a): constant-pressure funnel feeding, (b): constant-flow pump feeding).

Table 3. Calorimetric data from hundred-gram scale tests.

Trial Time	T_p^a (°C)	U^b ($W \cdot m^{-2} \cdot ^\circ C^{-1}$)	C_p^c ($kJ \cdot ^\circ C^{-1} \cdot kg^{-1}$)	Q (kJ)	ΔT_{ad} (°C)
Pre-reaction	44	235.89	2.0967	/	/
Post-reaction	44	91.96	1.9662	52.99	88.82

^a: System temperature before and after reaction; ^b: heat transfer coefficient; ^c: specific heat capacity.

The nitration synthesis of HMX via the acetic anhydride method employed a two-stage feeding approach, resulting in a clearly biphasic exothermic profile. As shown in Figure 5a, the dropwise feeding method caused significant fluctuations in the heat release curve, characterized by prominent, sharp peaks with high instantaneous heat output. This phenomenon may be attributed to non-uniform feeding during the dropwise addition process. Figure 5b presents the results obtained using constant-flow pump feeding. The nitration process exhibited significantly smoother heat release characteristics compared to the dropwise addition method, with no observed sharp thermal peaks or excessive instantaneous temperature spikes. The exothermic reaction was predominantly concentrated in the first stage. Based on the reaction mechanism of HMX synthesis via the acetic anhydride method, it is speculated that the reason is that the cleavage of N-N and C-N bonds in HA to form the intermediate DPT is primarily concentrated in the first feeding stage [23]. In contrast, the subsequent bond cleavage of DPT to generate HMX releases relatively less heat, resulting in a more stable heat release during the second feeding process.

The study revealed that the reaction system exhibits intense exothermic behavior during the feeding process, with temperature being highly sensitive to feeding rates. Different feeding methods will lead to significantly different heat release rates, and there is also a significant gap in the adiabatic temperature rise. Therefore, throughout the entire reaction process, the feeding rate should be controlled as stably as possible. The stable feeding rate process has a significant impact on the reaction safety assessment results, which can reduce the experimental and test errors and ensure the accuracy of the experimental results.

In order to obtain more accurate, comprehensive, and representative data on the heat release, the RC1 reaction calorimeter was employed to carry out the hundred-gram scale-up reaction calorimetry test, with the results presented in Figure 6 and Table 3.

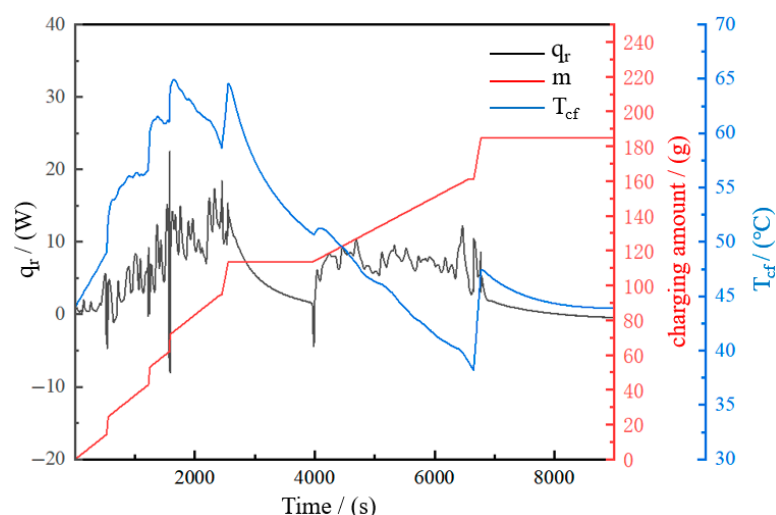


Figure 6. Calorimetric curve of the hundred-gram scale nitration synthesis of HMX.

As shown in Table 3, at the end of the first feeding stage, the calculated heat accumulation (X_{ac}) attained its maximum value of 24.83%, indicating that 24.83% of the raw materials remained unreacted. By the end of the second feeding stage, the peak value had decreased to 9.64%. The MTSR, determined via Equation (2), was found to be 65.68 °C. The selection of the maximum technical temperature (MTT) is dependent on the reaction system configuration: For the atmospheric pressure reaction system, MTT is the boiling point of the solvent or mixture of the reaction system, and the boiling point of acetic acid can be considered in this system; that is, the MTT is 117.9 °C. For closed systems, the MTT is the saturated vapor temperature corresponding to a pressure of 0.1 MPa, with an MTT of 90 °C assigned in this case. Given that industrial HMX production is carried out in a closed system, the MTT is accordingly set at 90 °C. Since the MTSR remains below this MTT threshold, the system exhibits minimal risk of violent boiling or material ejection due to reactant overheating. Therefore, under these process conditions, the temperature relationship during the reaction satisfies $MTSR < MTT$, thus precluding the occurrence of foaming and material ejection.

3.3. Adiabatic Calorimetry Test

The self-decomposition exothermic processes of AN-NA solution, HA-HAc solution, nitrification liquor, filtrate, crystal transformation solution, and its filtrate were studied using ARC. TMR_{ad} is the time required for the exothermic reaction from the start to the maximum reaction rate under adiabatic conditions. During the test, the heat released is absorbed by the material, and the device system also absorbs part of the heat. TMR_{ad} is calculated using Equation (4) [24,25]:

$$TMR_{ad} = \frac{c_p R T_0^2}{q_{T_0} E \varphi} - \frac{c_p R T_m^2}{q_m E \varphi} \quad (4)$$

where φ is the correction factor, R is the gas constant value = $8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{°C}^{-1}$, T_m is the temperature corresponding to the maximum value of the reaction rate, K; q_{T_0} is the exothermic rate of the reaction at the temperature of T_0 , $\text{W} \cdot \text{kg}^{-1}$; q_m is the exothermic rate of the reaction at the T_m temperature, $\text{W} \cdot \text{kg}^{-1}$.

The results of the adiabatic calorimetry experiment for the AN-NA and the HA-HAc solution are presented in Figure 7. As shown in Figure 7a, the AN-NA solution initiated thermal decomposition at 113 °C. Within 120 min, the temperature increased from 113.9 °C to 259.5 °C, while the pressure rose from 5 bar to 47.13 bar, with a maximum temperature

rise rate of $17.88\text{ }^{\circ}\text{C}/\text{min}$, manifesting distinct thermal explosion characteristics. This thermal behavior is ascribed to the diminished stability of ammonium nitrate in nitric acid and the relatively low boiling point of nitric acid; nitrogen oxides generated during nitric acid decomposition further accelerated the premature thermal decomposition of ammonium nitrate. In the ARC test of the HA-HAc solution (Figure 7b), two exothermic stages were observed, with the second stage exhibiting markedly intense reaction behavior. Over 180 min, the system temperature surged from $286.6\text{ }^{\circ}\text{C}$ to $400.1\text{ }^{\circ}\text{C}$, accompanied by a pressure elevation from 34.3 bar to 43.5 bar. These results demonstrate a significant risk of secondary decomposition in the event of cooling-system failure.

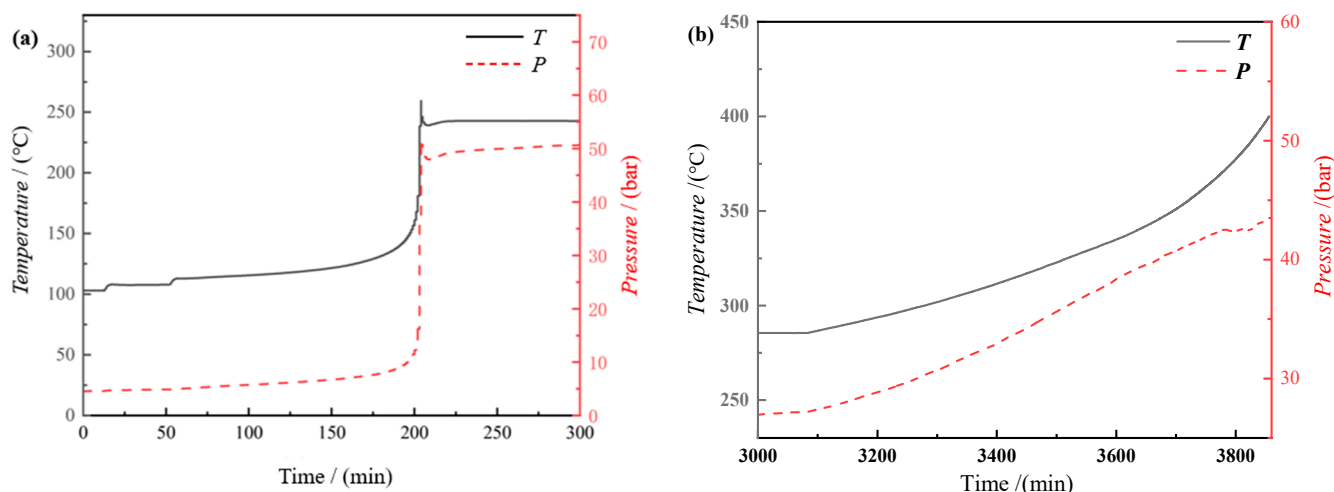


Figure 7. Two groups of solution ARC test curve ((a): AN-NA solution. (b): HA-HAc solution).

During the nitration reaction, the reaction solution collected after the second feeding stage (when all reactants had been added) was used as the nitration solution. ARC was performed on this nitration solution, with the test results shown in Figure 8. The test results indicate that the nitration solution exhibits multi-stage exothermic behavior, and its initial self-decomposition occurs at a relatively low temperature, which may pose potential safety hazards. Therefore, focused analysis was carried out on the first exothermic stage, which exhibits a temperature range of $125.92\text{--}147.96\text{ }^{\circ}\text{C}$ with an adiabatic temperature rise of $22.04\text{ }^{\circ}\text{C}$ and heat release of $181.17\text{ J}\cdot\text{g}^{-1}$. The nitration solution demonstrated $TMR_{ad} > 24\text{ h}$ at the MTSR temperature ($65.68\text{ }^{\circ}\text{C}$), with T_{D24} reaching $117.7\text{ }^{\circ}\text{C}$. Since T_{D24} exceeds the MTT ($90\text{ }^{\circ}\text{C}$), the system presents no risk of material ejection, as reaching MTT would require prolonged thermal accumulation under stagnant conditions. Even upon reaching MTT, the evaporative cooling effect of the solvent would provide an additional safety barrier.

The crude product from the acetic anhydride synthesis of HMX primarily consists of α -HMX crystals with minor amounts of the γ -HMX polymorph [26]. To obtain the more thermodynamically stable β -HMX crystal form, a polymorphic transformation process must be performed on the crude product. Common transformation methods include the ternary solvent method (acetone–ethyl acetate–water) and the nitric acid method, the latter offering advantages in terms of lower cost and shorter processing time. In this study, ARC was conducted on the nitric acid-mediated crystal transformation solution, with the results presented in Figure 9.

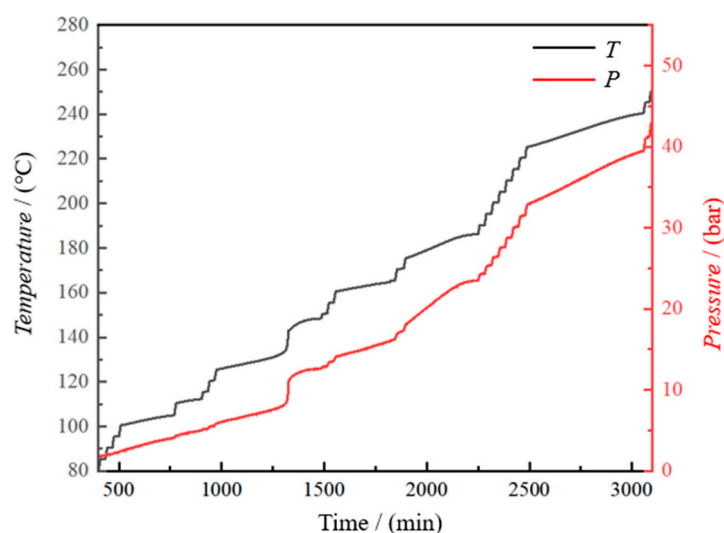


Figure 8. ARC test curve of nitration liquid.

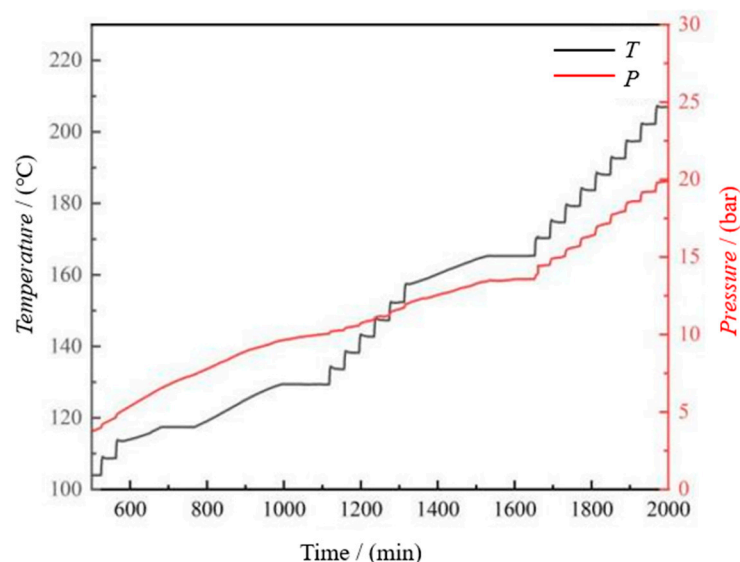


Figure 9. The ARC test curve of the nitric acid transformation solution.

The nitric acid-mediated crystal transformation solution exhibited three distinct exothermic intervals, with the first two intervals being nearly contiguous. The first interval displayed an exothermic heat of $47.32 \text{ J} \cdot \text{g}^{-1}$ with $T_{D24} = 105.9^\circ \text{C}$, followed by a second interval showing greater exothermicity ($128.61 \text{ J} \cdot \text{g}^{-1}$, $T_{D24} = 103.6^\circ \text{C}$). $TMR_{ad} > 24 \text{ h}$, indicating a low risk of self-decomposition.

The nitration and crystal transformation filtrates contain trace amounts of intermediates, impurities, byproducts, and products. As these residues pose potential explosion risks, thermal safety analysis was required for both solutions. ARC was employed to evaluate the two filtrates, with their exothermic profiles presented in Figure 10.

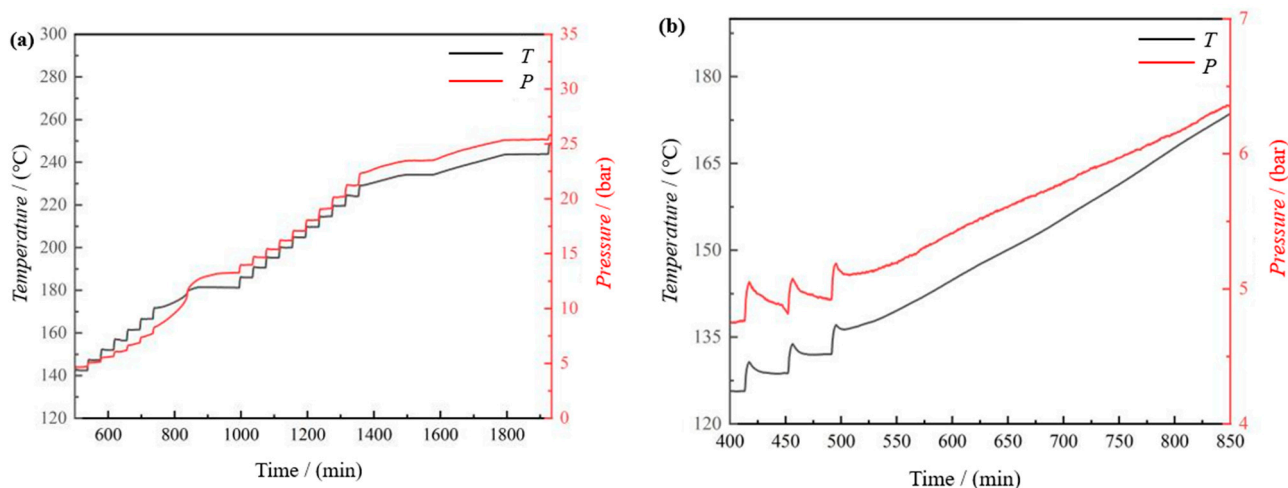


Figure 10. The ARC test curves of the two groups of filtrate solutions ((a): nitration filtrate; (b): crystal transformation filtrate).

As evidenced by the results in Figure 10, the nitration filtrate exhibits similar exothermic characteristics to the nitration solution, demonstrating multi-stage decomposition under elevated temperatures, with an initial exothermic onset temperature of 172.05 °C, heat release of 158.31 J·g^{−1}, T_{D24} = 146.1 °C, and TMR_{ad} > 24 h. The crystal transformation filtrate also displays comparable ARC exothermic behavior to the transformation solution, initiating thermal decomposition at 146.81 °C with an energy release of 70.66 J·g^{−1}, T_{D24} = 115.0 °C, and TMR_{ad} > 24 h. Under atmospheric pressure conditions, where MTT represents the boiling point of the solvent or mixture, the nitration filtrate (water/acetic acid mixture, MTT = 117.9 °C) and crystal transformation filtrate (nitric acid solvent, MTT = 83.0 °C) both present relatively low hazardous potential, though precautions should still be taken to prevent these systems from reaching their self-accelerating decomposition temperatures.

4. Conclusions

This paper investigated the thermal safety of the acetic anhydride-mediated HMX synthesis process. It was found that the decomposition temperatures of raw materials and products are all above 200 °C, indicating a low probability of decomposition during routine storage and handling. However, AN and HMX exhibit high decomposition enthalpies, so strict control of storage and operating conditions is essential. In contrast, the reaction solutions have relatively lower decomposition temperatures, meaning greater attention should be paid to their thermal safety. Although the preparation of the AN-NA solution and HA-HAc solution does not involve thermal decomposition risks, one-time feeding may cause bubbling or overflow. Multi-batch feeding can effectively mitigate this risk and is therefore safer. For the nitration process, a stable feeding rate must be maintained. During the nitration reaction, the temperature relationship satisfies $T_p < MTSR < MTT < T_{D24}$, which indicates a low risk of secondary decomposition and overflow. Given the high heat release rate during feeding, multi-point temperature sensors can be installed in the reactor, and an automatic control system should be adopted to precisely regulate the feeding rate. Two independent cooling systems (operated separately) are recommended to prevent cooling failure, with additional emergency cooling systems and emergency shutdown devices in place. The risk of thermal runaway during the polymorphic transformation process is low. Nevertheless, since the solution contains substances with explosion hazards, prolonged exposure to high-temperature environments must be avoided.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/eng7020072/s1>. References [18–20,27] are cited in supplementary file.

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