COMPATIBILITY STUDY OF OCTAHYDRO-1,3,5,7-TETRANITRO-1,3,5,7-TETRAZOCINE WITH SELECTED INSENSITIVE EXPLOSIVES USING MULTIPLE TECHNIQUES

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The present work explored the compatibility of octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) with selected insensitive explosives including nitroguanidine (NQ), 2,4,6-trinitrotoluene (TNT), 2,4,6-triamino-1,3,5-trinitrobenzene (TATB) and 2,6-diamino-3,5-dinitropyridine-1-oxide (ANPyO) through a combination of differential scanning calorimetry (DSC) with multiple heating rates, the vacuum stability test (VST) and X-ray diffraction (XRD) techniques. For DSC studies of 2℃/min heating rate based on STANAG 4147, HMX/ANPyO and HMX/TATB were regarded as compatible binary systems, while HMX/NQ and HMX/TNT had incompatible results. The thermal stability of the HMX/NQ and HMX/TNT mixtures were inferior to that of HMX, and the HMX/ANPyO and HMX/TATB mixtures became somewhat thermal stable compared to HMX. VST results further confirmed that HMX was incompatible with NQ and TNT, and compatible with ANPyO and TATB. Except for HMX/TATB, possible interaction was suspected for HMX/NQ, HMX/TNT and HMX/ANPyO binary systems from the analysis of XRD. The interaction may be the reflection of the peak temperature reduction for corresponding mixtures.

Keywords: compatibility; octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine; insensitive explosives

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1. Introduction

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX), an energetic material with excellent and comprehensive performance, has wide military applications as an explosive. Due to its relatively high sensitivity, HMX has been modified through various methods to become less sensitive [1-3]. An effective and common method is to add insensitive explosives into HMX [4-5]. As a cold explosive, the detonation properties of nitroguanidine (NQ) are slightly better than those of 2,4,6-trinitrotoluene (TNT). NQ has been applied to decrease the sensitivity of high explosives, gun propellants and low vulnerability of explosive ammunition [6-8]. However, the drawbacks of NQ like poor fluidity and loading, limit its use. Recently, 2,6-diamino-3,5-dinitropyridine-1-oxide (ANPyO) has emerged as a high energy density material with overall properties similar to 2,4,6-triamino-1,3,5-trinitrobenzene (TATB) and have attracted significant global attention [9]. Many investigations have been conducted for the mixed explosives containing ANPyO [10-11]. Despite their importance for its safe use, efforts related to the compatibility information for HMX with the above insensitive explosives are still limited.

Differential scanning calorimetry (DSC) with a 2℃/min heating rate and the vacuum stability test (VST) are recommended in STANAG 4147 for the investigation of compatibility. These methods are two of the most widely used thermal techniques [12-13]. Furthermore, in addition to DSC and VST, complementary tools, such as Fourier transform infrared spectroscopy, scanning electron microscopy and X-ray diffraction (XRD), have been applied for providing and exploring the possible interactions between the two components in a mixture with no change in chemical structures [14-17]. However, very few detailed research studies are available that refer to the compatibility of HMX with a selection of the above insensitive explosives. Therefore, in the present work, we investigate the compatibility of HMX with a selection of insensitive explosives using DSC, VST and XRD. The thermal ability of the mixtures was also studied by DSC method with four different heating rates of 2, 4, 8 and 10 ℃/min.

2. Materials and Methods

Materials

HMX, TNT, TATB and ANPyO were purchased from Gansu Yinguang Chemical Industry Group Co. Ltd. Baiyin, Gansu, China. NQ with chemical purity of 98% was abtained from the Aladdin Reagent Co. Ltd., Shanghai, China. Binary mixtures (BMs) of HMX/NQ, HMX/TNT, HMX/TATB and HMX/ANPyO were prepared with a mass ratio of 1:1 (w/w) through grinding with an agate mortar. This ratio can maximize the probability of observation of interactions between components in an investigated mixture and is commonly used for investigating compatibility and thermal stability issues [18-20].

Experimental conditions

DSC

The DSC measurements were conducted by a SETARAM DSC 131. Closed stainless steel crucibles of 30 μL were used for packing samples with ~0.7 mg for testing. The DSC tests for HMX and its mixtures were under 50 mL/min pure nitrogen with heating rates of 2, 4, 8 and 10°C/min.

VST

VST was performed through ADX-3 vacuum stability apparatus with a pressure transducer. The pure energetic component and BM consisting of an equal mass of individual materials were tested, respectively. The heating time was 40 h under 100 °C for each vacuum stability test. The equation for VST is:

$$V_R = V_C - (V_A + V_B) \tag{1}$$

Where V_R is the volume of gas generated as a result of the reaction between the components of the investigated mixture, V_C is the volume of gas from the mixture, V_A and V_B are the volumes produced by the pure contact materials.

XRD

The XRD patterns of the individual components and the prepared BMs were obtained using a Bruker D8 ADVANCE X-ray diffractometer with Cu Kα radiation. The applied scan range 2θ was 5–60° at 40 kV and 200 mA.

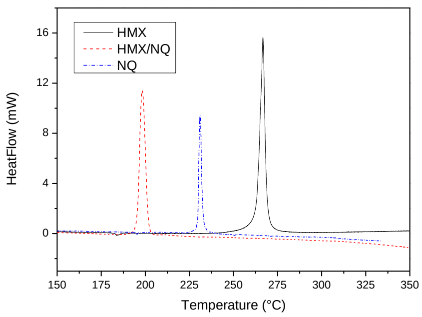
3. Results and Discussions

DSC analysis

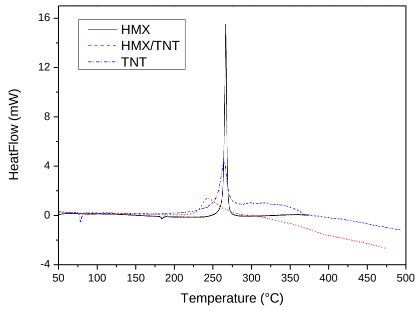
Compatibility assessment

The compatibility judgment by DSC was based on the analysis of the 2 °C /min heating rate according to STANAG 4147; however, 5 and 10 °C /min have also been chosen in some studies [21-22]. In the STANAG 4147 standard, the peak decomposition temperature obtained from the DSC curves of the BM ($T\text{p}_2$) and the main explosive ($T\text{p}_1$) was applied. The difference values $\Delta T\text{p}$ ($\Delta T\text{p} = T\text{p}_1 - T\text{p}_2$) of 4°C and 20°C are the threshold of compatibility and incompatibility, respectively. The mixed system can be regarded as compatible or incompatible in terms of the $\Delta T\text{p}$ value is less than 4 °C or above 20 °C, respectively. Between 4 and 20 °C, there exists some incompatibility for the investigated mixture.

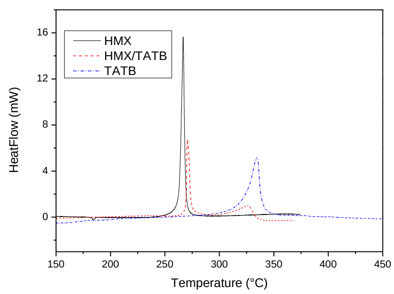
Fig. 1 and Table 1 present the DSC curves of the investigated BMs with heating rate β of 2 °C/min. As shown in Fig. 1, there exists one exothermic peak in the DSC record of the tested individual explosive. The DSC profile behaves differently when HMX is mixed with various insensitive explosives. For HMX/NQ and HMX/TNT mixtures, the decomposition processes obviously shift to an earlier event compared to HMX. From Table 1, the calculated ΔTp values of the two mixtures are 68.4 and 33.4°C, respectively. Therefore, according to the STANAG standard, HMX/NQ and HMX/TNT are considered incompatible BMs as the differences in peak temperature exceed 20 °C.



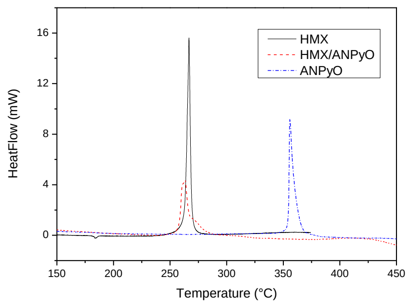
(a) HMX/NQ



(b) HMX/TNT



(c) HMX/TATB



(d) HMX/ANPyO

Fig.1 DSC records of tested mixtures at 2 °C/min heating rate

Table 1

Compatibility results by DSC with 2 °C/min heating rate

BMs	T_{p1}/℃	T_{p2}/℃	$\triangle T_p$/℃	Compatibility judgment
HMX/NQ	266.7	198.3	68.4	Incompatibility
HMX/TNT	266.7	242.9	23.8	Incompatibility
HMX/TATB	266.7	269.5	-2.6	Compatibility
HMX/ANPyO	266.7	263.5	3.2	Compatibility

There are two decomposition events for HMX/TATB and HMX/ANPyO BMs, which can be attributed to the decomposition processes of HMX and TATB or ANPyO, respectively. For the HMX/ANPyO mixture, the decomposition processes slightly shift to an earlier event compared to HMX, and the $\Delta T\text{p}$ value between HMX and HMX/ANPyO is 3.2 °C. For the HMX/TATB mixture, compared to HMX, the peak temperature of the mixture moves to a slightly higher value with an increase of 2.6 °C. Therefore, HMX/TATB and HMX/ANPyO are considered compatible based on the STANAG standard because the difference in the exothermic peak is below 4°C.

Kinetic parameter and thermal stability

In order to explore the influence of selected insensitive explosives on the thermal stability of HMX, multiple heating rates of 2, 4, 8 and 10 ℃/min were applied. Kinetic analysis was carried out using the Kissinger method to explore the dynamics from DSC data [23, 24], according to Equation (2).

$$\ln \frac{\beta_i}{T_{pi}^{\ 2}} = \ln\left(\frac{AR}{E_a}\right) - \frac{E}{RT_{pi}} \quad (2)$$

Where E represents the apparent activation energy, A the frequency factor, T_{pi} is the peak temperature obtained from DSC curves at different heating rates β and R the gas constant.

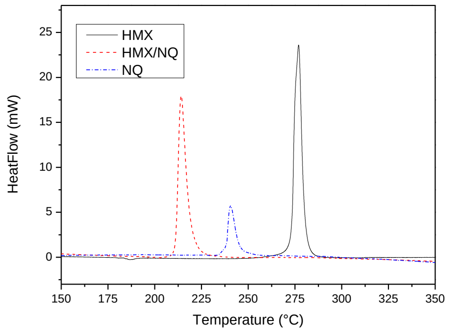
The thermal explosion critical temperature (T_b) is of importance for evaluating thermal stability of explosives, which can be calculated from [25]:

$$T_{pi} = T_{p0} + b\beta_i + c\beta_i^2 + d\beta_i^3 \quad i = 1, 2, 3, 4 \quad (3)$$

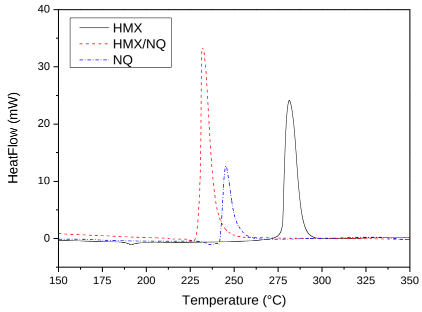
$$T_b = \frac{E - \sqrt{E^2 - 4ERT_{p0}}}{2R} \quad (4)$$

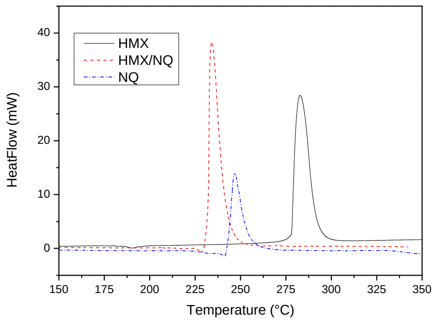
Where b, c and d are coefficients, T_{p0} the value of T_p referring to $\beta \to 0$.

Figures 2-5 show the DSC curves of HMX and its mixture with selected insensitive explosives at different heating rates. The peak temperatures and calculated $\Delta T\text{p}$ values of the investigated BMs by DSC with different heating rates are listed in Table 2. The computed kinetic parameters and T_{pi} values are listed in Table 3. As clearly observed from Table 2, the T_{pi} values increase with increasing heating rate.



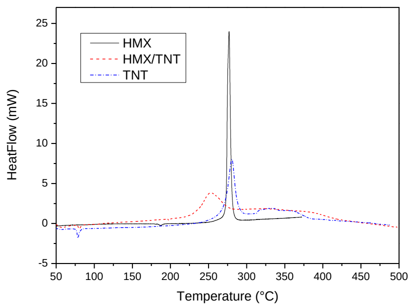
(a) 4 °C/min



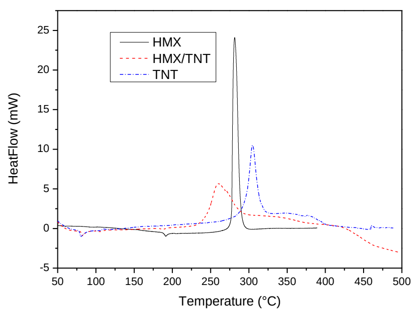


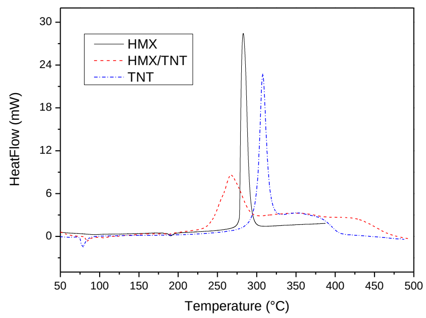
(b) 8 °C/min (c) 10 °C/min

Fig. 2 DSC records of HMX/NQ mixtures at various heating rates



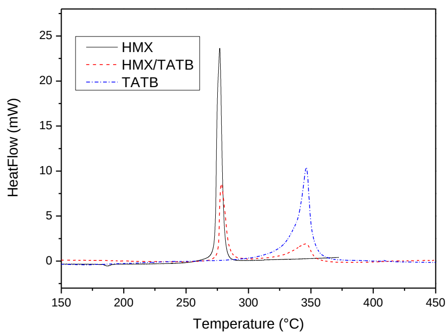
(a) 4°C/min



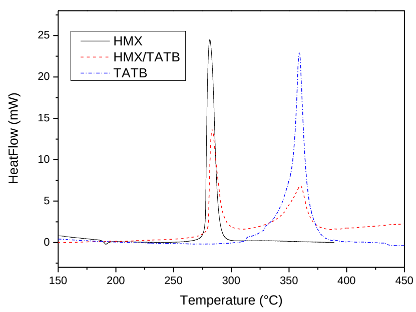


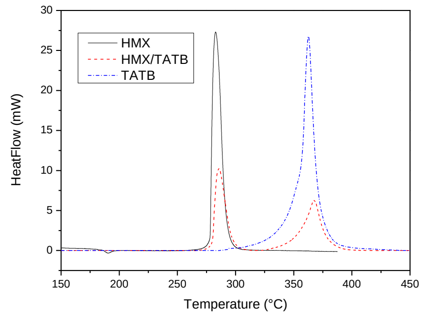
(b) 8°C/min (c) 10°C/min

Fig.3 DSC records of HMX/TNT mixtures at various heating rates



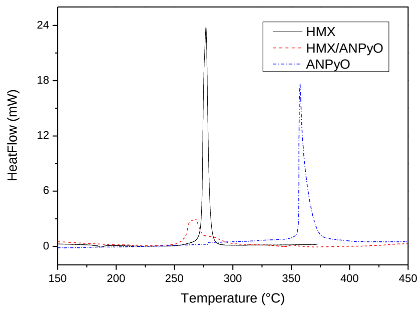
(a) 4 °C/min



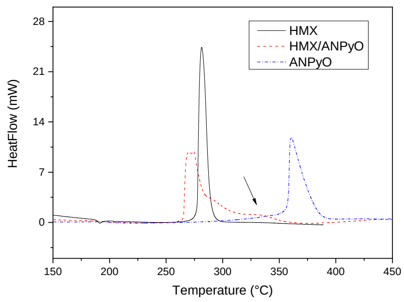


(b) 8 °C/min (c) 10°C/min

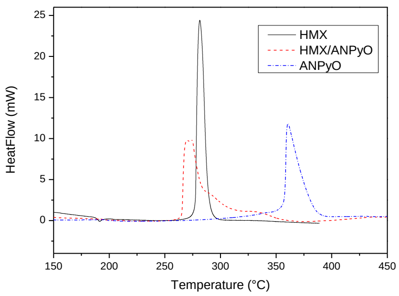
Fig. 4 DSC records of HMX/TATB mixtures at various heating rates



(a) 4 °C/min



(b) 8 °C/min



(c) 10 °C/min

Fig. 5 DSC records of HMX/ANPyO mixtures at various heating rates

Table 2

Peak temperatures by DSC with different heating rates

BMs	Heating rate/°C·min^{-1}	T_{p1}/°C	T_{p2}/°C	ΔT_p/°C
HMX/NQ	4	277.0	214.2	62.8
	8	281.4	232.1	49.3
	10	282.8	234.1	48.7
HMX/TNT	4	277.0	253.1	23.9
	8	281.4	259.9	21.5
	10	282.8	266.9	15.9
HMX/TATB	4	277.0	278.3	-1.3
	8	281.4	283.4	-2.0
	10	282.8	285.6	-2.8
HMX/ANPyO	4	277.0	268.3	8.7
	8	281.4	274.4	7.0
	10	282.8	275.5	7.3

Table 3

Kinetic parameter and thermal stability of tested BMs

BMs	E/kJ·mol^{-1}	ln$A$/s^{-1}	r	T_b /°C
HMX	238.3	57.7	0.9736	248.3
HMX/NQ	81.9	24.6	0.9964	181.1
HMX/TNT	158.5	41.6	0.9873	223.9
HMX/TATB	272.0	94.9	0.9950	258.6
HMX/ANPyO	313.5	75.2	0.9981	259.5

It can be found that from Table 2 the calculated ΔTp values for the HMX/NQ BM at heating rates of 4, 8 and 10 °C/min are all above 20 °C and even more than 40 °C, indicating that HMX and NQ have strong chemical interactions with each other. Therefore, some incompatibility results can be expected at higher heating rates. For the HMX/TNT BM, the difference in the peak temperature is ~20 °C at various heating rates, indicating some chemical interactions and potential incompatibility therefore are suspected. For the HMX/TATB mixture, the exothermic peak temperatures at various heating rates are all slightly higher by ~2°C than that of pure HMX, which suggests that the addition of TATB effectively stabilizes HMX and results in good compatibility. When HMX and ANPyO are mixed and heated at tested heating rates, the ΔTp values are ~7–8 °C and chemical interactions are suspected. Therefore, lack of compatibility is suggested for HMX/ANPyO BM at heating rates higher than 2 °C /min.

From Table 3, the E values of the HMX/NQ and HMX/TNT mixtures are lowered by 156.4 and 79.8 kJ·mol^{-1} than that of HMX (238.3 kJ·mol^{-1}), respectively. The T_b values of the HMX/NQ and HMX/ANPyO mixtures are decreased by 67.2 and 24.4 °C compared to that of HMX (248.3°C). The addition of NQ and TNT can therefore lead to a drop in the thermal stability of HMX. The decrease in peak temperature, activation energy and thermal stability can be indicative of incompatibility between HMX and NQ or TNT.

For HMX/ANPyO and HMX/TATB BMs, the E values are 75.2 and 33.7 kJ·mol^{-1} higher than that of HMX (238.3 kJ·mol^{-1}), respectively. The T_b values of the HMX/ANPyO and HMX/TATB mixtures are close to each other and 11.2 and 10.3 °C higher than that of HMX, respectively. Therefore, ANPyO and TATB can somewhat increase the thermal stability of HMX.

The reasons why the addition of NQ and TNT can significantly advance the decomposition process of HMX are associated with the decomposition mechanism of the components [26]. There exists a competition mechanism in the thermal decomposition process of HMX, in which the C-N and N-N bond can break simultaneously, as illustrated in Equations (5)–(7) [27,28]. Low temperature and low heating rate are beneficial to the breaking of C-N bonds and higher concentrations of CH_2O and N_2O can be generated, while high temperature and higher heating rate are conducive to the breaking of N-N bonds.

$$HMX \rightarrow 4(CH_2O+N_2O)\ ;\ \Delta H = -212.3 kJ\cdot mol^{-1} \quad (5)$$

$$HMX \rightarrow 4(HCN+HONO) \text{ or } 4(HCN+H\cdot + NO_2)\ ;\ \Delta H = +117.2 kJ\cdot mol^{-1} \quad (6)$$

$$5CH_2O+7NO_2 \rightarrow 7NO+3CO+2CO_2+5H_2O \quad \Delta H = -1350.0 kJ\cdot mol^{-1} \quad (7)$$

For the HMX/NQ BM, the decomposition mechanism of NQ is relatively complex. Nitroamide (NH_2-NO_2) and cyanamide (NH_2-CN) are produced in the initial decomposition of NQ due to the breaking of the C-N bond in the molecule. The decomposition process where the intermediate product NH_2-NO_2 produce H_2O and N_2O requires the combination of OH and hydrogen radicals and is irreversible [29,30]. When the HMX/NQ mixture is heated, the dehydration of NH_2-NO_2 in NQ promotes the generation of hydrogen radicals during the decomposition of HMX. Therefore, the decomposition process of HMX/NQ BMs could be accelerated in comparison with HMX.

For the HMX/TNT BM, it is believed that the initial thermal decomposition of TNT is mainly the oxidation reaction of methyl (- CH_3) [31]. When the HMX/TNT mixture is heated, the hydrogen radicals generated in the thermal decomposition of HMX could result in the acceleration of the oxidative dehydration of methyl of TNT, thereby potentially resulting in an accelerated thermal decomposition event of the BM.

Regarding the degree of incompatibility of HMX/ANPyO BM at higher heating rates, the result may be due to the improvement in the thermal effect per unit time [32]. Furthermore, there may exist an interaction between the decomposition products between HMX and the complex multi-step decomposition reaction of ANPyO [33].

VST results

The sample quality used in VST is more representative that in DSC. As an accepted method, VST have been applied to confirm the compatibility assessments by DSC [34-35]. The compatibility criterion from VST in the STANAG is the amount of evolved gas related to the mixture and the pure components in the mixture. The investigated mixture can be regarded as a compatible system when the evolved gas volume is less than the critical value of 5 mL; otherwise, incompatible results can be concluded. It is obvious from Table 4 that the compatibility issue of the HMX with NQ is unsatisfied as the V_R value is ~10.35 mL, i.e., higher than the critical value of 5 mL, which raises a potential safety issue once NQ is in the HMX mixture. With the exception of the HMX/NQ BM, the V_R values of HMX with selected insensitive explosives are below 5 mL.

Therefore, HMX/TNT, HMX/ANPyO and HMX/TATB are compatible, while HMX/NQ shows incompatibility according to the STANAG. The compatibility findings, except for HMX/TNT BM, show consistency with DSC and VST and the inconsistency is also available in released research [36,37]. The results based on both the DSC and VST indeed reflect the compatibility from a different perspective. The different temperature coverages for the two tested methods can be supposed to account for the contrasting and even contradictory conclusions.

Table 4

The generated gas volume of tested BMs

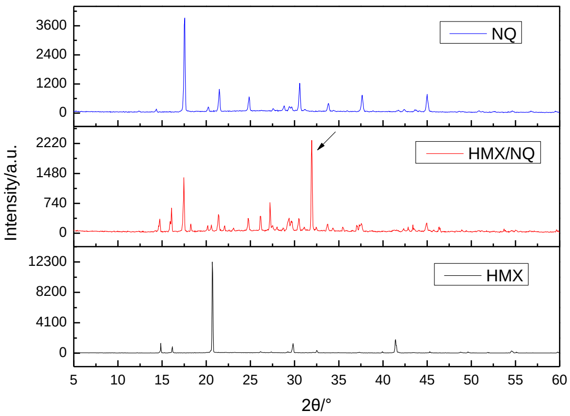
BMs	HMX/NQ	HMX/TNT	HMX/TATB	HMX/ANPyO
V_R value/mL	10.35	0.90	1.16	4.68

XRD analysis

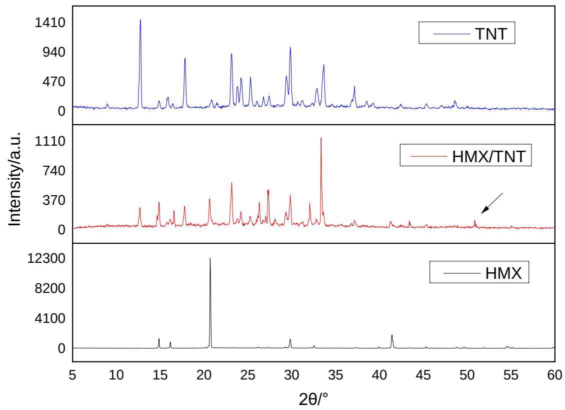
As an effective supplementary method, XRD has recently been applied to explore the possible compatibility or interaction between high explosive and contact materials, although no related standard has been released [38-39]. The XRD patterns of the tested BMs are presented in Fig. 6.

As shown in Fig. 6, several changes are easily observed. Except for the new peaks (2θ between 31° and 33°) of HMX/NQ and (2θ between 50° and 51°) of HMX/TNT, the XRD patterns of the HMX/TATB can be considered as a combination of those of pure HMX and NQ. The results, therefore, evidently suggests interactions [40] for the HMX/NQ and HMX/TNT BMs. For HMX/ANPyO BM, there also exists evidence of substantial increased intensity (2θ between 32.0° and 33°) of the mixture compared to that of pure HMX and ANPyO. These results clearly suggest interactions in HMX/NQ, HMX/TNT and HMX/ANPyO BMs.

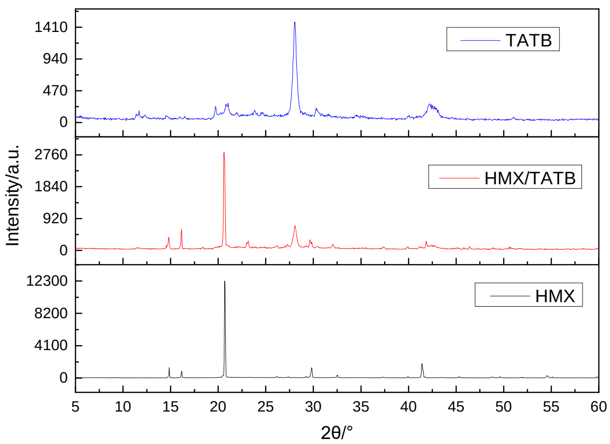
The interactions suggested in the HMX/NQ, HMX/TNT and HMX/ANPyO BMs by the XRD record may not necessarily result in some degree of incompatibility between the components in the BMs. In fact, these interactions may cause the reduction of peak temperature of HMX with the addition NQ, TNT and ANPyO, as partially indicated in the DSC results.



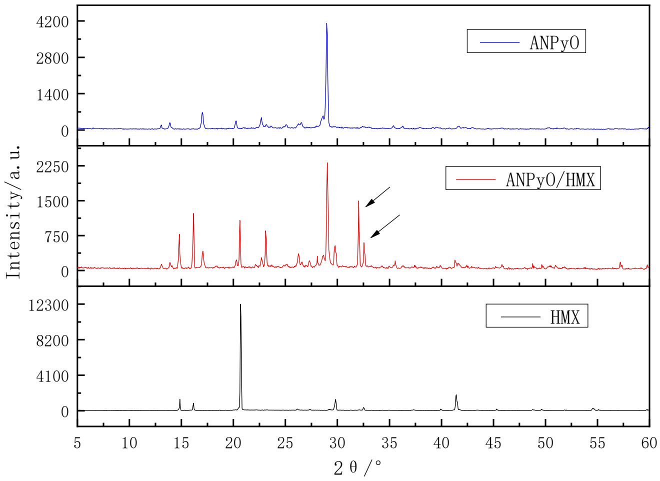
(a) HMX/NQ



(b) HMX/TNT



(c) HMX/TATB



(d) HMX/ANPyO

Fig. 6 XRD records of investigated BMs

4. Conclusions

The compatibility of HMX with selected insensitive explosives was investigated by thermal techniques (DSC and the VST) based on STANAG 4147, and supplementary techniques XRD. The results by DSC showed that HMX/NQ and HMX/TNT binary systems are incompatible. The VST further confirmed that HMX/TNT, HMX/ANPyO and HMX/TATB are compatible, while HMX/NQ is incompatible. The thermal stability of the HMX was decreased by the addition of NQ and TNT, and somewhat increased by ANPyO and TATB. Supplementary analytical technique XRD provide evidence of possible interaction for investigated mixtures, which may partially verify the data derived from DSC findings. Further investigations would be needed in order to explore the definite mechanism for compatibility related to high explosives.

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