



Pre-formulation Compatibility Studies of 5-Amino-1H-tetrazole Nitrate with Several Typical Materials by Thermal and Non-thermal Techniques

Wanxiao Guo, Zhiwei Han,* Qiuhan Lin, Boliang Wang

Nanjing University of Science and Technology,

Xiaolingwei 200, 210094 Nanjing, China

**E-mail: hanzhiwei@njust.edu.cn*

Abstract: The aim of the present study was to assess the physicochemical compatibility of a promising energetic salt, 5-amino-1H-tetrazole nitrate (5-ATN), with some typical materials. Thermal techniques (differential scanning calorimetry (DSC) and vacuum stability test (VST)) and non-thermal techniques (X-ray diffractometry (XRD) and Fourier Transform Infrared Spectroscopy (FTIR)) were applied. Five energetic materials (TNT, RDX, HMX, CL-20 and AP) and three common additives (Al, DOS and F₂₆₀₄₋₂) were tested to evaluate their compatibility with 5-ATN. Based on the DSC results, except for AP that was only partially compatible with 5-ATN, all of the selected materials exhibited good compatible with 5-ATN. The VST test further confirmed the compatibility of the 5-ATN/AP mixtures. Combined with the thermal methods, the FTIR results agreed with the DSC findings. The XRD results showed some differences.

Keywords: 5-ATN, compatibility, energetic materials

1 Introduction

As a key branch of material science, energetic materials continue to be a considerably explored area of great importance [1]. Improved energetic materials have been given unprecedented attention by scientists around the world. Highly energetic ionic salts represent a unique class of energetic molecules, which have attracted substantial interest in the last decade because of their distinctive properties, such as low vapour pressures, high densities and enhanced thermal

stabilities [2-5]. Among these new molecules, tetrazole-based energetic species have drawn particular attention due to the high heat of formation (237 kJ/mol) for the five-membered nitrogen-rich ring of tetrazole (CH₂N₄) [6-9]. Previous studies of these compounds have focused on the synthesis of novel derivatives, some of which have high detonation properties and superior stabilities, or on the development of new synthetic pathways [10-13].

5-Amino-1*H*-tetrazole hydronitrate (5-ATN), a typical tetrazole-based energetic salt, has potential applications in propellants and gun powders due to its excellent properties: measured density 1.847 g·cm⁻³; nitrogen content 56.76%; oxygen balance -18%, molar heat of formation 87 kJ·mol⁻¹; detonation velocity 8900 m·s⁻¹; friction sensitivity >324N [6, 13]. Moreover, the synthetic process for 5-ATN is simple and easy to industrialize. The preparation, crystal structure, detonation properties and thermal behaviour of 5-ATN have been intensively studied [9, 14-16]. However, to our knowledge, few studies have concentrated on basic research for its applications, such as compatibility.

It is well known that energetic materials are rarely used separately. Interactions arising from the incompatibility between the energetic material and the contacted components may further accelerate the rate of ageing, alter the shelf life and impair the chemical nature of the energetic material. Consequently, unexpected explosions due to decomposition reactions could result [17]. Therefore, the compatibility of new explosives is the first issue that should be investigated before formulation studies. Thermal methods, such as DSC and VST, have been outlined by the NATO Standardization Agreement STANAG 4147 for compatibility assessment. DSC has been proposed to be a rapid and efficient method to initially assess the compatibility of mixtures containing the active component and various contacted ingredients [18, 19]. Despite the stated advantages of the DSC method, one of its main drawbacks is the small amount of sample required (milligram scale). Interpretations deduced from DSC may not be suitable for larger samples [20]. VST is used routinely for compatibility assessment [21, 22]. The sample quantity (gram scale) used in a VST test is much more representative. However, the application of VST is limited partly because it is time consuming. Consequently, it is recommended that DSC and VST should be combined with each other for exploring compatibility issues.

However, information from thermal studies should be interpreted carefully in order to avoid erroneous judgements and conclusions [23]. Therefore, some non-thermal techniques have been frequently used to expand on the thermal behaviour that is not observed at room temperature. Besides the thermal methods, XRD and FTIR have contributed significantly in the search for possible interactions in pharmaceutical chemicals [24-26]. To our knowledge, the

published literature and standard guidelines concerning the use of FTIR and XRD in assessing compatibility for energetic materials remain limited. Therefore, the experimental results have depended mainly on DSC and VST. XRD and FTIR were supplementary methods.

The aim of the present work was to obtain valuable compatibility data that has not been released for interactions between 5-ATN and its typical contacted materials, by thermal techniques (DSC and VST) combined with supplementary methods (XRD and FTIR). Knowledge of such information will be helpful in designing and optimizing the 5-ATN-based formulations required for future applications.

2 Experimental

2.1 Materials

5-ATN was prepared in our laboratory according to the existing method. The structural formula is shown in Figure 1 and its purity was 99.5%. The 5-ATN prepared was dried in an oven before use.

Cautionary note: 5-ATN samples should not be exposed to prolonged daylight in order to prevent photolytic decomposition in the presence of bright light.

TNT, RDX, HMX, CL-20 and AP were used as the contacted energetic materials. TNT, RDX HMX and CL-20 were purchased from Gansu Yin Guang Chemical Industry Group Co., Ltd. (Gansu, China), their chemical purities were greater than 99%. AP was produced industrially and was used without further processing; its chemical purity was 98%. F₂₆₀₄₋₂ (a kind of fluorinated rubber) and DOS (dioctyl sebacate) used as common additives were all industrially produced.

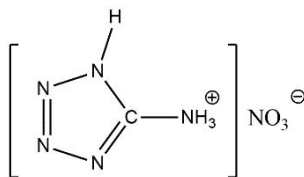


Figure 1. Structural formula of 5-ATN

2.2 Thermal and non-thermal techniques

2.2.1 Thermal techniques

Differential scanning calorimetry

A SETARM DSC 131 differential scanning calorimeter (SETARAM Instrumentation, Caluire, France) with a stainless steel crucible was used for thermal analysis of 5-ATN, contacted individual materials alone and their binary mixtures (BMs). BMs of 5-ATN with each selected compounds were prepared by grinding in an agate mortar and pestle at a mass ratio of 1:1 before DSC testing. Approximately 1 mg sample mass was weighed from the mixture. The DSC test of each sample was under a flow of nitrogen at a rate of 50 mL/min and a heating rate of 10 °C/min. From STANAG 4147, the criteria of compatibility by DSC are based on the following: when the shift towards lower temperature is by 0-4 °C, or 4-20 °C, or more than 20 °C, the tested materials are regarded as compatible, a degree of incompatibility (further investigation required), or incompatible, respectively.

Vacuum stability test (VST)

The volume of evolved gas, when 5-ATN, the contacted compound and their BMs are heated separately at 100 °C for 40 h in an initial vacuum, is measured by a pressure transducer (PVST) according to STANAG 4147. The measuring principle is based on the additional gas liberated from the tested mixture [27]. In the sense of the STANAG, incompatibility is judged if the additional gas evolved for the tested mixture of compounds against the same parameters for the pure components is above 5 mL/g.

2.2.2. Non-thermal techniques

X-ray diffractometry

The XRD patterns of the samples were recorded using a Bruker diffractometer (model D8 ADVANCE, Germany) with measurements of 2θ in the range from 5° to 60° using Cu K α radiation.

Fourier transform infrared spectroscopy

The apparatus used to conduct FTIR experiments was a Thermo Fisher FTIR spectrometer in the transmittance mode. The spectra were recorded at room temperature using 32 scans, a resolution of 0.5 cm⁻¹ and a range from 4000 cm⁻¹ to 500 cm⁻¹.

3 Results and Discussion

3.1 DSC analysis

All of the selected DSC curves of single components and BMs measured at a heating rate of 10 °C/min are shown in Figures 2a-e. The thermal decomposition of 5-ATN exhibited a single exothermic peak at 177.6 °C due to its rapid decomposition. The peak temperature value was chosen to calculate the shift in the peak temperature of 5-ATN (T_{dE}) and its admixtures (T_{dM}).

The temperatures of the decomposition peaks for all of the BMs and their temperature differences are listed in Table 1. According to the recommendations of STANAG 4147, the following preliminary observations can be deduced.

The DSC curve of the 5-ATN/TNT exhibits one exothermic peak. This sharp exothermic peak was attributed to the decomposition of 5-ATN and the temperature peak increased by -1.8 °C, meaning that TNT is compatible with 5-ATN according to STANAG 4147.

Table 1. Data summary of the dynamic DSC results

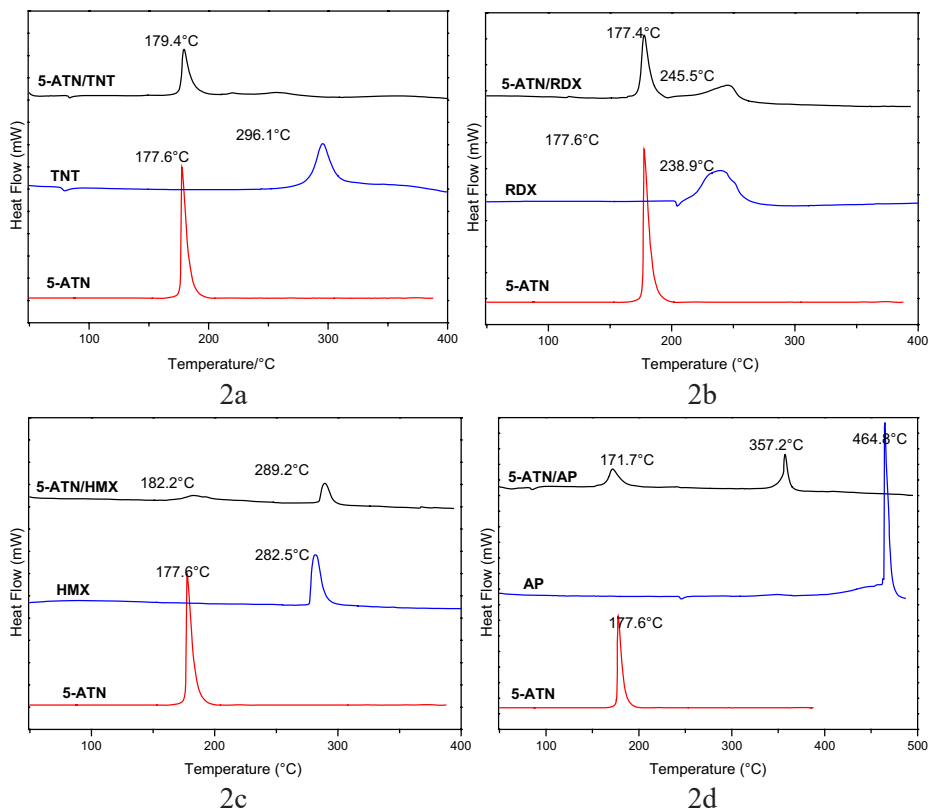
BMs	T_{dE} [°C]	T_{dM} [°C]	ΔT_p^a [°C]	Compatibility
5-ATN/TNT	177.6	179.4	-1.8	Compatible
5-ATN/RDX	177.6	177.4	0.2	Compatible
5-ATN/HMX	177.6	182.2	-4.6	Compatible
5-ATN/AP	177.6	171.7	5.9	Partially compatible
5-ATN/CL-20	177.6	176.9	0.7	Compatible
5-ATN/Al	177.6	174.8	2.8	Compatible
5-ATN/F _{2604.2}	177.6	176.0	1.6	Compatible
5-ATN/DOS	177.6	174.2	3.4	Compatible

$$^a \Delta T_p = T_{dE} - T_{dM}$$

Figures 2b-2e showed that all of the other BMs had two peaks; the peaks of 5-ATN/RDX, 5-ATN/HMX, 5-ATN/AP and 5-ATN/CL-20 in the DSC curves were reduced by 0.2 °C, -4.6 °C, 5.9 °C and 0.7 °C respectively, in contrast to the T_{dE} of 5-ATN. The second peak of the BM 5-ATN/RDX was 6.6 °C higher than that of neat RDX. It could be inferred that the presence of 5-ATN may stabilize RDX. Similarly, the presence of 5-ATN also made HMX more stable, but HMX had a small negative influence on the stability of 5-ATN. Figures 2d and 2e showed that the exothermic peaks at 357.3 °C and 234.1 °C for 5-ATN/AP and 5-ATN/CL-20 respectively, were lower than those of the corresponding reference materials (AP and CL-20). Concerning ΔT_p of the corresponding

BM, it may be concluded that AP and CL-20 mixed with 5-ATN could reduce their stability; moreover AP could promote the decomposition of 5-ATN, while CL-20 had no effect on the thermal behaviour of 5-ATN. Consequently, RDX, HMX and CL-20 were compatible with 5-ATN, while 5-ATN/AP was partially compatible according to STANAG 4147. It was therefore necessary to perform further tests to determine the compatibility of the BM 5-ATN/AP.

The DSC patterns of the BMs 5-ATN/Al, 5-ATN/F₂₆₀₄₋₂ and 5-ATN/DOS were similar to the behaviour of 5-ATN alone. These DSC curves (Figures 2f-2h) exhibited one exothermic peak. The ΔT_P values for these mixtures were 2.8 °C, 1.6 °C and 3.4 °C, respectively. Therefore, 5-ATN/Al, 5-ATN/F₂₆₀₄₋₂ and 5-ATN/DOS are compatible with 5-ATN, according to the criteria stated in STANAG 4147.



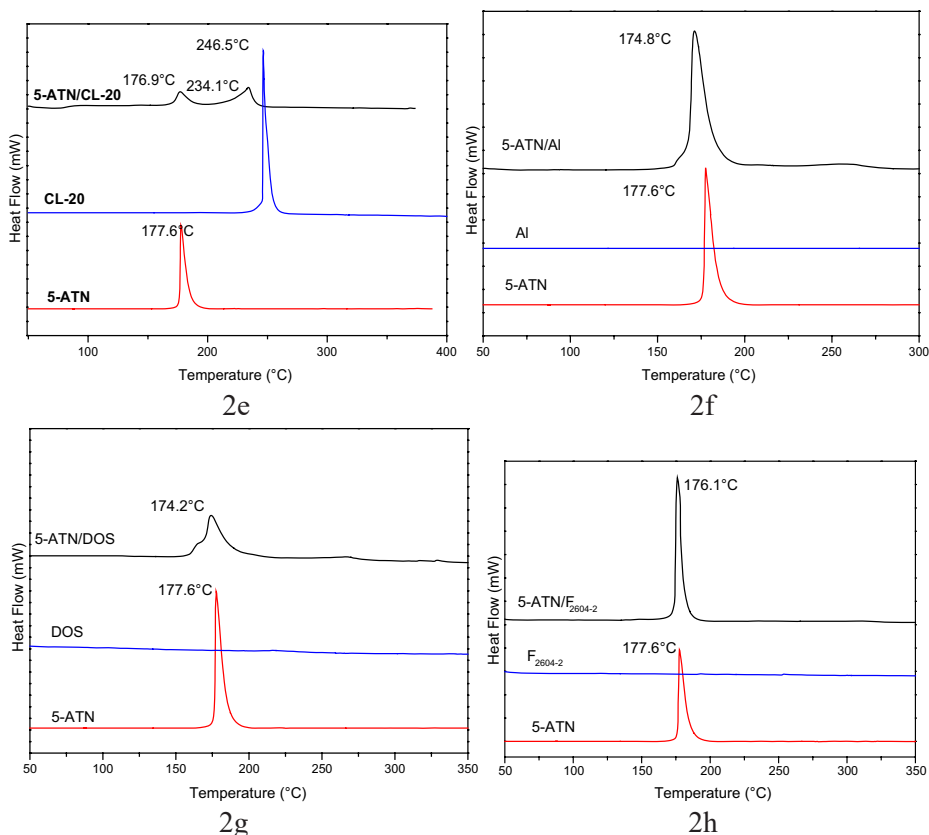


Figure 2. DSC curves of all of the binary systems

3.2 VST results

STANAG 4147 suggested one more independent measurement, based on the involvement of a different principle, for confirmation of compatibility, especially for those systems judged incompatible by DSC [28, 29]. Compared with DSC, the operating temperature of the VST is much closer to that of the practical conditions for the storage of mixtures containing the active compound.

Table 2 lists the additional volume of gas released, and compatibility judgements based on the VST results according to STANAG 4147. It may be seen that all of the VST values of the tested mixtures were well below 5 mL. Notably, the VST results of the mixtures with negative values were also considered as compatible in many investigations [30, 31]. Therefore, AP was compatible with 5-ATN according to STANAG 4147. Additionally, this mixture was determined to be compatible by VST, which was in agreement with the judgement by DSC.

Table 2. Volume of evolved gas in VST

BM	Evolved gas [mL/g]	Compatibility judgement
5-ATN/TNT	-1.58	Compatible
5-ATN/RDX	-6.49	Compatible
5-ATN/HMX	-6.11	Compatible
5-ATN/CL-20	0.43	Compatible
5-ATN/AP	-5.05	Compatible
5-ATN/Al	-2.40	Compatible
5-ATN/F ₂₆₀₄₋₂	-2.30	Compatible
5-ATN/DOS	-1.62	Compatible

3.3 XRD studies

The XRD (X-ray diffractometry) analytical technique is a direct measurement of the crystal forms that contain the fingerprints of materials, and has been proven to be of immense help in understanding incompatibilities between ingredients and confirming the observations obtained by thermal methods [32-34]. Possible chemical interactions are assumed to be absent in the absence, shift or substantial broadening of the XRD peaks for the tested mixture of compounds against those for the individual components used in the tested mixture [35]. Since F₂₆₀₄₋₂ and DOS are amorphous structures, there were no characteristic peaks, and the presence of these diffraction peaks may mask the 5-ATN diffraction peaks, so there is no representative data.

The XRD patterns are shown in Figure 3a-3f. 5-ATN/AP showed partial compatibility according to the DSC results. The XRD pattern of the 5-ATN/AP mixture (Figure 3e) showed that the peaks at 31.5° and 32.6° (attributed to the pattern of 5-ATN) disappeared in the XRD pattern of the mixture. However the characteristic diffraction peaks (8.42°, 10.83° and 16.41°) of 5-ATN, remained practically unchanged in this BM. This observation implied that possible chemical interactions were suspected between 5-ATN and AP. But this interaction may not necessarily induce incompatibility between the two explosives.

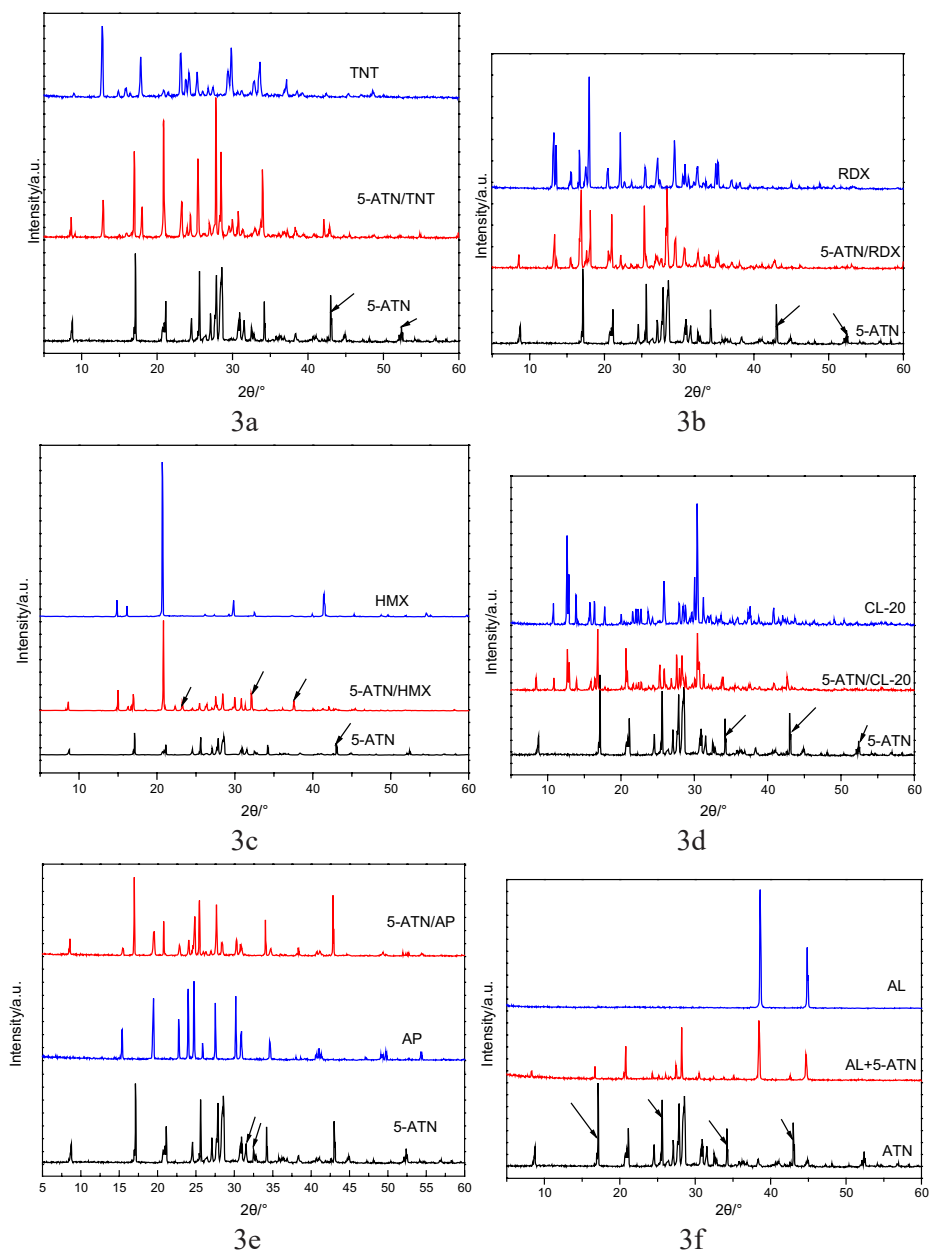


Figure 3. XRD patterns of all of the mixtures

By contrast, the characteristic peaks of 5-ATN/TNT at 43.1° and 52.4° also exhibited some changes in intensity compared with pure 5-ATN. In fact, those changes might cause improved thermal stability, as partially indicated in the DSC results [36]. Similar phenomena were observed with the other BMs, such as 5-ATN/RDX, 5-ATN/HMX and 5-ATN/CL-20. Therefore, combined with the DSC and VST results, the XRD patterns of 5-ATN/TNT, 5-ATN/RDX, 5-ATN/HMX, 5-ATN/CL-20 and 5-ATN/AP could confirm the compatibility between the two individual components within each BM.

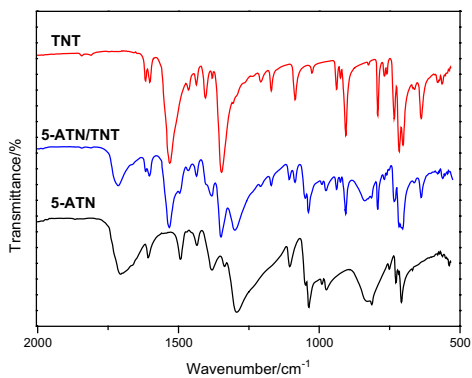
In a similar manner to 5-ATN/AP, a remarkable change occurred in the XRD pattern of 5-ATN/Al (Figure 3f) compared with those of 5-ATN, and exhibited the disappearance of several important peaks (*i.e.* 26.1° and 33.8°) and a substantial decrease in the intensity of the other major peaks (*i.e.* 16.7° and 42.5°). Thus, the XRD pattern of 5-ATN/Al suggested possible chemical interactions between the two individual components. The disappearance and reduction of the characteristic peaks requires further study.

3.4 Infrared analysis

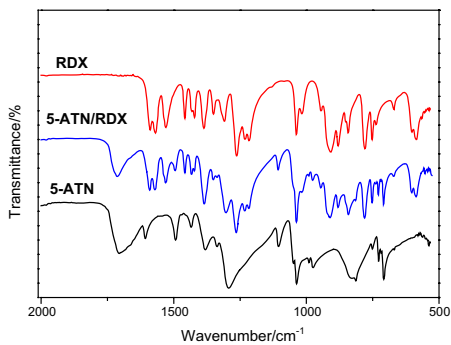
The FTIR method always acts as a non-destructive complementary method, for the detection of possible interactions between the components. If the FTIR measurement for a mixture were a combination of the characteristic peaks, without any absence, shift or substantial broadening of the peaks, of the individual components, then no interactions could be inferred [37, 38].

The FTIR spectra of the mixtures are shown in Figures 4a-4e. The FTIR spectra of the physical mixtures 5-ATN/TNT, 5-ATN/RDX, 5-ATN/CL-20, 5-ATN/Al, 5-ATN/F₂₆₀₄₋₂ and 5-ATN/DOS clearly showed that there was no change in the spectra of the BMs. Therefore, it was concluded that there was no chemical interaction between these pairs of components.

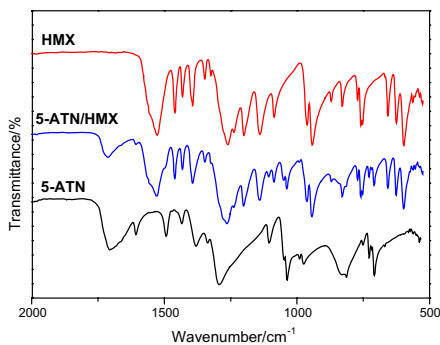
The FTIR spectral peaks of 5-ATN/HMX at 1606 cm⁻¹ (NO³⁻ vibrations) and 5-ATN/AP at 1107 cm⁻¹ (C–N vibrations) were somewhat different to those of 5-ATN. But all of the other characteristic peaks were not changed. This inferred that the presence of HMX and AP caused chemical interactions when mixed with 5-ATN. Therefore, the FTIR test indicated that all of the selected materials were compatible with 5-ATN, which agreed with the DSC results.



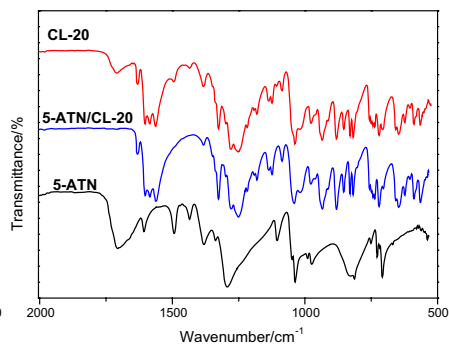
4a



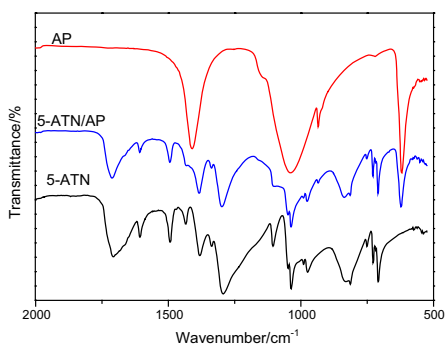
4b



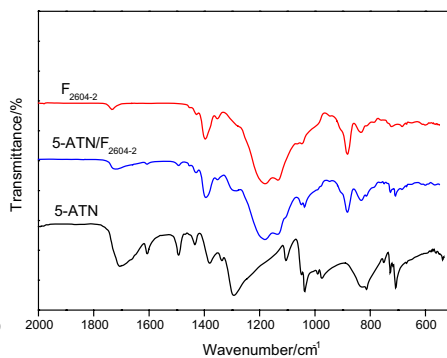
4c



4d



4e



4f

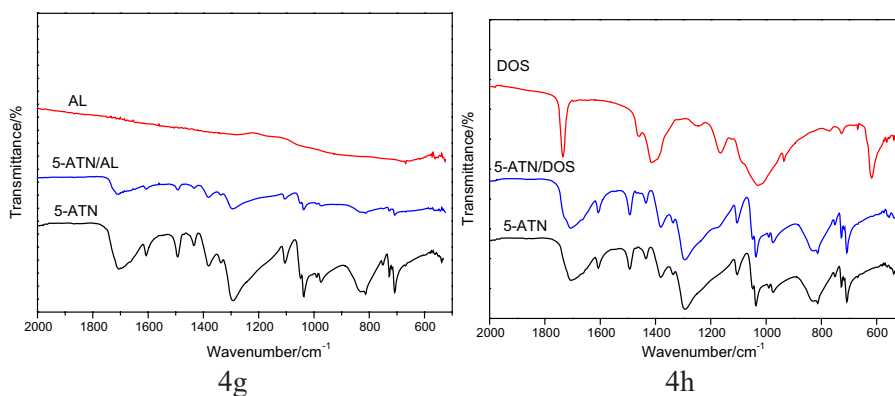


Figure 4. FTIR spectra of all of the binary systems

4 Conclusions

The compatibility of 5-ATN with some typical energetic materials and three commonly used additives were investigated in this study using different physicochemical methods, including thermal techniques (DSC and VST) and supplementary non-thermal techniques (XRD and FTIR). In the DSC compatibility study, 5-ATN showed great compatibility with all of the selected materials except AP, which was partially compatible. Further exploration by VST confirmed the compatibility of 5-ATN with all of the selected materials. Combined with the results of the thermal methods, the non-thermal FTIR method did not show any typical trace that could be used to conclude incompatibility. Some changes in the XRD patterns of the mixtures 5-ATN/AP and 5-ATN/Al suggested possible interactions within these BMs, which disagreed with the DSC observations. Traditionally, compatibility criteria have been mainly based on the results of the thermal analyses. All of the selected materials were compatible with 5-ATN by the thermal techniques (DSC and VST), according to STANAG 4147. But what is interesting is that some other non-thermal techniques suggested that possible interactions might exist between the components of the BMs. Although these interactions could not lead to safety problems directly, this phenomenon probably decreases the shelf life of the explosives containing those components. The mechanism of that phenomenon is worth pursuing in the future.

Acknowledgements

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