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# Compatibility study of *trans*-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin (TNAD) with some energetic components and inert materials

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#### ABSTRACT

The compatibility of *trans*-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin (TNAD) with some energetic components and inert materials of solid propellants was studied by using the pressure DSC method where, cyclotetramethylenetetranitroamine (HMX), cyclotrimethylenetrinitramine (RDX), 1,4-dinitropiperazine (DNP), 1.25/1-NC/NG mixture, lead 3-nitro-1,2,4-triazol-5-onate (NTO-Pb), aluminum powder (Al, particle size = 13.6  $\mu$ m) and *N*-nitrodihydroxyethylaminedinitrate (DINA) were used as energetic components and polyethylene glycol (PEG), polyoxytetramethylene-*co*-oxyethylene (PET), addition product of hexamethylene diisocyanate and water (N-100), 2-nitrodianiline (2-NDPA), 1,3-dimethyl-1,3-diphenyl urea (C<sub>2</sub>), carbon black (C.B.), aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), cupric 2,4-dihydroxy-benzoate ( $\beta$ -Cu), cupric adipate (AD-Cu) and lead phthalate ( $\varphi$ -Pb) were used as inert materials. It was concluded that the binary systems of TNAD with NTO-Pb, RDX, PET and Al powder are compatible, and systems of TNAD with DINA and HMX are slightly sensitive, and with 2-NDPA,  $\varphi$ -Pb,  $\beta$ -Cu, AD-Cu and Al<sub>2</sub>O<sub>3</sub> are sensitive, and with PEG, N-100, C<sub>2</sub> and C.B. are incompatible. The impact and friction sensitivity data of the TNAD and TNAD in combination with the other energetic materials under present study was also obtained, and there was no consequential affiliation between sensitivity and compatibility.

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#### 1. Introduction

Compatibility is an important safety aspect related to the production and storage of energetic materials. To test different combinations of materials, a simple test method with clear criteria is advisable. In the last ESTAC (Environmental Science and Technology Alliance Canada) conference, the use of microcalorimetry and the vacuum stability test for the compatibility testing of propellants were presented. In an investigation [1], it was shown that the results obtained in pressure DSC and TG/DTA measurements and those obtained using vacuum stability tests were very similar for the same combinations of materials. The trans-1,4,5,8tetranitro-1,4,5,8-tetraazadecalin (TNAD) was firstly synthesized by Willer [2] and it is an energetic compound containing two nitrous rings, which can be used as a main ingredient in cast explosives and propellants [3–8]. Reactivity or compatibility of TNAD with some energetic components and inert materials is one of the most stringent aspects of TNAD in practical application. Liu et al. [9,10] theoretically studied the detonation properties of energetic TNAD molecular derivatives and Prabhakaran et al. [11] studied the kinetics and mechanism of thermal decomposition of TNAD, while Skare [12] and Svatopluk [13.14] had done the investigation on the thermal behavior of nitroamines including TNAD. Moreover, Ling Qiu et al. [15,16] theoretically studied the structure of crystalline trans-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin by Ab Initio and Molecular Dynamics methods, seldom, however, has the compatibility of it with some energetic components and inert materials used in propellants been reported. The compatibility of 1,3,3trinitroazetidine (TNAZ) with some energetic components and inert materials of solid propellants was successfully studied by using the PDSC method [17]. Actually, some energetic materials such as DNP will melt and volatilize without decomposition at ambient pressure [18] and it is likely to be unreasonable to judge the compatibility under that condition [19]. So in this work, the compatibilities of trans-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin with some energetic components and inert materials under non-isothermal conditions investigated by means of pressure DSC [20-24] are reported. In addition, in order to make our conclusion more convictive, the investigation on impact and friction sensitivity properties of the trans-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin and its combination with the other energetic materials was also carried out according to the existing methods [25,26].

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#### 2. Experimental

#### 2.1. Materials

The *trans*-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin was synthesized by our work group according to the existing method [7], and the purity was more than 99.5%. The following is the molecular structure of it:



Cvclotetramethylenetetranitroamine (HMX. >99.5%). cvclotrimethylenetrinitramine (RDX. >99.6%). 1.4dinitropiperazine (DNP, >99.7%), 1.25/1 (mass rate)-NC/NG mixture (NC, ≥99.5%; NG, ≥99.2%), lead 3-nitro-1,2,4-triazol-5onate (NTO-Pb, >99.3%), aluminum powder (13.8 µm, >99.9%) and N-nitrodihydroxyethylaminedinitrate (DINA, >99.5%) used as energetic components and polyethylene glycol (PEG, M = 10,000), polyoxytetramethylene-co-oxyethylene (PET, M = 4000), addition product of hexamethylene diisocyanate and water (N-100), 2nitrodianiline (2-NDPA, >99.5%), 1,3-dimethyl-1,3-diphenyl urea  $(C_2, \geq 99.0\%)$ , carbon black (C.B., >99.8%), aluminum oxide (Al<sub>2</sub>O<sub>3</sub>,  $\geq$ 99.8%), cupric 2,4-dihydroxybenzoate ( $\beta$ -Cu,  $\geq$ 99.8%), cupric adipate (AD-Cu, >99.4%) and lead phthalate ( $\varphi$ -Pb, >99.2%) used as inert materials were industrially procured. Mixtures of TNAD and energetic components or inert materials were prepared according to mass rate 1:1.

All the sample systems involved in this investigation, which were purified by different solvents and then dried for  $48 h (60 \circ C)$ , were premixed symmetrically and defined as follows (the boldface number in the parentheses are their code designation):

TNAD (1); DNP (2); 1/1-TNAD/DNP (3); RDX (4); 1/1-TNAD/RDX (5); HMX (6); 1/1-TNAD/HMX (7); NTO-Pb (8); 1/1-TNAD/NTO-Pb (9); NC+NG (10); 1/1-TNAD/NC+NG (11); 1/1-TNAD/AI (12); 1/1-TNAD/DINA (13); PET (14); 1/1-TNAD/PET (15); PEG (16); 1/1-TNAD/PEG (17); 1/1-TNAD/2-NDPA (18); 1/1 TNAD/N-100 (19); 1/1-TNAD/Al\_2O\_3 (20); 1/1-TNAD/C.B. (21);  $\beta$ -Cu (22); 1/1 TNAD/ $\beta$ -Cu (23); 1/1-TNAD/AD-Cu (24); AD-Cu (25);  $\varphi$ -Pb (26); 1/1-TNAD/ $\varphi$ -Pb (27); 1/1-TNAD/C\_2 (28); AI (29); DINA (30).

#### 2.2. Equipment and experimentation

All the PDSC measurements were carried out by NETZSCH DSC 204 HP instrument. The conditions of PDSC were as follows: sample mass, TNAD (about 0.38–0.46 mg) and mixture system (0.52–0.78 mg); heating rate, 10 °C min<sup>-1</sup>; atmosphere, dynamic atmosphere of nitrogen with a flow rate of 50 ml min<sup>-1</sup> and pressure of 0.1 and 1 MPa. The TNAD, energetic component or inert material or mixture of 1/1-TNAD/energetic component and 1/1-TNAD/inert material was sealed in an aluminum cell, and all the samples were premixed symmetrically.

The impact sensitivity test was carried out on the drop-weight impact machine. The drop-weight impact machine used here is based on a design that the Explosives Research Laboratory at Bruceton, Pennsylvania (USA), developed during World War II [26]. The sample is placed on a fixed roughened anvil. A hammer of known contact area is positioned above the sample and the weight is raised to a predetermined height and dropped. The conditions of this test were as follows: temperature, 18 °C; free-falling height, 25 cm; hammer weight, 2.0 kg; sample mass, about 40 mg; experiment repeat, 25 times for probability calculations.

The friction sensitivity test was carried out with the method of ABL (Above Base Line) [27]. In the ABL friction test, the sample is placed on an anvil, and a known force is applied hydraulically through a stationary wheel. A pendulum is used to propel the sliding anvil at any of several standard velocities perpendicular to the force vector. Sample initiation is detected by visual means (spark or flame). The conditions of this test were as follows: temperature,  $18 \,^{\circ}$ C; pendulum angle,  $90^{\circ}$ ; sample mass, about 50 mg; experiment repeat, 25 times for probability calculations.

#### 3. Results and discussion

#### 3.1. Compatibility of TNAD with some energetic components

The DSC curves (Fig. 1) of pure TNAD obtained at the pressure of 0.1 and 1 MPa showed a sharp exothermic change, respectively, and the exothermic peak became very sharp when the pressure was changed to 4 MPa. The onset of the exotherms was noticed at 227.09 °C with the peak temperature of 238.6 °C at 0.1 MPa or 239.3 °C at 1 MPa. The DSC curves showed no endothermic change before the onset of exothermic change, it was indicated that this compound was decomposed in solid state. Typical DSC or PDSC curves of systems **1–13** are shown in Figs. 2 and 3, and their maximum exothermic peak temperatures are shown in Table 1, and the evaluated standards of compatibility for explosive and contacted materials [25] are listed in Table 2. In fact, Beach and Canfield gave the test method of compatibility of explosives with polymers in 1971 [26]. From Fig. 1 and Table 1, the following observations can be obtained.

The DSC curve of the TNAD–DNP (1,4-dinitropiperazine) mixture shows no endothermic change and one sharp exothermic change. The endothermic change of 44.82 °C without change in composition is the eutectic point for the system TNAD/DINA at 1 MPa. When TNAD and *N*-nitrodihydroxyethylaminedinitrate (DINA) were mixed, the binary eutectic solid system came into being, where melting point is little higher than that of pure DINA. The melting point of pure DINA was 50.87 °C [17]. Because of the large quantity of heat that TNAD emitted, it was too late to melt for TNAD before decomposition, and there was no endothermic peak before exothermic change.



**Fig. 1.** The DSC curves of TNAD at pressure of 0.1, 1 and 4MPa (heating rate: 10 K/min; sample mass: 0.38–0.46 mg).



Fig. 2. The DSC curves of TNAD with some energetic components at 0.1 or 1 MPa (heating rate: 10 K/min; sample mass: 0.52–0.78 mg).



Fig. 3. The PDSC curves of TNAD with some energetic components at 1 MPa (heating rate: 10 K/min; sample mass: 0.52-0.78 mg).

#### Table 1

The evaluations of compatibilities for DSC and PDSC peak temperature (binary system of 50/50-energetic components/TNAD)

System Mixture system Single system		Pressure (MPa)	Peak tempera	Peak temperature			
			<i>T</i> <sub>p2</sub> (°C)	<i>T</i> <sub>p1</sub> (°C)	$\Delta T_{\rm p}$ (°C)	Rating	
1/1-TNAD/DNP ( <b>3</b> )	TNAD	1 0.1	211.2 210.6	239.3 238.9	28.1 28.3	D D	
1/1-TNAD/RDX ( <b>5</b> )	TNAD	1 0.1	242.2 241.9	239.3 238.9	-2.9 -3.0	A A	
1/1-TNAD/HMX ( <b>7</b> )	TNAD	1 0.1	227.3 226.9	239.3 238.9	11.9 12.0	C C	
1/1-TNAD/NTO-Pb ( <b>9</b> )	TNAD	1 0.1	235.5 234.8	239.3 238.9	3.8 4.1	B B	
1/1-TNAD/NC + NG ( <b>11</b> ) 1/1-TNAD/Al ( <b>12</b> ) 1/1-TNAD/DINA ( <b>13</b> )	NC + NG TNAD DINA	1 1 1	230.3 231.3 220.9	207.0 239.3 210.38(ref. [17])	-23.3 8.0 -10.52	A C A	

Mixture system, 1/1-TNAD/energetic component binary system; single system, system of the pure energetic component, where exothermic peak temperature is smaller of the two pure components;  $T_{p1}$ , the maximum exothermic peak temperature of single system;  $T_{p2}$ , the maximum exothermic peak temperature of mixture system;  $\Delta T_p = T_{p1} - T_{p2}$ .

-	2	2
5	3	2

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Evaluated standards of compatibility for explosive and contacted materials

Criteria ( $\Delta T_p$ (°C))	Rating <sup>a</sup>	
Less than or equal to 2	А	Compatible or good compatibility
3–5	В	Slightly sensitized or moderate compatibility
6–15	С	Sensitized or poor compatibility
15-above	D	Hazardous or bad compatibility

<sup>a</sup> A: safe for use in any explosive design; B: safe for use in testing, when the device will be used in a very short period of time, not to be used as a binder material, or when long-term storage is desired; C: not recommended for use with explosive items; D: hazardous, do not use under any conditions.

The DSC curve of HMX consists of two endothermic peaks and one exothermic peak. The first peak temperature at 247.1 °C is due to the crystal transformation from  $\alpha$  to  $\delta$ , more detail please see ref. [28]. The following endothermic peak at 280.5 °C is the phase change from solid to liquid. The exothermic peak at 282.7 °C is caused by the rapid decomposition reaction. The exothermic peak temperature of the TNAD–HMX mixture is higher 12.2 °C than that of pure TNAD and lower 55.4 °C than that of pure HMX, which suggests that the presence of HMX stabilized TNAD while TNAD made HMX more sensitive.

The endothermic peaks of RDX and DINA are caused by the phase change from solid to liquid. The endothermic peaks of binary systems **3**, **5**, **13** are due to the melting process of DNP, RDX and DINA.

The maximum exothermic peak temperature, different between TNAD and TNAD–RDX mixture ( $\Delta T_p$ ), is –2.9 °C. The value of  $\Delta T_p$  is 3.8 °C between TNAD and TNAD–NTO–Pb mixture. According to the standards of compatibility evaluated in Table 2, we think that the binary systems TNAD–RDX and TNAD–NTO–Pb have moderate compatibility because  $\Delta T_p$  is less than 5 °C.

The values  $\Delta T_{\rm p}$  of between TNAD and TNAD–HMX mixture and TNAD and TNAD–Al mixture are 11.9 and 8.0, respectively. The value of  $\Delta T_{\rm p}$  is –10.52 between DINA and TNAD–DINA. This phenomenon indicates that the chemical reaction between components of the mixture is ready to take place and the mixture has poor compatibility. The value of  $\Delta T_p$  between TNAD and TNAD–DNP mixture is 28.1, which is consistent with the destabilization of DNP by the presence of TNAD. This means an increase in reactive ability and a decrease in the thermal stability of the mixture. The compatibility of DNP with TNAD is incompatible. Because of the large quantity of heat that TNAD emitted, there was no endothermic peak before exothermic change for TNAD–DNP mixture, and it was too late for DNP to melt before decomposition.

The compatibility of binary systems TNAD/energetic component decrease in the order: **5** > **9** > **12** > **13** > **6** > **11** > **3**. The relative thermal stability of binary systems TNAD/energetic component decreases in the order: **5** > **9** > **12** > **11** > **6** > **13** > **3**.

#### 3.2. The compatibility of TNAD with some inert materials

Typical DSC curves of systems **1**, **14–28** are shown in Figs. 4 and 5. Their maximum exothermic peak temperatures are shown in Table 3. From Figs. 4 and 5 and Table 3, the following observations can be made.

The DSC curves of TNAD–PEG and TNAD–C<sub>2</sub> mixtures show one endothermic peak each. The endothermic peaks temperatures 61.5 and 121.1 °C are the eutectic points for the systems TNAD/PEG and TNAD/C<sub>2</sub>, respectively. The values of  $\Delta T_p$  between TNAD with TNAD–( $\beta$ -Cu), TNAD–(2-NDPA), TNAD–(AD-Cu), TNAD– $\varphi$ -Pb and TNAD–Al<sub>2</sub>O<sub>3</sub> are 11.4, 9.0, 13.6, 7.6 and 9.1 °C, respectively, show-



Fig. 4. The DSC curves of TNAD with some inert materials at 0.1 or 1 MPa (heating rate: 10 K/min; sample mass: 0.52-0.78 mg).



Fig. 5. The PDSC curves of TNAD with some inert materials at 1 MPa (heating rate: 10 K/min; sample mass: 0.52-0.78 mg).

ing that the increase of the rate of the decomposition reaction or reactivity and the poor compatibility of the mixture. The values of  $\Delta T_p$  between TNAD or C<sub>2</sub> and TNAD–PEG, TNAD–(N-100), TNAD–C<sub>2</sub> and TNAD–C.B. mixtures are greater than 15 °C, and it was indicated that these binary systems are extremely incompatible. To a certain extent, most of the inert materials examined herein can promote the decomposition of TNAD. However, PET as an exception has moderate compatibility with TNAD and the  $\Delta T_p$  is 2.78 °C. The compatibility of binary systems TNAD/inert material decrease in the order: **15** > **27** > **18** > **20** > **23** > **24** > **21** > **28** > **17** > **19**.

Lastly, one point we must emphasize is that when the system evaluated by PDSC is incompatible, we cannot affirm that it is indeed incompatible. Contrarily, when the system evaluated by PDSC is compatible, we can affirm that it is compatible at ambient pressure. Because the sample was tested under the condition of high temperature and high pressure, when it is compatible under that condition, it will be more compatible under lower temperature and pressure. In order to make our point, the compatibility of some selected systems of **3**, **5**, **7**, **9** and **20** involved herein were evaluated

again at the ambient pressure (see Figs. 2 and 4). From the results (Tables 1 and 3) of this experiment, it was indicated that the evaluation of the compatibility by the pressure DSC method was basically creditable. Whereas, when the system evaluated by PDSC is incompatible, we may use other evaluating methods such as DTA/TG or vacuum stability test (VST) to confirm it.

## 3.3. The impact and friction sensitivity of TNAD with some energetic materials

There are many uses for knowing the sensitivity of a material to impact and friction. These can purpose evaluation of material sensitivity over time. The sensitivity of the systems of TNAD with some other energetic materials can be confirmed this way. Comparison of sensitivity of two materials can determine if one material could replace another in a particular process.

According to Willer [2] and Helmut et al. [5], TNAD is insensitive, and it was approved by our test. From the results of the impact and friction sensitivity of TNAD with some energetic materials (Table 4),

#### Table 3

The evaluations of compatibilities for DSC and PDSC peak temperature (Binary system of 50/50-inert materials/TNAD)

System		Pressure (MPa)	Peak temperature			
Mixture system	Single system		<i>T</i> <sub>p2</sub> (°C)	<i>T</i> <sub>p1</sub> (°C)	$\Delta T_{\rm p}$ (°C)	Rating
1/1-TNAD/PET (15)	PET		174.9	176.02	2.78	В
1/1-TNAD/PEG (17)	TNAD		207.7	239.3	31.6	D
1/1-TNAD/2-NDPA (18)	TNAD	I	230.3	239.3	9.0	С
1/1-TNAD/N-100 ( <b>19</b> )	TNAD		200.9	239.3	38.4	D
1/1-TNAD/Al <sub>2</sub> O <sub>3</sub> ( <b>20</b> )	TNAD	1	230.2	239.3	9.1	С
		0.1	229.7	238.9	9.2	С
1/1-TNAD/C.B. ( <b>21</b> )	TNAD		220.7	239.3	18.6	D
1/1-TNAD/β-Cu ( <b>23</b> )	TNAD		227.9	239.3	11.4	С
1/1-TNAD/AD-Cu (24)	TNAD	1	225.7	239.3	13.6	С
1/1-TNAD/φ-Pb ( <b>27</b> )	TNAD		231.7	239.3	7.6	С
1/1-TNAD/C <sub>2</sub> (28)	C <sub>2</sub>		207.3	235.7(ref. [17])	28.4	D

The meanings of marks in Table 3 are same as in Table 1.

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Single system	Impact (%)	Friction (%)	Mixture system	Impact (%)	Friction (%)
TNAD (1)	0	8	1/1-TNAD/DNP ( <b>3</b> )	0	0
DNP ( <b>2</b> )	0	0	1/1-TNAD/HMX (6)	96	92
RDX ( <b>4</b> )	0	0	1/1-TNAD/RDX (5)	0	4
HMX (6)	100	100	1/1-TNAD/NTO-Pb (9)	0	0
NTO-Pb (8)	0	0	1/1-TNAD/NC + NG (11)	76	80
NC + NG (10)	84	92	1/1-TNAD/Al (12)	0	4
DINA ( <b>30</b> )	0	0	1/1-TNAD/DINA (13)	0	0

the following conclusions can be made. The TNAD in combination with some energetic materials are insensitive except for **6** and **11**. The HMX and NC+NG in systems **6** and **11** are very sensible, and it makes the system more sensitive. Contrasted this results with compatibility experiments data, there was no consequential affiliation between sensitivity and compatibility. System **3** is incompatible while its impact and friction sensitivity is very low. Binary system **13** is compatible while its impact and friction sensitivity is very low. Binary system **13** is whereas, if the energetic materials can react with each other when they are mixed, they must be incompatible and very sensitive. Binary systems **5**, **9** and **11** are of great compatibility and low sensitivity.

#### 4. Conclusions

- (1) The systems of TNAD–RDX, TNAD–(NTO–Pb), TNAD–PET and TNAD–(Al powder) binary mixtures have moderate compatibility, and the TNAD–DINA and TNAD–HMX binary mixtures have poor compatibility according to the evaluated standard of compatibility.
- (2) The compatibilities of the TNAD– $(\beta$ -Cu), TNAD–DNP, TNAD–(AD-Cu), TNAD– $\phi$ -Pb and TNAD– $Al_2O_3$  binary mixtures are also poor, but the compatibilities of the TNAD–(N-100), TNAD–(NC+NG), TNAD– $C_2$  and TNAD–C.B. mixtures are incompatible.
- (3) There was no consequential affiliation between sensitivity and compatibility. Whereas, if the energetic materials can react with each other when they are mixed, they must be incompatible and very sensitive. Binary systems 5, 9 and 11 are of great compatibility and low sensitivity.

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