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Ternary phase diagrams of DNTF and TNAZ and their eutectics

Ying Hui Shao · Xiao Ning Ren · Zi Ru Liu · Xiang Zhang

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Abstract The eutectic ternary phase diagrams of some typical volatilizable energetic materials have been investigated by high pressure differential scanning calorimeter (PDSC). The ternary *H–X* phase diagrams for TNT/TNAZ/DNTF (TTD) and TNAZ/DNTF/RDX (TDR) systems were constructed by the correlation of the apparent fusion heat with the composition (*H–X* method). And, the ternary *T–X* phase diagrams (the temperature dependence on composition) for the two ternary systems were constructed by calculating from the data of the five *T–X* binary phase diagrams. The eutectic compositions (mol%) of TTD and TDR ternary systems were obtained to be 52.3/27.3/20.4 (*H–X* method), 53.2/25.8/21.0 (*T–X* method) and 54.9/39.6/5.5 (*H–X* method), 55.1/42.2/2.7 (*T–X* method), respectively. The eutectic temperatures of the ternary systems were obtained by PDSC determination and *T–X* method calculation to be 76.5 and 76.7 °C, 47.5 and 50.2 °C, respectively. It is shown that the results obtained by two methods are in agreement and the error in measuring or calculating eutectic compositions and temperatures for the two ternary systems are within allowable ranges of ±3 mol% and ±3 °C, respectively. Moreover, by means of constructing two ternary *H–X* phase diagrams with different fixed composition of a component and comparing the apparent fusion heat of eutectics with calculated one, the results obtained from *H–X* method for TTD system were proved. The results showed that the gasification or volatilization of easy volatile materials could be efficiently restrained by high pressure atmosphere, and the perfectly and ideally *H–X* ternary phase diagrams can be constructed. In comparison with *T–X* method, *H–X* method has as a virtue of being quick and simple, especially on constructing ternary phase diagram.

Keywords Physical chemistry · PDSC · Volatility materials · Ternary system · Phase diagram

Y. H. Shao · X. N. Ren (✉) · Z. R. Liu · X. Zhang
Xi'an Modern Chemistry Research Institute, Xi'an 710065, China
e-mail: xueyexy@163.com

Introduction

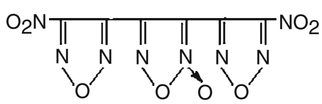
3,4-Dinitrofurazanfuroxan (DNTF) is a kind of new high energy density material [1, 2]. Its energy is more excelled than that of cyclotetramethylenetetranitramine (HMX) and close to that of 2,4,6,8,10,12-Hexanitrohexaazaisowurtzitane (CL-20). It has some traits such as low melting point, high density, good stability, seemly sensitivity, simple synthesize technics, and so on [3]. The pilot study indicated that there is a widest prospect, a new kind of excellent integration performance high energy explosive, and it could form eutectic liquid carrier. The energy and liquefy temperature for the eutectic mixtures could be adjusted yet and the different performance required casting explosive ingredients also could be confected. The ternary eutectic explosive systems have a more castability and a more adjustable energetic performance, therefore, hold many research worker interests [4–7]. The ternary explosive systems that formed by DNTF with TNT and other energy materials have been already studied, however, the same as TNAZ, DNTF, and TNT all take on serious volatility [8, 9], could not be make theirs contents accurate in systems at ambient pressure, so the melting and liquefying process of the two ternary eutectic systems TNT/TNAZ/DNTF and TNAZ/DNTF/RDX have been determined, as similar to the binary systems with volatilizable materials in previous study

[10], under pressure ambience by High Pressure Differential Scanning Calorimetry (PDSC) in this study. The ternary phase diagrams for the apparent fusion heat with the composition (ternary *H–X* phase diagrams) have been constructed. The ternary *T–X* phase diagrams (the temperature dependence on composition) for the two ternary systems have been constructed by calculating from the data of the five *T–X* binary phase diagrams [11–13].

Experimental

Materials

The four energetic materials used in this study are 3,4-dinitrofurazanfuroxan (DNTF) 1,3,3-trinitroazetidine (TNAZ), 2,4,6-trinitrotoluene (TNT), and cyclotrimethylenetrinitramine (RDX) and the structural formula DNTF and TNAZ are as following:



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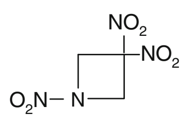

These four samples were all prepared by *Xi'an Modern Chemistry Research Institute* and their purity quotients were determined by liquid chromatogram to be higher than 99.9%.

In *H–X* phase diagrams constructed by the correlation of the apparent fusion heat with the composition (*H–X* method), the composition of a component must be fixed and the apparent fusion heat of ternary or binary eutectics for ternary systems with various composition ratios of other two components. Therefore, following samples were prepared as:

In TNAZ/DNTF/RDX systems (TDR system), the mass percent of TNAZ (X_{TNAZ}) has been fixed to be 35% and the mass ratios of DNTF and RDX are to be 60:1, 60:2, 60:3, 60:4, 60:5, 55:10, 50:15, 48:17, 45:20, 42:23, 40:25, 35:30, 30:35, 20:45 and 10:55, respectively.

In (TNT/TNAZ/DNTF)-1 system (TTD-1 system), the mass percent of TNT (X_{TNT}) has been fixed to be 40% and the mass ratios of TNAZ and DNTF are to be 55:5, 50:10, 45:15, 40:20, 35:25, 30:30, 25:35, 20:40, 15:45, 10:50, and 5:55, respectively.

In (TNT/TNAZ/DNTF)-2 system (TTD-2 system), the mass percent of TNT (X_{TNT}) has been fixed to be 50% and the mass ratios of TNAZ and DNTF are to be 44:6, 42:8, 35:15, 30:20, 22:28, 20:30, 10:40, and 5:45, respectively.

The above samples have been mixed mechanically and heated until the temperature that higher liquefying temperatures but lower decomposition temperatures, after natural cooling these samples have been measured by PDSC.

Apparatus and experiment conditions

TA Model DSC 910S differential scanning calorimeter has been applied to measure the apparent fusion heats of the three systems with various mixing ratios. All DSC tests were carried out in static atmosphere of 1 MPa, using sample size of 8.00 ± 0.1 mg and aluminum crucible. The heating rates for TTD and TDR systems are to be 5 and 10 K min^{-1}, respectively.

The methods used in this studies were reported previously in which the ternary *T–X* phase diagram is constructed by the data of the three binary *T–X* phase diagrams, viz. *T–X* method [6]. The computer software of the ternary *T–X* phase diagram were manufactured by *Xi'an Modern Chemistry Research Institute* and the calculation equations are taken from the literature [6, 7].

Results and discussion

PDSC curves of TDR and TTD ternary systems

As the melts of DNTF, TNAZ, and TNT are too easy to volatilize or gasify materials at ambient pressure, all DSC tests were carried out in static atmosphere of 1 MPa. PDSC curves of TDR systems (TNAZ = 35 mass%) and TTD systems (TNT = 40 mass%) are shown as in Figs. 1 and 2, respectively. The characteristic values of the melting and liquefying processes for TDR, TTD-1, and TTD-2 are shown as in Tables 1, 2, and 3. In Tables 1, 2, and 3 the symbols: ΔH_{eu}—eutectic fusion heat and T_e—eutectic temperature.

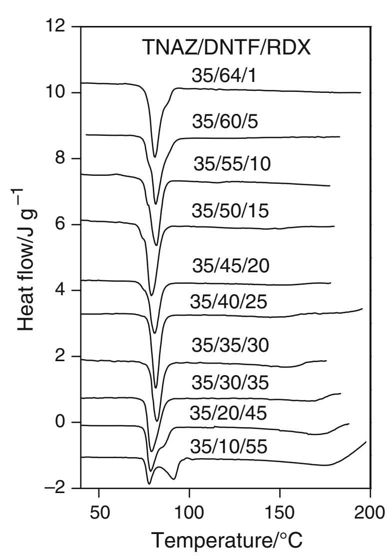

Fig. 1 PDSC curves of TNAZ/DNTF/RDX systems at 1 MPa and X_{TNAZ} = 35 mass%

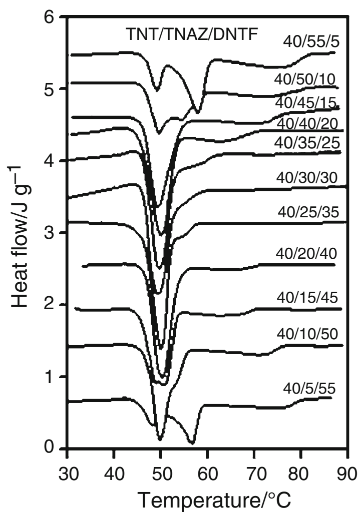

Fig. 2 PDSC curves of TNT/TNAZ/DNTF systems at 1 MPa and X_{TNT} = 40 mass%

Table 1 PDSC characteristics of TDR system at 1 MPa

$X_{TNAZ}/X_{DNTF}/X_{RDX}$		ΔH_{eu}/J g^{-1}	T_e/°C
mass%	mol%		
35/64/1	46.5/52.3/1.2	107.2[a]	76.5
35/63/2	46.4/51.3/2.3	58.1	77.0
35/63/2	46.4/51.3/2.3	104.0[a]	75.0
35/61/4	46.1/49.4/4.5	101.2[a]	76.8
35/60/5	45.9/48.4/5.7	99.50	74.6
35/55/10	45.2/43.7/11.1	102.9	78.2
35/50/15	44.4/39.1/16.5	103.1	77.2
35/48/17	44.2/37.3/18.5	101.0	77.4
35/45/20	43.8/34.6/21.6	101.1	76.8
35/42/23	43.4/32.0/24.6	99.14	76.2
35/40/25	43.1/30.3/26.6	100.1	76.1
35/35/30	42.4/26.1/31.5	87.17	78.0
35/30/35	41.8/22.1/36.1	70.66	75.7
35/20/45	40.6/14.3/45.1	51.66	75.5
35/10/55	39.5/6.9/53.6	24.43	75.1

[a] These values are the sums of fusion heats of the ternary and binary eutectics

It can be seen from Figs. 1 and 2 that there is an endothermic peak, on which the onset temperature (i.e., T_e) is independent on the mass ratios of the compositions, on all PDSC curves the three systems and there are the second or third endothermic peaks on some PDSC curves. It is indicated that the three ternary systems are the eutectic systems with the eutectic temperature average of 76.5 °C or 349.7 K (for TDR) and 47.5 °C or 320.7 K (for TTD). The second endothermic peak is a binary eutectic peak for other two remaining components and the third one is a liquidus process of the third remaining component.

Table 2 PDSC characteristics of TTD-1system at 1 MPa

$X_{TNT}/X_{TNAZ}/X_{DNTF}$x		ΔH_{eu}/J g^{-1}	T_e/°C
mol%	mol%		
40/55/5	36.8/59.9/3.3	17.71	46.9
40/50/10	37.6/55.6/6.8	37.42	48.2
40/45/15	38.4/51.1/10.5	53.80	46.5
40/40/20	39.3/46.4/14.3	72.51	46.7
40/35/25	40.1/41.6/18.3	88.70	46.5
40/30/30	41.1/36.5/22.4	81.30	46.9
40/25/35	42.1/31.1/26.8	84.44	46.5
40/20/40	43.1/25.5/31.4	86.62	47.8
40/15/45	44.2/19.6/36.2	71.51	46.0
40/10/50	45.3/13.4/41.3	47.01	48.0
40/5/55	46.5/6.9/46.6	26.72	47.6

Table 3 PDSC characteristics of TTD-2 system at 1 MPa

$X_{TNT}/X_{TNAZ}/X_{DNTF}$		ΔH_{eu}/J g^{-1}	T_e/°C
mass%	mol%		
50/44/6	47.0/48.9/4.1	20.05	48.2
50/42/8	47.4/47.1/5.5	32.29	48.0
50/40/10	47.8/45.2/7.0	40.56	46.9
50/35/15	48.9/40.4/10.7	62.30	48.8
50/30/20	50.0/35.5/14.5	81.29	47.2
50/25/25	51.1/30.3/18.6	101.47	47.2
50/22/28	51.9/27.0/21.1	100.59	48.3
50/20/30	52.3/24.8/22.9	92.06	47.4
50/10/40	55.0/13.0/32.0	47.52	48.5
50/5/45	56.4/6.7/36.9	25.37	46.2

Ternary *H–X* phase diagrams for TDR and TTD systems

On the basis of the method described by the literatures [5, 7], using the data in Tables 1, 2, and 3, the ternary *H–X* phase diagrams for TDR and TTD systems were constructed by the correlation of the apparent fusion heat with the composition (*H–X* method), as shown in Figs. 3 and 4.

When the composition of RDX component is small in the TDR ternary systems, the ternary eutectic peak cannot be separated from the binary ones formed from the remainder of the TNAZ and DNTF. Therefore, in order to obtain the value of *X* axis for intersecting point *B* in Fig. 3, the sums of the fusion heats of the ternary and binary eutectics for TDR systems are also used to construct the ternary *H–X* phase diagram, as shown the *BC* line in Fig. 3. The following equations can be found by a least-squares regression from the data in Table 1, *r* is the regression correlation coefficient:

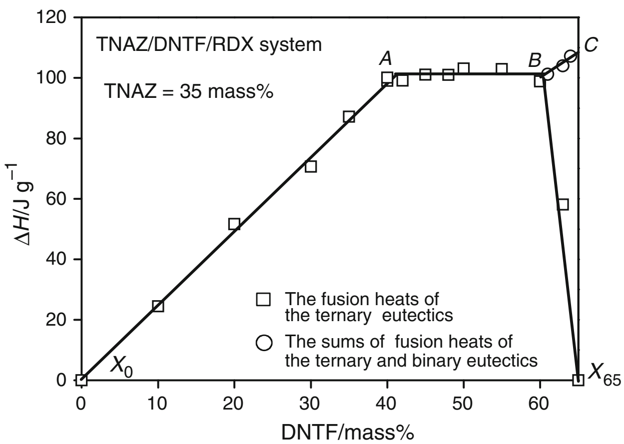

Fig. 3 Ternary phase diagram for TDR system

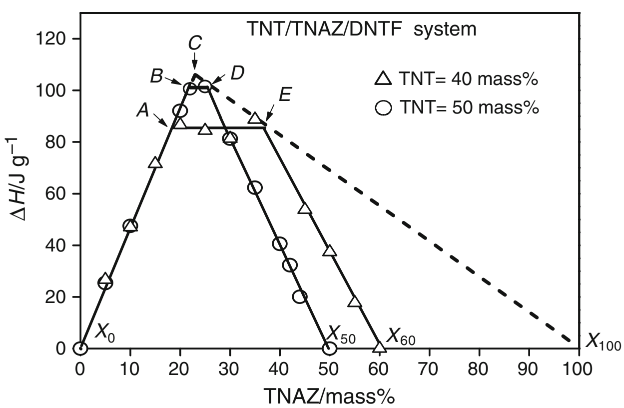

Fig. 4 Ternary phase diagram for TTD system at 1 MPa

For $X_{\mathrm{DNTF}} = 0$–40 mass% (X_0A line), we have

$$\Delta H_{\mathrm{eu}} = 2.464 \cdot X_{\mathrm{DNTF}} \quad (r = 0.9975) \tag{1}$$

For $X_{\mathrm{DNTF}} = 40$–60 mass% (AB line), we take an average value of ΔH_{eu} in the range of 40–60 mass%

$$\Delta H_{\mathrm{eu}} = 101.0 \tag{2}$$

For $X_{\mathrm{DNTF}} = 60$–65 mass%, we have

$$BC\text{line}: \Delta H_{\mathrm{eu}} = 1.820 \cdot X_{\mathrm{DNTF}} - 9.865 \quad (r = 0.9852) \tag{3}$$

where, ΔH_{eu} are the sums of the fusion heats of the ternary and binary eutecticsor

$$X_{65}B\text{line}: \Delta H_{\mathrm{eu}} = 1269 - 19.42 \cdot X_{\mathrm{DNTF}} \quad (r = 0.9777) \tag{4}$$

The values of A and B on X axis in Fig. 3 can be calculated by Eqs. 1, 2 and 3 or 4, i.e., the compositions of DNTF at A and B points are obtained to be $X'_{\mathrm{DNTF}} = 40.98$ and $X''_{\mathrm{DNTF}} = 60.91$ (from Eqs. 2 and 3). On the basis of the formulae described by the literatures [5, 7], for the eutectic compositions (mass%) of TNAZ/DNTF/RDX ternary systems, $X^0_{\mathrm{TNAZ}}/X^0_{\mathrm{DNTF}}/X^0_{\mathrm{RDX}}$, we have

$$X^0_{\mathrm{TNAZ}} = 35/(100 + X'_{\mathrm{DNTF}} - X''_{\mathrm{DNTF}}) \tag{5}$$

$$X^0_{\mathrm{TNAZ}} = X'_{\mathrm{D}}/(100 + X'_{\mathrm{DNTF}} - X''_{\mathrm{DNTF}}) \tag{6}$$

$$X^0_{\mathrm{RDX}} = (100 - 35 - X''_{\mathrm{DNTF}})(100 + X'_{\mathrm{DNTF}} - X''_{\mathrm{DNTF}}) \tag{7}$$

Substituting the above values of X'_{DNTF} and X''_{DNTF} into Eqs. 5–7, the eutectic compositions, $X^0_{\mathrm{TNAZ}}/X^0_{\mathrm{DNTF}}/X^0_{\mathrm{RDX}}$, would be found and their units have been converted to mol%, as shown in Table 4.

The two trapezoids (i.e., ternary H–X phase diagrams) in Fig. 4 have been formed from the two set of the PDSC data for $X_{\mathrm{TNT}} = 40$ and 50 mass%. It is seen that the left sideline of a trapezoid are superposed on that of another trapezoid, indicating that the slope for linear relation of ΔH_{eu} with X_{TNAZ} is independent upon the fixed values of X_{TNT} on small X_{TNAZ}. The right sidelines of two trapezoids are almost a parallel relationship, showing that the two lines have the approximate slopes.

In a way similar to that for TDR system, the following equations can be found by a least-squares regression from the data in Tables 2 and 3.

For X_0A and X_0B lines, we have

$$\Delta H_{\mathrm{eu}} = 4.708 \cdot X_{\mathrm{TNAZ}} \quad (r = 0.9975) \tag{8}$$

for A_0E line (TTD-1) and B_0D line (TTD-2)

$$\Delta H_{\mathrm{eu}} = 85.27 \tag{9}$$

and

$$\Delta H_{\mathrm{eu}} = 101.0 \tag{10}$$

for $X_{60}E$ line (TTD-1) and $X_{50}D$ line (TTD-2)

$$\Delta H_{\mathrm{eu}} = 215.2 - 3.582 \cdot X_{\mathrm{TNAZ}} \quad (r = 0.9997) \tag{11}$$

and

Table 4 The eutectic compositions and temperatures for TDR and TTD systems

Systems	Eutectic compositions/mol %		Eutectic temperature/°C	
	HX-method	*TX*-method	Found by DSC	Calc. by *TX*-method
TNAZ/DNTF/RDX	54.9/39.6/5.5	55.1/42.2/2.7	76.5	76.7
TNT/TNAZ/DNTF	52.3/27.3/20.4	53.2/25.8/21.0	49.3	50.2

$$\Delta H_{\mathrm{eu}} = 205.2 - 4.123 \cdot X_{\mathrm{TNAZ}} \quad (r = 0.9984) \tag{12}$$

The values of *A*, *B*, *D*, and *E* on *X* axis in Fig. 4 can be calculated by Eqs. 8–12 to obtain X'_{DNTF} and X''_{DNTF}, the eutectic compositions of TTD ternary systems, $X^0_{\mathrm{TNT}}/X^0_{\mathrm{TNAZ}}/X^0_{\mathrm{DNTF}}$, can be then found by the similar equations to above Eqs. 5–7 to be 51.1/27.4/21.5 mol% for TTD-1 and 53.5/27.2/19.3 mol% for TTD-2. It is shown that the results obtained from two TTD systems are very approximate. The average values of eutectic compositions for TTD-1 and TTD-2 systems are shown in Table 4. In reality, the data used to calculate the ternary eutectic compositions may be enough obtained from a ternary *H*–*X* phase diagram with a fixed composition of component and here the *H*–*X* phase diagrams of two TTD systems have been constructed so as to prove the results with each other.

It is noticeable that *D* and *E* points with X_{100} point ($X_{100} = X_{\mathrm{TNAZ}} = 100$ mass%) in Fig. 4 can be linked up through a straight line and a triangle, i.e., the principle graph of *H*–*X* method in the literatures [5, 7], can be formed from the straight line, the extended line of EX_{100} or EX_{100} line and *X* axis. The values of *X* and *Y* axes for the top point *C* of the triangle must be the TNAZ composition X^0_{TNAZ} (22.2 mass%) in the ternary eutectic and the apparent fusion heat of the ternary eutectic ΔH^0_{eu} (104.7 J g^{-1}), respectively. The apparent fusion heat of the eutectic prepared according to the result from determination of the above *H*–*X* phase diagrams and one calculated from the apparent fusion heat ($\Delta H^0_{\mathrm{TNT}} = 92.6$ J g^{-1}, $\Delta H^0_{\mathrm{TNAZ}} = 135.0$ J g^{-1} and $\Delta H^0_{\mathrm{DNTF}} = 96.8$ J g^{-1}) of the three pure materials according to eutectic compositions from determination of *H*–*X* method are to be 102.8 and 103.3 J g^{-1}, respectively, showing that these results are in good agreement with the value of *Y* axis at *C* point in Fig. 4, ΔH^0_{eu} (104.7 J g^{-1}). These results show that the data obtained from *H*–*X* method are reliable for ternary systems and the volatilization or gasification of molten component in mixed system could be efficiently restrained by high pressure atmosphere, which provides for the exact measures of the apparent fusion heats of the eutectics.

Ternary *T*–*X* phase diagrams for TDR and TTD systems

In order to proof the results obtained from *H*–*X* method, the ternary *T*–*X* phase diagram for TDR and TTD systems was constructed from the three binary *T*–*X* phase diagrams by using the *T*–*X* method proposed in the literatures [6, 7]. The data used to construct the ternary *T*–*X* phase diagrams of TDR and TTD systems have been calculated from the thermodynamic parameters of the five binary systems, including TNT/TNAZ, TNT/DNTF, TNAZ/DNTF, TNAZ/RDX, and DNTF/RDX, in previous study [11–13]. These parameters are shown in Table 5. In Table 5 the melting points T_i^0 were determined by using DSC or PDSC and the ΔH_{ij} (i, $j = 1, 2, 3$) were obtained from following CSL equation by constructing binary *T*–*X* phase diagrams [6, 7, 14]:

$$\ln X_i = \frac{\Delta H_{ij}}{R}\left(\frac{1}{T_i^0} - \frac{1}{T_i}\right) \tag{13}$$

where X_i is the mole fraction of component *i*, T_i^0 and T_{i} are the melting point of pure component *i* and its liquidus temperature in binary system, respectively, ΔH_{ij} is the apparent fusion heat of component *i* in the presence of another component *j*, and *R* is the gas constant.

The XMCI-ZX01 software used to calculate the ternary *T*–*X* phase diagrams was manufactured by this institute. The ternary *T*–*X* phase diagrams of TDR and TTD systems were constructed, as shown in Figs. 5 and 6. The eutectic compositions and temperatures for TDR and TTD ternary systems are also shown in Fig. 4.

The results shown in Fig. 4 show that the eutectic compositions and temperatures for TDR and TTD ternary systems obtained from *H*–*X* method and DSC are in good agreement with those of the *T*–*X* method. The error in measuring or calculating eutectic compositions and temperatures for the two ternary systems are within allowable ranges of ±3 mol% and ±3 °C, respectively [5]. However, as previous described in the literature [5, 6], *H*–*X* method has as a virtue of being quick and simple by comparison with *T-X* method, especially on constructing ternary phase diagram.

Table 5 The thermodynamic parameters of binary systems for some energetic materials

Materials	Melting points T_i^0/°C	Mixed systems	ΔH_{ij}/J mol^{-1}	ΔH_{ji}/J mol^{-1}	References
TNT	80.0	TNT/TNAZ	22.10	26.86	[11]
TNAZ	99.4	TNT/DNTF	17.68	24.41	[13]
DNTF	108.0	TNAZ/DNTF	29.41	34.37	[11]
RDX	204.3	TNAZ/RDX	16.60	36.66	[11]
–	–	DNTF/RDX	6.90	51.98	[12]

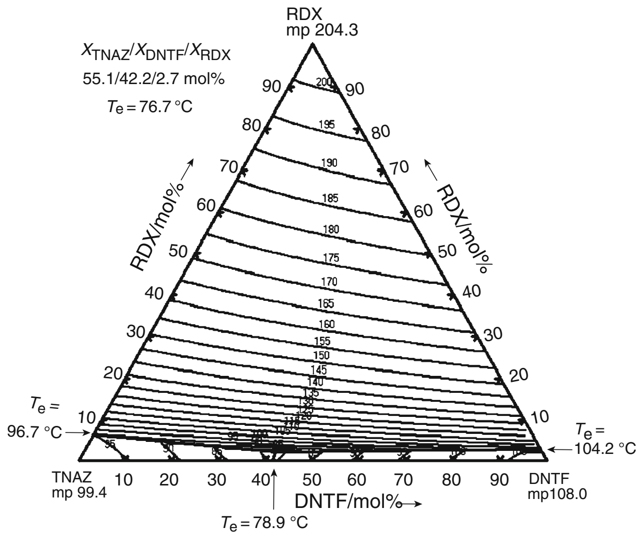

Fig. 5 *T–X* phase diagram of TDR system

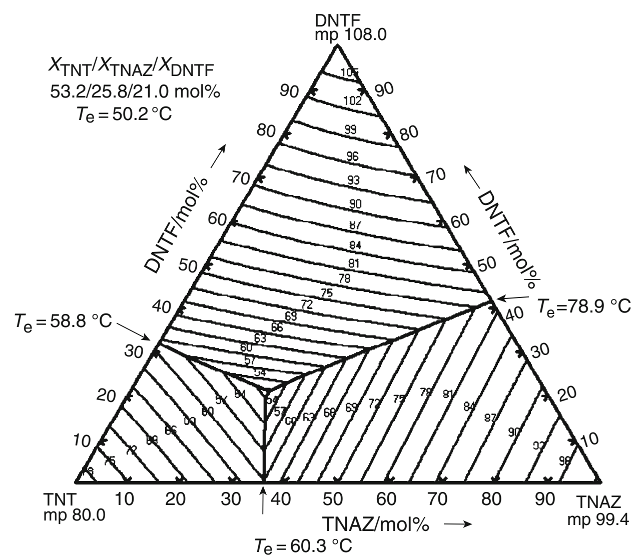

Fig. 6 *T–X* phase diagram of TTD system

Conclusions

The ternary *H–X* phase diagrams of TNAZ/DNTF/RDX (TDR) and TNT/TNAZ/DNTF (TTD) systems were quickly constructed from the PDSC data by using *H–X* method, the eutectic compositions (mol%) for the two ternary systems were obtained to be 54.9/39.6/5.5 and 52.3/27.3/20.4, respectively, and their eutectic temperatures were determined to be 76.5 and 47.5 °C. Simultaneously, the results of TTD system were confirmed by constructing two *H–X* phase diagrams with different fixed compositions of a component and by comparison of measuring eutectic fusion heat with calculating value. The results show that the data obtained from *H–X* method are reliable for ternary systems and the volatilization or gasification of molten component in mixed system could be efficiently restrained by high pressure atmosphere, which provides for the exact measures of the apparent fusion heats of the eutectics.

The two ternary *T–X* phase diagrams of TDR and TTD systems were constructed from the thermodynamic parameters of the five binary *T–X* phase diagrams which were published in previous articles by *T–X* method. The eutectic compositions (mol%) and temperatures for TDR and TTD systems were calculated to be 55.1/42.2/2.7 and 53.2/25.8/21.0, 76.7 and 50.2 °C, respectively. The results obtained from *T–X* method are in good agreement with those of the *H–X* method. The error in measuring or calculating eutectic compositions and temperatures for the two ternary systems are within allowable ranges of ±3 mol% and ±3 °C, respectively. However, *H–X* method has as a virtue of being quick and simple by comparison with *T–X* method, especially on constructing ternary phase diagram.

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