

# The Synthesis of Energetic Compound on 4,4'-((2,4,6-trinitro-1,3-phenylene)bis(oxy))bis(1,3-dinitrobenzene)(ZXC-5): Thermally Stable Explosive with Outstanding Properties

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**Abstract:** The novel, thermally stable explosive 4,4'-((2,4,6-trinitro-1,3-phenylene)bis(oxy))bis(1,3-dinitrobenzene) (Be referred to as ZXC-5 in our laboratory) has been reported. ZXC-5 can be synthesized by a simple synthetic method (The total synthesis of ZXC-5 requires only two steps and the total yield of ZXC-5 is more than 89%) and shows the superior detonation performances (detonation pressure, detonation velocity, sensitivity toward mechanical stimuli, and temperature of decomposition). The structure of ZXC-5

was characterized by multinuclear (<sup>1</sup>H, <sup>13</sup>C) NMR and mass spectrometry. The structure in the crystalline state was confirmed by low-temperature single-crystal X-ray diffraction. From the calculated standard molar enthalpy of formation and the measured densities, the detonation properties were predicted by using the EXPLO5 V6.01 thermochemical computer code. The sensitivity of ZXC-5 towards impact, electrostatic discharge, and friction were also measured.

**Keywords:** energetic materials · thermochemistry · facile synthetic route · structure elucidation · aromaticity

## 1 Introduction


Thermally stable (heat-resistant) explosives are a kind of explosives with high thermal stability, high melting point and low vapor pressure. These explosives must possess targeted performance properties (detonation velocity, specific energy), but, at the same time, show safety in handling and practicability for deep sea oil and gas exploitation (e.g., thermally stable perforators) as well as space exploration (e.g., in achieving stage separation in space rockets). Synthesis of heat-resistant explosive based on a benzene backbone has had a long tradition since the discovery of trinitrotoluene (TNT) in 1863 [1–2]. Because of its important property as a melt-castable explosive it is still in use nowadays, even though it is highly toxic for the environment [3–4]. As heat-resistant explosives, the following explosives: 1,3,5-triamino-2,4,6-trinitrobenzene (TATB), 2,2',4,4',6,6'-hexanitrostilbene (HNS), 2,6-bis(picrylamino)-3,5-dinitro pyridine (PYX) and tetranitrodibenzo-1,3a,4,6a-tetrazapentalene (TACOT) should be received special attention.

As a heat-resistant explosive, TATB shows a high density ( $\rho_{\text{TMD}} = 1.93 \text{ g cm}^{-3}$ ) [3], has a high thermal stability (350 °C) [4] and a high Chapman–Jouguet (C–J) detonation velocity and detonation pressure ( $D_{\text{C–J}} = 7.66 \text{ km s}^{-1}$ ,  $P_{\text{C–J}} = 28.7 \text{ GPa}$  at a density of  $1.854 \text{ g cm}^{-3}$ ; cylinder test) [5]. TATB is insensitive (Impact sensitivity: IS = 50 J; friction sensitivity: FS = 353 N and electrostatic discharge ignition: ESD = 2.56 J) [6]. TATB is difficult to initiate, because its initiation sensitivity is relatively high [7]. HNS has only a moderately high theoretical maximum

density ( $\rho_{\text{TMD}} = 1.745 \text{ g cm}^{-3}$ ), has high thermal decomposition temperature ( $T_{\text{dec}} = 318 \text{ °C}$ ), and shows low values for the detonation parameters ( $D_{\text{C–J}} = 7000 \text{ m s}^{-1}$  at  $\rho = 1.70 \text{ g cm}^{-3}$ ;  $P_{\text{C–J}} = 20.0 \text{ GPa}$  at  $\rho = 1.60 \text{ g cm}^{-3}$ ). PYX is characterized by an excellent thermal stability ( $T_{\text{dec}} = 373 \text{ °C}$ ), a theoretical maximum density that is slightly higher than that of HNS ( $\rho = 1.757 \text{ g cm}^{-3}$ ), and good detonation parameters ( $D_{\text{C–J}} = 7500 \text{ m s}^{-1}$ ,  $P_{\text{C–J}} = 25.19 \text{ GPa}$  at  $\rho = 1.75 \text{ g cm}^{-3}$ ), but the post processing of the product is complex. Also PYX shows only moderate sensitivity towards IS, FS, and ESD (IS = 8 J, FS = 360 N, and ESD = 1.00 J, respectively) [7]. TACOT ( $T_{\text{dec}} = 401 \text{ °C}$ ) shows higher thermal stability than PYX ( $T_{\text{dec}} = 373 \text{ °C}$ ), shows a high density ( $\rho_{\text{TMD}} = 1.750 \text{ g cm}^{-3}$ ), but it only shows low values for the detonation parameters ( $D_{\text{C–J}} = 7200 \text{ m s}^{-1}$ ;  $P_{\text{C–J}} = 24.5 \text{ GPa}$ ). The density ( $\rho_{\text{TMD}} = 1.837 \text{ g cm}^{-3}$ ), enthalpy of formation ( $\Delta_f H = 197.6 \text{ kJ mol}^{-1}$ ), detonation velocity ( $D_{\text{C–J}} = 8030 \text{ m s}^{-1}$ ) and detonation pressure values ( $P_{\text{C–J}} = 27.3 \text{ GPa}$ ) of 5,5'-bis(2,4,6-trinitrophenyl)-2,2'-bi(1,3,4-oxadiazole) (TKX-55)

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are highest than those of the commonly used heat resistant explosives (PYX, HNS, TACOT), but its synthetic route is still very long (about 5 steps from toluene) and its sensitivity value is also very low (impact sensitivity: IS = 5J)<sup>[8]</sup>

Because PYX, HNS, TACOT and TKX-55 as heat-resistant explosives possess the unique combination of very high thermal stability and high initiation sensitivity, they are the very desirable explosives for military and commercial applications. Today, they have been synthesized in the laboratory, but they still can't be produced on a large scale because of the complexity of the synthetic routes or the insecurity in the process of production (For example, NaN<sub>3</sub> was used. In the synthesis process of TACOT, there are two steps that need to be operated by diazotization).. Therefore, the research on heat-resistant explosive has attracted more and more interest in chemists.

## 2 Experimental Section

### 2.1 Caution!

We have synthesized all compounds in millimolar amounts and have experienced no difficulties with temperature. However, appropriate safety precautions should be taken, especially when these compounds are prepared on a large scale. The use of appropriate safety precautions (safety shields, face shields, leather gloves, protective clothing, such as heavy leather welding suits and ear plugs) is mandatory. All compounds should be stored in explosive cases. Ignoring safety precautions can lead to an accident or serious injury.

### 2.2 Material and Instruments

The reagents were available commercially and were used as purchased without further purification. Reactions were monitored by TLC analysis, by using precoated silica gel TLC plates obtained from Haiyang of Qindao.<sup>1</sup>H and <sup>13</sup>C NMR spectroscopic data were recorded on a Bruker Avance 300 or 500 MHz FT NMR spectrometer respectively with tetramethylsilane (TMS) as an internal standard using [D<sub>6</sub>]DMSO as the solvent. Decomposition temperatures were determined by DSC-TGA using TA instruments SDT Q 600 instrument. Elemental analyses were performed on a flash EA 1112 full automatic trace element analyser.

**4,4'-((2-nitro-1,3-phenylene)bis(oxy))bis(1,3-dinitrobenzene) (3)** 2-Nitro-benzene-1,3-diol (15.5 g, 0.10 mol, 1.0 eq) was dissolved in anhydrous acetonitrile (150 mL) and triethylamine (30.3 g, 6.0 eq) was added under vigorous stirring. The resulting solution was allowed to stir at ambient temperature for 15 min before 1-chloro-2,4-dinitrobenzene (44.56 g, 0.22 mmol, 2.2 eq) was added. The reaction mixture was heated at 80 °C for 6 h after 1-chloro-2,4-dinitrobenzene was added. Then the mixture was allowed to

cool to room temperature and dump into the ice water (500 g). The solid was collected by filtration, washed with hot water and absolute ethanol in sequence to obtain compound **3** (orange solid, 16.91 g, Yield: 96.07%). <sup>1</sup>H NMR (300 MHz, [D<sub>6</sub>] DMSO): δ = 8.922–8.913 (d, *J* = 2.7 Hz, 2H), 8.562–8.522 (dd, *J*<sub>1</sub> = 9.3 Hz, *J*<sub>2</sub> = 2.7 Hz, 2H), 7.844–7.787 (m, 1H), 7.645–7.614 (d, *J* = 9.3 Hz, 2H), 7.474–7.446 (d, *J* = 8.4 Hz, 2H); <sup>13</sup>C NMR (75 MHz, [D<sub>6</sub>] DMSO): δ = 153.179, 147.095, 143.444, 140.099, 130.290, 122.453, 121.340, 116.909; Elemental Analysis calcd (%) for C<sub>18</sub>H<sub>9</sub>N<sub>5</sub>O<sub>12</sub> (487.29): C, 44.29; H, 1.78; N, 14.25; found: C, 44.37; H, 1.86; N, 14.37; O, 39.40

**4,4'-((2,4,6-trinitro-1,3-phenylene)bis(oxy))bis(1,3-dinitrobenzene) (ZXC-5)** 4,4'-((2-nitro-1,3-phenylene)bis(oxy))bis(1,3-dinitro-benzene) (**3**) (9.74 g, 20 mmol) was dissolved in concentrated sulfuric acid (100 mL, 95–98%) and fuming nitric acid (15 mL) was added drop wise under vigorous stirring at the ice water bath. Then the resulting solution was allowed to warm to ambient temperature for 2 h before being poured into ice water (500 mL) and stirred vigorously until all the precipitates were formed. the precipitates were collected by filtration and washed with water and absolute ethanol in sequence to obtain ZXC-5 (orange solid, 10.74 g, Yield: 93.07%). <sup>1</sup>H NMR (300 MHz, [D<sub>6</sub>] DMSO): δ = 9.507 (s, 1H), 8.946 (s, 2H), 8.565–8.595 (m, 2H), 7.792–7.822 (m, 2H); <sup>13</sup>C NMR (125 MHz, [D<sub>6</sub>] DMSO): δ = 145.353, 141.803, 140.215, 131.791, 130.088, 124.153, 120.205. Elemental Analysis calcd (%) for C<sub>18</sub>H<sub>7</sub>N<sub>7</sub>O<sub>16</sub> (577.29): C, 37.31; H, 1.12; N, 16.75; found: C, 37.45; H, 1.22; N, 16.98; O, 44.34.

**2,2'-((2-nitro-1,3-phenylene)bis(oxy))bis(1,3,5-trinitrobenzene) (7)** 2-Nitro-benzene-1,3-diol (3.1 g, 20 mmol, 1.0 eq) was dissolved in anhydrous acetonitrile (50 mL) and triethylamine (12.12 g, 120 mmol, 6.0 eq) was added under vigorous stirring. The resulting solution was allowed to stir at ambient temperature for 15 min before 2-chloro-1,3,5-trinitrobenzene (10.89 g, 0.44 mmol, 2.2 eq) was added. The reaction mixture was heated at 80 °C for 6 h after 2-chloro-1,3,5-trinitrobenzene was added. Then the mixture was allowed to cool to room temperature and dump into the ice water (500 g). The solid was collected by filtration, washed with water and purified by chromatography column to obtain compound **7** (yellow solid, 8.60 g, Yield: 74.52%). <sup>1</sup>H NMR (300 MHz, [D<sub>6</sub>] DMSO): δ = 9.330 (s, 4H), 7.520–7.463 (m, 1H), 7.032–7.003 (d, *J* = 8.7 Hz, 2H); <sup>13</sup>C NMR (125 MHz, [D<sub>6</sub>] CD<sub>3</sub>COCD<sub>3</sub>): δ = 150.85, 146.512, 145.907, 145.368, 133.629, 127.448, 111.902; Elemental Analysis calcd (%) for C<sub>18</sub>H<sub>7</sub>N<sub>7</sub>O<sub>16</sub> (577.29): C, 37.23; H, 1.16; N, 16.76; found: C, 37.45; H, 1.22; N, 16.98; O, 44.34.

**2,2'-((2,4-dinitro-1,3-phenylene)bis(oxy))bis(1,3,5-trinitrobenzene) (8)** Potassium nitrate (KNO<sub>3</sub> 10.1 g, 0.1 mmol) was dissolved in concentrated sulfuric acid (100 mL, 95–98%) under vigorous stirring at the ice water bath, before compound **7** was added in batches. The resulting solution was allowed to stir at ambient temperature for 10 hr after compound **7** (5.77 g, 10 mmol) was added. Then the mixture was allowed to dump into the ice water (300 g). The

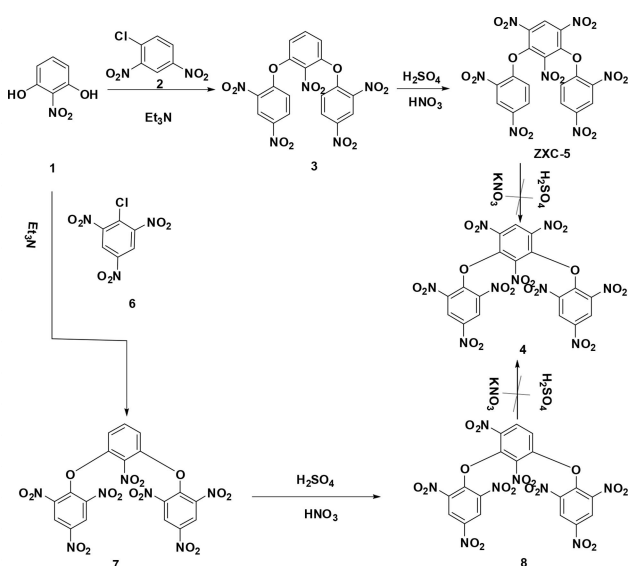
solid was collected by filtration, washed with water and purified by chromatography column to obtain compound **8** (pale yellow solid, 1.45 g, yields: 23.3%).  $^1\text{H NMR}$  (300 MHz,  $[\text{D}_6]$  DMSO):  $\delta = 9.336(\text{s}, 2\text{H}), 9.234(\text{s}, 2\text{H}), 8.481\text{--}8.450(\text{d}, J = 9.3\text{ Hz}, 1\text{H}), 7.479\text{--}7.447(\text{d}, J = 9.6\text{ Hz}, 1\text{H})$ ; Elemental Analysis calcd (%) for  $\text{C}_{18}\text{H}_6\text{N}_8\text{O}_{18}$  (622.28): C, 34.65; H, 0.94; N, 17.89; found: C, 34.74; H, 0.97; N, 18.01; O, 46.28.

## 3 Results and Discussion

### 3.1 Synthesis

It is worth pointing out that we originally intend to prepare 2,2'-((2,4,6-trinitro-1,3-phenylene) bis(oxy)) bis(1,3,5-trinitrobenzene) (compound **4**) by treatment of mixed acid (consisting of concentrated sulfuric acid and fuming nitric acid) and compound **3**. However, only 4,4'-((2,4,6-trinitro-1,3-phenylene)bis(oxy))bis(1,3-dinitrobenzene) (ZXC-5) was obtained because of the influence of the strong steric hindrance. The synthesis and investigation into the novel, thermally stable ZXC-5 is presented for the first time. The synthesis of ZXC-5 is achieved in only two steps from commercially available compounds of 2-Nitrobenzene-1,3-diol and 1-chloro-2,4-dinitrobenzene (Scheme 1). The synthetic route presented herein results in high yields of ZXC-5 and selectivity of the desired compound. The intermediates and the final products are precipitated from the reaction mixture without involving a time-consuming and costly purification process.

In addition, by treatment of mixed acid (consisting of concentrated sulfuric acid and fuming nitric acid) and compound **7**, only compound **8** is obtained instead of compound **4** (Scheme 1). In this manuscript, compound **8** has



Scheme 1. Synthesis of ZXC-5

not been discussed, because of its many shortcomings (For example, its yield is low, and the purification of the product is also difficult).

The first step in the synthesis of ZXC-5 is the coupling reaction between one molecule of 2-Nitrobenzene-1,3-diol and two molecules of 1-chloro-2,4-dinitrobenzene in the presence of alkali (triethylamine) as absorb acid agent (yield 96%). An excess of 1-chloro-2,4-dinitrobenzene was easily removed by thermal filtering because of its low melting point (The melting point of resorcinol is below  $55^\circ\text{C}$ ). In the final step, ((4,6-dinitro-1,3-phenylene)bis(oxy))dibenzene (compound **3**) is dissolved in the concentrated sulfuric acid (95–98%) and stirred at ice water bath, then the fuming nitric acid is slowly added to the mixture through a constant pressure drop funnel. After all the fuming nitric acid has been added, the mixture is then warmed to room temperature for 50–60 minutes and continued stirring for 10 h before the reaction mixture is poured onto crushed ice to yield ZXC-5 (yield 93%). ZXC-5 is a pale yellow solid and insoluble in water.

**Single Crystal X-ray Structure Analysis.** The structure of ZXC-5· $\text{CH}_3\text{CO}_2\text{Et}$  in the crystalline state was determined by means of low-temperature single-crystal X-ray diffraction. Selected crystallographic data, measurement parameters, and refinement details are given in the Supporting Information. ZXC-5· $\text{CH}_3\text{CO}_2\text{Et}$  crystallizes from the hot mixed solvents of ethanol and ethyl acetate with one solvent molecule per ZXC-5· $\text{CH}_3\text{CO}_2\text{Et}$  molecule in the triclinic space group P-1, with a density of  $1.608\text{ g cm}^{-3}$  at 173 K (when ZXC-5 does not contain the molecule of  $\text{CH}_3\text{CO}_2\text{Et}$ , its measured density is  $1.771\text{ g cm}^{-3}$  at 298 K) and five molecules in the unit cell. The molecular structure of ZXC-5 is shown in Figure 1, and the packing diagram of ZXC-5 is shown in Figure 2.

**Physicochemical Properties.** ZXC-5 shows the desired properties required for a heat-resistant explosive: good detonation parameters, low sensitivity values, and a high decomposition temperature. The thermal stability of ZXC-5 was measured by using differential scanning calorimetry (DSC), which showed that it decomposed above  $288.61^\circ\text{C}$  and melted above  $271^\circ\text{C}$  (Figure 3).

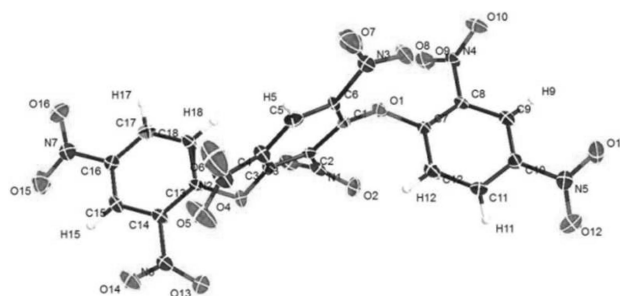


Figure 1. Molecular structure of ZXC-5 shown with thermal ellipsoids at the 50% probability level. Cocrystallized solvent molecules are removed for clarity



Figure 2. Packing diagram of ZXC-5 viewed along the *c* axis.

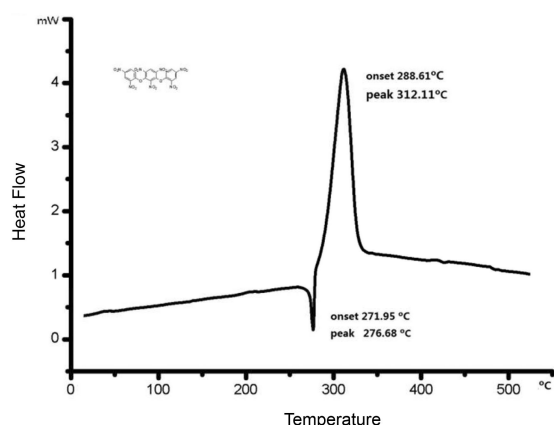


Figure 3. DSC diagram of ZXC-5 (5 K/min)

The measured density of ZXC-5 is  $1.771 \text{ g cm}^{-3}$  (obtained from a pycnometer measurement at 298 K), which is higher than the densities of both HNS ( $\rho_{\text{TMD}} = 1.740 \text{ g cm}^{-3}$ ) and PYX ( $\rho_{\text{TMD}} = 1.757 \text{ g cm}^{-3}$ ).<sup>[9]</sup> The calculated standard molar enthalpy of formation for ZXC-5 ( $\Delta_f H = 268.2 \text{ kJ mol}^{-1}$ ) is more than 3.4 times higher than that of HNS ( $\Delta_f H = 78.2 \text{ kJ mol}^{-1}$ ), and more than 6.1 times higher than that of PYX ( $\Delta_f H = 43.7 \text{ kJ mol}^{-1}$ ). The high density and high heat of formation result in very good detonation parameters for ZXC-5. The Chapman–Jouguet (C–J) characteristics of ZXC-5: detonation velocity ( $V_{\text{C–J}} = 7917 \text{ m s}^{-1}$ ) is highest than the corresponding values for PYX ( $V_{\text{C–J}} = 7757 \text{ m s}^{-1}$ ) and HNS ( $V_{\text{C–J}} = 7612 \text{ m s}^{-1}$ ), and is approximately equal to that of TKX-55 ( $V_{\text{C–J}} = 8030 \text{ m s}^{-1}$ ) [10–12]. The detonation pressure of compound ZXC-5 ( $P_{\text{C–J}} = 28.17 \text{ GPa}$ ) is the highest than those of PYX ( $P_{\text{C–J}} = 25.1 \text{ GPa}$ ), HNS ( $P_{\text{C–J}} = 24.3 \text{ GPa}$ ) and TKX-55 ( $27.3 \text{ GPa}$ ). The impact sensitivity (IS) of ZXC-5 is 39 J (Table 1), which is more than 3.9 times higher than that of PYX (IS = 10 J), and more than 7.8 times higher than those of HNS and TKX-55 (IS = 5 J). The friction sensitivity of ZXC-5 is higher than the measuring range of the friction tester apparatus (according to the STANAG regulations). ZXC-5 is also

Table 1. Physicochemical properties of PYX, HNS, and ZXC-5.

	PYX	HNS	TKX-55	ZXC-5
formula	$\text{C}_{17}\text{H}_7\text{N}_{11}\text{O}_{16}$	$\text{C}_{14}\text{H}_6\text{N}_6\text{O}_{12}$	$\text{C}_{16}\text{H}_4\text{N}_{10}\text{O}_{14}$	$\text{C}_{18}\text{H}_7\text{N}_7\text{O}_{16}$
IS <sup>[a]</sup> [J]	10	5	5	39
FS <sup>[b]</sup> [N]	360	240	> 360	> 360
ESD <sup>[c]</sup> [J]	0.5	0.8	1.0	1.0
N <sup>[d]</sup> [%]	24.80	18.67	25	16.98
OB <sup>[e]</sup> [%]	−55.36	−67.6	−57	−65.1%
$T_{\text{dec}}$ <sup>[f]</sup> [°C]	360	318	335	288.6
$\rho$ <sup>[g]</sup> [ $\text{g cm}^{-3}$ ]	1.75	1.74	1.83	1.77
$\Delta_f H$ <sup>[h]</sup>	43.7	78.2	197.6	268.2
EXPLOS V6.01				
$\Delta_E U$ <sup>[i]</sup>	−4870	−5142	4961	−6413
$P_{\text{C–J}}$ <sup>[j]</sup> [GPa]	25.1	24.3	27.3	28.17
$V_{\text{C–J}}$ <sup>[k]</sup> [ $\text{m s}^{-1}$ ]	7757	7612	8030	7917

[a] IS: impact sensitivity (BAM drop hammer, method 1 of 6). [b] FS: friction sensitivity (BAM drop hammer, method 1 of 6). [c] ESD: electrostatic discharge device (OZM research). [d] Nitrogen content. [e] Oxygen balance. [f] Temperature of decomposition. [g] Density at 298 K. [h] Standard molar enthalpy of formation is calculated ( $[\text{kJ mol}^{-1}]$ ). [i] Heat of detonation is calculated ( $[\text{kJ kg}^{-1}]$ ). [j] Detonation pressure is calculated. [k] Detonation velocity is calculated.

less sensitive toward electrostatic discharge (typical values for the human body are within the range 0.005–0.02 J) than PYX and HNS (Table 1). The Oxygen balance of ZXC-5 (OB = −57%) is also higher than that of HNS (OB = −67.6%)

## 4 Conclusions

The synthetic route for the preparation of 4,4′-((2,4,6-trinitro-1,3-phenylene)bis(oxy))bis(1,3-dinitrobenzene) (ZXC-5) with detailed characterizations has been reported. The crystal structure for ZXC-5 was determined using low-temperature single-crystal X-ray diffraction. It is surprising, ZXC-5 shows lower sensitivities toward impact and friction than the HNS and PYX. Moreover, ZXC-5 also shows a good thermal stability ( $T_{\text{dec}} = 288.61 \text{ °C}$ ). The detonation performances (enthalpy of formation, density, detonation velocity, and detonation pressure values) of ZXC-5 are higher than those of the currently used heat-resistant explosives (HNS and PYX). Thus, it can be concluded that ZXC-5 is one of the most thermally stable explosives. In fact, ZXC-5 can be prepared by using a facile synthetic route (The total synthesis of ZXC-5 requires only two steps and the total yield of ZXC-5 is more than 89%). ZXC-5 is insoluble in water, and its preparation can avoid the toxic effects on the environment. This combination of properties makes ZXC-5 unique among thermally stable explosives.

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