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Explosive Nitrotriazolone Formulates

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ABSTRACT

Nitrotriazolone has been synthesised and fully characterised. This explosive was found to exhibit self-binding properties forming pellets at different loads. The compression strength of these pellets were in the range 80–128 kgf/cm^2 under one to three tonne/cm^2 load. Other pressable formulations containing nitrotriazolone have also been successfully prepared. A composition comparable to composition B was also prepared using nitrotriazolone and trinitrotoluene (60:40). Mechanical properties and insensitivity of this new composition were found to be superior.

Keywords: Nitrotriazolone, explosive formulations, NTO, 3-nitro-1,2,4-triazol-5-one, castable charges, plastic-bonded explosive, pellets, insensitive-explosive

1. INTRODUCTION

Problems associated with catastrophic mishaps, sympathetic detonations and dimensional instability at elevated temperatures have made common explosives such as 1,2,4-trinitrotoluene (TNT), 1,3,5-trinitro hexahydro-s-triazine (RDX) and 1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) less attractive in certain applications where materials are subjected to extreme conditions while being transported or stored. In this context, 3-nitro-1,2,4-triazol-5-one (NTO) has emerged as a vital explosive molecule combining high performance (close to RDX) and insensitivity [comparable to 1,3,5- triamino-2,4,6-trinitrobenzene (TATB)] coupled with good thermal stability. A great deal of the sensitivity and performance data on NTO has been published[1-5] and its applications, such as a potential replacement for RDX in bomb fill have been reported[6]. However, there are only a few references where formulates based on NTO appear.

The objective of the present study is to synthesise and characterise this important explosive molecule and to evaluate it as an explosive. Since NTO has self-binding properties, it has been explored to make pressable and castable charges containing NTO.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

Semicarbazide hydrochloride (laboratory grade) and formic acid and nitric acid (both analytical grade) were obtained from trade and used as such. Ethanol was distilled before use.

2.2 Synthesis of NTO

Synthesis of NTO[7] was carried out in two steps: Condensation of semicarbazide hydrochloride and formic acid forming triazolone (TO), and (ii) nitration of TO to form NTO (Scheme 1).

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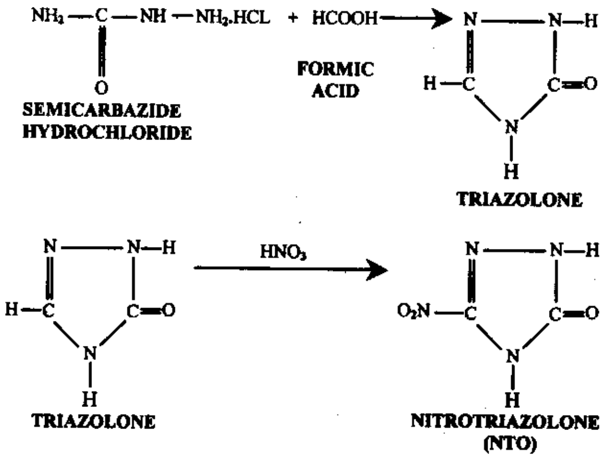

Scheme 1. Synthesis of NTO

2.2.1 *Synthesis of Triazolone*

Semicarbazide hydrochloride (300 g) was added to 300 ml of formic acid in a three-necked flask. The mixture was heated with stirring until all semicarbazide hydrochloride was dissolved completely. The excess of formic acid was removed by distillation until crystallisation occurred. Water (675 ml) was then added and distillation continued to dryness. The contents were filtered and crystallised from ethyl alcohol. The yield was 70 per cent; mp, 232 °C (reported, 232 °C).

2.2.2 *Synthesis of* NTO

Nitric acid (900 ml) was added to 150 g of TO maintaining the temperature between 0 °C and 5 °C. The mixture was heated to 60–70 °C with constant stirring. The reaction was exothermic and brown fumes evolved. After the reaction was complete, the NTO precipitated out. The contents were chilled (0–5 °C) in an ice-bath, filtered and washed with water to remove excess nitric acid. Pure NTO was obtained by crystallisation from water. The yield was 80 per cent.

2.3 Explosive Formulation

NTO without any binder was pressed under different loads. Pellets of different diameters between 10 mm and 30 mm were obtained under different loads and the compression strength determined using universal testing machine (Instron, model-1185, UK). Pellets (30 mm) pressed under optimal conditions were subjected to velocity of detonation (VOD) by the probe method. NTO was evaluated in castable plastic-bonded explosive (PBX) formulation with hydroxyl-terminated polybutadiene (HTPB) as the binder. The processing was carried out by mixing the components in a sigma mixer for about 3 hr under vacuum at 45 °C. The formulation NTO/HTPB (80:20) was cast and cured with methylene diisocyanate (MDI) at ambient temperature. NTO loading was bi-modal in particle size i.e., 300–355 μm (75 %) and 63–75 μm 25 %). Theoretical performance parameters of the NTO/HTPB cast composition have also been computed. NTO/TNT (60:40) castable compositions have been prepared by the method established similar to RDX/TNT[8].

Since it was not possible to make pellets using pure RDX or pure HMX, HMX was coated with 3 per cent polyurethane binder, using slurry method. This composition was mixed with 25 per cent pure NTO and the pellets were made to determine the various characteristics like explosive, mechanical and sensitivity. Properties like thermal stability, sensitivity and vacuum stability of all the formulations have also been determined by standard methods.

The IR spectra were recorded on a Perkin-Elmer FTIR spectrophotometer model-1605 using smear technique. DTA was carried out in static air, using a locally fabricated DTA apparatus at a heating rate of 10 °C/min. The impact sensitivity test was carried out on an impact sensitivity apparatus of the fall hammer type with a falling weight of 2 kg. The friction sensitivity was measured using a Julius Peters friction sensitivity apparatus.

3. RESULTS & DISCUSSION

The IR spectrum of TO (Fig. 1) shows carbonyl absorption at 1696 cm^{-1}, *N-H* stretching at 3090 cm^{-1}. The IR spectrum of NTO shows NO_2 stretching vibrations at 1544 cm^{-1} and 1354 cm^{-1} and *C=O* at 1716 cm^{-1}. The spectrum is presented in Fig. 2. Figures 3(a) and 3(b) show the IR spectra of NTO polymorphs[6] α and β . By comparison, it may be inferred that NTO synthesised is the stable α-polymorph.

DTA curve of NTO shows no melting, but an exotherm at 258 °C (T_i) and T_{max} at 264 °C (Fig. 4). Impact and friction sensitivities of NTO were determined. The values are presented in Table 1 along with the physical and explosive properties of NTO in comparison with RDX. It is clear from these values that NTO is an insensitive explosive.

3.1 Explosive Formulations

The thermal properties and vacuum stability data for the compositions prepared are presented in Table 2. All NTO containing formulations are thermally stable. This is due to the high thermal stability of NTO compared to TNT, which exhibits a phase change at 80 °C. The good storage life

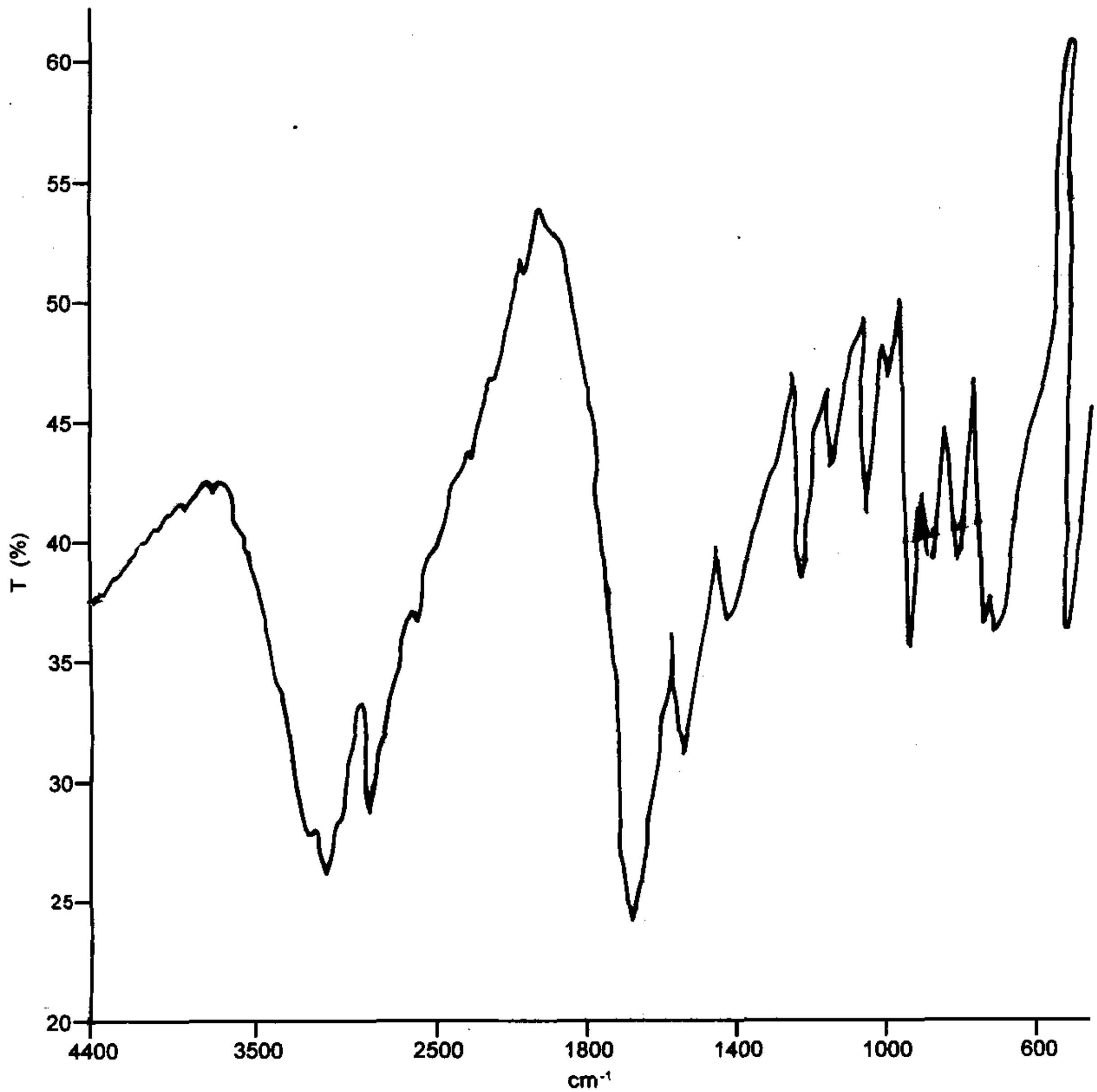

Figure 1. IR spectrum of triazolone

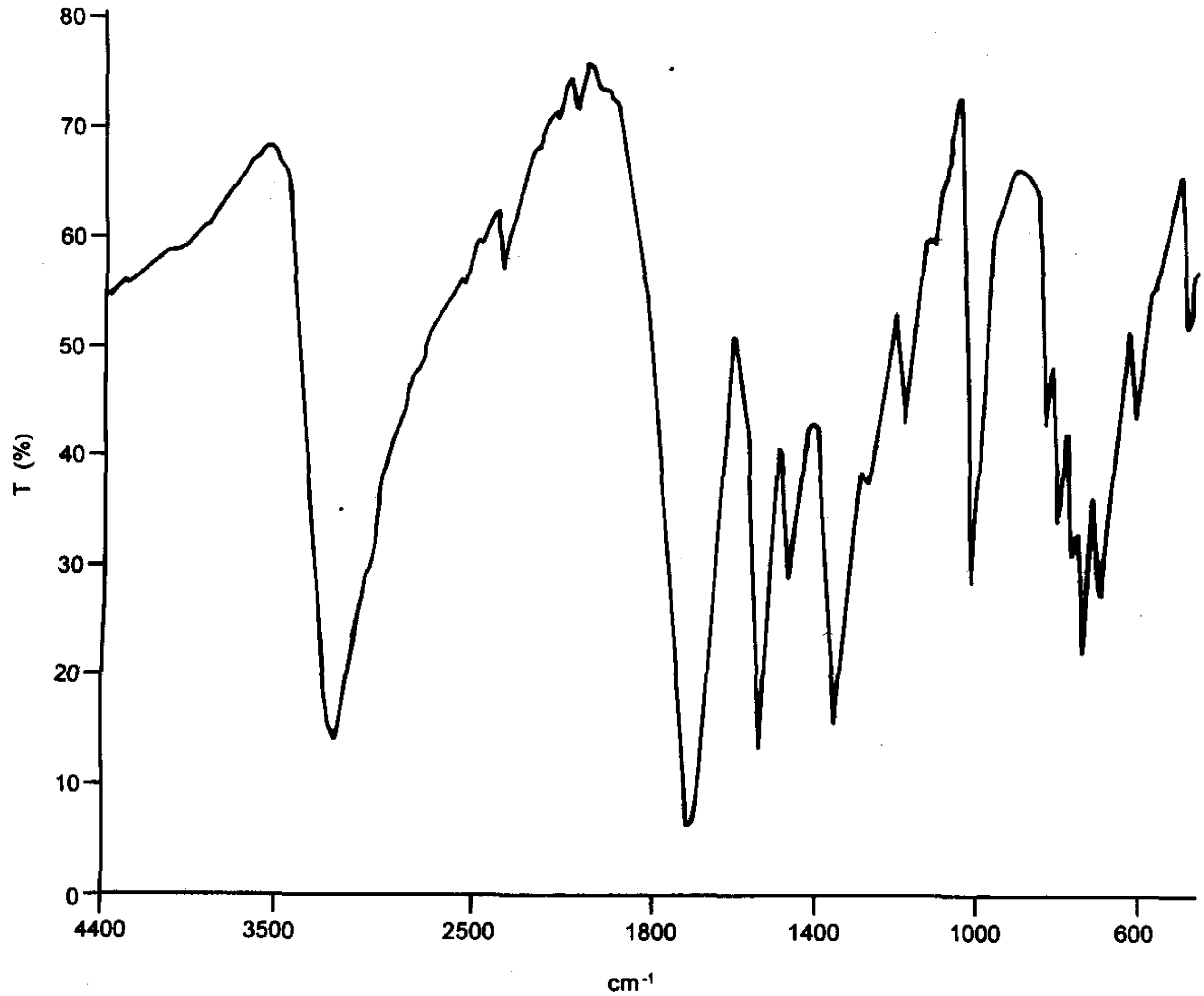

Figure 2. IR spectrum of NTO

of these compositions is indicated by the results of the vacuum stability test. The composition, density, VOD and sensitivity characteristics of the formulations are given in Table 3. Density is a measure of the distribution of a solid in the polymer matrix and depends on solid loading, density of explosive used, particle size distribution of the filler crosslink density of the polymer among other factores. Density observed was around 95 per cent of the theoretical maximum density.

The sensitivity of NTO is reduced considerably compared to pure compound by its incorporation in the polymer matrix. The improvement in the sensitivity is ascribed to the perfect coating of the explosive by the stable polymer matrix. Compound to RDX/TNT (60:40), which is a well-known castable high explosive filling, NTO with 80 weight per cent loading is less sensitive and thus more safe for handling and processing. The calculated VOD values using BKW code are matching with the experimentally obtained VOD data.

Compression strength is a measure of particle-to-particle bonding and the bonding between the explosive and the polymer matrix. It was observed that NTO has self-binding properties and forms good pellets under different loads, which is not possible with RDX or HMX. The compression strength of NTO pellets were in the range 80–128 kgf/cm^2 under loads of 1–3 tonnes/cm^2. The NTO/HMX-PU pellets were also pressed under optimal loads. The data on these pellets are presented in Table 4.

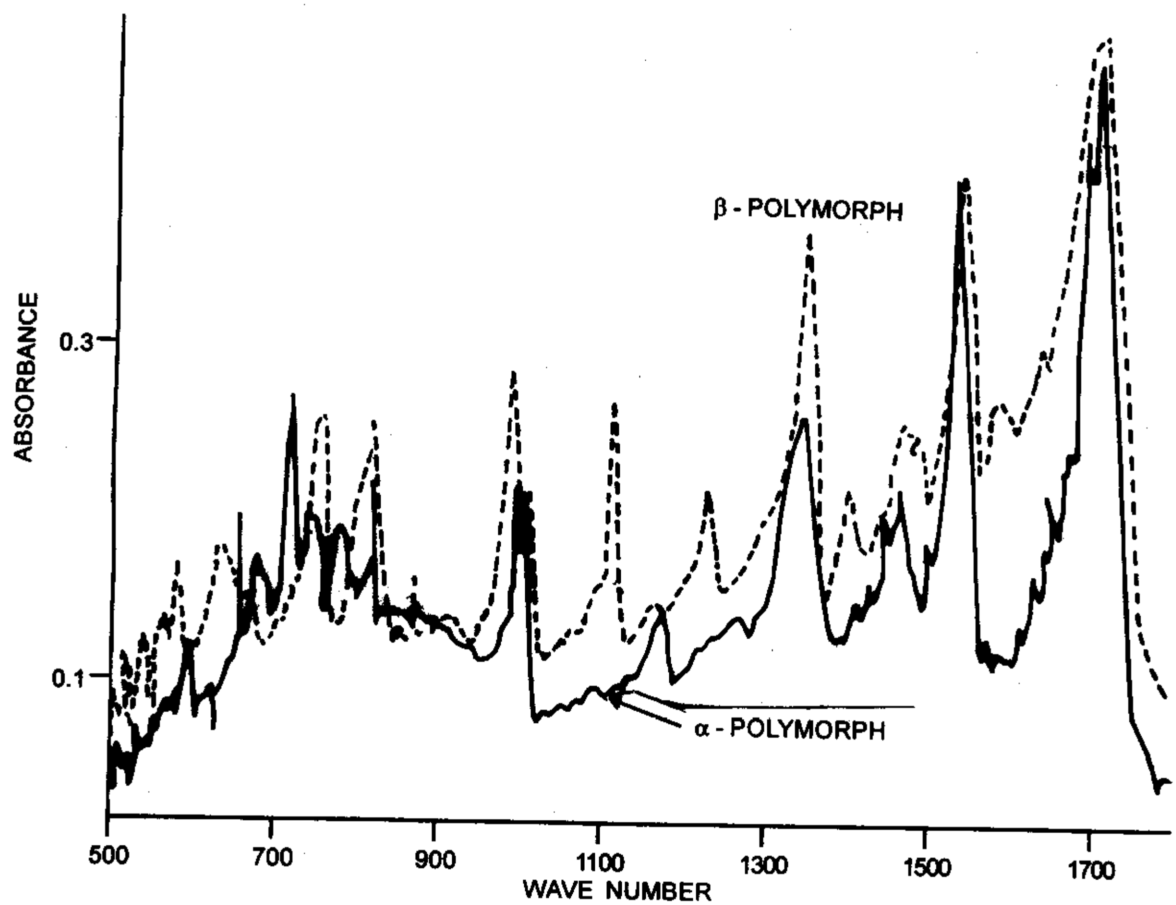

Figure 3. IR spectra of NTO polymers

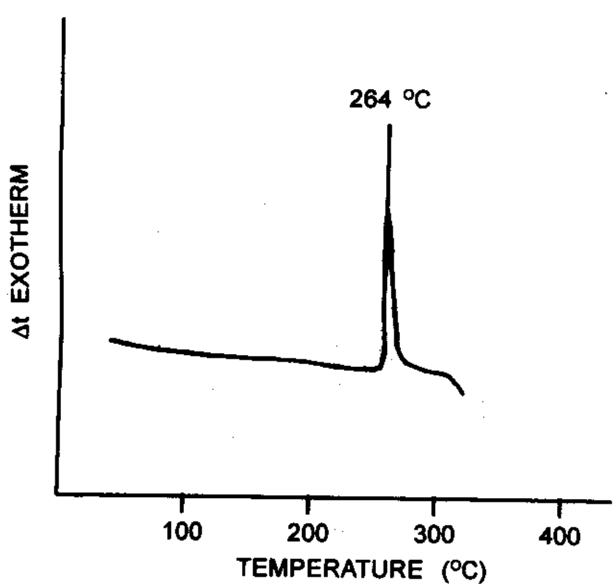

Figure 4. DTA curve of NTO

Table 1. NTO properties and comparison with RDX

	NTO		RDX
	Observed	Theoretical	
Molecular formula	$C_2H_2N_4O_3$		$C_3H_6N_6O_6$
Crystal density (g/cm³)	1.84	1.93	1.80
DTA exotherm (°C)	264	> 252	210
VOD (m/s)	7992	8510	8700
C-J pressure (kbar)	—	349	348
Impact sensitivity (cm) (Ht. of 50 % explosion)	87	—	21
Friction sensitivity (kg) (Not sensitive up to)	36	—	36
Vacuum stability (ml) at 120 °C for 48 hr	0.49	—	0.12–0.90

Table 2. Stability characteristics of NTO-based formulations

Explosive formulations	Thermal stability (onset of thermal change) (°C)	Vacuum stability (volume of gases at 120 °C for 48 hr, ml)
NTO (pure)	254	0.49
NTO/HTPB (80:20)	250	0.90
NTO/TNT (60:40)	080	0.92
NTO/HMX (25:75)	250	0.80
RDX/TNT (60:40)	080	0.95

Table 3. Composition and sensitivity parameters of NTO-based formulations

Composition	Density	VOD		Impact[a] sensitivity	Friction sensitivity
		Expt	Calc		
	(g/cm^3)	(m/s)		$h_{50\%}$ (cm)	(kg)
NTO (pure)	1.82	7992	8000	078	>36
NTO/HTPB (80:20)	1.59	7150	7180	127	>36
NTO/TNT (60:40)	1.62	7134	7200	164	>36
NTO/HMX (25:75)	1.76	8427	8450	040	16
RDX/TNT (60:40)	1.67	7500	7600	095	24

Table 4. Mechanical properties of explosive formulations

Explosive formulation	Compression strength (kgf/cm^2)	Compression (%)
NTO	128.0	03.0
NTO/HMX (25:75)	069.4	02.5
NTO/TNT (60:40)	101.2	01.2
NTO/HTPB (80:20)	016.0	12.0
RDX/TNT (60:40)	015.3	16.6

4. CONCLUSION

NTO synthesised was found to be the stable α-polymorph. The compound shows good thermal and chemical stability. It forms self-binding pellets of good compression strength. The NTO-based pressable and castable explosive formulations exhibited superior mechanical and thermal properties and were insensitive. These data qualify the new explosive formulations to be included in the Lovex class of explosives.

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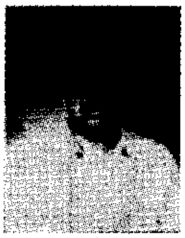

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