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Synthesis, characterization, thermolysis and performance evaluation of mercuric-5-nitrotetrazole (MNT)

M.B. Talawar*, J.S. Chhabra, A.P. Agrawal, S.N. Asthana,
K.U.B. Rao, Haridwar Singh

High Energy Materials Research Laboratory, Pune 411 021, India

Abstract

Mercuric-5-nitrotetrazole (MNT) was synthesized on using a reported method. The product having bulk density of 1.5 g/cm^3, was obtained during this work using mercuric nitrate doped with additives such as cephol/dextrin in the process. Synthesized MNT was characterized by metal content analysis, IR and ESCA. The DTA profile indicated the thermal stability of MNT up to 200 °C. It revealed its higher thermally sensitive [thermal sensitive figure (S) ∼0.8] in comparison to that of service lead azide (SLA) [$S \sim 0.4$]. Percussion sensitivity data also showed higher sensitivity of MNT. However, it was found less friction sensitive than SLA. The chemical stability of MNT in a carbon dioxide environment was evaluated in comparison to SLA by determining mercury (gravimetrically) and lead azide (volumetrically) contents respectively. Results obtained indicated that no discernable changes occurred in MNT, even after storage for 90 days while in case of SLA, drastic change in lead azide content was observed. IR spectra of MNT sample stored in a closed aluminum dish for 5–10 years could be superimposed on that of the freshly prepared MNT sample. The performance of MNT filled detonator no. 27 assessed in terms of extent of damage on a witness plate was found equivalent to that of the standard ASA (azide, styphynate and aluminium) composition filled detonator.
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1. Introduction

Lead azide is a primary explosive, which is ranked top amongst the conventional initiators, and is being most widely used in service detonators. In spite of its best initiating and filling properties, it suffers from certain drawbacks like, (a) high friction sensitivity, (b) tendency to undergo hydrolysis in the presence of moisture, (c) rapid deterioration in a carbon dioxide environment, and (d) incompatibility with copper (a component of the detonator fuze casing). The search [1–3] is on for superior replacement of lead azide in primary explosive compositions. Among lead free primary explosives, the most promising initiator is silver azide [4]. Although silver azide has far better shelf life [4] in comparison to lead azide, it is much more sensitive to friction stimuli. Further, it is difficult to obtain silver

azide in free flowing form with high bulk density. Moreover in view of the high cost, silver azide is being used only for specialized applications. Recently, co-ordination compounds [5–8] containing near stoichiometric fuel and oxidizer fractions have evinced great interest. The extensive study on the relationship between structure of co-ordination compound and explosive properties has been reported by various research groups [9–11]. Nickel and cobalt complexes appear suitable for detonator applications. Nickel hydrazinium nitrate (NHN) [12–14] may find wide ranging applications in conventional detonators whereas bis-(5-nitrotetrazoleto-N^2)-tetraamine cobalt perchlorate (BNCP) has emerged as an energy producing component for semi conducting bridge (SCB) initiator applications [15–18]. Mercuric salt of nitro tetrazole (MNT) is being investigated [19–21] as primary explosive filling in UK [2,3,22], USA [20,23] and Australia [21]. Von Herz [24] reported the synthesis of MNT from Cu(NT)$_2$HNT·4H$_2$O by the diazotisation of 5-aminotetrazole monohydrate (AT) in presence of excess of nitrite and cupric sulphate. The

* Corresponding author. Tel.: +91 20 25869303;
fax: +91 20 25869316.
E-mail address: mbtalawar@yahoo.com (M.B. Talawar).

original patented [24] process of MNT reported by Herz suffered from the drawbacks such as low yields and the poor physical form rendering the filtration of the product difficult. The process was also beset with hazards of occasional minor explosions. Bates et al [25] reported higher yields of MNT by altering the reaction parameters. It is also reported [22–23,26] that MNT has shown to be very effective single component detonant with greater output than lead azide. It is claimed that in contrast to lead azide, the MNT [27] possess the advantage of excellent inflammability even in a moist environment. Further, its stability is not influenced by the presence of carbon dioxide and it may be stored without deterioration in tropical climates. Its sensitivity to electrostatic initiation is also relatively less than that of the primary explosives in use. Unlike mercury fulminate, MNT does not get dead pressed [27].

As detailed information on MNT is not readily available in open literature, the work on it was continued in addition to research on energetic co-ordination compounds [28–30] by the authors particularly from the point of data generation with respect to its application. The multifaceted studies on MNT encompass synthesis in high bulk density, characterization, thermolysis, stability and performance evaluation in detonator no. 27.

2. Experimental

2.1. Materials

The starting material, 5-aminotetrazole was prepared in the laboratory by interaction of dicyandiamide with sodium azide and hydrochloric acid. Sodium nitrite, ethylene diamine, mercuric nitrate and copper sulfate (A.R. grade) obtained from the trade were used as such without further purification.

2.2. Synthesis of MNT

The MNT was prepared according to the method reported by Redman and Spear [20]. Thus, 5-aminotetrazole was treated with sodium nitrite and copper sulfate to obtain $Cu(NT)_2HNT \cdot 4H_2O$ (where NT: nitrotetrazole) [31]. The copper salt was subsequently converted to ethylene diamine complex The MNT was obtained by treating the complex with mercuric nitrate in HNO_3 medium. The precursors and final product were air dried. An outline of the reaction is given in Scheme 1 The synthesis of the compounds was carried out in fuming hood behind protective polycarbonate shield in stainless steel reaction vessel.

2.2.1. Preparation of MNT with additives

A scheme [20] similar to that described above was followed for preparing MNT incorporated with additives (cephol/dextrin). A concentrated nitric acid ($2 \, cm^3$) diluted with water ($5 \, cm^3$) was added over a period of 15 m to a mechanically stirred solution of 0.0025 g cephol/dextrin and 2.5 g of $Cu(en)_2(NT)_2$ in $30 \, cm^3$ water, It was followed by the addition of aqueous solution of mercuric nitrate ($7 \, cm^3$). The reaction mixture was heated at 65–70 °C with constant stirring for nearly 30 m and then allowed to cool down to 55 °C. Subsequently, the supernatant solution was decanted, and the product obtained was washed with water ($5 \times 10 \, cm^3$). The compound was filtered and air dried.

Scheme 1. Synthesis of MNT, where en: ethylene diamine; NT: nitrotetrazole.

2.3. Characterization

2.3.1. Metal content analysis

The purity of the MNT was checked by determining the mercury content by gravimetric method [32]. The experimentally determined metal content of MNT (Hg: 46.54 %) was found to be in close agreement with the theoretical value (Hg: 46.85 %).

2.3.2. IR spectral analysis

FT-IR spectra of the synthesized compound and samples drawn at regular intervals were recorded on a Perkin-Elmer IR Spectrophotometer Model 457.

2.3.3. Electron spectroscopy for chemical analysis (ESCA) studies

The ESCA of the samples was measured on ESCA 3000, (VG Microteck, UK) instrument, using MgK_2 (1253.6 eV) as radiation source. The binding energies of all the elements were corrected by considering C_{1s} binding energy $= 285.0$ eV as internal standard. The analysis of each complex was carried out under high vacuum of the order of 10^{-9} torr.

2.3.4. Bulk density

Bulk density of MNT was determined by standard procedure adopted for initiators i.e. measuring of volume of solution in butanol in a standard graduated tube, every 30 m until two successive readings remained unchanged.

2.3.5. Thermal studies

A micro DTA apparatus designed and fabricated in the laboratory was used for thermal analysis. The temperature measurements were made using Pt/Pt–Rh (13%) thermocouple in conjunction with universal temperature programmer (Precision ± 0.1 °C) at the heating rates of 5 and 15 °C/min. Ignition temperature was determined by DTA as well as by using woods metal bath at the heating rate of 5 °/m.

2.3.6. Sensitivity characterization

Percussion sensitivity was measured using ball drop apparatus fabricated in the laboratory. Friction sensitivity measurements were made on a Julius Peters Friction sensitivity apparatus for primary explosives. The sample was kept between a fixed corrugated (rough) plate and a movable stainless steel plate. The movable plate was pulled at varying lever loads to subject sample to different levels of friction.

2.3.7. Chemical stability studies

The chemical stability of MNT was compared with that of service lead azide (SLA) by monitoring chemical changes in the compounds exposed to an atmosphere of carbon dioxide saturated with moisture created in desiccators containing a saturated solution of sodium carbonate. The samples of MNT and SLA were withdrawn periodically. The mercury and lead azide content were determined to assess the degradation of MNT vis-à-vis SLA. Samples were also placed on copper foils in similar environment to observe the visual changes in foil at its point of contact with the compounds.

2.3.8. Performance evaluation in detonator no. 27

MNT (100/200/300 mg) was filled into the stem of tubular detonator no. 27 and pressed at 30 MPa pressure with dwell time of 45 s to assess its potential as primary explosive for detonator application. Safety fuse with a 20 cm length was inserted into the tubular detonator touching the filled MNT layer and crimped softly. Relative humidity (R_H) of <55% was ensured in the process room throughout the filling operation. The MNT filled tubular detonator was held vertically on a 3 mm lead plate and the safety fuse was initiated by safety match. Damage to the lead plate, if any, was measured in terms of the diameter of the neatly punched crater.

In the next set of experiments, secondary explosive tetryl/pentaerthritol tetranitrate (550 mg) was filled into tubular detonator no. 27 and pressed at 10 MPa pressure with dwell time of 45 s. Subsequently, MNT was introduced and pressed as in previous instance. The safety fuse of 20 cm length was inserted into the filled tubular detonator touching the layer of MNT and crimped softly. The evaluation was carried out as above. A minimum of 10 detonators were tested for each experimental composition.

The standard detonator no. 27 containing ASA [SLA: 65%, lead styphnate (LS): 32.5% and Al: 2.5%] pressed above the initially pressed base charge of high explosives CE/PETN/RDX was used as a reference.

2.3.9. Method of disposal

MNT could be disintegrated on adding to 20% sodium hydroxide solution with stirring and can be disposed off after 48 h. This method may be used for the safe destruction of these materials.

3. Results and discussions

3.1. Infrared spectral studies

The MNT showed the IR frequencies at 680 and 800 cm^{-1} due to Hg–N linkage. The absorption assignable to tetrazole ring appeared at 1460 cm^{-1}. The two strong absorptions at 1565 and 1340 cm^{-1} exhibited the characteristic frequencies for asymmetric and symmetric vibrations due to NO_2 group of nitrotriazole. The other vibrations observed in the IR spectrum at 860, 1040, 1100 and 1190 cm^{-1} may be attributed to heterocyclic ring frequencies.

3.2. Electron spectroscopy for chemical analysis (ESCA) studies

Electron spectroscopy for chemical analysis (ESCA) data of the precursor [Cu (en)$_2$(NT)$_2$] and the final product [MNT] studied during this work is given in Table 1 The

Table 1
ESCA analysis results of complexes [Cu(en)$_2$(NT)$_2$and MNT]

Nomenclature	Binding energy (eV)					
	Carbon (C$_{1S}$)	Carbon (C$_{1S}$)	Oxygen (O$_{1S}$)	Nitrogen (1, N$_{1S}$)	Nitrogen (2, N$_{1S}$)	Metal ion
Cu(en)$_2$(NT)$_2$	285.0	287.3	532	399	401	933.2 (Cu 2p$_{3/2}$)
MNT	285.0	287	534.5	400	406.8	1037 (Hg 4f$_{7/2}$)

Where en: ethylene diamine; NT: nitrotetrazole; MNT: mercuric-5-nitrotetrazole.

presence of nitrogen in two different environments (i.e. nitrogen in nitro group and tetrazole ring) was indicated by the presence of two sharp peaks in the binding energy range of 399–400 and 401–407 eV, respectively. The prominent peak in the binding energy range of 532–534.5 eV confirms the presence of oxygen in the precursor as well as final product. The binding energies of central metal atoms were observed at 1037 and 933.2 eV due to Hg and Cu of MNT and Cu(en)$_2$(NT)$_2$, respectively. The binding energies observed for different elements are in agreement with the standard values (Table 1) and those of Cu and Hg matched with the stable oxidation state of II.

3.3. Synthesis of MNT having bulk density 1.5 g/cm^3

Two routes are reported [20] for the preparation of MNT, one via sodium salt of nitro tetrazole and the other via copper salt of bis ethylene diamine complex of 5-nitrotetrazole. The second route is preferred during this work (preparation scheme) in view of the extremely high sensitivity of sodium salt of nitrotetrazole formed as precursor in the first route, introducing an element of hazard of explosion during dehydration process. The physical appearance and the bulk density of the product realized with different starting and dopant materials are summarized in Table 2. Mercuric nitrate yielded product of higher bulk density (1.25 g/cm^3) compared to that obtained with mercuric oxide (0.92 g/cm^3). The additives are known to modify the crystal morphology with

appreciable change in bulk density. Thus, cephol was found effective in obtaining MNT with improved bulk density of the order of 1.5 g/cm^3 with change in crystal shape from granular to hexagonal whereas dextrin doped mercuric nitrate yielded non-uniform product. Hao [33] and McGuchan [2] reported the crystal modified forms of MNT. Researchers [21] from Australia also made little mention about the preparation of MNT with bulk density of 1.18 g/cm^3 without disclosing the process details including the nature of additive used.

3.4. Thermal studies

The thermal profiles of MNT at different heating rates are shown in Fig. 1. It gave an exotherm with T_{max} of 228 and 230 °C at the heating rate of 5 and 10 °C/min respectively. No thermal effects were observed in DTA output till the onset temperature of 200 °C. The SLA also did not undergo thermal changes till the onset temperature of 300 °C. The relatively lower decomposition temperature of MNT than that of SLA is indicative of its ease of thermal initiation than that of latter. The ignition temperature of MNT (228 °C) in conventional woods metal bath was also found lower than that for SLA. The relatively higher sensitivity of MNT to thermal stimuli compared to SLA was also revealed by the observation that the DTA of SLA could be recorded with samples as large as 10 mg and at heating rate of 10 °C/min whereas MNT detonated during DTA conducted at the heating rate of 5 °C/min with the sample weight as low as 3–4 mg. The thermal sensitivity value (S) may also be defined in terms of the ratio of height of DTA profile to half its width. It was also found higher for MNT (0.8) despite smaller sample size (3 mg) than that (0.4) for SLA (10 mg) (Table 3).
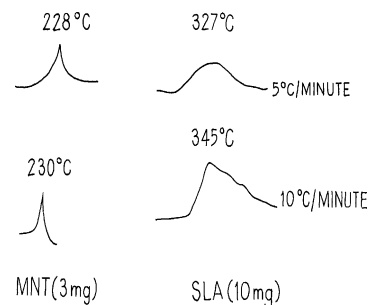
Table 2
Bulk density and the nature of crystals obtained for MNT batches

Serial number	Mercury salt/dopant used	Physical appearance	Bulk density (g/cc)
1	HgNO$_3$	Uniform granules	1.25
2	HgNO$_3$	Uniform round granule	1.20
3	HgO	With plate form	0.92
4	HgNO$_3$	Uniform granules	1.20
5	HgO	Uniform granules with plate form	0.92
6	HgO	Uniform granules with plate form	0.85
7	HgNO$_3$/dopant	Hexagonal in shape	1.5
8	HgNO$_3$/dopant	Not uniform, truncated, uneven crystals	1.15
9	HgO/dopant	Uniform hexagonal particles with few plates	0.9

Fig. 1. Thermal profiles of MNT and SLA.

Table 3
Thermal and mechanical sensitivity data for SLA and MNT

Serial number	Properties	SLA	MNT
1	Ignition temperature (°C) DTA (5 °C/min) (sample weight, mg)	327 (10)	228 (3)
2	Thermal sensitivity (S) DTA 5 °C/min (sample weight, mg)	0.4 (10)	0.8 (3)
3	Percussion sensitivity (ft lbs/in.^{2})	9.7	2.0
4	Friction sensitivity (50% E, g)	30	500

Table 4
Comparative stability of MNT and SLA under an atmosphere of carbon dioxide saturated with moisture

Serial number	Storage period (days)	Percentage of Hg in MNT	Percentage of lead azide in SLA
1	0	46.54	97.1
2	4	46.11	93.2
3	11	46.33	90.2
4	24	46.11	72.1
5	31	45.45	70.2
6	40	45.67	53.5
7	60	46.33	41.7
8	90	46.47	30.2

3.5. Chemical stability

Results of the chemical stability studies on SLA and MNT under an atmosphere of carbon dioxide saturated with moisture are given in Table 2 The variation of the lead azide content or the mercury content of MNT in the two samples with storage time is depicted in Fig. 2 It can be inferred that no discernable change occurred in the Hg content of MNT even after storage for 90 days whereas lead azide content of SLA decreased to about half within 50 days (Table 4).

The comparative stability of the two compounds (MNT and SLA) under similar conditions was also monitored by IR spectroscopy. The IR spectra of the samples withdrawn at regular intervals are presented in Fig. 3 and Fig. 4 The intensities of the absorptions of MNT withdrawn at different intervals did not exhibit appreciable change, whereas in case of SLA, a considerable decrease in the intensities of
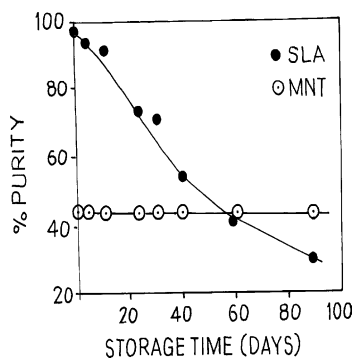
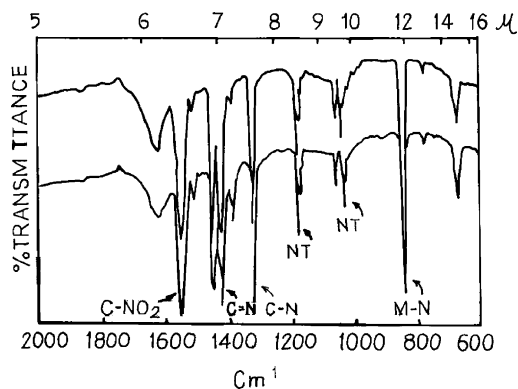Fig. 2. Effect of carbon dioxide atmosphere on MNT and SLA.

Fig. 3. IR spectra of fresh and stored samples of MNT.

absorption corresponding to azide group (4.9 μm) was observed. The appearance of absorption bands with increasing intensities observed at 7.2, 12 and 15 μm may be attributed to the formation of the carbonates. It is also worth to mention an interesting observation that IR spectra (Fig. 3) of MNT stored in an aluminum dish for 5 and 10 years superimposed over the IR spectra of freshly prepared compounds. Compatibility of SLA and MNT with copper was also assessed by visually observing change in color of copper foil having sample placed on it in desiccators. In case of MNT, no visual change in the copper foil was observed even after 90 days, whereas considerable blackening of the copper foil was visible in case of SLA within 50 days indicating the greater compatibility of MNT with copper under these conditions. The blackening of the copper foil housing SLA is indicative of the formation of sensitive copper azide due to the reaction of hydrazoic acid liberated from the decomposition of latter (SLA) with Cu. These results bring out that MNT is relatively more stable than SLA.

The higher stability of MNT in carbon dioxide atmosphere may be due to higher ionization potential (IP) [34] of mercury (10.4 eV) than that of lead (7.4 eV). Further, higher dispersal of π electronic density in the aromatic nitrotetrazole ring compared to that in azide group of SLA is also anticipated to confer higher stability on the MNT.

A highly strained molecular configuration of the azide ions in asymmetric arrangement around lead atom, may also contribute towards the possibility of its reaction with the moieties capable of forming less strained structures.

3.6. Sensitivity to mechanical stimuli

The results of the percussion and friction sensitivity studies are presented in Table 3 The height for 50% probability of percussion of MNT using a ball of 3/4″ diameter was 12 in. whereas SLA gave corresponding value of 14.5″ with the ball of 7/8″ diameter. These results bring out that MNT is more sensitive to percussion than lead azide. The impact sensitivity values reported by Redman and Spear [21,35] using ball and disc method were in the range of 4–8 cm for the detonation of all MNT samples. Interestingly, the fric-
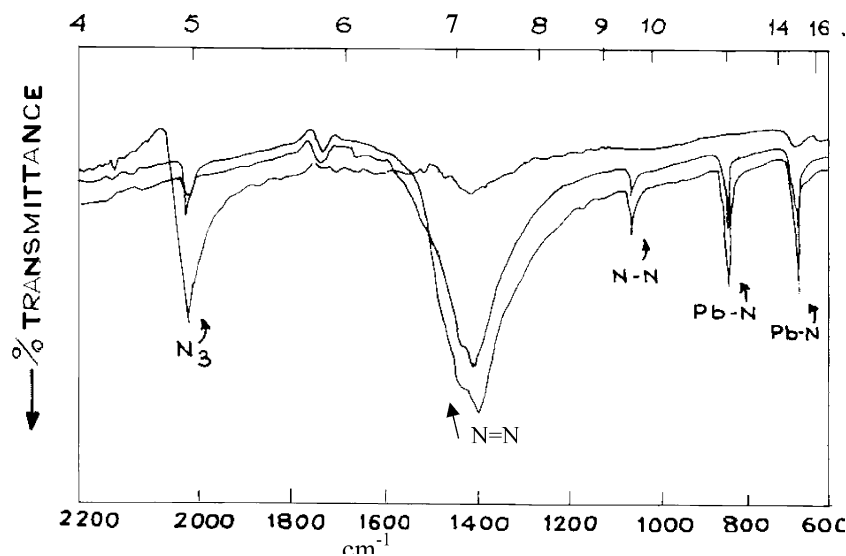
Fig. 4. IR spectra of fresh and stored samples of SLA.

tion test results suggest its less sensitivity (insensitive up to 500 g) in comparison to SLA (insensitive up to 30 g).

3.7. Performance evaluation studies

MNT (100/200/300 mg) pressed in detonator no. 27, punctured hole on witness plate of dia equivalent to that caused by standard ASA composition (9 mm). A combination of MNT (350 mg) with tetryl/PETN (550 mg) pressed in the stem of detonator no. 27 also punched hole (9–10 mm) on witness plate equivalent to that in the case for standard detonator no. 27, establishing that MNT is capable of transmitting shock waves through the booster charge. The reported performance evaluation [21,36] of MNT in copper ICI no. 8 flat detonator tubes (initiated electrically, 18 V capacitor) mentions the crater of the order of 0.48–0.58 mm on aluminium witness block.

4. Conclusion

The method for the preparation of mercury salt of 5-nitrotetrazole in free flowing form with bulk density of 1.5 g/cm^3 has been established on the lab scale by incorporating cephol as dopant. Thermal studies and percussion test results reveal higher sensitivity of MNT than SLA, whereas it was found less sensitive to friction stimuli. The study bring out that MNT is superior to SLA in terms of chemical stability and compatibility. The limited performance evaluation of MNT alone as well as in combination with secondary explosives in detonator no. 27 indicated its potential for detonator applications. The newly generated data on the thermolysis, sensitivity characteristics, chemical stability aspects and the performance evaluation can enrich the existing scanty technical data base of MNT to assess its suitability as a possible replacement for service lead azide.

Acknowledgements

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