Defence Science Journal, Vol. 52, No. 2, April 2002, pp. 135-146

# Hexanitrohexaazaisowurtzitane or CL-20 in India: Synthesis and Characterisation

A.K. Sikder , Nirmala Sikder, B.R. Gandhe, J.P. Agrawal and Haridwar Singh
*High Energy Materials Research Laboratory, Pune*- 411 021

## ABSTRACT

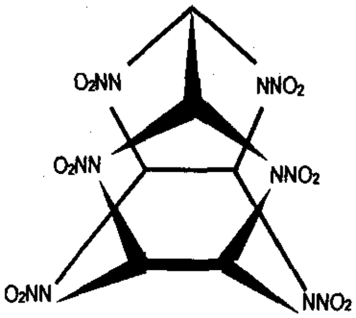
Hexanitrohexaazaisowurtzitane (HNIW) more commonly called CL-20, the highest density and the most powerful real world explosive, has been made with very high yield and a high product purity. CL-20 is thoroughly characterised by spectral data (IR, NMR and mass) along with electron spectroscopy for chemical analysis (ESCA) and X-ray diffractogram. Furthermore, small-scale sensitivity tests have also been carried out.

**Keywords:** Hexanitrohexaazaisowurtzitane, HNIW, CL-20, synthesis, explosive, characterisation, ESCA, electron spectroscopy, chemical analysis

## 1. INTRODUCTION

Current high performance, minimum-signature propellants, such as cyclotetramethylenetetranitramine (HMX), which are detonable (hazard class 1.1), use high levels of nitramines. Less-sensitive, minimum-signature propellants using other energetic solids have been formulated, but these suffer from reduced performance.

For many years, the three Services have been challenged to replace these sensitive, high energy propellants with equally high energy, less-sensitive formulations that would perform over a wide range of temperature. In 1987, a major scientific breakthrough came when a China Lake researcher, Nielsen synthesised the energetic solid hexanitro hexaazaisowurtzitane (HNIW)[1] more commonly called CL-20. CL-20 belongs to the family of polycyclic-caged nitramines, and is superior to HMX or RDX wrt higher crystal density, higher heat of formation and a better oxidiser-to-fuel ratio[2] (Table 1).



**Caged structure of CL-20**

Because of this high chemical energy, propellants and explosives using CL-20 deliver superior performance (up to 14 % more than HMX) over conventional high energy propellants and explosives[3-5]. On the other hand, propellant formulations containing CL-20 have the potential for remarkable stability, resistance to external stimuli (insensitive, hazard class 1.3) and burns cleanly, making it more environmental-friendly as well as reducing a missile's plume signature without encountering combustion stability problems[6-8]. Hence,

Revised 19 December 2001

**nitrohexaazaisowurtzitane (HNIW) or CL-20**

| HNIW | Properties |
|---|---|
| Melting point | : 228 °C ( Decomposition with explosion without a liquid phase) |
| *VOD | : 9.4 km/s |
| *Density | : 2.04 g/cm$^3$ |
| *Pc-j | : 419 kbar |
| *$\Delta H_f$ | : + 99.3 kcal/mol |
| *OB (%) | : −10.95 |
| Particle size | : 11 μm |
| Drop Ht. ( 50 % explosion) | : 22 cm |
| Friction ( insensitive up to) | : 14.4 kg |
| F of I | : 33 (CE, 76) |
| *T*(decomp.), DSC | : 220 °C |

* Reported values[2]

CL-20 displays extraordinary attributes and has been described as the world's most powerful non-nuclear explosive and is the greatest promise for major technological advances in future weapon systems.

CL-20 is polymorphic having four stable polymorphs* (α, β, ϒ and ε) which are known to exist at ambient conditions. The ε-polymorph has higher symmetry, thus morphologically stable at room temperature and has the highest density (2.04 g/cm$^3$) than the other three polymorphs[9]. But the application of ε-HNIW in energetic formulations (eg as a substitute for RDX or HMX) requires reliable information on its thermal and energetic properties.

In the years since the original elegant route of Nielsen, dramatic improvements[10-14] have been made for the synthesis of CL-20. These parallel routes have resulted the intermediates that can be completely nitrated with standard nitrating agents. However, the synthetic strategies and the experimental conditions for making this mighty molecule are still well guarded in literature.

able to synthesise CL-20 in a small-scale laboratory method and the results on the synthesis and characterisation of the HNIW or CL-20 have been reported here.

## 2. EXPERIMENTAL PROCEDURE

Melting points were determined on open capillary and are not corrected. The IR spectra were recorded on Perkin-Elmer infrared spectrometer using *KBr* matrix. $^1H$-NMR and $^{13}C$-NMR spectra were recorded on Brucker 300 MHz instrument, model WG-300 and chemical shifts were recorded in δ units (parts per million) with reference to tetramethylsilane (TMS) as internal standard. Elemental analyses were performed on Carlo-Erba elemental analyser EA-1108. Mass spectra were determined by electron ionisation at 70 eV on JEOL-DS mass spectrometer.

Differential thermal analysis (DTA) was recorded on the micro-DTA apparatus fabricated in the laboratory by heating 10 mg of sample at 10 °C/min in the presence of static air. The impact sensitivity was determined by fall hammer method using 2 kg drop weight and friction sensitivity was determined on Julius Peter's apparatus by following standard methods[15].

### 2.1 Materials

Unless otherwise stated, all common reagents and solvents were used as supplied from commercial sources without further purification. Glyoxal and benzylamine were supplied by E. Merck (India). Formic acid, acetonitrile, hexane, acetone, acetic anhydride, chloroform and ethyl acetate were purchased. Pearlman's palladium hydroxide on charcoal catalyst, bromobenzene and $BF_4$-based nitrating agents, such as $NOBF_4$ and $NO_2BF_4$ were used as obtained.

### 2.2 Synthesis

#### 2.2.1 *Preparation of* HBIW

Preparation of 2,4,6,8,10,12-hexabenzyl-2,4,6,8,10,12-hexaazatetracyclo- $[5.5.0.0^{5,9}.0^{3,11}]$ dodecane (2,4,6,8,10,12-hexabenzyl -2,4,6,8,10,12-hexaazaisowurtzitane) (HBIW) 1 was prepared by the method as described by Niesen[16], *et al.* and was recrystallised

---

* Polynitramines are powerful explosives and have to be handled with care. All standard explosive safety procedures were observed in the experimental operations involving these materials.

yield, 80-83 %, mp, 151-154 °C. The compound was homogeneous on silica gel thin layer chromatographic plate, *Rf*, 0.65 (hexane: ethylacetate, 6:4) and the purity was further confirmed by HPLC (Rt, 4.6 min; solvent system, *MeOH*: triple distilled water, 61:39; flow rate, 1.5 ml/min; UV, 298 nm).

IR (*KBr*) shows the absence of *NH* and *C=O* bands. The *CH* bands are found at 3024 $cm^{-1}$, 2832 $cm^{-1}$, 2750 $cm^{-1}$. $^1H$-NMR($CDCl_3$/TMS) δ ppm : 7.20-7.24 (s, Ph), 3.57 (s, 2*H*,-*CH*-),4.03(s, 4*H*, -*CH*-), 4.09 ( s, 8*H*, -$CH_2$), 4.16 (s, 4 *H*, -$CH_2$-).

Mass spectrum (EI), m/e ( rel. Int.) 70 eV: 708(M+, 30 %), 617(100 %), 525(15 %), 435(20 %), 236, 91 (30 %).

Analysis calculated for $C_{48}H_{48}N_6$ ; *C*, 81.32 ; *H*, 6.83 ; *N*, 11.86; Found : *C*, 81.49 ; *H*, 7.01 ; *N*, 11.78.

Preparation of 4,10-dibenzyl-2,6,8,12-tetraacetyl-2,4,6,8,10,12-hexaazaisowurtzitane (4,10-dibenzyl-2,6,8,12-tetraacetyl 2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.$0^{5,9}$.$0^{3,11}$] dodecane-2 was synthesised as described[1], and was recrystallised from acetonitrile afforded small colourless prism; yield, 63.1 per cent, mp, 306–310 °C, DTA: 315 °C (endotherm).

IR (*KBr*) γ $cm^{-1}$ : 3026, 1652, 1410, 1306, 1132, 706. $^1H$-NMR ($CF_3COOD$) δ ppm : 7.25–7.6 (m, 10*H*, Ph), 6.8 (*Br*, 2*H*, *CH*), 5.4–5.8 (*Br*, 4*H*, *CH*), 4.2–4.4 (*Br*, 4*H*, $CH_2$), 2.0 – 2.65 (*Br*, m, 12*H*, $CH_3$). 57.11 (2*C*, $CH_2$), 70.34 (4*C*, *CH*), 77.4 (2*C*, *CH*), 135.3 (6*C*, Ph), 173 (4*C*, *CO* $CH_3$).

Analysis calculated for $C_{28}H_{32}N_6O_4$ (Mol. Wt. 516) : *C*, 65.1; *H*, 6.20; *N*, 16.3; Found : *C*, 64.15; *H*, 6.24 ; *N*, 16.27.

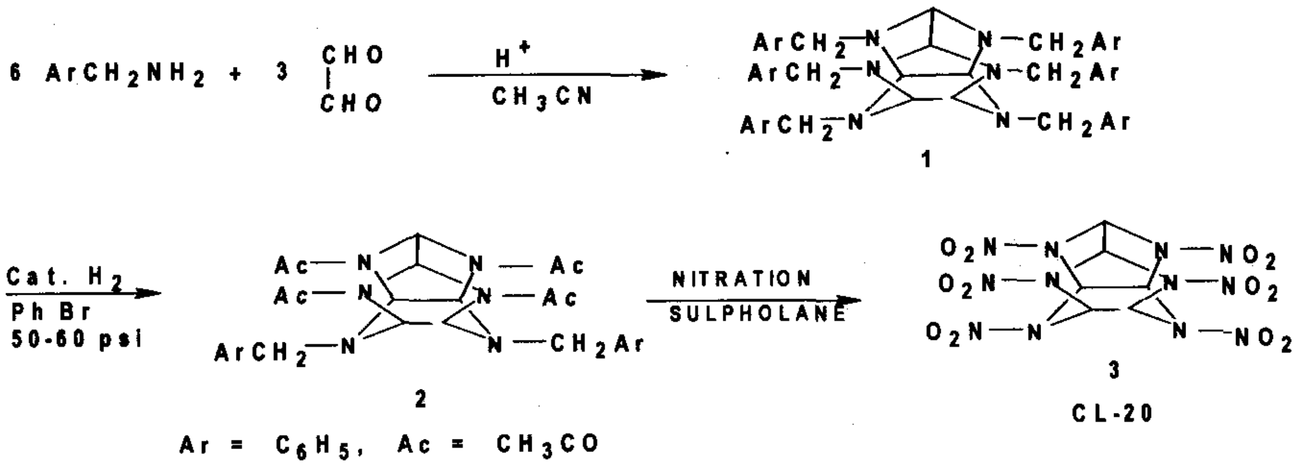
2.2.2 *Preparation of* HNIW

Preparation of 2,4,6,8,10,12-hexanitro-2,4,6, 8,10,12 hexaazatetracyclo (5.5.0.0,$^{5,9}$ 0,$^{3,11}$) dodecane or 2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaaza isowurtzitane (hexanitrohexaazaisowutzitane) (HNIW or Cl-20) 3 was prepared by adopting the method of Nielsen[1], obtained as white solid ; yield, 90-97 per cent ; recrystallised from ethylacetate followed by precipitation with chloroform (recovery 90 %), mp, 228 °C (explodes with brownish fumes); DTA, 220 °C (d). Elemental analysis calculated for $C_6H_6N_{12}O_{12}$ (Mol. Wt. 438): *C*, 16.44 ; *H*, 1.36 ; *N*, 38.30. Found: *C*, 17.03; *H*, 1.0; *N*, 37.40.

## 3. RESULTS & DISCUSSION

### 3.1 Synthesis of CL-20

The route adopted for the preparation of CL-20 is depicted in Scheme I. The precursor, hexabenzylhexaazaisowurtzitane 1 (Scheme I) or HBIW is a well-known material and easily prepared by acid catalysed condensation of benzylamine with aqueous glyoxal (40 %) with 80 per cent yield and product purity more than 95 per cent. Different batches of HBIW have been prepared using a variety of acid catalysts, such as formic acid, acetic acid and sulphuric acid. The reaction appeared to be rapid and



Scheme I

nearly complete when formic acid was used as catalyst in acetonitrile maintaining the p*H* of the reaction mixture approximately 9.5.

The replacement of benzyl groups by nitro groups is achieved by subjecting HBIW to a reductive acetylation strategy in acetic anhydride under atmospheric pressure of hydrogen to replace four of six benzyl groups to give tetraacetyldibenzyl hexaazaisowurtzitane 2 (Scheme I) with 65–70 per cent yield. The production of 2 is critically dependant upon the quantity and the activity of the catalyst, and the concentration of the acetylating agent. Nitrosation of 2 (Scheme I) results in the formation of tetraacetyldinitroso hexaazaisowurtzitane which was further nitrolysed *in-situ* to obtain CL-20 or HNIW. The protecting group used to replace the benzyl group should be readily replaceable by the nitro group. In this process, CL-20 was prepared in four batches in the laboratory at a 10 g level batch size with high yield (90–96 %) and excellent purity. The compound was homogeneous on silica gel thin layer chromatographic plate, *Rf*, 0.8 (heptane/hexane: ethyl acetate, 6:4) and the purity ( > 95 %) was confirmed by high performance liquid chromatography (HPLC).

## 3.2 Characterisation of CL-20

### 3.2.1 *Determination of Purity by Chromatography*

The product obtained from the reaction mixture was purified through a short silica gel column with the eluent, ethylacetate; followed by thin layer chromatography (TLC, silica gel) with heptane/hexane:ethylacetate (6:4) as solvent system. HPLC was used to confirm further purity of CL-20. The mobile phase comprised acetonitrile-water-tetrahydrofuran (40:50:10) at a flow rate 1 ml/min and injection volume 20 µl in $C_{18}$ µBP columns with UV detector at 232 nm. The total analysis time was less than 15 min. The retention time of CL-20 under this condition was found to be 11.72 min with purity more than 95 per cent (Fig. 1 ).

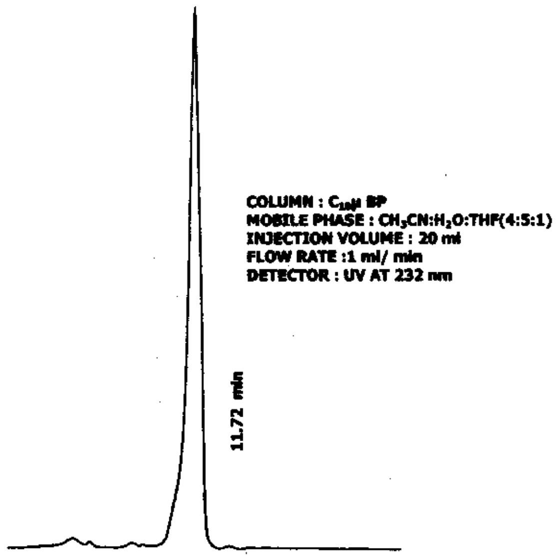


**Figure 1. High performance liquid chromatography of CL-20.**

### 3.2.2 *Vibrational Spectroscopy*

IR spectra were recorded on Perkin-Elmer FTIR usually with *KBr* as pellets. IR spectrum of CL-20 (Fig. 2) is dominated by the strong absorptions of the asymmetric and symmetric stretching vibration of $NO_2$ groups at 1612–1557 $cm^{-1}$ and 1332–1266 $cm^{-1}$, respectively. The cage ring *CH* stretchings appear at 3034–2924 $cm^{-1}$. The other characteristic bands at 1168 $cm^{-1}$, 1032 $cm^{-1}$, 950 $cm^{-1}$, 882 $cm^{-1}$ and 750 $cm^{-1}$ correspond to the cyclic nitramine structure (*N-N* stretching, *N-O* str., *C-N* bond, etc). The absence of *NH* and *CO* bonds further confirms the conversion of acetyl group to nitramine groups.

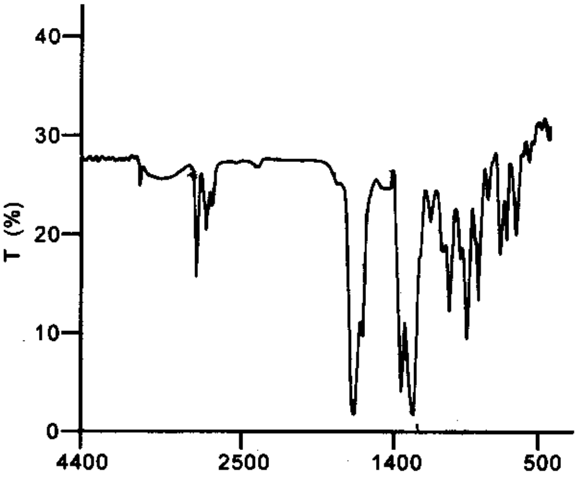


**Figure 2. IR (*KBr*) spectrum of HNIW (CL-20)**

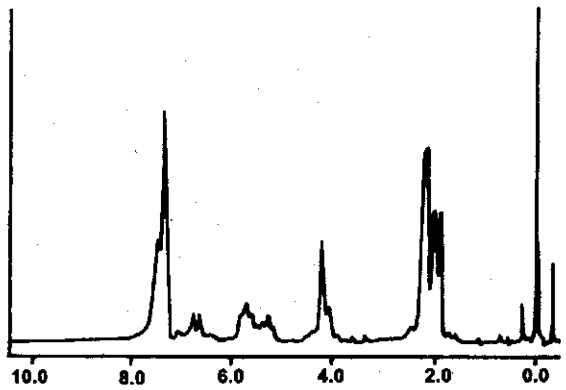


**Figure 3. $^1H$-NMR spectrum (300 MHz) of 2 in $d_1$-TFA.**

### 3.2.3 NMR *Spectroscopy*

The $^1H$-NMR spectrum of 2 (Scheme I) (solvent, $CF_3COOD$) (Fig. 3) causes the acetyl methyl groups to appear as a group of broad singlets at δ 1.8–2.7. The six hydrogen atoms of the ring methine (-*CH*-) protons absorb as a series of non-integral multiplets at δ 5.2–7.0 in the ratio 2*H*:4*H* . The absorption of methylene protons of the benzyl groups appears as broad multiplets at δ 3.8–4.5.

In addition, 2 (Scheme I) exhibits a broad multiplets at δ 7.2–7.8 for aromatic proton absorption. The $^{13}C$-NMR spectrum (Fig. 4) correlates well with the structure. The acetyl carbonyl carbons and methyl carbons appeared at δ 173 and δ 21.3, respectively. The ring cage *CH* carbons exhibit at δ 70.34 and δ 77.4, while benzyl $CH_2$ carbons resonate at δ 57.11 and that of phenyl carbons at δ 135.3, respectively.

The $^1H$ and $^{13}C$ spectra (Figs 5 and 6) of HNIW were recorded in deuterated acetone at 30 °C with TMS as internal standard. The cage ring methine protons (-*CH*-) displayed two sharp singlets at δ 8.34 and δ 8.21, respectively in the ratio 2:1. The $^{13}C$-NMR spectrum shows two sharp singlets at δ 75.08 (2*C*) and δ 72.13 (4*C*), respectively. Satisfactory protons integrals were obtained in all cases and the values reported herein are in excellent agreement with the values found in the literature.

### 3.2.4 *Mass Spectrometry*

The electron impact mass spectrum (EIMS) was recorded on JOEL-DS double focussing mass spectrophotometer at 70 eV using direct insertion

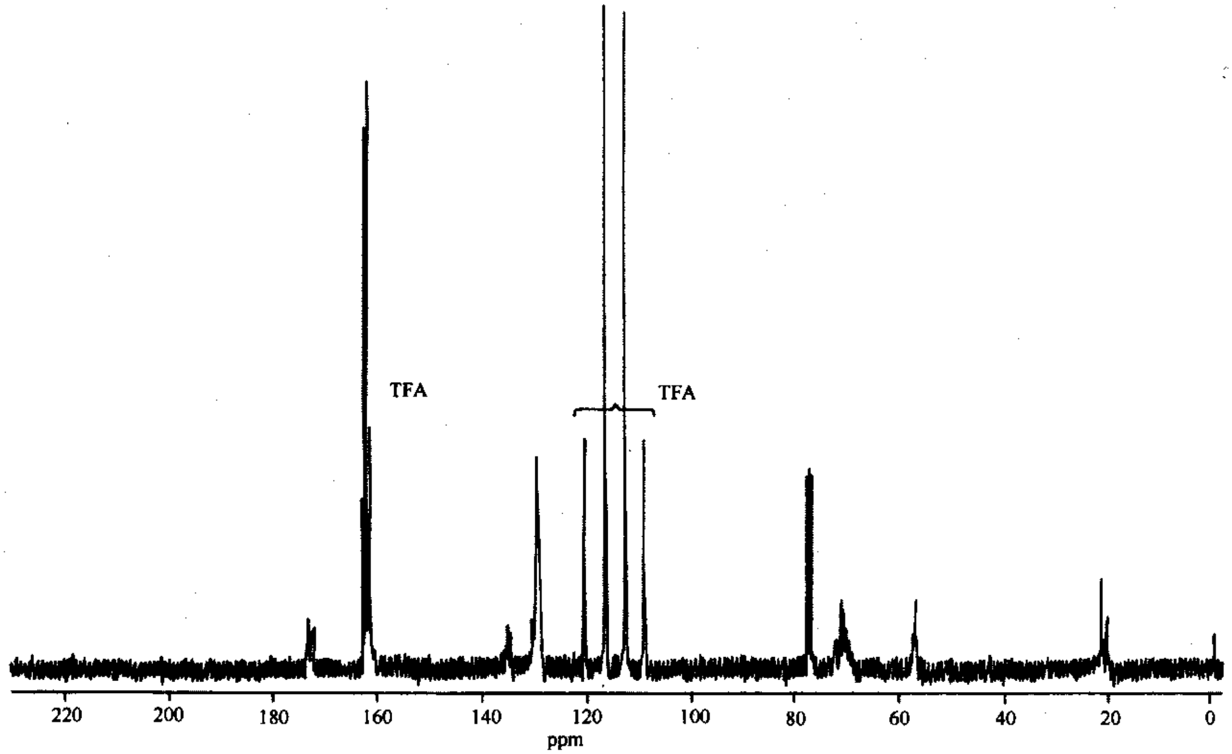


**Figure 4. $^{13}C$–NMR spectrum in $d_1$–TFA of TADB isowurtzitane, 2**

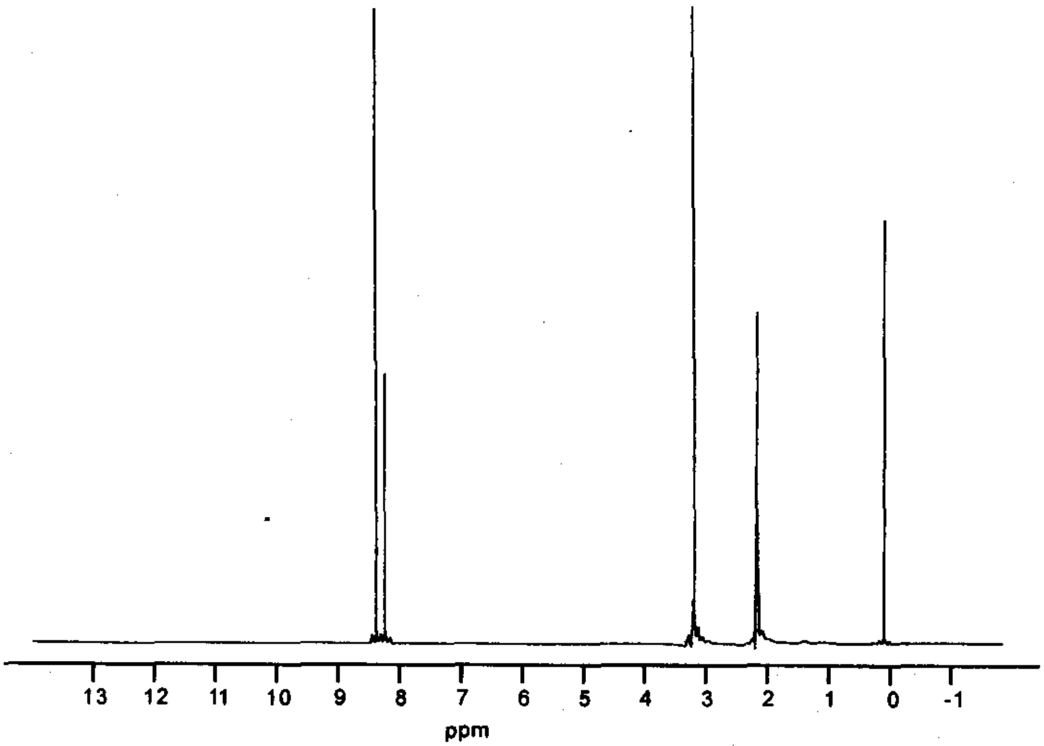


**Figure 5.** $^{1}H$-NMR spectrum of CL-20 (solvent : acetone–$d_6$)

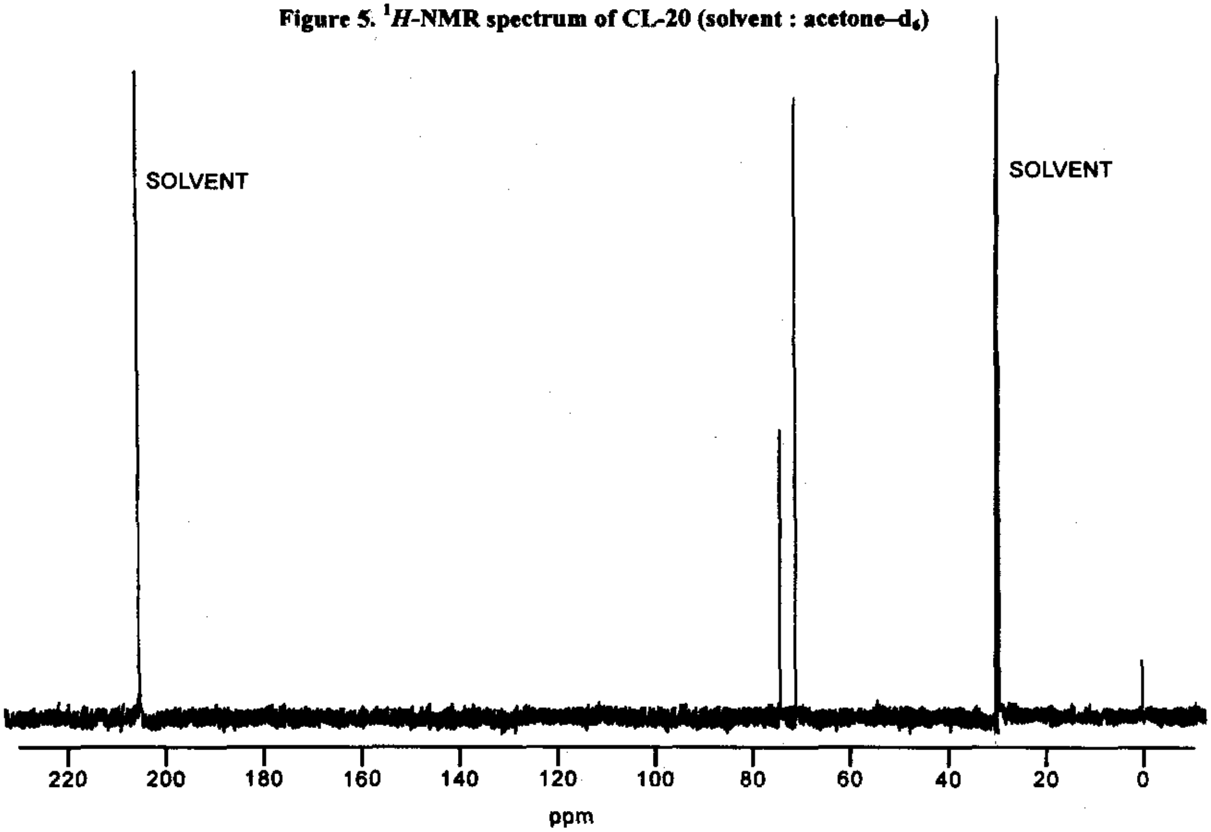


**Figure 6.** $^{13}C$-NMR spectrum of CL-20 (solvent : $CD_3COCD_3$)

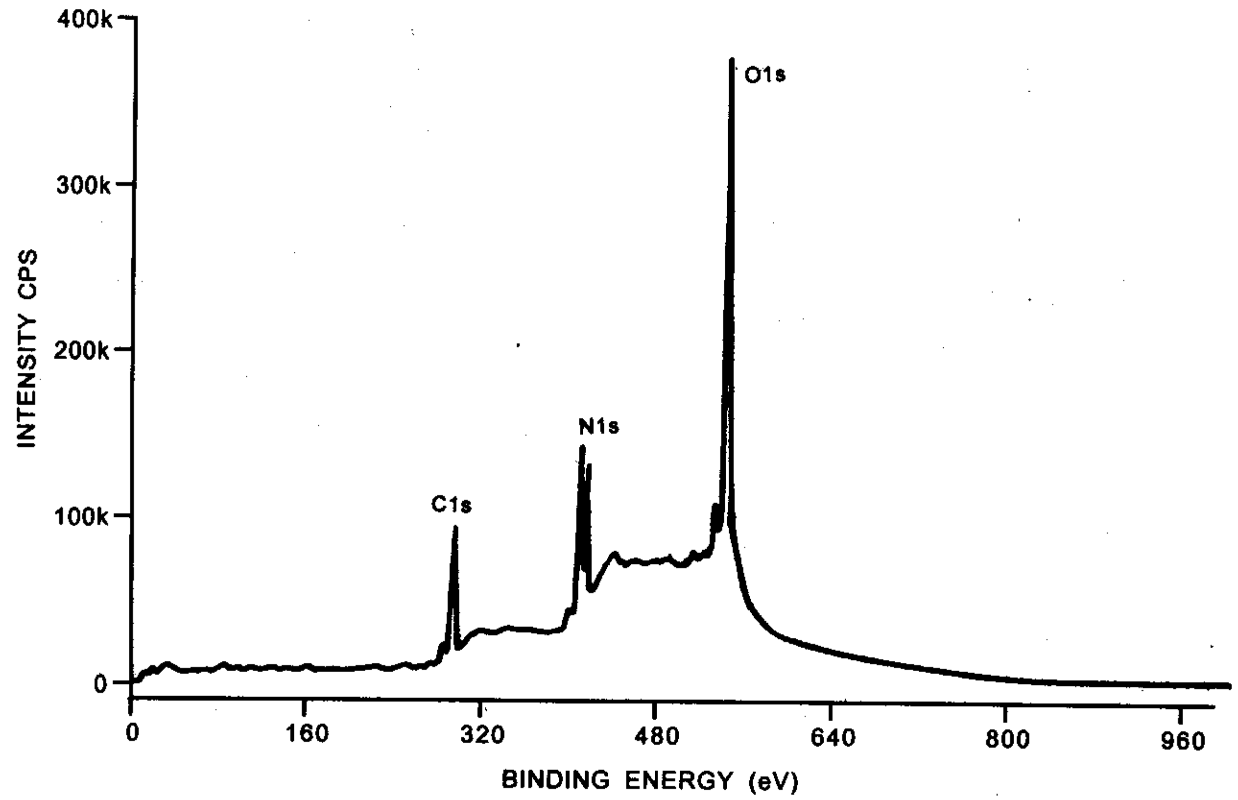
Table 2. XPS results of CL-20

| Atom | Band number | Binding energy (eV) | Corresponding atom |
|---|---|---|---|
| Carbon 1s | 1 | 295.77 | Carbon |
| Nitrogen 1s | 2 | 415.17 | Cage $N-N$ |
| | 3 | 409.34 | $\underset{\underset{O}{\downarrow}}{-N}=O$ |
| Oxygen 1s | 4 | 541.65 | $-NO_2$ |

probe. The spectrum showed no molecular ion peak but nevertheless, some interesting ions which are of diagnostic value for structural information were observed at m/z 54 ($C_2H_2N_2$), m/z 81, 82 ($C_3H_3N_3$/$C_3H_4N_3$), m/z 108 ($C_4H_4N_4$), 127, 135, 150, 178, 209, 213 ($C_5H_5N_6O_4$), 224, etc. In the low mass region it is seen that m/z 46 dominates the spectrum (100 % base peak). The chemical ionisation mass spectrum (CIMS) in methane as reagent gas (CIMS, $CH_4$) showed m/z 439 (M + 1) along with m/z 342, 301 and 209 which further confirms the structure of the compound.

### 3.2.5 *Electron Spectroscopy for Chemical Analysis*

Electron spectroscopy for chemical analysis (ESCA) was used to determine the structure of CL-20 along with IR, NMR and mass spectrometry. The XPS spectra of CL-20 has not been found in the literature. The spectra were measured with a Philips PW 5300 ESCA instrument. The intensity versus binding energy (eV) of different atoms ($C$, $N$, $O$) of CL-20 are represented in Fig. 7, and the results are given in Table 2.



The binding energy of nitrogen atom in isowurtzitane ring is found to be different from that of nitrogen atom in $NO_2$ group, and the difference of binding energies of two nitrogen atoms (endocyclic *N* atom and nitro group *N* atom) is about 6 eV.

### 3.2.6 *X-Ray Diffraction Analysis*

Philips automatic X-ray diffractometer (model, PW1710) using *Cu*-anode operated at 40 kV and 30 mA along with the wide angle X-ray diffraction (WAXRD) are used for diffraction studies on powder sample of CL-20. The WAXRD patterns of 47 peaks as a function of the Bragg angle, 2θ taken at wavelength $X\alpha_1$ = 1.540 Å and 1.544 Å of sample is shown in Fig. 8. The d-spacings calculated from angular positions the reflections and from reflection broadening (2*θ*) are also obtained in the tabulated form. The spectrum of compound shows a typical amorphous structure with a tail towards large value of 2*θ*

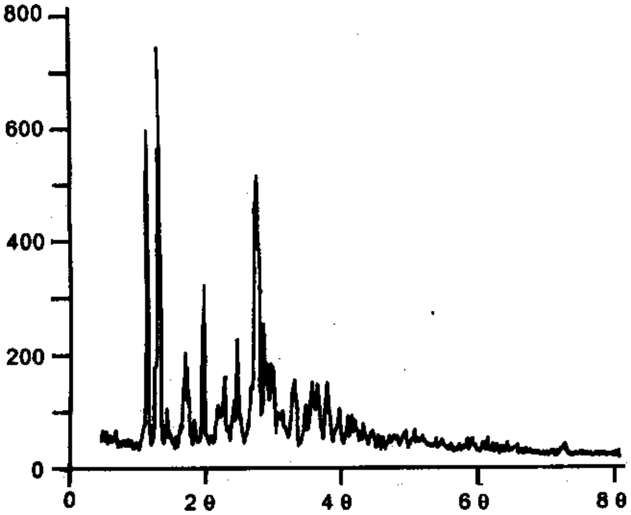


**Figure 8. X-ray diffractogram (counts versus 2*θ*) of CL-20.**

### 3.2.7 *Scanning Electron Microscopy*

Scanning electron microscopy (SEM) of CL-20 was carried out on Philips model SEM-XL30 instrument. Four magnifications of SEM photographs are presented in Fig. 9. All the agglomerated micrographs have amorphous, sub-micron and low tap density of nano-particle crystals. However, on 4000-time magnification, a cluster of layers of small needle like crystals are observed.

### 3.2.8 *Thermal Analysis*

The thermal properties of CL-20 have been determined with combined thermogravimetry (TG)/ DTA and differential scanning calorimetry (DSC). The DTA was recorded by taking 10 mg of sample at 10 °C/min. in the presence of static air. The DTA results show that there is a sharp exotherm ($T_{max}$) at 220 °C, which corresponds to the first-stage degradation.

Perkin-Elmer DSC-7 was employed for DSC studies. For this, 2-3 mg of sample was crimped in an aluminium cup which was heated against blank cup at different heating rates, i.e., 5 °C/min, 7.5 °C/min, 10 °C/min, 12.5 °C/min, 15 °C/min, 20 °C/min and 25° C $min^{-1}$. The data in Table 3 show that it exhibits exothermic peaks, increasing at different heating rates and the area under the curve decreases with increasing heating rate. It seems that chemical reactions take place with structural changes at higher heating rates.

**Table 3. Maximum exotherm responds as a function of 4.8 scan speed and activation energy of CL-20**

| Heating rate (β)*, (°C/min) | $T_m = t + 273$ (K) | log β | ln $(\beta/T_m^2)$ | Activation energy (kcal/mole) Ozawa | Activation energy (kcal/mole) Kissinger |
|---|---|---|---|---|---|
| 5.0 | 510.77 | 0.6989 | -10.86 | 36.43 | 38.58 |
| 7.5 | 516.79 | 0.8750 | -10.48 | | |
| 10.0 | 519.77 | 1.0000 | -10.20 | | |
| 12.5 | 522.21 | 1.0969 | -9.99 | | |
| 15.0 | 524.87 | 1.1760 | -9.81 | | |
| 20.0 | 524.07 | 1.3010 | -9.52 | | |
| 25.0 | 526.60 | 1.3979 | -9.31 | | |

* Heating rate is denoted by the symbol β. The values of peak maxima, $T_m$, log β and ln $(\beta/T_m^2)$ correspond to the different heating rates

The reaction becomes violent at 251 °C (20 °C/min) and the reaction proceeds with the pronounced self-accelaration. The activation energy of decomposition was calculated following the methods of Kissinger and Ozawa, and was found to be 36.43 kcal/mole and 38.58 kcal/mole, respectively which is in excellent agreement with the reported values of ε-polymorphs of CL-20 (41.0 ± 6.2 kcal/mole)[17,18]. As compared with RDX and HMX, ε-CL-20 exhibits slightly low activation energy.

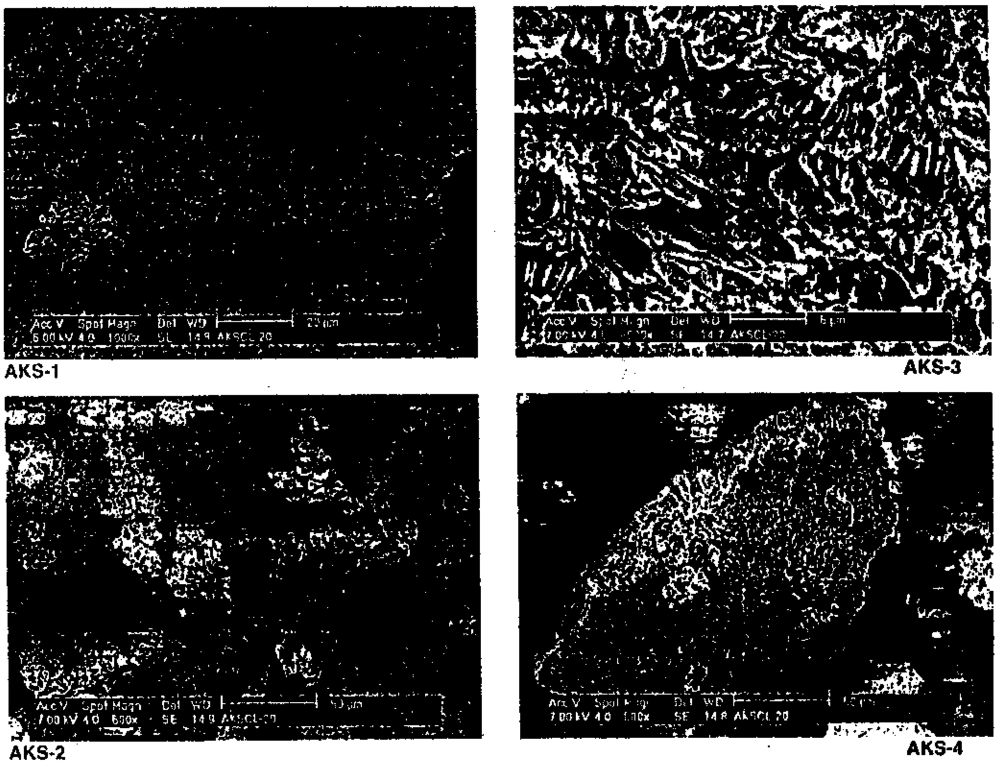


**Figure 9. Four magnifications of SEM photographs of powder CL-20 (AKS-1: Fibrous porous layer on 1000-time magnification; AKS-2: Amorphous nano particles ( 10-40 μm) of irregular shape clusters of CL-20; AKS-3: 4000-times magnification shows layers of needle crystals of cauliflower structures; AKS-4:500-time magnification of a single amorphous lump of particle size 500-600 nm).**

This indicates that ε-HNIW is slightly more thermally unstable than conventional nitramine explosives.

Figure 10 shows DTA, TG and DTA curves of CL-20. The TG curve reveals one-stage sharp decomposition of HNIW at 215 °C with weight loss of 72 per cent after 20 min and in the subsequent stages, weight loss is very slow. The mass loss is found to be 12.5 per cent over the temperature range 215 °C to 580 °C. Thus, calculated weight loss due to overall reactions is approximately 85 per cent up to 580 °C and beyond 580 °C, HNIW produces approximately 15 per cent of solid residue.

### 3.2.9 *Sensitivity Tests*

The powder sample of HNIW was subjected to impact sensitivity and friction sensitivity tests. The results (Table 1) show that CL-20 is sensitive to impact ($h_{50}$), 22 cm; F of I, 33 ( CE, 76 ) and friction, insensitive up to 14.4 kg as compared to HMX and RDX ( F of I, 80 ). These values are very close to the reported values[18,19] of ε-polymorph of CL-20. This high sensitivity of ε-polymorph is probably due to the less favourable particle shape (more irregular).

## 4. CONCLUSION

HNIW was prepared in four batches on a small scale in the laboratory with high yield (96 %) and high purity (> 95 %). Column chromatography and HPLC techniques were used to determine the purity of HNIW. CL-20 was characterised satisfactorily by spectral data (IR, NMR and mass) along with ESCA, SEM and X-ray diffraction techniques.

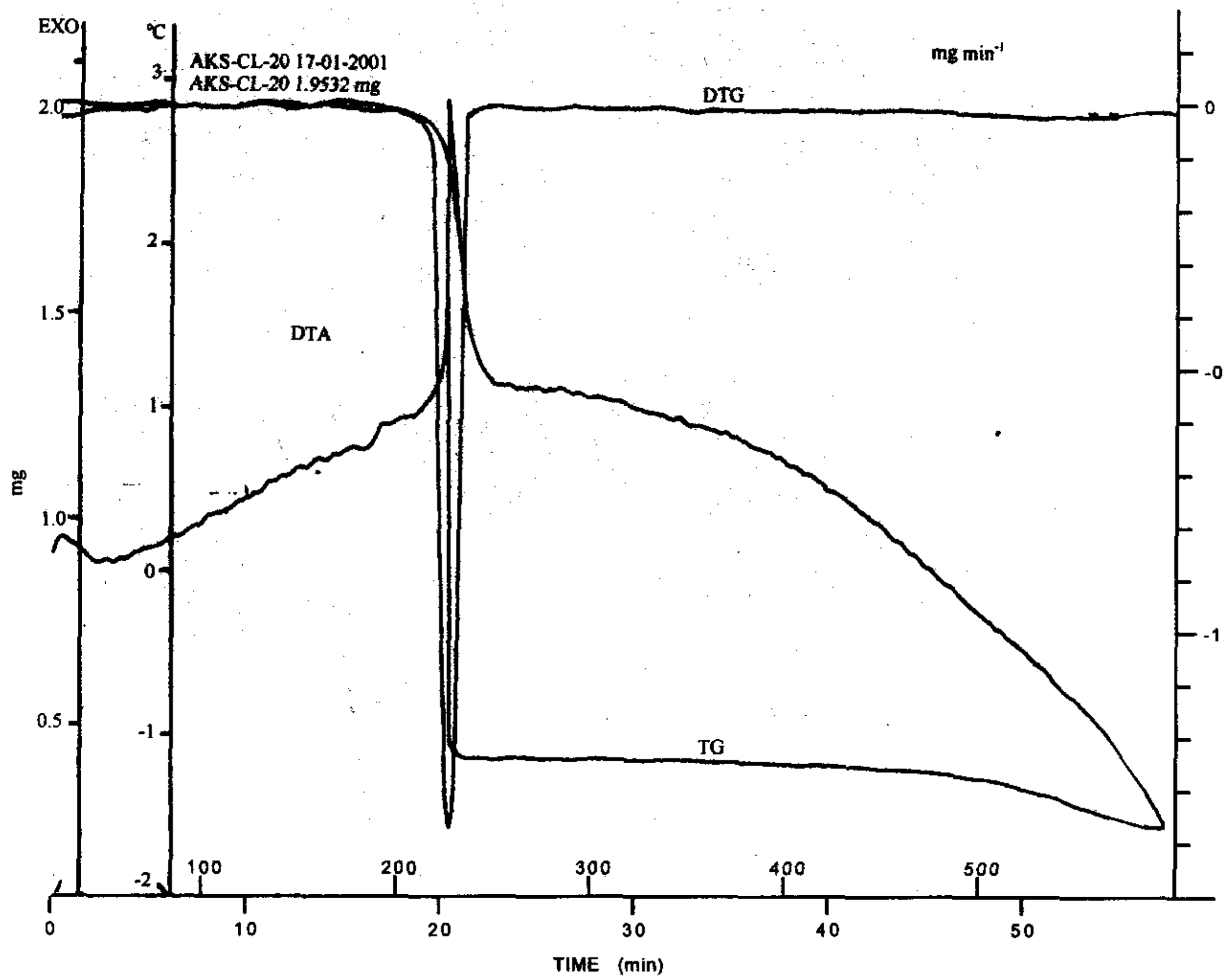


**Figure 10. DTA/DTG of CL-20**

## REFERENCES

1. Nielsen, A.T. US Dept of Navy, US Patent Office Application Case No.70631, 24 June 1987.
2. Olah, G.A, & Squre, D.K. Chemistry of energetic materials. Academic Press Inc., San Diego, USA, 1991. p.94.
3. Simpson, R.L. ; Urtiew, P.A.; Ornellas, D.L.; Moody, G.L.; Scribner, K.J & Hoffman, D.M. CL-20 performs exceeds that of HMX and its sensitivity is moderate. *Propell. Explo., Pyrotech.*, 1997, **22**, 249.
4. Borman, Stu. Advanced energetic materials emerge for military and space applications. *Chemical Engineering,* 1994, 18-21.
5. Bottaro, J.C. Recent advances in explosives and solid propellants. *Chemistry Industry,* 1996, 249-53.
6. Aerotech news and review. Development on CL-20. *J. Aeros. Def. Indust. News*, July 2000.
7. Golfier, M.; Graindorge, H.; Longevialle, Y. & Maee, H. New energetic materials and their applications in energetic materials. *In* 29th International Annual Conference of ICT, Karlsruhe, Federal Republic of Germany, June 30-July 03, 1998. pp. 3.

8. Braithwaite, P.C.; Hatch, R.L.; Lee, K. & Wardle, R.B. Devlopment of high performance CL-20 explosive formulations. *In* 29th International Annual Conference of ICT, Karlsruhe, Federal Republic of Germany, June 30- July 03, 1998. pp.4.

9. Yuanhe, H. ; Xingi, Z. & Wenhur, W. Investigation of structures and properties of HNIW. *In* Proceeding of the 3rd Symposium on Phrotechnics and Explosives. Beiging Institute, Beiging, China, 1995. pp.167.

10. Nielsen A.T. US Patent Office Application No. 253, 106, 30 September 1988.

11. Nielsen, A.T. Synthesis of caged nitramine explosive. *In* Joint Army, Navy, NASA, Air Force (JANNAF) Propulsion Meeting, San Diego, CA, 17 December 1987.

12. Kodama, T. Hexaquis (trimethyl-silylethyl-carbamyl) hexa azaiso-urutitanium (Urutitan). Japanese Patent No. 0632 1962, 14 May 1993.

13. Kasei, A. Hexaazaisowurtzitane derivatives and process for producing the same. Japanese Patent No. 0820 8655, 13 August 1996.

14. Bellamy, A. J. Polynitrohexaazaisowurtzitane derivatives related to hexanitroexaazaisowurtzitane (HNIW). 31st International Annual Conference of ICT, Karlsruhe, Fed. Rep. of Germany, 27-30 June 2000. pp.109/1-109/9.

15. Arvami, L. & Hutchinson, R. The sensitivity to impact and friction. *In* Energetic Materials, Vol. 2: Technology of the inorganic azide Plenum Press, New York, 1977. pp.111-59.

16. Nielsen, A.T.; Nissan, R.A. & Vanderah, D.J. Polyazapolycyclics by condensation of aldehydes and amines. *J. Org. Chem.*, 1990, **55**, 1459-466.

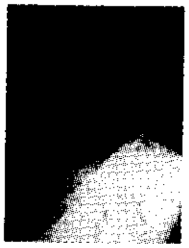
17. Larikova, A. Kinetics of thermal decomposition of hexanitro hexaazaisowurtzitane. *In* 29th International Annual Conference of ICT, Karlsruhe, Federal Republic of Germany, 27-30 June, 2000. pp. 64.

18. Foltz, M.F.; Clifford, L.; Garcia, C.F. & Nicols, A.L. III. The thermal stability of the polymorphs of hexanitrohexaazaisowurtzitane, part I. *Propell. Explos. Pyrotech.*, 1994, **19**, 133-145.

19. Ostmark, H.; Bergman, H. & Sjoberg, P. Sensitivity and spectroscopicproperties of the β and ε-polymorphs of HNIW. *In* Proceedings on Energetic Materials Technology, Arizona, 24 –27 September 1995. pp.75-81.

## Contributors

**Dr AK Sikder** joined DRDO at the Defence Research & Development Establishment (DRDE), Gwalior, in 1983 and continued his work till 1995 on high organophosphorus toxic compounds which are used as chemical warfare agents and their antidotes . From 1995, he has been working at the High Energy Materials Research Laboratory (HEMRL), Pune. His research work includes high energy materials in a wide spectrum of subjects of defence interest. He has published about 39 research papers in national/international journals.



**Dr (Mrs) Nirmala Sikder** obtained her PhD from Jiwaji University, Gwalior, in 1996. She joined DRDE, Gwalior, in 1985 and continued her research till 1996 on synthesis, characterisation and hydrolysis studies on toxic organophosphorus compounds. From 1996, she has been working at the HEMRL, Pune. Her research includes high energy materials. She has published more than 15 papers in national/international journals.