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Closed-Vessel and Thermal Studies on Triple-Base Gun Propellants Containing CL-20

C. N. Divekar,* R. R. Sanghavi,† U. R. Nair,‡ T. K. Chakraborthy,§ A. K. Sikder,¶ and Amarjit Singh**
High Energy Materials Research Laboratory, Pune 411 021, India

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This study was undertaken to see the effect of the replacement of picrite by 2,4,6,8,10,12-hexanitro–2,4,6,8,10,12-hexaazaisowurtzitane (CL-20) (5, 10, and 15%) in triple-base gun propellants. Experimental results of a closed-vessel test at a loading density of 0.2 g/cm^3 indicate improvement in energetics of the CL-20-incorporated compositions. The force-constant values obtained were 1046, 1071, and 1095 J/g for 5, 10, and 15% CL-20-based formulations, compared with 1028 J/g for conventional picrite composition as the control. Similar trends were observed for linear burning-rate coefficient β_1 values. The selected energetic plasticizers N-(n-butyl)-N-(2-nitroxyethyl) nitramine (Bu-NENA) and bis-2,2 dinitro propyl formal/acetal (BDNPF/A) were also evaluated for their effectiveness in CL-20-containing gun-propellant compositions. Thermal analysis was carried out using differential scanning calorimeter and thermogravimetric analyzer. Replacement of picrite by CL-20 resulted in an increase in ΔH values (by differential scanning calorimeter) compared with that of control composition. Weight-loss data (from thermogravimetric analyzer) showed a similar trend. The CL-20-based compositions were found relatively more sensitive to friction stimuli (12–17 kg) compared with control composition (19.2 kg).

I. Introduction

TO MEET the futuristic requirements of both the U.S. Army and U.S. Navy, researchers are working to develop new and improved gun propellants. One of the methods to achieve this objective is the incorporation of new energetic compounds that impart enhanced performance and reduced vulnerability to explosives and propellants.

Conventional triple-base gun propellants contain nitrocellulose (NC), nitroglycerine (NG), and picrite (NQ), along with other ingredients. This class of propellants is currently used for tank and field gun ammunition. They have several advantages such as low flame temperature, flash less combustion, low barrel erosion, long shelf life, etc. However, triple-base propellants have reached the saturation limit in terms of energy output and ballistic performance. Hence, further improvement in energy output of triple-base propellants is a challenging task. There is scope for enhancing the performance of triple-base propellants with respect to energy level by partial replacement of NQ by energetic oxidizers.

One of the methods currently employed for improving the ballistic performance of triple-base propellants is the use of cyclic nitramines such as 1,3,5-trinitro–1,3,5-triazacyclohexane (RDX) or 1,3,5,7-tetranitro–1,3,5,7-tetraazacyclooctane (HMX) as an energetic component [1–4]. Further research in this direction led to the use of high-energy dense materials such as 2,4,6,8,10,12-hexanitro–2,4,6,8,10,12-hexaazaisowurtzitane (CL-20 or HNIW) [5,6], as an alternative to RDX and HMX. Eisele and Menke [7] reported the probability of application of CL-20-based compositions in rocket motors for high-velocity ballistic/guided tactical missiles (high-velocity missile or hyperschall-flugkörper). Golfier et al. [8] reported that CL-20 propellants offer ~7% higher energy compared with corresponding RDX-based formulations and ~35–110% higher burning rates than HMX-based propellants. The triple-base gun propellants containing bis-azido methyl oxetane (BAMO)-azidomethyl methyl oxetane (AMMO), BAMO-nitratomethyl-methyl oxetane (NIMMO), bis-ethyl methyl oxetane (BEMO)-NMMO, CL-20, RDX, and 1,3,3-trinitroazetidine (TNAZ) are the futuristic energetic propellants. However, limited information on ballistic parameters of CL-20-based formulations is reported [9]. A patent [10] described desensitization of CL-20 compositions by use of energetic ingredients such as glycidyl azide polymer (GAP)/bis-2,2 dinitro propyl formal/acetal (BDNPF/A). Gore et al. [11] studied the effect of BDNPF/A on burning rates of propellants. Nair et al. [12] conducted studies on CL-20-based aluminized composite modified double-base (CMDB) propellants. They found that CL-20-based aluminized CMDB propellants offer burning rates superior to RDX incorporated propellants and are less vulnerable to friction stimuli than ammonium perchlorate (AP)-CMDB formulations. Mueller [13] described a CL-20-based gun propellant containing RDX, NC, and BDNPF/A, which gave a force constant of 1253 J/g at a combustion temperature of 3698 K with energy output of 4830 J/g. He concluded that the batch-processed (kneader and ram) propellant is safe to load in the closed vessel and gun simulator. However, details of percentage ingredients of the compositions are not reported. Haaland et al. [14] patented the process for the manufacture of high-performance gun propellant containing an energetic thermoplastic elastomeric binder from the group consisting of poly-NMMO, GAP, poly-BAMO, poly-AMMO, poly-NAMMO (15–30%), and high-energy oxidizers such as CL-20, RDX, HMX, 3-nitro–1,2,4-triazole-5–1 (NTO), NQ, 1,3,5-triamino–2,4,6-trinitrobenzene (TATB), TNAZ, and 70–85% ammonium dinitramide (ADN).

Harris et al. [15] recommended Hytrel (copolymer of polybutylene terephthalate-polyether glycol) and BAMO-AMMO copolymer-based CL-20-containing propellants as a superior alternative to M-30 composition. Wardle et al. [16] reported a higher impetus of oxetane-based CL-20 gun propellant (1297 J/g) compared with RDX propellant (1182 J/g).

Recently, energetic polymers and plasticizers (compounds with nitro, nitroso, fluoro, fluoronitro, azide, or fluoroanion groups) are reported for application in propellants [17]. Energetic plasticizers such as alkylnitrato ethylnitramines (methyl-NENA, ethyl-NENA, and butyl-NENA), nitrate esters, [NG, diethylene glycol dinitrate, triethylene glycol dinitrate, butane triol trinitrate, BDNPF/A, and GAP] have also been mentioned in a recent patent [18]. A solid rocket

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*Technical Officer B, Department of Gun Propellants.
†Scientist E, Deputy Director, Department of Gun Propellants.
‡Scientist D, Assistant Director, Department of Technical Services.
§Scientist F, Joint Director, Department of Gun Propellants.
¶Scientist F, Joint Director, Department of Energetic Materials.
**Scientist G, Associate Director.

Table 1 Theoretical thermochemical data of triple-base gun propellants containing CL-20[a]

Parameters	Picrite 40, CL-20:0	Picrite 35, CL-20:5	Picrite 30, CL-20:10	Picrite 25, CL-20:15
Force constant, J/g	1032	1062	1092	1138
Flame temperature, K	2937	3073	3211	3480
P_{max}, MPa	254	261	268	279
Covolume, cm^3/g	0.9300	0.9286	0.9274	0.9246
Gamma	1.242	1.240	1.238	1.233
Sp. energy, $F/\gamma - 1$	4264	4425	4588	4884
Cal val, cal/g	924.5	965	1005.5	1082.2
Mol. wt, g/mol	23.65	24	24.4	25.4

[a]Control composition, %: NC 30 ± 1, NG 25 ± 1, picrite 40 ± 1, DBP 2 ± 0.1, K_2SO_4 2 ± 0.1, and carbamite 1 ± 0.05.

propellant based on GAP binder plasticized with nitrate esters and oxidized by ammonium nitrate and triaminoguanidine nitrate is reported by Judge and Lessard [19]. The propellant is reported to offer nontoxic exhaust, low smoke signature, and good tensile properties.

CL-20 has several positive features, such as high density (2.04 g/cm^3), high heat of formation ($\Delta H_f + 108$ kcal/mol), and high oxygen balance (−11%), as compared with HMX (density 1.9 g/cm^3, $\Delta H_f - 17.9$ kcal/mol, and oxygen balance −22%) and RDX (density 1.8 g/cm^3, $\Delta H_f - 14.7$ kcal/mol, and oxygen balance −22%) [20]. As the information on CL-20-incorporated gun propellants is scanty, the present study was carried out to evaluate CL-20 in triple-base propellants to generate data. A triple-base propellant containing 40% NQ was selected as the control composition in view of its optimum energy level in terms of force constant. Thermal studies were undertaken to gain insight into the chemical process occurring in the condensed phase during combustion.

II. Experimental

A. Formulations

The CL-20 used for this study was synthesized by the authors on the lines of the method reported [6,21]. The average particle size of characterized CL-20 was 7 ± 2 μm.

The control composition of TBPs comprise 30 ± 1% NC (12.2% N_2), 25 ± 1% NG, 40 ± 1% NQ of particle size 25–30 μm, 2 ± 0.1% K_2SO_4, 1 ± 0.05% N-N′ diphenyl diethyl urea (carbamite), and 2 ± 0.1% dibutyl phthalate (DBP). Picrite was replaced by 5, 10, and 15% CL-20 (particle size 7 ± 2 μm) step by step in the first set of experiments. In the second set of experiments, the inert plasticizer DBP was totally replaced by energetic plasticizers: namely, Bu-NENA and BDNPF/A in a 5% CL-20-containing TBP.

Six compositions of triple-base propellant were prepared. The propellant dough was made in a sigma blade incorporator (1 kg capacity) by solvent method. The solvent used was acetone alcohol in 80:20 ratio [22]. The propellant dough was extruded in a hydraulic press (60 t) to get strands in a heptatubular rosette-shaped configuration using a suitable die-pin assembly (11.6/6/0.8/0.8). Extruded strands were predried (about 10–15 min) and then cut into grains of suitable length (21 ± 0.5 mm) using a Bowas rotary-cutting machine. The propellant grains were dried at 40 ± 2°C in a hot-air blower oven for 45–50 h (volatile matter ≤ 0.3%). The grains were

Table 2 CV firing results of triple-base gun propellants containing CL-20 at loading density 0.2 g/cm^3[a]

Parameters	Picrite 40, CL-20:0	Picrite 35, CL-20:5	Picrite 30, CL-20:10	Picrite 25, CL-20:15
Force constant, J/g	1028	1046	1071	1095
Pressure index, α	0.70	0.73	0.77	0.80
P_{max}, MPa	247	250	257	263
β_1, (cm/s)/MPa	0.124	0.131	0.153	0.169
Dp/dt_{max}, MPa/ms	31.2	32.1	36.5	41.8
Rise time, ms	12.6	12.1	11.5	10.3

[a]Control composition, %: NC 30 ± 1, NG 25 ± 1, picrite 40 ± 1, DBP 2 ± 0.1, K_2SO_4 2 ± 0.1, and carbamite 1 ± 0.05.

subjected to differential scanning calorimeter (DSC), thermogravimetric analyzer (TGA), closed-vessel (CV), and friction- and impact-sensitivity tests.

B. Closed Vessel

The propellants were evaluated in a CV (700 cm^3 capacity, operates at a maximum pressure of 280 MPa) at loading density 0.2 g/cm^3. The ballistic parameters such as force constant F, chamber pressure P_{max}, pressure exponent α, and linear burning-rate coefficient β_1 were obtained from the CV test.

To minimize oscillations due to pressure waves and the effect of cooling, the chamber length-to-diameter ratio was maintained as 2:1. The temperature of the vessel was maintained by circulating water through the jacket continuously at the time of firings. The propellant charge was ignited by passing a current (~3–5 A) through a short length of fine Nichrome wire soldered across the firing pins in the electrode. The pressure transducer containing a tourmaline piezoelectric gauge was used to measure chamber pressure P_{max}.

C. Sensitivity

Sensitivity of the propellant compositions to impact stimuli was determined applying the fall-hammer method (2 kg drop weight) as per the Bruceton staircase [23] approach, and the results are given in terms of statistically obtained 50% probability of explosion ($h_{50\%}$). Friction sensitivity was measured using standard methodology.

D. Thermal Analysis

Thermal analysis of propellants was carried out on a DSC (Pyris-7) and TGA (Mettler Toledo model 8551) at the heating rate of 10°C/ min under N_2 atmosphere. For DSC experiments, 0.5–1 mg samples were used and for TGA experiments, 2.5–2.8 mg samples were tested. Two experiments were carried out for each sample to confirm the reliability of data. The accuracy of the instrument was 0.2%.

III. Results and Discussion

A. Closed Vessel

In the first set of experiments, the compositions were evaluated by replacing picrite by 5, 10, and 15% CL-20 in the control composition. The thermochemical data was theoretically determined using a program developed at High Energy Materials Research Laboratory (Pune, India) that is not published elsewhere. The theoretical

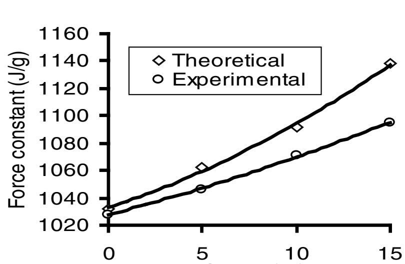


Fig. 1 Force constant vs percent of CL-20 in triple-base propellant system.

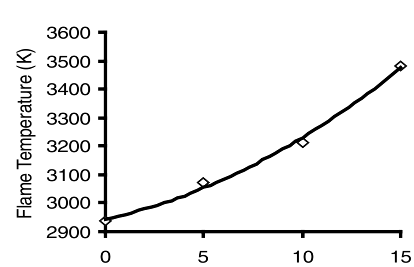


Fig. 2 Flame temperature vs percent of CL-20 in triple-base propellant system.

Table 3 Theoretical thermochemical data of triple-base gun propellants containing CL-20 and energetic plasticizers[a]

Parameters	Picrite 35, CL-20:5, DBP 2	Picrite 35, CL-20:5, BU-NENA 2	Picrite 35, CL-20:5, BDNPF/A 2
Force constant, J/g	1062	1089	1090
Flame temperature, K	3073	3195	3225
P_{max}, MPa	261	267	267
Covolume, cm^3/g	0.9286	0.9224	0.9205
Gamma	1.240	1.237	1.235
Cal val, cal/g	965	1012	1021
Mol. wt., g/mol	24	24.4	24.6

[a]Control composition, %: NC 30 ± 1, NG 25 ± 1, picrite 35 ± 1, CL-20:5, DBP 2 ± 0.1, K_2SO_4 2 ± 0.1, and carbamite 1 ± 0.05.

Table 4 CV firing results of triple-base gun propellants containing CL-20 with energetic plasticizers at loading density 0.2 g/cm^3[a]

Parameters	Picrite 35, CL-20:5, DBP 2	Picrite 35, CL-20:5, BU-NENA 2	Picrite 35, CL-20:5, BDNPF/A 2
Force constant, J/g	1046	1064	1066
Pressure index, α	0.73	0.73	0.73
P_{max}, MPa	250	263	252
β_1, (cm/s)/MPa	0.131	0.137	0.144
Dp/dt_{max}, MPa/ms	32.1	38.5	38.1
Rise time, ms	12.1	11.1	10.8

[a]Control composition, %: NC 30 ± 1, NG 25 ± 1, picrite 35 ± 1, CL-20:5, DBP 2 ± 0.1, K_2SO_4 2 ± 0.1, and carbamite 1 ± 0.05.

thermochemical data indicate that the force constant increased from 1032 to 1138 J/g when replacing picrite by 5–15% CL-20 (Table 1 and Fig. 1).

The gun propellants were also subjected to a CV test. Figure 1 depicts theoretical and experimental values of the force constant for the propellants with varying percentages of CL-20. The control composition gave a force-constant value of 1028 J/g (Table 2), whereas the compositions containing 5–15% CL-20 gave the force-constant values of 1046–1095 J/g, indicating an increase in force constant by 2–6% compared with control composition. The flame temperature (theoretical) of CL-20-containing compositions was higher (3073–3480 K) compared with that of control composition (2937 K) (Table 1and Fig. 2). The increase in energy level may be due to positive heat of formation ($\Delta H_f + 108$ kcal/mol) and higher oxygen balance (−11%) of CL-20 compared with picrite ($\Delta H_f -$ 22 kcal/mol and oxygen balance −31%).

The gamma values ($\gamma = Cp/Cv$) obtained (Table 1) for CL-20-based compositions were on the lower side, compared with the base composition. These values reduced from 1.242 to 1.233 for 5–15% CL-20-containing formulations. It may be attributed to an increase in specific energy ($F/\gamma - 1$) from 4425–4884 J/g on replacement of 5–15% of picrite by CL-20 in triple-base formulations. Chamber pressure P_{max} marginally increased from 247 to 263 MPa (Table 2).

Table 5 Impact- and friction-sensitivity test results of triple-base gun propellants containing CL-20[a]

	Impact test, cm[b]	Friction test, kg[c]
Picrite, 25–30 μm	170	36
Cl-20, 7 ± 2 μm	38	10
Picrite 40, base	29	19.2
Picrite 35 and CL-20:5	25	17
Picrite 30 and CL-20:10	23	14
Picrite 25 and CL-20:15	21	12

[a]Control composition, %: NC 30 ± 1, NG 25 ± 1, picrite 40 ± 1, DBP 2 ± 0.1, K_2SO_4 2 ± 0.1, and carbamite 1 ± 0.05.
[b]Probability of explosion $h_{50\%}$.
[c]Insensitive to friction up to levels shown.

Table 6 DSC results of picrite and CL-20 and triple–base gun propellants containing CL-20[a]

Composition, %	Peak decomposition I, endo	Temperature, °C II, Exo	$-\Delta H$, J/g I	$-\Delta H$, J/g II
Picrite, 25–30 μm (alone)	247.7	251.3	69.2	1304.2
CL-20, 7 ± 2 μm (alone)	——	253.7	——	3419.2
Picrite 40 (base)	——	194.1	——	1976
Picrite 35 and CL 20:5	——	195.2	——	2090.2
Picrite 30 and CL-20:10	——	193.3	——	2176.8
Picrite 25 and CL-20:15	——	193.6	——	2332.4

[a]Control composition, %: NC 30 ± 1, NG 25 ± 1, picrite 40 ± 1, DBP 2 ± 0.1, K_2SO_4 2 ± 0.1, and carbamite 1 ± 0.05.

Pressure exponent values increased from 0.7 to 0.8, which is well within the acceptance limits for gun propellants.

In the second set of experiments, the inert plasticizer (2% DBP) was replaced by energetic plasticizers (namely, Bu-NENA and BDNPF/A), which were found to effectively increase energy as well as mechanical strength for triple-base propellant in previous study carried out by the authors [24]. The plasticization study was undertaken using a base formulation of 5% CL-20 and 35% picrite and incorporated triple-base composition, as it has a lower flame temperature (3073 K) compared with 10 and 15% CL-20-based composition (Table 2). The replacement of DBP by Bu-NENA and BDNPF/A resulted in an increase of theoretical flame temperature by 122–152 K (Table 3) and an increase in force constant by 18–20 J/g (Table 4).

Linear burning-rate coefficient β_1 also increased from 0.131 to 0.137–0.144 (cm/s)/MPa. However, pressure index values did not change (0.73) (Table 4).

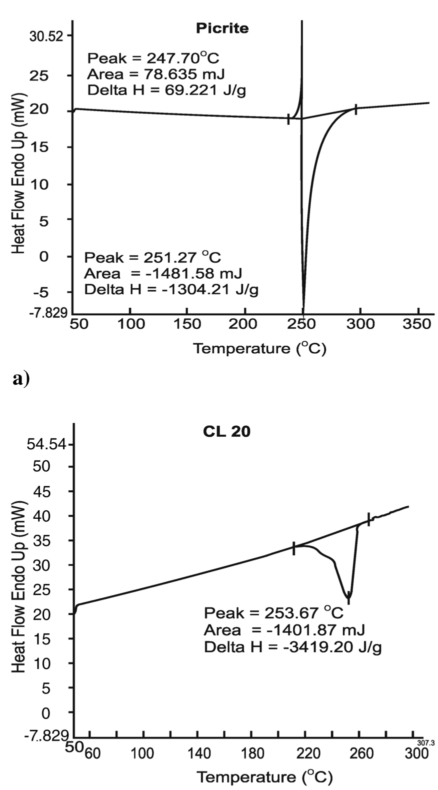
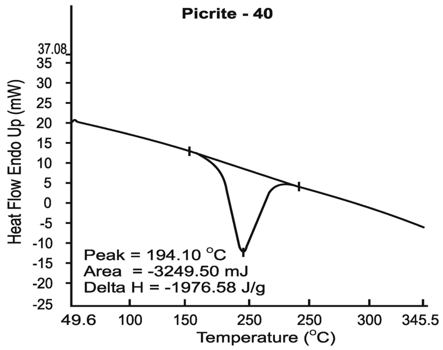
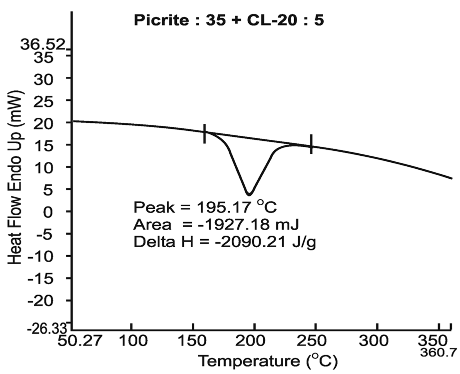


Fig. 3 Plots of a) DSC profile of picrite (alone) and b) DSC profile of CL-20 (alone).



a)



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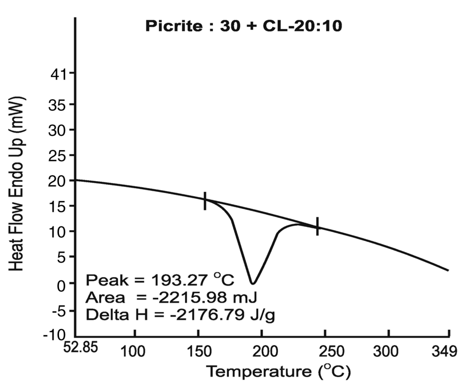

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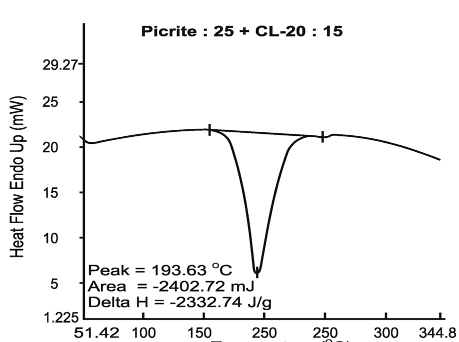

d)

Fig. 4 Plots of a) DSC profile of triple-base propellants, b) DSC profile of 5% CL-20-containing triple-base propellants, c) DSC profile of 10% CL-20-containing triple-base propellants, and d) DSC profile of 5% CL-20-containing triple-base propellants.

B. Sensitivity

The picrite alone was found to be insensitive for mechanical stimuli (friction 36 kg and impact $h_{50\%}$ 170 cm) compared with CL-20 (friction 10 kg and $h_{50\%}$ 38 cm) (Table 5). The triple-base gun propellant containing 40% picrite (base) gave $h_{50\%}$ of 29 cm and it was friction-sensitive up to 19.2 kg. The results bring out that the CL-20-incorporated compositions are relatively sensitive to mechanical stimuli, compared with the control. This may be attributed to a higher oxygen balance of CL-20 (−11%), compared with picrite (−31%). The results obtained are in close agreement with CL-20 propellant developed by Mueller [13] (friction 16 kg and impact insensitivity 4 Nm).

Table 7 TG results of triple-base gun propellants containing CL-20[a]

Composition, %	Temperature range, °C I	Temperature range, °C II	Weight loss, % I	Weight loss, % II
Picrite 40, Base	121–181	181–204	21.9	55.6
Picrite 35 and CL-20:5	115–180	180–243	29.7	58.8
Picrite 30 and CL-20:10	111–186	186–246	31.7	63.4
Picrite 25 and CL-20:15	108–186	186–250	32.3	64.4

[a]Control composition, %: NC 30 ± 1, NG 25 ± 1, picrite 40 ± 1, DBP 2 ± 0.1, K_2SO_4 2 ± 0.1, and carbamite 1 ± 0.05.

C. Thermal Decomposition

In DSC, picrite (alone) exhibited endotherm at 247.7°C with ΔH of 69.3 J/g and exotherm 251.3°C with ΔH of 1304.2 J/g (Table 6 and Fig. 3a). CL-20 (alone) gave one exotherm (unlike picrite) at 253.7°C with a ΔH value of 3419.2 J/g (Fig. 3b). Thus, the decomposition temperatures of CL-20 (253.7°C) and picrite (251.3°C) are close to each other.

The control composition gave a peak temperature of 194°C with ΔH of 1976 J/g (Table 6 and Fig. 4a). Incorporation of 5–15% CL-20 in triple-base formulations resulted in an increase in the ΔH value to 2090–2334 J/g (Figs. 4b–4d), whereas it did not influence decomposition temperature (193–195°C).

Relatively higher values of ΔH for CL-20-containing formulations in comparison to picrite composition can be explained on the basis of the greater compensating heat feedback due to combustion of CL-20 (oxygen balance −11%), compared with picrite (−31%).

CL-20 particles are reported to leave the burning surface virtually unreacted and burn only in gas phase [25], where highly exothermic oxidative reactions are taking place, which are likely to result in more heat feedback to the burning surface. This may be the reason to increase the linear burning-rate coefficient of CL-20-containing compositions, compared with that of picrite-based composition.

The TGA of the picrite and picrite and CL-20-based propellants revealed a two-stage weight-loss pattern (Table 7). The base composition revealed 22% weight loss in the temperature range of 121–181°C for first-stage decomposition and 55.6% weight loss for second-stage decomposition in the temperature range of 181–204°C. The first stage decomposition temperature 121–181°C is close to that obtained for double-base matrix (199°C). The propellant composition containing 5–15% CL-20 increased the weight loss from 21.9 to 29.7–32.3% in a temperature range similar to that in case of base composition. The similar pattern was also exhibited in second-stage decomposition. The decomposition temperatures obtained in second stage suggests that CL-20 and picrite (180–250°C) decompose as a homogeneous mass. It is probable that the increased weight loss in first- and second-stage decomposition was facilitated by the decomposition of condensed-phase reactions (decomposition of double-base matrix) due to heat feedback caused by oxidative decomposition of CL-20 in gas phase, as compared with decomposition of picrite.

IV. Conclusions

The partial replacement of picrite with CL-20 (5, 10, and 15%) led to an increase in force constant, flame temperature, specific energy, and linear burning-rate coefficient β_1, with a marginal rise in pressure exponent and sensitivity, without any effect on the decomposition temperature. However, replacement of 15% picrite by CL-20 resulted in an increase in flame temperature, β_1, friction, and impact sensitivity beyond the acceptable limit of gun propellant. Hence, the replacement of picrite with CL-20 to the extent of 10% may be considered from the application point of view.

It was found that the replacement of nonenergetic plasticizer DBP with Bu-NENA and BDNPF/A increases the ballistic performance of the triple-base propellant. Incorporation of BDNPF/A led to marginal increases in the β_1 value (0.144), as compared with Bu-NENA (0.137). However, both plasticizers Bu-NENA and BDNPF/A exhibited relatively similar force constants (1089 and 1090 J/g).

Thermal studies carried out by DSC and TGA indicate an increasing trend of exothermicity ($-\Delta H$) and percent of weight loss, respectively.

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S. Son
Associate Editor