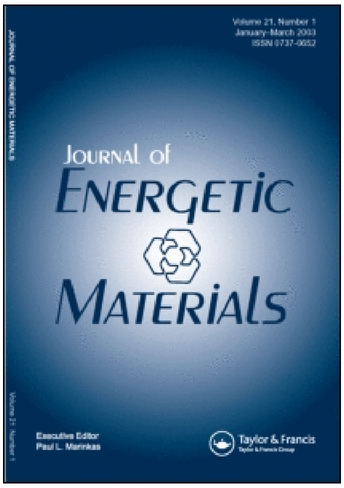



**Journal of Energetic Materials**
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713770432

# Combustible cartridge case formulation and evaluation

G. R. Kurulkar[a]; R. K. Syal[a]; Haridwar Singh[a]
[a] Development Laboratory, Explosives Research, Pune, India

**To cite this Article** Kurulkar, G. R. , Syal, R. K. and Singh, Haridwar(1996) 'Combustible cartridge case formulation and evaluation', Journal of Energetic Materials, 14: 2, 127 — 149
**To link to this Article: DOI:** 10.1080/07370659608216061
**URL:** http://dx.doi.org/10.1080/07370659608216061

# COMBUSTIBLE CARTRIDGE CASE FORMULATION AND EVALUATION

G.R. Kurulkar, R.K. Syal* and Haridwar Singh
Explosives Research and Development Laboratory,
Sutarwadi, Pune - 411 021, INDIA

## ABSTRACT

The formulation of combustible cartridge case was studied by changing the percentage of ingredients namely; nitrocellulose (NC), nitroguanidine (NiGu), cellulosic fibres, dibutylphthalate (DBP) and diphenylamine (DPA). A composition containing around 55% nitrocellulose & balance cellulosic fibers and nitroguanidine along with 3.5 parts dibutylphthalate & 1 part of diphenylamine was found compatible with triple base multi-tubular propellant, besides having reasonably good mechanical and ballistic properties.



* Author to whom correspondence should be addressed.

Journal of Energetic Materials Vol. 14, 146- (1996)
Published in 1995 by Dowden, Brodman & Devine, Inc.

# INTRODUCTION

In 60s few advanced countries had started R & D work to replace conventional brass cartridge case by combustible cartridge case (CCC) for their tank/artillary gun ammunition. Use of the combustible cartridge case (CCC) saves metal, ensures light weight ammunition, eliminates the disposal of spent cases and simplifies automatic firing equipment. They function to contain the main propellant charge and since ccc adds energy to propulsion system, there is saving on propellant mass. The cases get consumed on firing and leave no smouldering or burnt residue. In view of strategic and tactical importance of ccc for tank/mortar and other ammunition, most of the work done on formulation, processing of ccc is either classified or patented[1-22] and very little information is available in the open literature.[23-24] Hence, present study was undertaken on the formulation and evaluation of CCC. The results of various studies conducted on formulation and evaluation of CCC for tank gun application are discussed in this paper.

The basic formulation of CCC is based on the combustible ingredients nitrocellulose(NC), and cellulosic fibre, gas generator like nitroguanidine, plasticizer (dibutylphthalate) and stabilizer (diphenylamine) etc.

## Criteria for Selection of CCC Ingredients

The formulation of CCC is based on the energetic material to provide energy for complete combustion and a binder to provide mechanical strength. The processing of composition needs a plasticizer and a stabilizer for safe storage life. Nitrocellulose, a well known ingredient of gun propellants was chosen due to following considerations. The mechanical properties of CCC depend on the percentage and the nitrogen content of NC used. A higher nitrogen content of NC has lower mechanical property. Though lower nitrogen content of NC has lower calorimetric value as well as an influence on the burning rate and the energy of CCC, its mechanical properties are better than the CCCs made from higher nitrogen content. In view of this, and taking into consideration the use of high and low nitrogen content NC being used in the manufacture of propellants, a 12.6% nitrogen content NC was used to strike a balance between mechanical properties and energy.

It is well known that fibres which do not have hydroxyl groups and lack other mutually attracting groups cannot be made into a satisfactory/cohesive sheet. Since NC has less hydroxyl groups, to make a sheet of pure NC is impracticable. Secondly, during pulping the molecular weight and thereby the fibre length is reduced. In order to get required mechanical properties of the ccc, cellulosic fibres were used. Although use of cellulosic materials improves the mechanical properties, the combustibility of CCC is affected. To improve the combustibility of CCC which is affected due to the use of cellulosic material, incorporation of other energetic ingredient was thought of. The use of well known ingredient of triple base propellant i.e. picrite (nitroguanidine) was thought of to improve the combustibility. The thermochemical behaviour of picrite (nitroguanidine) is characterised by a high amount of combustion gases, connected with relatively low heat of explosion, a high force factor and a relatively low flame temperature. Further, picrite acts as a coolant. On decomposition it produces large amount of nitrogen gas, which helps in suppressing the flash at muzzle end. DBP was used as a non-explosive plasicizer, whereas DPA was used as a stabilizer to take care of ageing problem of NC.

The cccs used for evaluation during the course of present study were made by felting process. The flow sheet of the process is given in Fig - 1.

## EXPERIMENTAL

### Materials

Nitrocellulose (12.6% N), picrite (nitroguanidine) were obtained from Ordnance Factory and dibutylphthalate (DBP), cellulose, triacetin, acetone, ethyl alcohol, diphenylamine (DPA), and carbamite of desired purity were obtained from trade sources & their purity was checked before use. Analysis results of these ingredients are given in Table- 1.

### Equipments

The various equipments used for processing include beater, felting machine, water squeezing unit, steam heated dryer and isostatic press etc. Closed vessel[25] (C.V.), instron and methyl violet test apparatus were used for ballistic evaluation, mechanical properties determination & thermal stability of ccc respectively.

## Composition/Formulation

The five basic compositions consisting of variations in nitrocellulose and picrite were prepared for making combustible cartridge case. Table - 2 includes the details of various formulations of ccc studied.

# PROCESSING

## Slurry Preparation

To a predetermined amount of water in the beater, required quantity of cellulose is added, and the beating is continued till the desired freeness (CSF 200 ± 10) is attained. A weighed quantity of NC (on dry basis) and picrite are then added and mixing is carried out for 30 minutes. DBP, alcoholic solution of DPA, are added subsequently. While mixing the ingredients, the slurry contains 10% solids approximately. The mix is diluted with water to reduce the slurry consistency to approximately 1% solids. The slurry mix thus obtained is fed to the felting machine.

## Felting Operation

Preforms are made in the felting machine with the application of vacuum. The thickness and weight of the preform is controlled by feeding known amount of slurry. The preform or felt is loosely woven matrix (60% of water) with a wall thickness 3-4 times greater than the finished product. Water from felted preform is removed by pneumatic rubber balloon inflated at pressure of about 3 Kg/cm$^2$. Preform is dried in hot air oven at a temperature of 60 ± 2°C, till the moisture content is brought down to about 6 percent.

## Solvent Treatment and Processing

The dry preform is dipped in acetone for a predetermined time, allowed to dry in hot air oven till acetone percentage is reduced to 20% by weight (approx). Pressing is carried out by applying isostatic pressure of 30 kg/cm$^2$. CCCs are dried with hot air at temperature of 50 ± 2°C for about six hours and are coated with a thin film of air drying varnish and trimmed to required size. Table-4 shows the effect of acetone on mechanical properties and burning characteristics of CCC.

# EVALUATION

## Closed Vessel Firing

Sample pieces of size 100mm X 60mm were prepared from ccc. Samples of charge mass 190g were weighed and used for close vessel firing.

In case of the propellant, standard triple base propellant (NQ/M 047)charges of 140g were weighed and used for evaluation. 5g gun powder (G-12) was used for ignition of 140g of propellant charge. 1.3g of gun powder was tied in a cambric cloth bag and 3.7g of gun powder was distributed loose on the propellant charge. The same procedure was followed for the samples prepared from cccs.

The out put of CV data aquisition system was fed to P.C. and the ballistic parameters Pmax and rise time were calculated. A typical P-T profile is shown in Fig. 2. The output was printed with the help of printer provided with P.C. The burning time was considered from 5% Pmax to Pmax. The results are given in Table-3.



## Mechanical Properties

Test specimens were prepared by cutting strips of size 100mm X 20mm X 2mm. Five specimens were tested with Instron machine for tensile strength and % elongation (Model- 1185, Make- Universal Testing Machine, Britain). For recording the events on pen chart recorder, the speed of the cross head was 50 mm/min and speed of chart was 100 mm/min. The results of mechanical properties are presented in Table - 4.

### Thermal Stability

Samples were evaluated by methyl violet test apparatus. Results are given in Table-5.

## RESULTS AND DISCUSSION

### Effect of Picrite on Burning Characteristics

Typical P-T curve is shown in Fig. 2. It was found that 15% picrite with 70% NC produced higher Pmax and burning time than the standard propellant, whereas 35% picrite with 50% NC gave lower values of Pmax and burning time. However, results of 30% picrite with 55% NC were comparable with these of standard multi-tubular triple base propellant.

### Effect of Acetone on Burning Characteristics

Table-4 shows the C.V. firing results obtained from ccc, which were gelatinised with acetone under different time varying from 30 sec. to 90 sec. Results obtained indicate that there is a marginal effect on Pmax and burning time. Hence, dipping time in acetone is more relevant for processing purpose than to burning characteristics. Further, the amount of acetone present in ccc at the instance of pressing is more important than the time of dipping. If acetone is more than 35% by weight at the time of pressing, ccc becomes more gelatinised and there is a difficulty in removal of CCC from pressing mould.

## Compatibility of Stabilizers

NC has been used as the main energetic material for CCC which is known to undergo degradation on storage by auto catalytic reaction. Hence, the effect of two wellknown stabilizers for NC namely, DPA and carbamite were studied on thermal stability of ccc.

It was observed (Table-5) that 1 part DPA gave heat test value of 50 minutes. The increased concentration of DPA from 1 to 2 parts and finally to 3 parts increased the thermal stability to 55 and 60 minutes respectively. On the other hand use of 1 to 3 parts of carbamite gave heat test value of 32-35 minutes. From these results it can be concluded that for ccc composition, DPA appears to be a better stabilizer than carbamite. As expected, combination of DPA and carbamite gave intermediate values of thermal stability.

## CONCLUSION

CCC composition consisting about 55% NC, 15% cellulosic fibre, 30% picrite, 3.5 parts DBP,and 1 part stabilizer is suitable and compatible with triple base multi-tubular propellant. Increase in concentration of picrite lowers the Pmax and the burning time.

The suitability of cccs for various applications could be confirmed by conducting actual firings in the gun vis-a-vis the brass cartridge case and comparing the ballistics.

TABLE 1 : Analysis results of raw materials

| Material | Results | Specified Limits |
|---|---|---|
| **Nitrocellulose (NC)** | | |
| a) Nitrogen content | 12.7% | 12.6 ± 0.1% |
| b) Mineral matter | 0.80% | 0.8% max |
| c) Alkalinity (calculated as $CaCO_3$ | 0.3% | 0.4% max |
| d) Organic matter insoluble in acetone | Traces | 0.5% max |
| e) Heat test at 80°C | 16 minutes | 10 minutes min |
| **Nitroguanidine (picrite)** | | |
| a) Volatile matter | 0.24% | 0.25% max |
| b) Picrite content | 98.85% | 98.5% min |
| c) Acidity(Calculated as $H_2SO_4$) | 0.005% | 0.06% max |
| d) Water soluble impurities | 0.06% | 1.0% max |
| e) Ash content | 0.13% | 0.3% max |
| **Cellulose** | | |
| a) Organic matter soluble in ether, alcohol | 0.20% | 0.4% max |
| b) Ash content | 0.90% | 1.0% max |
| c) Degree of freeness | 185 | 180 min<br>200 max |
| d) α- Cellulose content | 95.4% | > 95% |
| **Di-Butyl Phthalate (DBP)** | | |
| a) Ester content (as DBP) | 99.15% | 99% min |
| b) Water content | 0.0106% | 0.1% max |
| c) Acidity (as phthalic acid) | 0.004% | 0.01% max |
| **Diphenylamine (DPA)** | | |
| a) Moisture (over caustic potash in a vacuum desiccator) | 0.032% | 0.5% max |
| b) Reaction | | |
| i) Alkalinity | Nil | Nil |
| ii) Acidity as $H_2SO_4$ | Nil | 0.005% max |
| c) Organic matter insoluble in ether alcohol | Nil | 0.02% max |
| d) Mineral matter | 0.0048% | 0.05% max |
| e) Setting point | 53°C | 51.5°C min<br>53.0°C max |

TABLE 2 : Formulations of CCC

| Formula No. | NC % | Picrite % | Cellu-lose % | DBP | DPA |
|---|---|---|---|---|---|
| | | | | Parts/100parts | |
| C-1 | 70 | 15 | 15 | 3.5 | 1.0 |
| C-2 | 65 | 20 | 15 | 3.5 | 1.0 |
| C-3 | 60 | 25 | 15 | 3.5 | 1.0 |
| C-4 | 55 | 30 | 15 | 3.5 | 1.0 |
| C-5 | 50 | 35 | 15 | 3.5 | 1.0 |

TABLE 3 : Effect of picrite on burning characteristics

| Formulae No. | Pmax (MPa) | *Burning time (ms) |
|---|---|---|
| C - 1 | 274 | 16.4 |
| C - 2 | 269 | 14.2 |
| C - 3 | 269 | 12.8 |
| C - 4 | 260 | 8.6 |
| C - 5 | 247 | 6.5 |
| ** Propellant NQ/M 047 | 262 | 10.8 |

NOTE : * Burning time was measured in ms from 5% Pmax to Pmax.

** Propellant composition in percentage. NC 21, NG 21, Picrite 55, carbamite 3 and $K_2SO_4$ traces.

TABLE 4 : Effect of acetone on mechanical properties & burning characteristics of CCC*

| Dipping Time seconds | T.S. $Kg/cm^2$ | % Elongation | Pmax (MPa) | Burning Time to Pmax (ms) |
|---|---|---|---|---|
| 30 | 121 | 4.8 | 250 | 5.6 |
| 45 | 134 | 5.0 | 250 | 5.7 |
| 60 | 160 | 5.8 | 250 | 6.5 |
| 90 | 178 | 5.9 | 253 | 7.2 |

*CCC formula No. C-4.

TABLE 5 : Compatibility of stabilizers with CCC composition.

| Diphenylamine (by parts) | Carbamite (by parts) | M.V. Test (minutes) |
|---|---|---|
| 1 | Nil | 50 |
| 2 | Nil | 55 |
| 3 | Nil | 60 |
| Nil | 1 | 32 |
| Nil | 2 | 35 |
| Nil | 3 | 35 |
| 1 | 2 | 35 |
| 2 | 1 | 45 |

CCC formula No.C-4 except stabilizer.

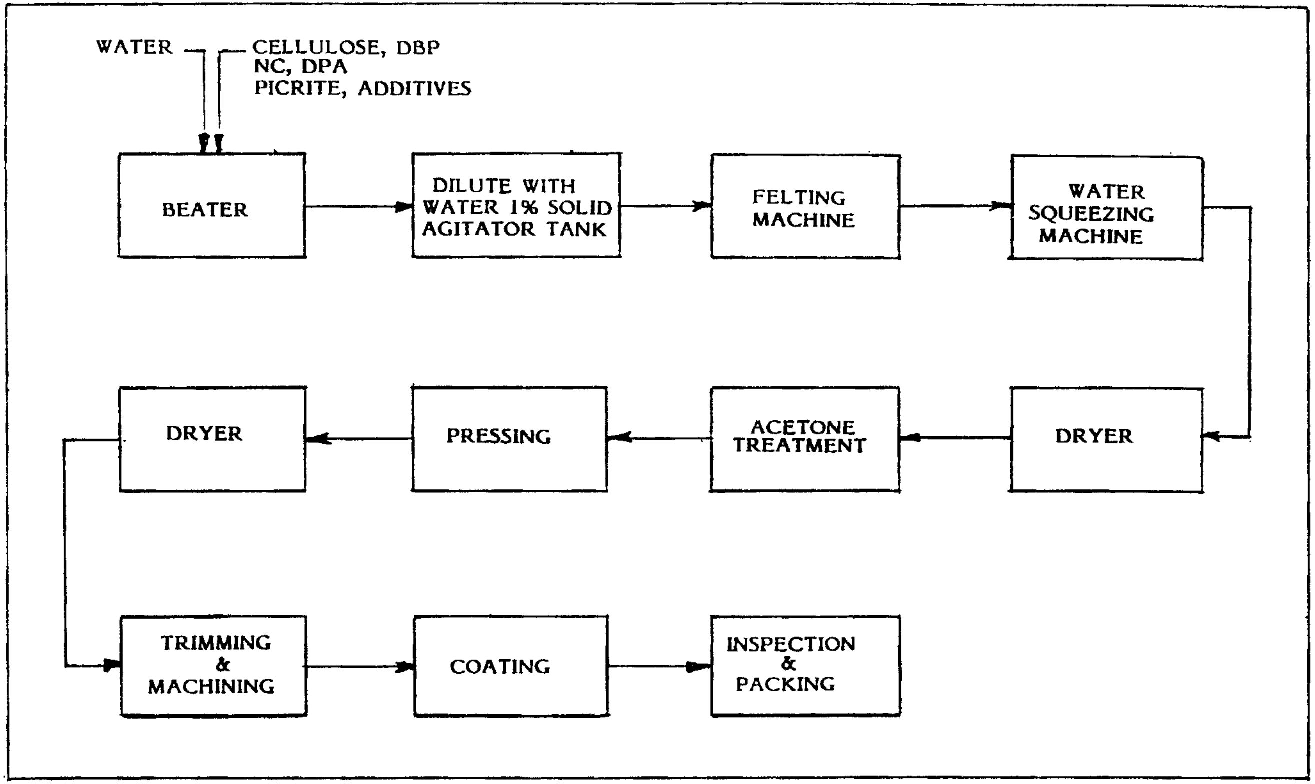
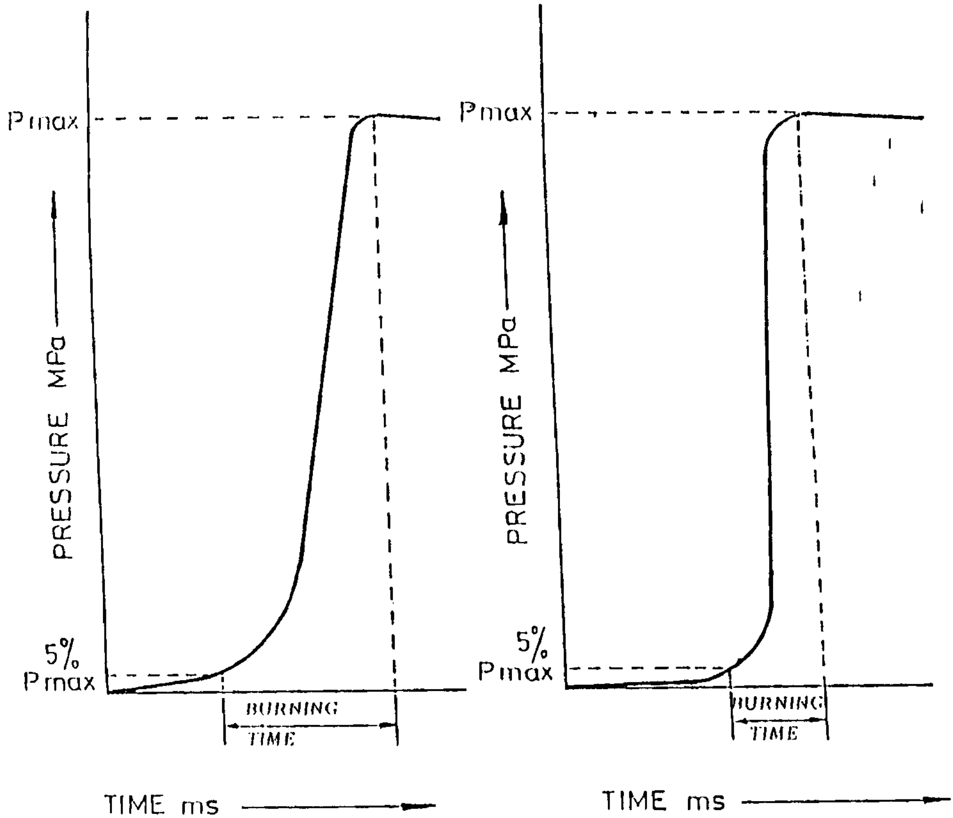



FIGURE 1 : PROCESS FLOW SHEET FOR MANUFACTURE OF CCC.




(A) (B)

FIGURE 2

(A) Typical P-T curve for standard propellant.

(B) Typical P-T curve for CCC.

## REFERENCES


1. K.F. Beal, W.D. Schmidt, N.J. Emerson, United States Dept. of the Army, Combustible cartridge case and method of making same, US pat. 2,982,211, 2 May 1961.
2. S. Axelrod, G. De mitraek, United States Dept. of the Army, Combustible cartridge cases and the like, US pat. 3,236,704, 22 February 1966.
3. J.N. Godfrey, M.G. De Fries, Atlantic Research Corporation, Combustible cartridge case, US pat. 3,260,203, 12 July 1966.
4. M.G. DeFries, J.N. Godfrey, Atlantic Research Corporation. Combustible cartridge case compositions, US pat. 3,264,993, 9 August 1966.
5. G.E. Brown, Atlantic Research Corporation, Combustible cartridge case, US pat. 3,280,74, 25 October 1966.
6. W.S. Baker, United State Dept. of the Army, Combustible cartridge cases, US Patent 3,282,146, 9 March 1966.

7. P. De Luca, Hawley Products Co. Combustible cartridge cases from nitrocellulose and kraft pulp, US patent 3,320,886, 23 May 1967.

8. M. Plunguian, Hercules Inc. Molding compositions from fibrous materials impregnated with nitro-cellulose and a plasicizer. French pat.1,552,875, 10 January 1969.

9. R.F. Remaly, W.P. Shefcik, M.B. Nelson, United States Dept. of the Army, Felting process for making combustible cartridge cases, US pat. 3,474,702, 28 October 1969.

10. J. Bobinski, J.P. Picard, United States Dept. of the Army. Combustible cartridge case, US pat. 3,706,280, 19 December 1972.

11. J.D. Kiles, R.C. Hartlein, W.D. Larson, C.R. Olson, Dow Corning Corporation, Combustible cartridges,Canadian pat.962,841, 18 February 1975.

12. K. Jacobsen, E. Troen, Raufoss Ammunisjon fabrikker A/S, Combustible cartridge cases, Norway pat. 133,382, 21 April 1976.

13. French Corporate Body, Mouldable compositions comprising polyvinyl nitrate, French pat. 1,443,289, 21 July 1976.

14. W. Oversohl, Wasag Chemie AG, Method for the production of combustible ammunition containers and product thereof, US pat. 4,068,589, 17 January 1978.

15. W. Oversohl, Wasag Chemie AG, Apparatus for production of combustible ammunition containers, US pat. 4,178,207, 11 December 1979.

16. W. Oversohl, Wasag Chemie Gesellschaft Mit Beschrankter, Production of combustible ammunition mouldings, German pat. 1,570,084, 25 June 1980.

17. W. Oversohl, German Offen, Method and apparatus for preparing combustible munitions moldings, German pat. 3,008,999, 24 Sept 1981.

18. A. Mengelle, J. Plazanet, Societe Nationale des Poudres et. Explosifs, Combustible tubes containing nitrocellulose manufactured by paper making technique and combustible or semicombustible tubular casing containing these tubes, French pat. 2,485,182, 24 December 1981.

19. A. Brazquies, M. Maures, Societe Nationale des Poudres et. Explosifs, Combustible or semicombustible cartridge casings comprising multi-ply combustible paper, Belgium pat. 901,070, 17 May 1985.

20. D. Mueller, F. Volk, Fraunhofergesellschaft zer Foerderung der Angewandten Forschung e.V. Combustible, nitrocellulose-based cartridges for tube weapons, German pat. 3,500,067, 10 July 1986.

21. M. Maures, J. Pasdeloup, P. Lespinasse, Societe Nationale des Poudres et Explosifs, Combustible articles by stamping combustible paper, French pat. 2,570,645, 28 March 1986.

22. M. Haider, J. Ulrike, J. Knobloch, W. Oversohl, WNC Nitrochemie G.m b H., Aschau, Oversohl, Elisabeth Christine, Combustible molded ammunition components, PCT, Int.Appl.WO 8,605,175, 12 September 1986.

23. Howard C. Appleman, "Combustible Ordnance in the United States" 2nd Australian Gun Propellant Conferance Toward 2000, Mulwala, N.S.W. Australia, (1986).

24. J.L. Simonotti, "Combustible cartridge cases : Products and Processes", Military Technology MILTECH, 9/86.

25. H.J. Scholz, "Burning behaviour of combustible explosive charge case material in a closed vessel" Internationale Jahrestagung (1983).