

# Evaluating Class B Fire Extinguishing Efficacies of Organic Nitro Compounds Based Aerosol Forming Compositions

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**Abstract** Every year, increasing fire accidents worldwide have directed our attention towards an effective fire suppression system. For many decades, Halons have been used as effective fire extinguishing agents. Vienna convention (1985) and Montreal protocol (1987) determined Halons as ozone-depleting agents, and subsequently, their manufacturing is banned by the U.S. Environmental Protection Agency. Thus, against the urgent background of this elimination of Halons, aerosol-based extinguisher resulted as one of the significant Halons substitute technology. The Ozone Depletion Potential and Global Warming Potential values of aerosol extinguishing agents are nearly zero. This has provided thrust for various Universities and research institutions for undertaking numerous projects to develop aerosol-forming pyrotechnic composites for fire fighting applications. In the present work, novel fire extinguishing pyrotechnic compositions were fabricated, in which four different organic nitro compounds, 3,5-dinitrosalicylic acid, 3,5-dinitrobenzoic acid, *m*-nitrobenzenesulphonate and 3-nitrophthalic acid, were used as the reductant. These nitro compounds undergo rapid redox reaction with strong oxidants to produce aerosol fire extinguishing agents, extinguishing 4.57 KW of small scale *n*-heptane pool fires in 3 seconds. The fire extinguishing efficacy and thermal characteristics of novel compositions were evaluated against potassium nitrate and phenol formaldehyde resin-based conventional pyrotechnic composition.

**Keywords:** aerosol, class B fire, pyrotechnic, fire suppression, organic nitro compounds, halon alternatives

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## 1. Introduction

Halon based extinguishants have been used as effective fire extinguishing agents for many decades. Halon extinguishers can produce significant quantities of life-threatening and ozone-depleting halogen acid gas and aerosol during fire suppression. The thermal degradation of halon can produce HF (hydrogen fluoride), COF<sub>2</sub> (carbonyl fluoride), HCl (hydrogen chloride), HBr (hydrogen bromide), or HI (hydrogen iodide) gases [1]. Therefore, the Vienna convention (1985) and the Montreal protocol (1987) declared the phase-out movement against halon extinguishants. Many countries have joined the protocol and are committed to exploring halon alternatives. The traditional halon alternatives like dry powder, foam, inert gas and water mist are usually not as efficient as halon extinguishers. These agents have high minimum extinguishing concentration, require pressurized cylinders,

huge storage area, complex piping installation and high cost of maintenance.

Aerosol forming pyrotechnic compositions (AFPCs) for firefighting application has gained much attention in recent years as halon alternatives. Unlike traditional halon alternatives, aerosol extinguishing agents do not require pressurized gases to be driven out, occupies minimum space and has smooth installation without piping. These aerosol-based fire extinguishing agents have nil environmental impact as they possess almost zero ozone depletion potential (ODP), global warming potential (GWP) and atmospheric lifetime (ALT) values [2].

Several AFPCs have been reported in the patents and literature, wherein chlorate, perchlorate or nitrates of potassium and strontium have been used as oxidants [3-9]. Various synthetic organic resins like melamine-formaldehyde, phenol-formaldehyde, epoxy resin, polyurethane etc., are used as reductants [10,11,12]. Such combination on actuation undergoes rapid redox reaction to produce hot condensed aerosol extinguishing agents,

which can effectively extinguish class A, B, C, D and K fires. Available aerosol generators containing AFPCs of mentioned ingredients are associated with thermal and health hazards. During pyrotechnic combustion, the flame temperature may reach up to 2100°C with the generation of heat and flame up to 1m from the nozzle and rendered metal surface red hot of those aerosol generators [13,14]. It may cause secondary fire risk in explosive platforms, mainly oil-producing platforms and ship engine rooms which contain diesel vapours self-igniting at 330°C in their atmosphere.

Further, exposure to melamine, phenol, polyurethanes, and epoxy resins are considered a life-threatening occupational hazard. Phenolic resins have both acute as well as chronic effects on the pulmonary function of the human mucous membrane [15,16,17,18]. Formaldehyde based organic resins release carcinogenic formaldehyde [19,20,21]. On the basis of the aforementioned ground, this research study focuses on developing novel flameless resin-free AFPCs which show enhanced cooling efficiency for its discharged aerosol. It can simplify the complex structure and reduce the high cost of the hot aerosol generators by eliminating any physical cooling pellets installed in front of the generators to reduce the temperature of released aerosol and hot gases. In the present work, we have prepared four improved resin-free novel pyrotechnic compositions (PyCs) using 3,5-dinitrosalicylic acid (3,5-DNS), 3,5-dinitrobenzoic acid (3,5-DNB), *m*-nitrobenzenesulphonate (*m*-NBS) and 3-nitrophthalic acid (3-NPA) as reducer and potassium nitrate, potassium chlorate, potassium perchlorate or their combination as an oxidant to overcome the drawbacks and disadvantages of the known such AFPCs. Due to the presence of the strong electron-withdrawing nitro group, it facilitates rapid flameless aerosolization of AFPCs without adding any modifiers and additives. The prepared novel AFPCs were assessed for their class B fire extinguishing efficacies, calorific values, burn rate, ignition temperature and combustion flame temperature against conventional potassium nitrate and phenolic resin-based AFPCs.

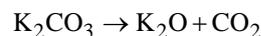
## 2. Aerosol Flame Interaction

Pyrotechnic combustion of AFPCs generates condensed micro particles of potassium bicarbonate, which further undergo endothermic decomposition in the fire zone and causing the removal of heat from the flame. For example, bicarbonate produced subsequently decomposes between

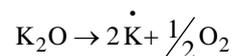
100 and 120°C to give potassium carbonate.



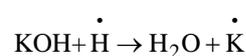
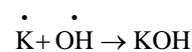
Potassium carbonate further decomposes to carbon dioxide and potassium oxide in the fire zone ( $T > 1200^\circ\text{C}$ ).



Potassium oxide produced subsequently decomposes ( $>1500^\circ\text{C}$ ) to free potassium radicals.



These potassium free radicals interact with flame propagating radicals to produce non-flammable neutral molecules of potassium hydroxide and water. Thus, acting as a chain termination step in flame propagation [22,23,24].



## 3. Experimental Work

### 3.1. Materials and Preparation

All the raw materials were of appropriate purity: source, grade and purity of the reagents are mentioned in Table 1. Formulations were prepared by grinding and mixing all ingredients according to their weight percentage mentioned in Table 2. All the ingredients were well-grounded and sieved through the sieve size of 74 and 53  $\mu\text{m}$  to get the particle size distribution of 74 to 53 microns. Water is used as a solvent for dough preparation. The prepared dough was finally cast into the cuboidal shape specimens (25 mm x 25 mm x 6 mm) and linear solid strands specimens (65 mm x 8 mm x 5 mm). The dough casting was carried out on a hydraulic press machine at 5000  $\text{kg}/\text{cm}^2$  pressure and maintained compression time for 60 seconds. To protect from moisture, casted samples were placed in a Memmert vacuum oven at 60 to 100°C and vacuum kept at 200 mbar for a minimum period of five hours and finally stored in a desiccator for further experimental studies. All experimental studies on a specific PyC were completed within one day to avoid the influence of changing temperature, humidity, and air pressure. For each PyC, three measurements were performed, and the average value was reported.

Table 1. Source and grade of raw ingredients

Raw material	Grade	Purity (%)	Source
3,5-Dinitrosalicylic acid	Analytical Reagent	98	Loba Chemie Pvt. Ltd.
3,5-Dinitrobenzoic acid	Analytical Reagent	99	Loba Chemie Pvt. Ltd.
<i>m</i> -Nitrobenzene sulphonic acid sodium salt	Analytical reagent	98%	Central Drug House (P) Ltd.
3-Nitrophthalic acid	Analytical Reagent	98%	Loba Chemie Pvt. Ltd.
Potassium nitrate	Analytical Reagent	99.5	S D Fine-Chem Ltd.
Potassium perchlorate	ACS reagent	$\geq 99$	Sigma Aldrich
Potassium chlorate	ACS reagent	$\geq 99$	Sigma Aldrich
Phenol formaldehyde resin	Commercial grade	99	Apex Plastic Pvt. Ltd.
<i>n</i> -Heptane	Laboratory Reagent	$\geq 90$	S.D. fine-chem limited

**Table 2. Formula and their chemical compositions**

Formula	Proportion by weight	
PyC-1	3,5-DNS : KNO <sub>3</sub> : KClO <sub>4</sub>	6:3:1
PyC-2	3,5-DNB : KNO <sub>3</sub> : KClO <sub>4</sub>	6:3:1
PyC-3	<i>m</i> -NBS : KNO <sub>3</sub>	1:1
PyC-4	3-NPA : KNO <sub>3</sub> : KClO <sub>3</sub>	6:3:1
PyC-5	KNO <sub>3</sub> : PFR	4:1

### 3.2. Experimental Analysis

The ignition temperature of PyCs was recorded from Differential Scanning Calorimetry (DSC) curves. These curves were acquired using a Perkin Elmer, Pyris Series instrument from 50°C to 700°C heating range at 10°C/min under a nitrogen atmosphere with a flow rate of 50mL/min. Bomb Calorimeter SDC 5015, from Sundy, China, was utilized for the determination of calorific value for 1g of samples in an atmosphere of oxygen at 3 Mbar of pressure.

Burn rate measurement of each PyC was carried out in accordance with the fuse wire method. For this purpose, two fuse wires were threaded into the linear solid strands of PyCs. The fusion of the first fuse wire during combustion provides a start signal for linear burn rate measurement, and fusion of the second wire provides a stop signal (Figure 1). By recording the time taken in the fusion of both wire and space between the wires, the linear solid strand burn rate can be determined.

The fire suppression experiments were conducted in a fire test chamber of stainless steel (SS) with nominal dimensions of 483 mm x 500 mm x 485 mm. A stainless-steel fuel pan with 49 mm depth and 96 mm diameter, used to produce 4.57 KW *n*-heptane pool fire as a threat. The pool was formed by pouring 92.5 mL of *n*-heptane over 185 mL of water in a fuel pan. A case containing cuboidal shape PyC embedded with nichrome loop was placed 200 mm away from fire threat. The PyC was actuated with the help of a nichrome loop acting as an igniter, which turned red hot on receiving the power of 12 V and 10 A D.C.

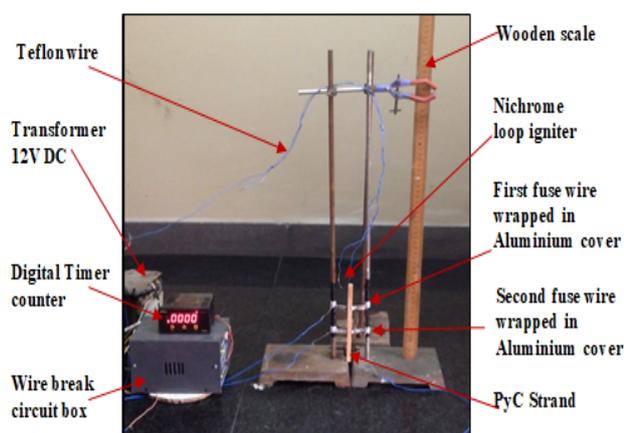
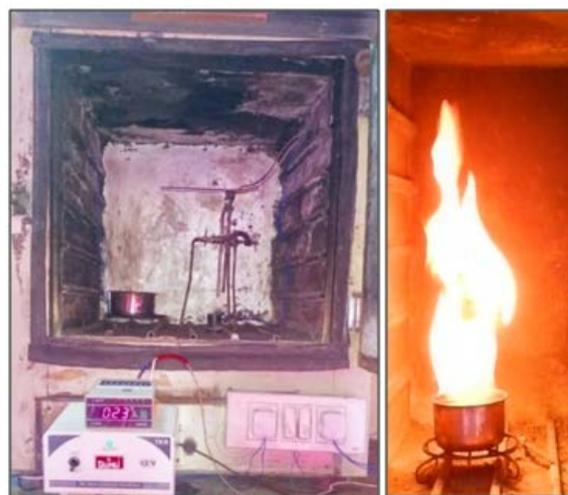
**Figure 1.** Linear solid strand burn rate measurement setup

Figure 2 shows the position of pool fire threat and thermocouples. Two K-type thermocouples of diameter 6 mm with a detection range of 0-1200°C were positioned horizontally and vertically. Averaging the data from thermocouples presented a complete temperature profile of the test chamber.

**Figure 2.** Fire test chamber

To record the combustion flame temperature of PyCs, cold discharge test was conducted. In cold discharge test, the sample was placed in a case, touching vertical thermocouple, and actuated in the absence of fire threat. *n*-Heptane pool fire was tested for its self-extinguishment under closed-door condition. It was found that for 10 minutes, the fire was not extinguished and burned naturally. After igniting the fuel, 30 seconds of pre-burn time was provided with open door condition to develop fully controlled steady fire. After that, the door was closed, and the fire was allowed to burn for a further period of 50 seconds. The aerosol was discharged by actuating PyC. After fire extinguishment, a re-ignition test was carried out to evaluate the effectiveness of PyC. In the re-ignition test, the chamber was kept sealed for 10 minutes, during which fuel was reignited at an interval of 5 minutes. After opening the door, the fuel pan was refuelled with a volume as mentioned above of *n*-heptane and water mixture. For each composition, three measurements were performed. To obtain fire extinguishment time and aerosol discharge duration, fire suppression experiments were recorded with the help of a digital video recorder.

The chemical nature of the discharged aerosol analyzed by XRD and FTIR. For this purpose, one gram of sample was burned in a 1000 mL glass beaker with mouth covered by aluminium foil. After settlement, the condensed aerosol particulate matter was collected in powder form. The FTIR spectra of condensed aerosol particulate matter were recorded on an IRAffinity-1S, SHIMADZU from 400 to 4000 cm<sup>-1</sup>. X-ray diffraction (XRD) pattern of the condensed aerosol particulate matter was recorded on a Panalytical, X'pert Pro MRD Diffractometer with Cu K $\alpha$  radiation ( $\lambda=1.5418 \text{ \AA}$ ).

## 4. Results and Discussion

The temperature profile of the test chamber (Figure 3) shows that during the pre-burn time, the temperature rose beyond 200°C because of *n*-heptane combustion and post-discharge of aerosol; a sharp decay in the temperature was observed. Hence, the temperature profile confirmed the fire extinguishment. The results of the *n*-heptane fire suppression experiment were determined experimentally and summarized in Table 3.

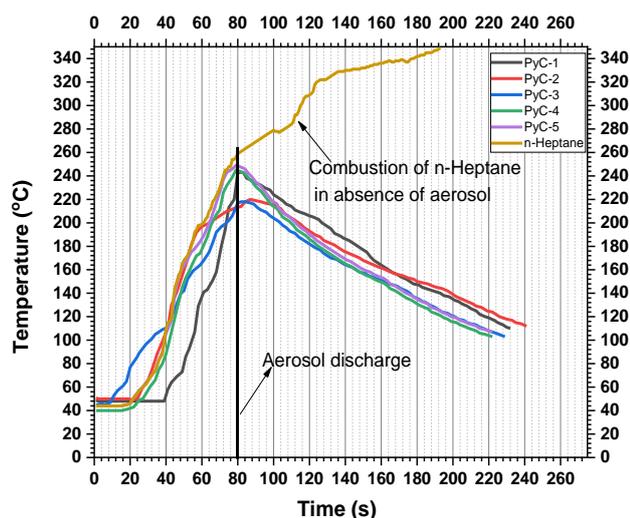


Figure 3. Temperature profile of test chamber during fire suppression experiment

Table 3. Results of *n*-heptane pool fire test

Formula	MEM (g)	CFT (°C)	FET (s)	Re-ignition test (min)	
				5	10
PyC-1	5.94	167	3	No	No
PyC-2	5.86	196	3	No	No
PyC-3	5.91	253	4	No	No
PyC-4	5.90	270	3	No	No
PyC-5	5.86	578	4	No	No

MEM, Minimum extinguishing mass; CFT, Combustion flame temperature; FET, Fire extinction time.

Though both the novel and conventional PyCs have shown similar fire extinguishing efficacy, extinguishing the fire within 3 to 4 seconds, from the cold discharge test, it was found that novel PyCs exhibited 53 to 71% lower combustion flame temperature than conventional PyC.

A faster burn rate of PyCs is a crucial factor that ensures a directional flow of the discharged aerosol towards the mouth of the fire. A faster burn rate of PyCs increases the effectiveness of aerosol extinguishing agents by reducing their MEM values. The data obtained from burn rate measurement (Table 4) showed that nitro compounds based PyCs have a much faster burn rate than the conventional PyC. A strong electron-withdrawing nitro group facilitates a rapid redox reaction with an oxidant to produce a significant amount of CO<sub>2</sub> and water vapours. These inert gases help in lowering the calorific value (Table 4) of PyCs and deliver the aerosol agents effectively into the fire zone. Due to rapid redox reaction, nitro compounds based PyCs showed a lower ignition temperature (Table 4 & Figure 4) resulted in immediate aerosol discharge (2-3 seconds) on thermal actuation (with a potential of 12V and 10A D.C.). Therefore, they showed a faster response during fire testing. On the other side, conventional PyC possesses a higher ignition temperature, thereby showed a delayed aerosol discharge (10 seconds) on thermal actuation.

Table 4. Exothermicity and burn rate values of PyCs

Formula	Burn rate (mm/s)	Calorific value (J/g)	Ignition temperature (°C)
PyC-1	17.02	5718	311
PyC-2	15.61	6651	346
PyC-3	16.51	5820	302
PyC-4	13.93	6017	284
PyC-5	10.13	7778	437

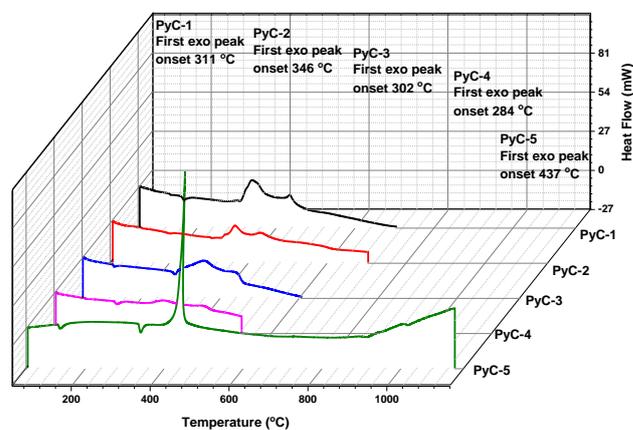


Figure 4. DSC curves of PyCs

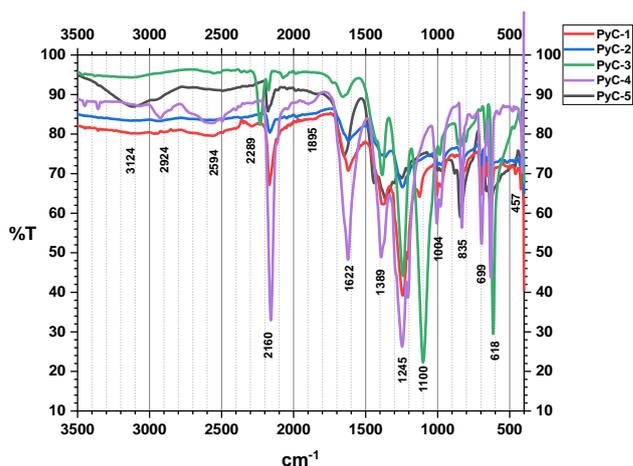


Figure 5. FTIR spectra of condensed aerosol particulate matter

To ascertain the chemical nature of extinguishing agents, condensed aerosol particulate matter was subjected to FTIR and XRD analysis. From FTIR spectra (Figure 5), common absorption bands correspond to potassium nitrite, unsaturated aliphatic hydrocarbon and potassium bicarbonate were found in all samples. Potassium nitrite shows its characteristic absorption bands at 840 cm<sup>-1</sup> that is attributed to N=O stretching and 1244, 1361 cm<sup>-1</sup> attributed to N-O symmetrical stretching [25]. Potassium bicarbonate has its characteristic IR absorption bands that appeared at 2280-2960 cm<sup>-1</sup> correspond to O-H stretching, 1600-1650 cm<sup>-1</sup> correspond to C=O stretching, C-O stretching at 1361 cm<sup>-1</sup> and 1001, 968, 840, 696 cm<sup>-1</sup> correspond to bending vibrations in the finger-print region [25]. Unsaturated aliphatic hydrocarbons have their characteristics peaks at >3000 cm<sup>-1</sup> attributed to -C≡C-H stretching, 2800-3000 cm<sup>-1</sup> attributed to C-H stretching, 2174 cm<sup>-1</sup> attributed to -C≡C- symmetrical stretching, 1361 cm<sup>-1</sup> attributed to C-H bending vibrations, -C≡C-H

bend overtone at  $1244\text{ cm}^{-1}$  and  $\text{-C}\equiv\text{C-H}$  fundamental bend at  $630\text{ cm}^{-1}$  [26].

The absorption bands at  $3200\text{ cm}^{-1}$  attributed to aromatic C-H stretching, bands at  $1450\text{-}1650\text{ cm}^{-1}$  attributed to C=C ring stretching, bands at  $1000\text{-}1360\text{ cm}^{-1}$  attributed to in-plane bending of ring C-H and bands at  $627\text{-}999\text{ cm}^{-1}$  attributed to out of plane bending of ring C-H marked the presence of polynuclear hydrocarbons in condensed aerosol particulate matter collected from conventional PyC, which may raise the environmental safety concern. [26].

XRD patterns (Figure 6) of condensed aerosol particulate matter further confirmed the dominant phases present are potassium nitrite, potassium chloride and potassium bicarbonate [27,28,29].

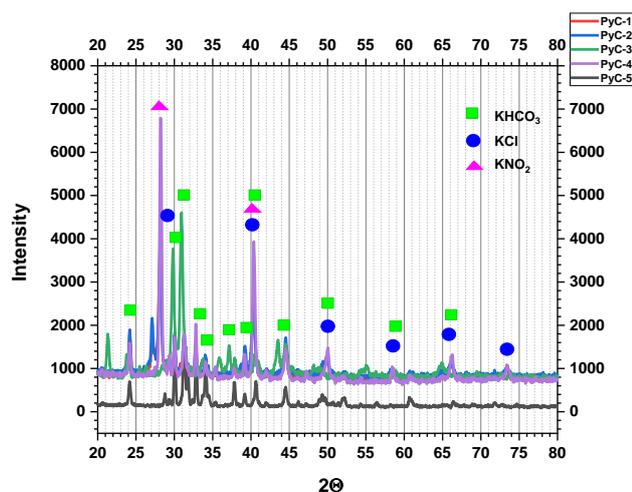


Figure 6. XRD patterns of condensed aerosol particulate matter

## 5. Conclusion

Fire suppression results showed that aerosol generated during the combustion of the aromatic nitro compounds-based PyCs have good fire extinguishing efficacy, extinguishing 4.57 KW *n*-heptane pool fire in 3 seconds. The aerosol discharged from newly developed PyCs possess 53-71% lower combustion flame temperature and lower calorific value ( $5718\text{-}6651\text{ J/g}$ ) than  $\text{KNO}_3$ -PFR based conventional PyC. Although conventional PyC showed similar fire extinguishing performance, it exhibits higher combustion flame temperature ( $578^\circ\text{C}$ ) and a higher calorific value of  $7778\text{ J/g}$ .

This enhanced cooling efficacy of newly developed PyCs can prevent secondary fire chances. By virtue of this, it can simplify the complex structure and reduce the high cost of the hot aerosol generators by eliminating any physical cooling pellets installed in front of the generators. The newly developed PyCs have shown a faster response in extinguishing the fire because of their lower ignition temperature ( $284\text{-}346^\circ\text{C}$ ) and faster burn rate ( $13.93\text{-}17.02\text{ mm/s}$ ) than conventional PyC having a  $437^\circ\text{C}$  ignition temperature and burn rate of  $10.13\text{ mm/s}$ .

These organic resin-free PyCs are also free from occupational hazards associated with the use of melamine-formaldehyde, epoxy, polyurethanes, phenol-formaldehyde resins etc., which are used as reducers in conventional AFPCs. The FTIR spectra with XRD analysis have

confirmed potassium nitrite, potassium chloride and potassium bicarbonate as the major ingredients of condensed aerosol particulate matter. From FTIR, the characteristic peaks of polynuclear hydrocarbons are observed in condensed aerosol particulate matter from conventional PyC, which may raise an environmental safety concern.

Hence, these nitro compounds based PyCs are promising candidates for substituting organic resin-based conventional PyCs used for fire fighting applications. However, detailed studies about the nature of gases discharge during pyrotechnic combustion and impact on visibility in the area of aerosol discharge are required to be pursued.

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