N-63-4-2

Contract No. DA 18-108-AMC-118(A)

QUARTERLY PROGRESS REPORT NO. 1

Covering the Period

18 March - 17 June 1963

INVESTIGATION OF THE FLASHING OF AEROSOLS

Prepared by

Alan R. Pittaway

25 July 1963

MRI

MIDWEST RESEARCH INSTITUTE

AD__________ Accession No.__________
Midwest Research Institute, Kansas City, Missouri
INVESTIGATION OF THE FLASHING OF AEROSOLS -
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Quarterly Report No. 1, 25 Jul 63, 33 pp - 3 illus, 3 tables, refs. Contract DA 18-108-AMC-118(A)

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2. Chemical warfare agents
3. Aerosol generation*
4. Explosive gases
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Alan R. Pittaway

25 July 1963

PREFACE

Work on this program is being accomplished under the supervision of Dr. R. W. Shortridge, Assistant Director, Chemistry Division. Mr. Alan R. Pittaway is the principal investigator. Mr. Roger Schroeder, Mr. Richard Fetter, Mr. Dale Brees, and Mr. Fred Bergman have made significant contributions to the design and fabrication of the experimental equipment during this report period. Mr. Frank Brink has participated in its experimental work during the latter part of the period.

Approved for:

MIDWEST RESEARCH INSTITUTE

FV Morriss

F. V. Morriss, Director
Chemistry Division

July 25, 1963

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SUMMARY

This First Quarterly Progress Report on the "Investigation of Aerosol Flashing," Contract No. DA 18-108-AMC-118(A), covers the period 18 March through 17 June 1963.

The aerosol flashing problem can be analyzed as concerning one or more of the following combustion systems:

1. Combustion of the air-aerosol mixture but only during combustion of the detonation products with air.

2. Pyrolysis of the aerosol during combustion of the detonation products with air.

3. Combustion reaction of the aerosol with the detonation product gases.

4. Pyrolysis of the aerosol while in contact with the detonation product gases.

5. Combustion of the aerosol with air alone.

The ignition sources for these systems are shock waves, spontaneous ignition of either the detonation products or the aerosol, combustion of the detonation products, or contact with hot metal fragments. These ignition sources can be broken down as possibly acting in a sequential form rather than independently. Thus for number one above, a shock wave could ignite the detonation products which in turn ignites the aerosol, etc. The present evidence, subject to experimental verification, indicates strongly that the detonation product gases are intimately involved in the problem and that elimination of their combustion would greatly reduce or eliminate aerosol flashing.

There is no doubt that in some cases the agent aerosol does combust with air. When this occurs, the aerosol combustion can be simplified to two characteristic types, a diffusion flame with air surrounding each fuel droplet and/or a vapor phase "premixed flame" type system, both of which can vary from fuel-rich to fuel-lean.

Inhibition of combustion must take into consideration the peculiarities of the system and particularly the mode of ignition. Ideally, the

inhibitor should act to suppress the ignition of the detonation products (for case 1 above), but be capable of incorporation into the chemical agent for dispersion. Suppression of detonation product ignition would also reduce the exposure of the chemical agent to thermal decomposition processes.

Selection of an inhibitor also must be based on the fact that three different combustion systems are involved, i.e., detonation products with air, an agent diffusion flame, a rich and/or lean agent premixed flame, and various mixtures of all three.

This research program is aimed at determining the effects of particle size, aerosol concentration, vapor pressure, temperature, pressure, flammability limits, and the ignition source on the aerosol flashing problem. Experiments will be conducted in a constant pressure combustion chamber and a conventional shock tube. It will be possible from the data obtained to determine which of the above combustion systems is the most important, the ranking of the various ignition modes in order of their importance, and the influence of the aerosol physical factors on flammability.

I. INTRODUCTION

This is the First Quarterly Report on the "Investigation of the Flashing of Aerosols," Contract No. DA-18-108-AMC-118(A), MRI Project No. 2685-C, covering the period 18 March through 17 June 1963.

The purpose of this project is to determine additional information on the factors which influence aerosol flashing. Specifically, several possible ignition modes are to be studied and, in addition, the influence of the surrounding environmental gas. Additives which will tend to suppress either the ignition or the combustion of the aerosol are to be evaluated.

An analysis of the aerosol flashing phenomenon and a discussion of the method of approach to the problem are included as Appendix I. It is suggested that those who are unfamiliar with the problem review this material since it provides the background and justification for the experimental approach taken in this program.

II. PROGRESS TO DATE

A. Combustion Chamber Design

The combustion chamber to evaluate a number of the parameters involved in aerosol flashing has been designed and fabricated. The general specifications for the apparatus are as follows.

1. It must allow the creation of any desired combustible component mixture composition in a three-component system.

2. It must allow the combustion to be studied under constant pressure conditions.

3. It must be a totally-closed system to permit recovery of combustion products as desired.

To achieve these specifications the concept of a long cylindrical chamber, separated from a collapsed, flexible bag by a rupture diaphragm was adopted.

A photograph of the equipment is presented in Fig. 1. The main chamber consists of a 36-in. long x 5-1/2-in. ID clear plastic cylinder, set vertically in a stand. On a metal plate at the bottom (not shown) is mounted a diesel fuel injector for dispersing the liquid phase material. The bottom plate also has a provision for spray washing the interior, evacuating, and draining. A metal collar at the top of the cylinder contains a perforated plate, igniter access, connections for a gas expansion bag, and a spray nozzle for bag washing.

The perforated plate is a support for the rupture diaphragm and consists of 13/64-in. holes on 1/4-in. staggered centers. The open area is 60 per cent. Testing of several diaphragm materials (including several types of aluminum foil) led to the selection of Saran wrap as the most desirable. It is cheap, readily available, and uniform in characteristics. When supported by the perforated plate it will withstand a pressure differential greater than 20 psi. When unsupported as a 5-1/2-in. diameter diaphragm, it breaks at less than 1 psi pressure differential.

When the chamber is evacuated for filling with the combustible mixture, the perforated plate supports the rupture diaphragm, seals the system from the atmosphere, and prevents a combustible mixture from entering the expansion bag. When the mixture is ignited, the increased pressure in the chamber pushes the diaphragm away from the plate. Since it is unsupported across the 5-1/2-in. diameter of the tube, it ruptures at a very low pressure. Due to the large open area of the support plate, the expanding gas experiences no significant impediment to its flow into the bag.

The bag is designed to contain all of the volume increase caused by burning of the combustibles. It is constructed of polyethylene sheet, heat-sealed to form an air-tight bag. Provision has been made to inflate the bag with nitrogen gas after an experiment to facilitate washing.

Because of the nature of the materials to be studied, it was desirable to eliminate all rubber "O" rings and gaskets in the system. Gaskets between the Plexiglas tube and aluminum attachments were cast from Silastic RTV Silicone Rubber. Rubber "O" rings in the fuel injector pump are being replaced with Teflon. Replacement of the pump "O" rings has caused a program delay due to the special sizes involved.

A spark ignitor has been fabricated. The circuit is basically that of a capacitor bank fired on command by a 5C22 hydrogen thyratron tube. Pointed, 1/8-in. diameter tungsten rods are used as spark electrodes. The output is variable up to 160 joules maximum.

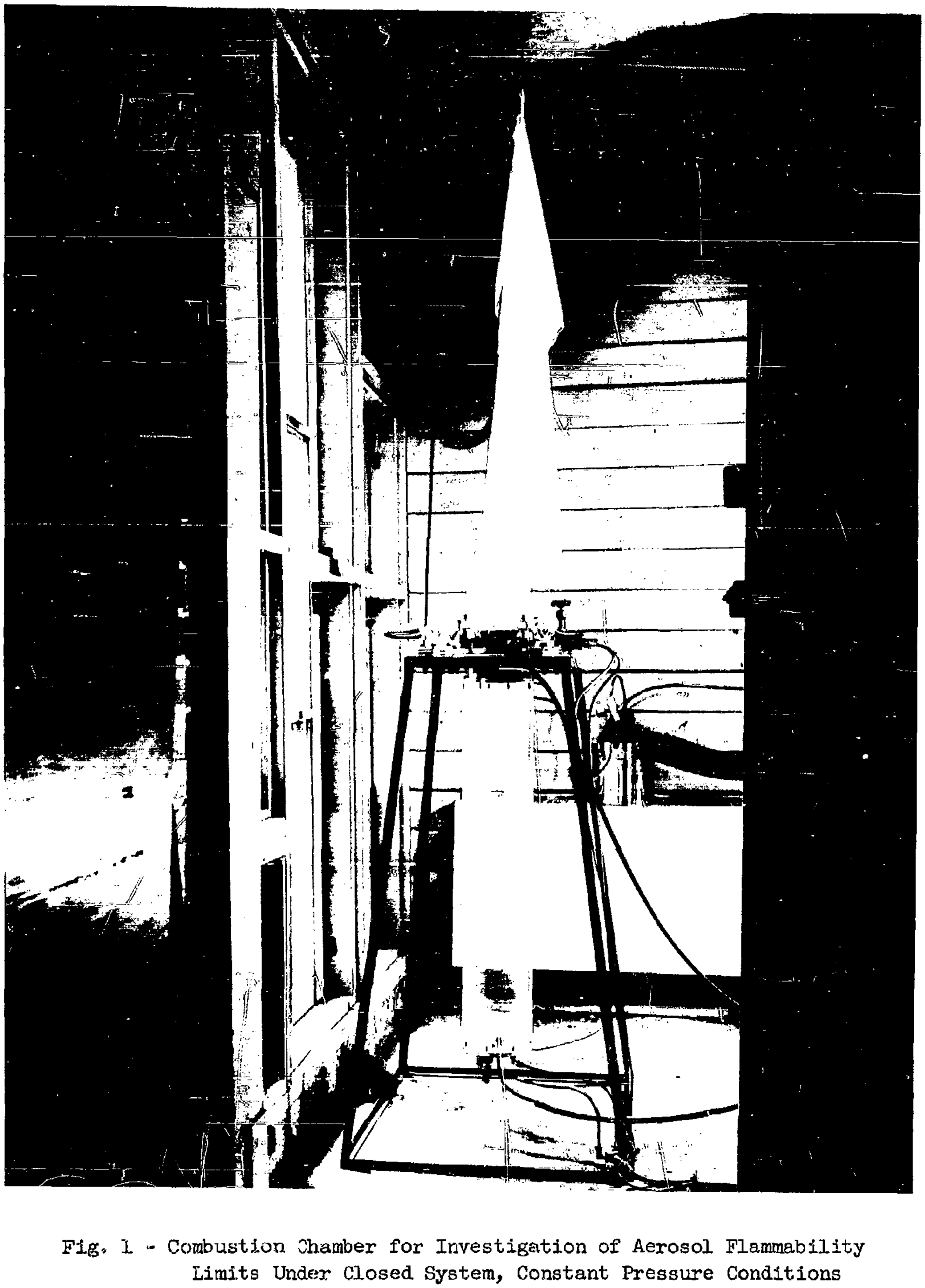


Fig. 1 - Combustion Chamber for Investigation of Aerosol Flammability Limits Under Closed System, Constant Pressure Conditions

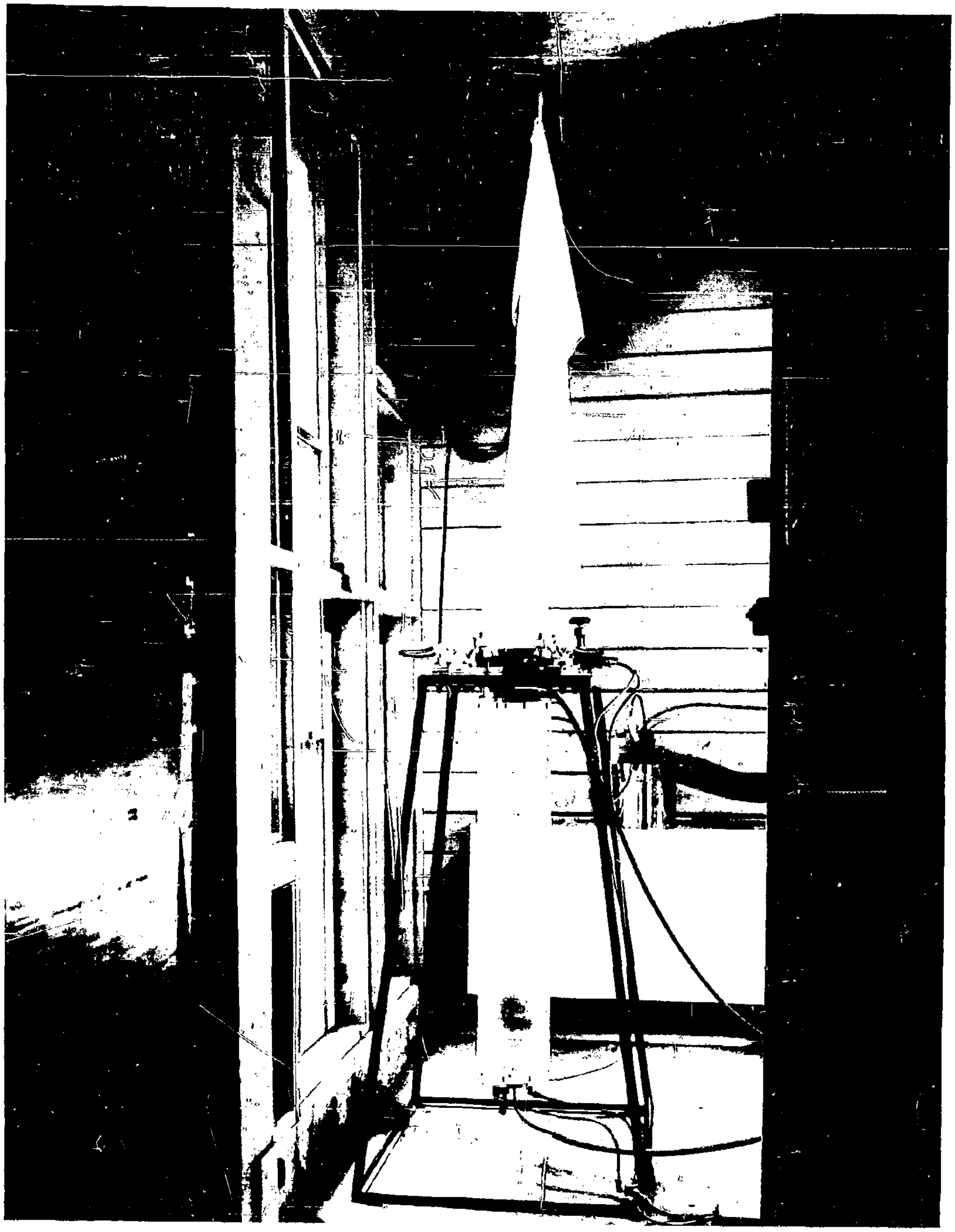


Fig. 1 - Combustion Chamber for Investigation of Aerosol Flammability Limits Under Closed System, Constant Pressure Conditions

The entire system has been test-fired using a methane-air gas mixture. The system performed as desired. The large diameter of the chamber creates an almost planar combustion wave which propagates smoothly down the tube. Two minor problems were encountered. The heat generated reduced the strength of the polyethylene bag where it is in direct contact with the aluminum collar. Two small stretch ruptures about 1 in. long occurred at this point. This problem was eliminated by constructing the mouth of the bag from a number of film plies, both to increase the mass and to reinforce the structure.

The second problem is the time necessary to get a uniform gas mixture after introducing the various mixture components. This problem is being solved by placing a small fan in the chamber. It will be driven through a leakproof seal in the chamber bottom. The blade is offset from the center to produce turbulence. The speed will be adjustable and care will be taken to keep the speed low enough to prevent de-aerosolization when a liquid phase is present in the system.

B. Detonation Product Gas Mixture

The current devices used to disseminate liquid aerosols explosively are the 155-mm. and 8-in. artillery shells and the 115-mm. M55 rocket. Drawings of these devices have been received from CRDL. Examination of these drawings disclosed that composition B-4 is used as a burster in all three. Composition B-4 consists of 60 per cent RDX and 40 per cent TNT, to which 0.5 per cent calcium silicate has been added. This material is vacuum cast to a loading density of about 1.72 g/cc. The explosive in the 155-mm. and 8-in. shell would be classified as under heavy confinement, according to Picatinny Arsenal. The explosive in the M55 rocket is much more lightly confined but it is difficult to assign a "degree of confinement" value to the liquid filling of this shell. Melvin A. Cook[1/] gives a complete calculation of the detonation products produced by Composition B at a loading density of 1.58 g/cc. A partial calculation for a loading density of 1.72 g/cc shows that the total moles of gas produced are the same at the higher density. This is in contrast to the usual case where an increase in loading density results in a decrease in the total moles of gas and an increase in the free carbon content. This different effect noted with Composition B may be caused by the better oxygen balance of this explosive.

The calculated composition at 1.58 g/cc is given below.

	Moles/kg	Per Cent by Volume
CO	8.0	25.55
CO_2	5.9	18.85
H_2	0.01	0.032
H_2O	0.1	0,32
N_2	10.6	33.85
NH_3	0.2	0.64
CH_4	0.6	1.92
CH_3OH	5.0	15.97
CH_2O_2	0.9	2.87
HCN	0	
C	0	
TOTAL	31.3	

Taking into account the facts that calculated values are subject to error because of lack of exact information regarding the correct equation of state, and Composition B is more fuel-rich than Composition B-4, it is believed that, for the purpose of this project, the above composition will serve adequately as a simulated detonation product gas mixture.

C. Candidate Aerosol Materials

While the intention of this project is to obtain some basic information on the phenomenon of aerosol flashing, it is desirable that the material aerosolized have characteristics of interest to CRDL. These general characteristics are (1) a high boiling point, (2) an intermediate spontaneous ignition temperature, and (3) a high flash point. A boiling point temperature on the order of 250°C, a spontaneous ignition temperature in the 300°C range, and a flash point of 175°C were selected as meeting typical requirements.

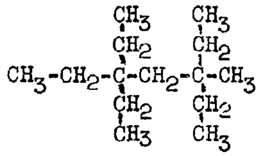
It was quickly realized that the scarcity of flash point and spontaneous ignition temperature data in these ranges would require a method of predicting values for compounds not listed in the literature. A literature search for existing data was made (covering the last 23 years) to provide the information for predicting values.

Data were obtained on four classes of compounds: pure aliphatic hydrocarbons, oxygenated hydrocarbons, aliphatic amines, and aromatic compounds.

The study made by Swartz and Orchin[7] shows that the spontaneous ignition temperature value is very dependent on the surface-to-volume ratio of the equipment used to measure it. The equipment used by CRDL is believed to be similar to that used by Frank and Blackham,[8] whereas most of the literature values were obtained in modifications of Bureau of Mines apparatus. By use of the information obtained by Swartz and Orchin, the value of 300°C in CRDL apparatus was graphically found to be equivalent to 262°C in Bureau-of-Mines-type apparatus.

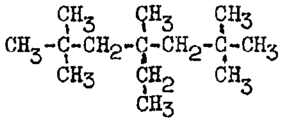
From the work of Zabetakis et al.,[9] it was found possible to predict the structure of an aliphatic hydrocarbon which would have the proper boiling point and spontaneous ignition temperature. By this method, for a spontaneous ignition temperature of 262°C, the average carbon chain length would have to be about 5.2 to 5.4 carbon atoms. For the correct boiling point, the hydrocarbon would have to contain about 14 carbon atoms.

The following type compounds fulfill these requirements:



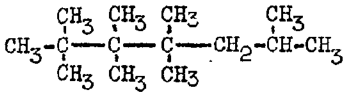
$$CH_3\text{-}CH_2\text{-}\overset{\overset{CH_3}{CH_2}}{\underset{\underset{CH_3}{CH_2}}{C}}\text{-}CH_2\text{-}\overset{\overset{CH_3}{CH_2}}{\underset{\underset{CH_3}{CH_2}}{C}}\text{-}CH_3$$

2,2,5,5-tetraethylhexane



$$CH_3\text{-}\overset{CH_3}{\underset{CH_3}{C}}\text{-}CH_2\text{-}\overset{CH_3}{\underset{\underset{CH_3}{CH_2}}{C}}\text{-}CH_2\text{-}\overset{CH_3}{\underset{CH_3}{C}}\text{-}CH_3$$

2,2,6,6-tetramethyl-4-methyl-4-ethylheptane



$$CH_3\text{-}\overset{CH_3}{\underset{CH_3}{C}}\text{—}\overset{CH_3}{\underset{CH_3}{C}}\text{—}\overset{CH_3}{\underset{CH_3}{C}}\text{—}CH_2\text{-}\overset{CH_3}{CH}\text{-}CH_3$$

2,2,3,3,4,4,6-heptamethylheptane

Unfortunately none of these compounds, or anything like them, can be obtained commercially as pure materials.

The work of Zabetakis et al. clearly illustrates the two types of ignition phenomena associated with hydrocarbons. When spontaneous ignition temperature is plotted vs. average carbon chain length, a modified Z-shaped curve is obtained which shows that the short chains have ignition temperature above 400°C and the long chains below 250°C. The straight, vertical portion of the Z falls within the 250° to 400° range.

Frank and Blackham point out that additives do not change the spontaneous ignition temperature of hydrocarbons which ignite by the high-temperature mechanism. The work of Zabetakis indicates that this fact would apply to hydrocarbons whose spontaneous ignition temperature was greater than 300° to 400°C.

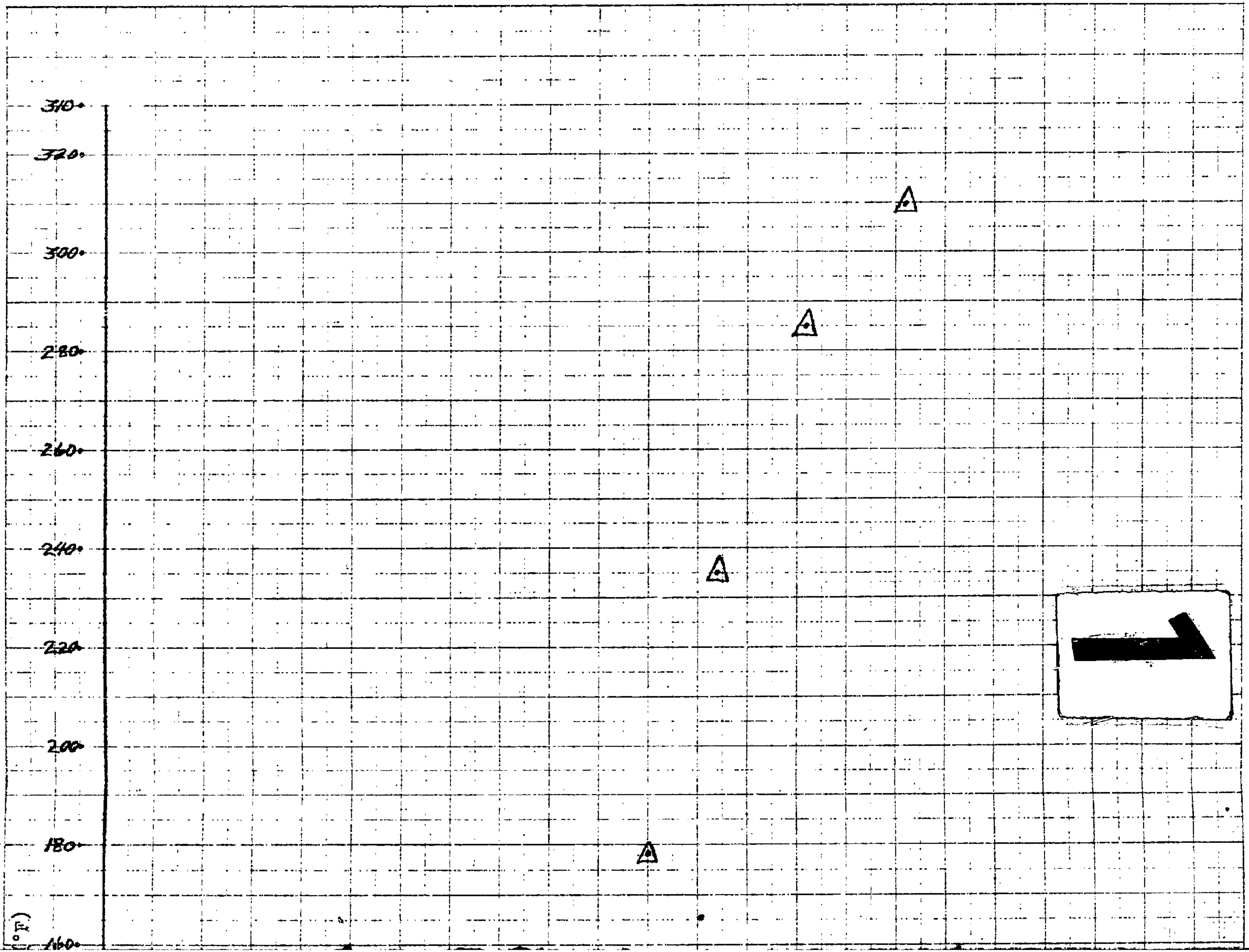
The literature data on the spontaneous ignition temperature (SIT) of oxygenated aliphatics, amines, etc., were insufficient to set up generalized rules regarding their behavior. Attempts are still being made to obtain additional data from manufacturers. In general, the successive substitution of OH groups on an aliphatic hydrocarbon molecule raises the SIT. The substitution of a ketone or aldehyde group lowers the SIT. The SIT values of amines are unusually high compared to corresponding hydrocarbons.

Flash point data are more readily available than SIT information. In a broad sense, flash points give a straight-line relationship with higher-range boiling points for any given class of compounds. A study was made of aliphatic hydrocarbons, aliphatic alcohols and glycols, aliphatic amines, and alkanol amines. These data are given in Figs. 2 and 3.

The curves indicate that in an aliphatic hydrocarbon the flash point is dependent on the boiling point. In general, it makes little difference whether or not part of the molecule is cyclic. The primary effect of an OH group is to raise the boiling point. Other than this, there is little effect, although the alcohol flash points tend to run slightly higher at a given boiling point. A large part of the scatter on both curves is believed to be due to the many sources of information used and the indiscriminate plotting of both open cup and closed cup data together, depending on which was available.

It is obvious that the proper combination of boiling point, flash point, and spontaneous ignition temperature cannot be obtained with any one of these series of compounds. It is obvious that a proper combination of boiling point and flash point alone cannot be obtained with these series of compounds.

In summary, it has been possible to find compounds which individually have the correct boiling point, or flash point, or spontaneous ignition temperature. There are some compounds which have both the proper flash point and boiling point, such as glycerin. The only compounds which are calculated to have both the correct boiling point and spontaneous ignition temperature are not available. The search for compounds which might have the desired spontaneous ignition temperature was severely hampered by lack of published data.



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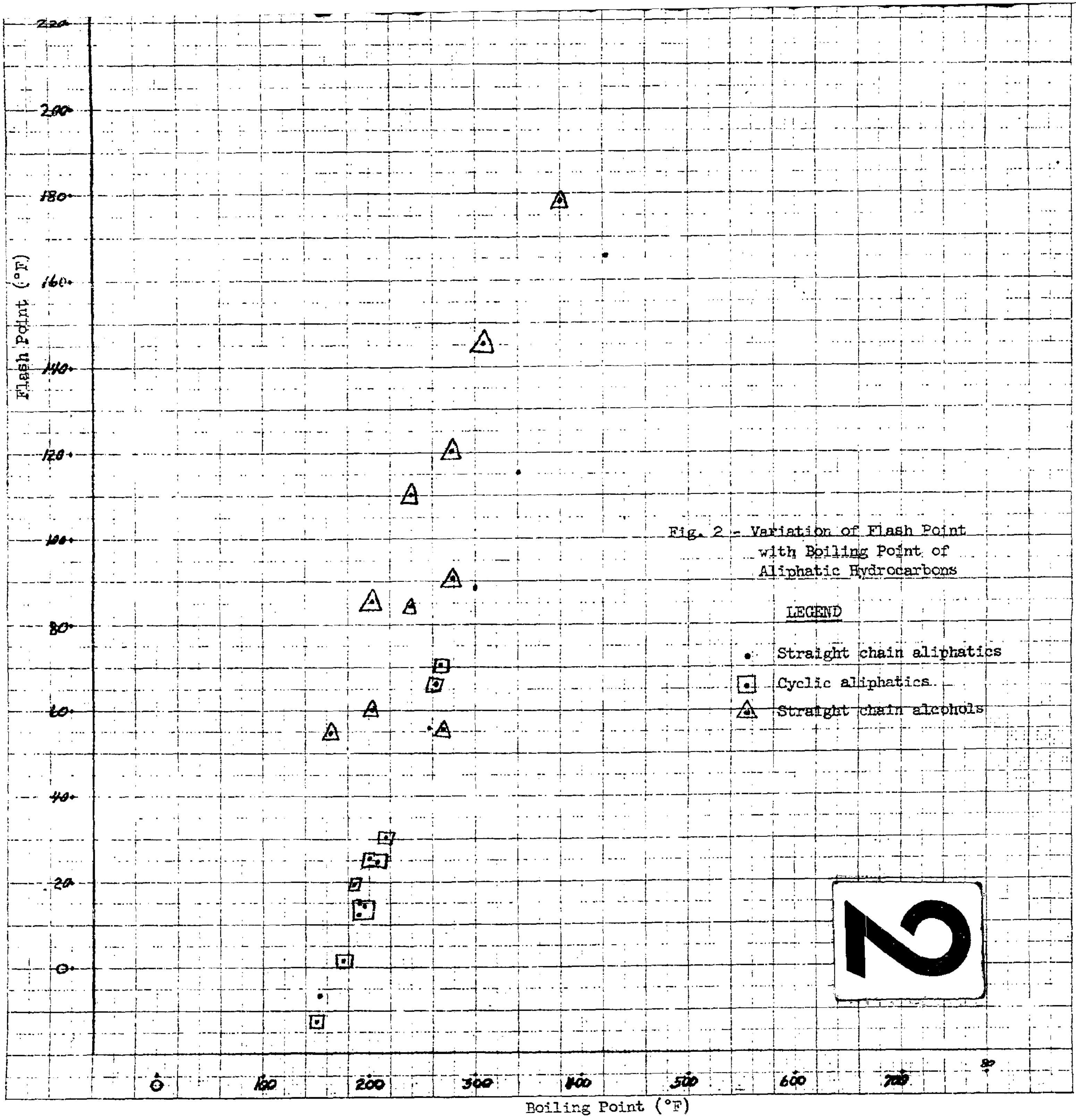

Fig. 2 - Variation of Flash Point with Boiling Point of Aliphatic Hydrocarbons

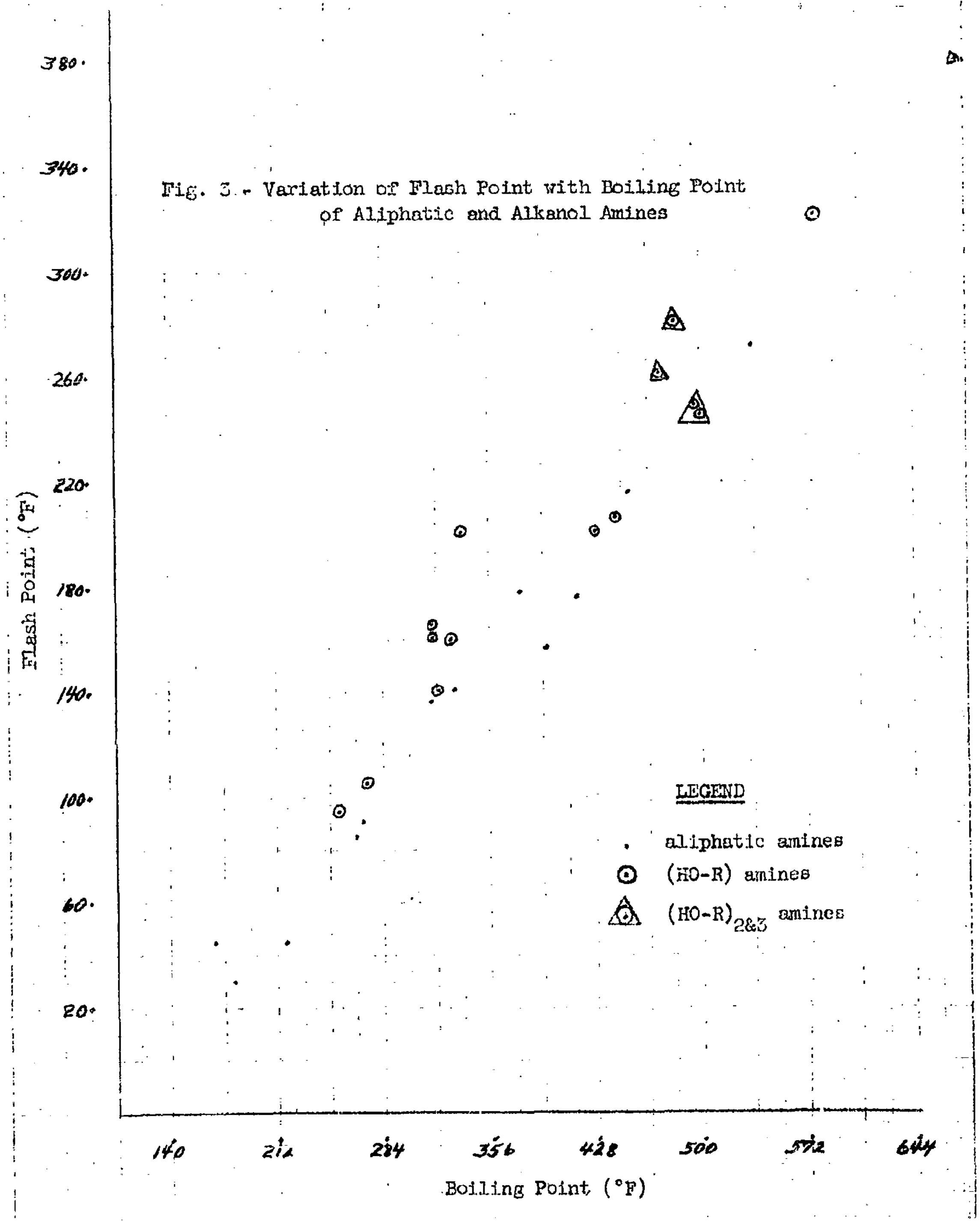

Fig. 3 - Variation of Flash Point with Boiling Point of Aliphatic and Alkanol Amines

A search of chemical warfare agents disclosed that compound QL comes close to the desired properties and is of low toxicity. This compound was proposed as a test material and has been accepted. The spontaneous ignition temperature of QL is high. Other materials to be evaluated will have to be selected with the thought of achieving a close match in spontaneous ignition temperature.

D. Shock Tube Fabrication

All basic design considerations for fabrication of a shock tube are complete. The important parameters which govern the operating conditions are being reduced to a set of charts. This will allow the operator to readily establish the conditions required to achieve a given thermal pulse in any aerosol-gas mixture.

The magnitude of the energy in the pressure wave shock at the moment of "breakaway" is being examined to establish guide posts for the shock tube experiments.

E. Project Status

The project is approximately 30 days behind the originally estimated time schedule. The delay has been caused by (1) delay in the starting date, (2) failure of supplier to deliver fuel injector pump on schedule and subsequent modification of the pump, and (3) revision in the requirements for an aerosol material. It is believed that this time can be made up later in the program within the original funds authorized.

F. Proposed Future Work

During the next report period we will determine the diesel fuel pump operating pressure required to produce the desired aerosol particle size. A stirring fan will be added to the bottom of the combustion chamber to aid mixing. Reports have been received detailing techniques for analysis of the test material. The equipment needed for this analysis is available. With the exception of obtaining experience with the procedures required to analyze for unconsumed aerosol, there is nothing which will further delay execution of the combustion chamber studies.

Design of the shock tube will be completed and the equipment will be installed in the near future. Complete information on all design parameters will be presented in the next letter report.

APPENDIX I

I. INTRODUCTION

This material was presented to the sponsor before the start of the program. It is included here for reference purposes only so that it will be available to orient those who are unfamiliar with the problem.

II. ANALYSIS OF THE PROBLEM

Aerosol combustion has been continuously studied for many years. The broad aspects of aerosol combustion include everything from studies of fuel oil burners to rocket motor combustion chambers, and from coal mine dusts to powdered detergent manufacture. The combustion of CW and BW agents is only one segment, but it is intimately related to the over-all field.

An analysis of aerosol flashing inhibition during explosive dissemination must be divided into two parts: (1) the ignition-source parameters, and (2) the suppression of aerosol combustion. These factors are inter-related, since the characteristics of the ignition may dictate the most favorable method for inhibition of the aerosol.

A. Potential Aerosol Ignition Sources

In the explosive dissemination of agents there are a number of possible ignition sources. All but one are related either to the explosive used or to the ammunition shell casing. These are in probable order of decreasing importance:

1. Ignition by combustion of the detonation products,

2. Ignition by contact with the detonation products,

3. Ignition by shock waves generated by:

    a. The burster charge,
    b. The explosion of an adjacent shell, or
    c. Shell fragments.

4. Ignition by electrical static discharge to relieve static potential developed when droplets move through the atmosphere, and

5. Ignition from hot shell fragments.

These are discussed below, at greater length, and in the same order.

1. Ignition by detonation product combustion: The burster charges used in chemical shell ammunition are usually Tetryol or Composition B. In a series of test firings of CW agents at Dugway Proving Ground, Tetryol was used in all but two tests, and Composition B was used once. Tetryol is composed of 35 per cent TNT and 65 per cent tetryl; Composition B of 55 per cent RDX, 40 per cent TNT, and wax.

a. Detonation products of explosives: Explosives can be classified in terms of their oxygen balance, i.e., the degree to which the detonation products are completely oxidized at the completion of the detonation process. TNT has a high negative oxygen balance. Under favorable conditions for a secondary explosion of the products of detonation of TNT with air, an explosion producing a heat of explosion of 2,500 kcal per gram of the original TNT might result. Hence, this secondary explosion could release nearly three times as much energy as the original primary explosion.1/ Secondary explosions of this type are well known. Tetryl also has a negative oxygen balance while that of cyclonite is slightly positive. The compositions of these three primary explosives, together with Tetryol and Composition B, are given in Table I in mole per cent.

TABLE I

COMPOSITION OF EXPLOSIVES IN MOLE PER CENT

Explosive	Carbon	Hydrogen	Nitrogen	Oxygen
RDX	16.20	2.70	37.80	43.20
Tetryl	29.28	1.74	24.36	44.64
TNT	36.96	2.20	18.48	42.24
Tetryol	31.97	1.90	22.30	43.80
Composition B	23.70	2.38	28.20	40.70

Tetryol and Composition B also have a negative oxygen balance, and Tetryol is more negative than Composition B.

The density to which an explosive containing TNT is compressed has a profound influence on the detonation products. This factor can be even more important than the oxygen balance in determining the combustibility of the products. Table II shows the wide variation obtainable.

TABLE II

VARIATION IN DETONATION PRODUCTS OF TNT AT THREE LOADING DENSITIES

	Loading Density, g/cc		
Moles/kg	0.6	1.0	1.6
CO	22.0	16.8	9.4
CO_2	1.5	3.5	6.8
H_2	5.1	2.5	0.6
H_2O	1.5	2.6	3.4
N_2	5.7	5.5	5.0
CH_4	1.3	1.9	2.2
NH_3	0.2	0.8	1.1
HCN	3.0	1.4	2.0
C	3.0	7.1	10.4

Table II indicates that, as the loading density increases, the CO_2 and C concentrations increase while the CO concentration decreases. Other factors being equal, increasing the loading density should reduce the ignitibility of the decomposition products and thus reduce secondary burning.

Tetryl and RDX exhibit similar changes in detonation products as a function of loading density.

In summary, all explosives with a negative oxygen balance produce very combustible detonation products. The combustibility, ignitibility, and composition of these products are strongly dependent on the loading density of the explosive charge. Table III illustrates further the magnitude of these phenomena by comparing TNT, tetryl, and RDX at a single loading density. Even though these materials differ significantly in oxygen balance, each produces large quantities of combustibles.

TABLE III

DETONATION PRODUCTS OF EXPLOSIVES AT A LOADING DENSITY OF 1.6 G/CC

Moles/kg	TNT	Tetryl	RDX
CO	9.4	10.6	8.0
CO_2	6.8	6.6	5.5
H_2	0.6	0.5	1.3
H_2O	3.4	2.2	8.0
N_2	5.0	7.0	12.1
NH_3	1.1	0.9	2.8
HCN	2.0	2.9	-
CH_4	2.2	1.6	-
C	10.4	2.7	-

The empirical assumption that improving the oxygen balance should reduce detonation product combustibility is only partly true. The loading density will determine the oxygen equilibrium between CO and CO_2 and will control the quantity of free carbon formed. Thus an oxygen-rich explosive at low loading density could produce as much combustible CO as an oxygen-poor explosive at a high loading density. On this basis, the tendency for secondary combustion of the detonation products would be the same.

There are two additional factors which influence the tendency for the detonation products to burn in a given system: the detonation temperatures produced and the brisance of the explosive. An increase in either increases the potential for ignition. Therefore, while it may be possible to lower the quantity of combustible products by changing the explosive oxygen balance, this may not eliminate the tendency of the products to burn if the detonation temperature or brisance are simultaneously increased.

In summary, the kind of explosive and the conditions under which it is detonated determine the tendency for the detonation products to burn with air after the explosion.

b. Summary of significant experimental data: In a recent series of firings at Dugway Proving Ground, attempts were made to correlate aerosol flashing to the time duration of the flash (as measured by IR sensors and photographically), the burster-to-agent weight ratio, and type of burster charge. Standardized bursters were apparently used in most instances.

In the majority of instances, when Tetryol bursters were used, the flash time varied from 0.002 to 0.007 sec. A second grouping of flash times occurred in the interval of 0.02 to 0.04 sec. The percentage of agent recovered did not correlate with the flash times in any manner, i.e., large recoveries were observed at flash times of 0.35 sec. and small recoveries at flash times of 0.002 sec. and vice versa. In a comparison test of Composition B and Tetryol, the flash times for Composition B varied from 0.6 to 0.06 sec. Compared to the Tetryol burster, the longer flash times did correlate with lower agent recovery, i.e., approximately 60 per cent for Tetryol and 10 per cent for Composition B.

In another series of tests, using a water-filled 115-mm. rocket with a Tetryol burster, the flash duration was 0.002 to 0.005 sec., indicating that the flash observed in the majority of instances with a Tetryol burster was primarily due to the burster alone.

A further observation on the results of the tests is that increasing the agent-to-burster weight ratio decreased the amount of agent lost.

Before a discussion of the above data in relation to the burster as the primary ignition source for aerosol flashing can be undertaken several other bits of information are needed. First, there is a relationship between agent loss and case strength. These data have appeared in a number of Army Chemical Center, Stanford, and Aerojet General reports. Second, agent loss can be minimized if the burster is surrounded by agent or if oxygen is excluded from the shell. These data appear both in Aerojet and Stanford reports. Third, high humidity inhibits combustion of the aerosol. Fourth, carbon tetrachloride around the burster tends to prevent flashing whereas brominated compounds are not as effective. This datum is from Aerojet General reports.

c. New interpretation and correlation of data: The explosion of burster charge explosives produces large quantities of combustible gases. The component of these gases, present in largest amount, is CO. When mixed with atmospheric oxygen, these gases form a highly combustible mixture which can be ignited with almost no added energy for activation. During the initial expansion of these gases and their mixture with air, they progress through a fuel/oxidant range from rich to lean. Unfortunately, CO is capable of forming explosive mixtures with air up to 100 per cent CO concentration,[2/] so that this gas is capable of combustion with air throughout the expansion of the detonation products until the CO concentration is less than about 12 per cent.

If it is assumed, for the sake of argument, that aerosol flashing is caused by the secondary burning of the burster explosive detonation products with air, then all of the above data fall neatly into place.

The flash duration observed when water was used as a filler is approximately the same as that observed when using an agent (both tests used a Tetryol burster). This fact indicates that the combustion of the agent occurs during the combustion of the detonation products, and that the aerosol cloud does not burn except when surrounded by the detonation product flame gases. Undoubtedly there are exceptions to this, but the normal loss of agent when Tetryol bursters are used occurs from this source. The Tetryol burster secondary combustion may produce aerosol combustion which is just below the critical value for ignition of total combustion of the aerosol-air cloud. This would tend to explain the wide variation in results experienced with Tetryol bursters.

The phenomenon observed when Composition B bursters are used presents a paradox. Composition B has a significantly lower negative oxygen balance than Tetryol. This should produce a smaller quantity of combustible detonation products and decrease agent loss. Since agent loss was increased with Composition B bursters, this suggests that one of two other phenomena may have been involved. The Composition B burster may have had a materially different loading density, thus changing the carbon monoxide to solid carbon ratio in favor of creating a more combustible mixture. On the other hand, this unusual behavior may signify that the chemical agent reacts with the oxygen in the detonation products and that agent destruction would be reduced by lowering the quantity of oxygen in the explosive.

The increased agent lost when the case strength (thickness) is increased is caused by the slightly longer time given to the detonation products before adiabatic expansion into the atmosphere. This slight delay allows the gases to come to better chemical equilibrium with the resultant production of additional CO. The greater CO concentration tends to produce a more severe secondary combustion with greater loss of agent.

If the burster is surrounded by agent, and/or oxygen is excluded from the shell, the expanding cloud of detonation products is prevented from mixing with oxygen until such time that the cloud expands to below either its self-ignition temperature or beyond its flammability limit. This physical configuration could also produce the phenomenon of direct inhibition of the CO-air flame by hydrocarbons of the CW agent system. This inhibition can be either chemical or physical since both forms are known. The increased loss of agent noted when the shell end closures are weaker than the shell casing is a related phenomenon. A weak end closure tends to cause separation of a portion of the detonation gases from the agent, allowing the gases to jet into the atmosphere from the case end in a manner most favorable for ignition of the gas-air cloud. Increasing the agent-to-burster weight ratio accomplishes the same thing as insuring that the agent surrounds the burster charge, i.e., it makes it easier to insure that combustion of the detonation gases is prevented.

As has been noted in the Aerojet reports, high atmospheric humidity tends to prevent aerosol flashing. Water vapor causes a rapid reduction in the upper flammability limit of CO with air,3/ thus tending to prevent secondary combustion of the detonation products. These same reports noted that CCl_4 was a better suppressing agent than bromine compounds. This conclusion is surprising since hydrocarbon combustion usually is inhibited much more by bromine than by chlorine. However, carbon monoxide-air combustion, as we have been discussing here, is inhibited more by chlorine than by bromine compounds, and CO is almost unique in this respect. For example, 1.16 per cent of CCl_4 will render CO nonflammable in air4/ but it requires 6.2 per cent methyl bromide5/ to accomplish the same task. Compare these data to that of a methane-air system; in this case it requires 13 per cent CCl_4 but only 4.7 per cent methyl bromide to prevent combustion.5/

d. Summary: There is a large body of evidence to indicate the burning of the burster detonation products as the chief culprit in aerosol flashing. The indications are that the combustion of the detonation products of the burster charge serves either to ignite the aerosol-air cloud, or to pyrolyze the agent during the combustion of the detonation products with air. This flashing can be prevented or minimized by

(1) Reducing the flammability of the detonation product gases,

(2) Changing the physical configuration of the agent and burster and/or the weight ratio, and

(3) Incorporating suitable inhibitors into the system.

This last factor is of major importance, and the reader is reminded that previous data show that the type of inhibitor used is of major importance.

2. Ignition by contact with detonation products: The ignition of high boiling-point hydrocarbon aerosols exhibits some interesting phenomena. There are a number of references to this but two bearing directly on the problem will serve to illustrate the point. Armour found that aerosols of moderately large particle sizes could not be ignited by spark energies up to 786 joules or exploding wire energies up to 468 joules. The aerosols were readily ignited by a 1-1/2 in. gas flame, a Nichrome wire shorted across 110 v.,

or a platinum or iron wire heated above 1016°C. Pistritto in an earlier investigation found essentially the same phenomena. The obvious trap of not allowing sufficient gap between the spark or exploding wire electrodes can be ruled out as Pistritto mentions this phenomenon in his report.

The data in the preceding paragraph imply a free radical mechanism of aerosol ignition and combustion. Pyrolysis of the simulants used, by sparks and exploding wires, did not produce sufficient quantities of molecular fragments to initiate the reaction. Flames produce the required chain carriers and initiators directly; therefore, a much lower rate of energy input, comparatively, is required for ignition. Hot wires produce pyrolysis products which can be significantly different from those of sparks and exploding wires. They can also be catalytic surfaces for reaction initiation; iron can be the equivalent of platinum in this regard.6/

Initially the explosive detonation products are rich in free radicals. There are considerable quantities of NO and OH radicals as well as H and O atoms. These are rapidly destroyed, within a few hundred microseconds of the detonation. Some of these, particularly OH, are remarkably long lived, requiring several milliseconds for complete recombination when the concentration is large. These radicals therefore have the potential of initiating aerosol-air combustion.

A second process can also occur. The detonation product gases initially are at a temperature of 2000°C to 3000°C. As these reacting gases move out through the chemical agent, direct reaction with the agent as well as pyrolysis of the agent can occur.

3. Shock wave ignition of aerosols: Shock waves are a potent source of thermal energy. The compressive force exerted is easily capable of causing pyrolysis of fuels and generation of free radicals.

There is no doubt that shock waves have the potential of causing ignition in a combustible system, and they are the proven cause of the phenomemon known as gun flash. When the propellent gases of a small arms or artillery weapon issue into the atmosphere, a standing shock wave is set up at the muzzle. This shock wave marks the line of demarcation between subsonic and supersonic flow. The gas in passing through this shock is strongly heated and compressed as kinetic energy is converted to heat. These gases expand, cool, and form an explosive mixture with the atmosphere, which is then ignited by hot gas and/or free radicals at the shock front. When this shock wave is destroyed, either by a nozzle or a bar-type suppressor, ignition is prevented and no flash occurs.

a. Shock waves from burster explosion: The most potent shocks available as ignition sources are those produced by the burster explosion. There are two shocks formed, the detonation shock and the "pressure wave" shock. Unfortunately, there is little information available at this instant to determine the relationship between the chemical agent filler and the movement of the detonation shock wave. If the agent moves an appreciable distance riding the surface of this wave, then there may be sufficient time to form an ignitible aerosol mixture. This does not seem to be too probable. The shock wave formed by the detonation has to be the agent responsible for the shell case rupture, and this action places it ahead of the agent filling at the moment of aerosol generation.

A different sequence of action occurs with the second shock wave. The detonation product gas molecules are initially receding from each other at sonic velocity, but are traveling at supersonic velocity with respect to the surrounding atmosphere and chemical agent. As these gases expand, they will overtake the surrounding agent filler, thus causing a shock wave to penetrate through the expanding agent after some mixture with air has occurred. This shock wave then could trigger aerosol flashing. The second shock, called a pressure wave, contains more net energy than the detonation wave.

The pressure-wave shock could also be the source of secondary ignition of the detonation products with air. The initial supersonic movement of the detonation product gases with respect to air does not allow penetration of the air across the shock front. The air essentially piles up, is heated by compression, and rides the surface of the wave. At about 20 to 50 times the original explosive diameter, the pressure in the product gases falls to one atmosphere and this shock front "breaks away" through the compressed air front causing additional heating in the initial product gas-air mixture, resulting in ignition. At properly chosen camera angles, high-speed photographs of the shell burst would show the expanding dark cloud (carbon particles) until its diameter was about 8 ft. At that point "break away" would occur and ignition could be observed at one edge, propagating through the remainder of the cloud.

There is a reservoir of untapped information available on the reaction of liquids under the influence of explosively generated shock waves, in the field of explosive forming of metals. An examination of these data should provide much fruitful information regarding the validity of the above hypothesis.

b. Shock wave from shell fragments: The initial burster explosion can produce shell fragments moving at velocities in excess of 2,000 ft/sec. The supersonic velocity of these missiles creates a shock wave which is equivalent to the bow wave of a small arms bullet. These shocks are energetically of low order and there are no known recorded examples of such a shock causing ignition of combustible gases, even under the most favorable circumstances. For example, a 0.30 caliber bullet fired through a balloon filled with hydrogen and oxygen will not cause ignition.

c. Shock waves from adjacent shell bursts: As noted in section (a) above, the burster charge creates a strong shock wave. If two or more charges are exploded simultaneously, as in a mine field, the shock wave created by an adjacent charge moving through the aerosol cloud could cause ignition. In addition, the line of intersection of two colliding shock waves would produce even more favorable conditions for ignition of an aerosol.

4. Ignition by static discharge: Dust and liquid droplets can acquire a high voltage charge by virtue of their movement in an air stream. Potential differences of 100,000 v. are possible. It is conceivable that a static discharge in an aerosol cloud could produce ignition. Ignition by static discharges in dust clouds is a well recognized hazard in many industries. However, it has been shown experimentally that aerosols of high-boiling liquids are difficult to ignite by spark discharges. Ignition by a static discharge is therefore considered possible but not probable.

5. Ignition by hot-shell fragments: Functioning of a chemical projectile or mine results in considerable mechanical work on the shell case. The detonation wave exerts force causing rupture and fragmentation. Much of this work then appears as heat in the shell fragments. After case rupture, these fragments are given considerable velocity and shock waves are formed as they move through the atmosphere.

It is difficult to see how these fragments could impart their latent heat to the aerosol cloud. The shock front effectively prevents any gaseous material from reaching the surface. Some particles with sufficient mass might have enough inertia to penetrate the front, but these would then be trapped inside the boundary layer surrounding the fragment.

Actually, the shock wave created by the fragment velocity is probably a much more potential ignition source than the heat from the fragment itself. Temperatures in this shock front should be much higher than the fragment temperature.

B. Aerosol Combustion Phenomena

1. Influence of drop size on combustion: There are two types of combustion involved in the burning of aerosols, (1) the diffusive burning of large drops, and (2) "premixed vapor" type burning of small drops.

Burgoyne and coworkers have identified the small droplet size as being below 10 microns and the large droplet size as above 40 microns, with a mixture of the two types of burning occurred for intermediate particle sizes. Work by Armour Research on higher boiling liquids than those used by Burgoyne indicated that the upper limit drop size tended to increase to between 50 and 100 microns. These data also suggest that the lower limit drop size was similarly suppressed.

The initiation of the diffusive burning of larger drops occurs in either a fuel-rich or a fuel-lean mixture. The droplet is surrounded by a vapor cloud and ignition occurs at the vapor-air mixture strength which is just able to support combustion. After ignition the subsequent combustion occurs in all mixtures within the flammability limits. For large drops, diffusion and not chemical kinetics is the rate-controlling process. Therefore, the burning of such drops produces merely a diffusion flame of vapor and air.

The burning of small drops can occur at any fuel/oxidant ratio from fuel-rich to lean since the combustion is primarily that of a premixed vapor in air. The burning rate process is controlled by chemical kinetics, and the flammability limits will be essentially the flammability limits of the pure vapor-air system.

C. Summary

The aerosol flashing problem can be analyzed as one or more of the following combustion systems:

1. Combustion of the air-aerosol mixture but only during combustion of the detonation products with air.

2. Pyrolysis of the aerosol during combustion of the detonation products with air.

3. Combustion reaction of the aerosol with the detonation product gases.

4. Pyrolysis of the aerosol while in contact with the detonation product gases.

5. Combustion of the aerosol with air alone.

The ignition sources for these systems are shock waves, spontaneous ignition of either the detonation products or the aerosol, combustion of the detonation products, or contact with hot metal fragments. These ignition sources can be broken down as possibly acting in a sequential form rather than independently. Thus for number one above, a shock wave could ignite the detonation products which in turn ignites the aerosol, etc. The present evidence, subject to experimental verification, indicates strongly that the detonation product gases are intimately involved in the problem and that elimination of their combustion would greatly reduce or eliminate aerosol flashing.

There is no doubt that in some cases the agent aerosol does combust with air. When this occurs, the aerosol combustion can be simplified to two characteristic types, a diffusion flame with air surrounding each fuel droplet and/or a vapor phase "premixed flame" type system, both of which can vary from fuel-rich to fuel-lean.

Inhibition of combustion must take into consideration the peculiarities of the system and particularly the mode of ignition. Ideally, the inhibitor should act to suppress the ignition of the detonation products (for case 1 above), but be capable of incorporation into the chemical agent for dispersion. Suppression of detonation product ignition would also reduce the exposure of the chemical agent to thermal decomposition processes.

Selection of an inhibitor also must be based on the fact that three different combustion systems are involved, i.e., detonation products with air, an agent diffusion flame, a rich and/or lean agent premixed flame, and various mixtures of all three.

III. METHOD OF APPROACH

An initial working concept of the flashing problem has been based on evidence available at this time. This concept states that aerosol ignition is a two-step phenomenon in which the detonation pressure (shock) wave ignites a detonation product gas-air mixture. The combustion of the product gases either ignites the agent aerosol or pyrolyzes the agent. The aerosol may or may not continue to burn after the combustion of the detonation products, depending upon circumstances at the time of ignition. This concept implies that aerosol flashing can be eliminated by (1) reducing concentration or changing the composition of the detonation product gases, (2) inhibiting the ignition and combustion of the detonation products, and (3) inhibiting the ignition and combustion of the aerosol. A successful attack on any one of these three items will break the chain of events which leads to aerosol flashing.

A. Influence of Detonation Product Combustion

1. Evaluation of pertinence: The working model states that the ignition cycle can be broken by reducing the quantity or changing the composition of the detonation products.

The ability of the detonation products to ignite an aerosol can be determined in the following experiment. A "synthetic" product gas composed of free carbon, carbon monoxide, carbon dioxide, hydrogen, water vapor, etc., will be charged into a large volume "combustion chamber", with air. A diesel fuel injector will squirt a known volume of fuel into the chamber, creating an aerosol. A spark igniter will initiate the combustion of the product gas-air mixture. The aerosol is not ignitible with a low energy spark, but the product gases are. By varying the product gas composition, aerosol particle size, and three concentration factors of product gases, air and aerosol, the following information can be obtained.

(1) The ability of the detonation products to ignite the aerosol.

(2) The ratio of product gas-air mixture to aerosol-air mixture required for ignition.

(3) Energy release required to obtain ignition and/or sustained combustion of the aerosol.

(4) The flammability limits of the combustible mixture.

(5) Relative ability of various product gas compositions to cause aerosol ignition.

(6) Effect of aerosol particle size on degree of total combustion.

Aerosol particle size can be varied by changing the diesel fuel injector operating conditions. Particles of 5 microns diameter are most conveniently produced but larger particles (variable up to 100 microns) can be created by decreasing the injection pressure, increasing the orifice diameter, etc.

2. Influence of solid detonation products: The solids produced by detonation of an explosive are primarily carbon, although in some instances nondetonated explosive particles also exist. To evaluate the effect of the detonation product gases on aerosol ignition requires inclusion of dispersed carbon particles in the synthetic gas mixtures used in Section III, A-1.

Incandescent solid carbon may itself be an ignition source. It can be conveniently studied experimentally as follows:

The combustion chamber to be used in the experiments of Section III, A-1, will be fitted with an electrically heated "puff box." High quality carbon black can be placed in this box, heated, and discharged as a solid aerosol by a puff of inert gas. The effect on the aerosol ignition can be observed as noted above. The experimental conditions to be varied would be the same as in part one above.

The injection of hot carbon particles into an air atmosphere may create experimental problems, in that ignition of the carbon cloud may occur directly. This phenomenon will not occur in the explosive dissemination of agents. For higher temperature work it may be necessary to "model" the carbon particle with an inert powder. This powder should approach the specific heat of carbon and its 3×10^4 to 4×10^4 Btu/hr radiative characteristics.

B. Shock Wave Ignition

It has been postulated that the initial factor in the ignition sequence is the shock (pressure) wave produced by detonation of the burster. This shock can ignite either a product gas-air mixture or the aerosol-air mixture directly. It is believed that a literature search and analysis will provide knowledge about the relative time history of the shock wave-agent-product gas cloud which will allow better interpretation of the significance of the shock as an ignition source.

The conditions required to achieve ignition by a shock wave can be experimentally determined in a shock tube. Three series of experiments can be performed. The first set involves creating shocks of varying magnitude in a series of synthetic product gas-air mixtures. The second set is the same as the first except that an air aerosol and an inert gas aerosol will be used. The third set will employ a series of three-component product gas-air-aerosol mixtures.

The following information can be obtained:

1. Ability of shocks to cause ignition of detonation product gases and aerosols.

2. Shock energy required to achieve ignition.

3. Relative ease of igniting a product gas-air mixture and an air aerosol.

4. Influence of product gas-air mixture on ignition ease of an air aerosol.

5. Flammability limits of the two combustible mixtures separately and of the combined three component mixture.

6. Ability of a shock wave to cause direct pyrolysis of an inert gas aerosol.

7. Differences in ability to achieve sustained aerosol combustion as a function of shock energy and the presence of detonation product combustion.

8. Influence of detonation product gas composition on ease of ignition by a shock wave and ability to ignite an aerosol.

C. Miscellaneous Ignition Sources

Hot metal fragments and static discharges are two other possible ignition sources.

Hot metal fragments are not believed to be capable of causing aerosol ignition for two reasons: (1) They probably are not able to contact the cloud directly due to the shock wave surrounding them, and (2) they are probably beyond the cloud at the time an aerosol is capable of being ignited. Initially the agent is in direct contact with the fragments at the time of case rupture. Small jets of liquid are expelled through the initial fissures. However, this is so early in the sequence of events that even a flame from ignition of these jets would be extinguished by the aerodynamic effects of particulate movement which occur shortly thereafter.

Some correlation exists between the material of the burster tube and occurrence of flashing, but this is not believed to be the same hot fragment ignition problem. The effect of burster tube construction material is probably related to (1) degree of explosive confinement, and/or (2) catalytic effects on the composition of the detonation product gases.

The problem can be studied experimentally by using the "puff box" technique of Section III, A-2, substituting various powdered metals for the carbon.

The ability of hot metal fragments to ignite an aerosol by the shock wave created by their high velocity can be determined by comparing shock data from experiments in Section III, B, to calculated shock energies for the fragments.

Ignition by a static discharge will be given further consideration but it is not believed that this is a cause of flashing. If the aerosol were generated by ejecting the liquid from a container via a nozzle, then it would be a probable cause. Aerosols generated by this technique can build up a charge between the cloud and the generator which can cause a "lightning flash" and aerosol ignition.

D. Use of Agent Combustion Inhibitors

From the agent standpoint, there is only one logical approach to the inhibition of aerosol flashing, the addition of a combustion inhibitor to the system. The combustion of an aerosol is of two types, diffusion or premixed, depending on the aerosol particle size. The actual combustion is between the vapor and air, never between the liquid and air.

3. Experimental approach:

a. Basic questions to be answered: A body of knowledge is needed on the inhibition of aerosol flashing which can be applied to the variety of problems that occur in different ammunition systems. Specifically, knowledge is required to answer the following questions:

(1) What is the best inhibitor to be added to the CW agent to prevent combustion of a CW agent-air aerosol under the conditions of (a) large droplet diffusion flame, and (b) small droplet vapor-type flame, at all fuel-air ratios?

(2) What is the best inhibitor to be added to the CW agent to prevent ignition of a CW agent-air aerosol when the droplets are (a) large, and (b) small, at all fuel-air ratios?

(3) What is the best inhibitor which can be added to a CW agent to prevent (a) ignition, and/or (b) combustion of the detonation products of the explosive?

(4) What effect does the presence of explosive detonation products have on the aerosol combustion when the products (a) do, or (b) do not burn?

b. Basic experiments to be performed: The questions require that the following be determined.

(1) The ignitibility of the CW agent in air, of the detonation products in air, and of the CW agent-detonation product-air mixture as a function of the fuel-air ratio and the type of inhibitor used.

(2) The combustibility (or flammability) of CW agent in air, of the detonation product in air, and of the CW agent-detonation product-air mixture, as a function of the fuel-air ratio and the type of inhibitor used.

IV. RESEARCH PROGRAM

A. Phase I

The factors involved in aerosol flashing are:

Particle size

Aerosol concentration

Vapor pressure

Temperature

Pressure

Flammability limits

Source of ignition

Each of these represents an experimental variable which could be investigated independently of the others. The problem is further complicated by the fact that the "source of ignition" represents a number of possible modes. This further multiplies the total number of variables to be studied, for each of the other six may be a dependent variable of the ignition source.

The ignition sources to be studied should be representative of the true situation as it exists in the field. One aim will be to determine how the ignition source influences the flashing problem. A secondary benefit of this approach will be "fall out" information which will determine the most probable ignition source in explosive dissemination.

1. Variables which will be studied: In the research to be conducted we will investigate the following variables.

a. The ability of four ignition sources to ignite aerosols. The four sources will be (1) ignition by combustion of the detonation product gases, (2) ignition by shock waves with and without the presence of detonation product gases, (3) ignition by hot metal fragments, and (4) ignition by hot solid carbon particles.

b. Aerosol particle size will be varied. The influence of this factor on each of the four ignition sources will be studied for nominal 5-micron and 100-micron size droplets. These sizes are representative of the two types of combustion which occur in aerosols.

c. The effect of aerosol vapor pressure will be studied on one or more liquids. Complete data, with variable particle size, all four ignition sources, and complete flammability limit data (as influenced by a and b above) will be obtained on one liquid. Sufficient data will be taken on the others to determine the influence of vapor pressure on these factors.

d. The influence of temperature and pressure will be confined to room temperature and atmospheric pressure. All equipment will be designed so that other temperatures and pressures can be studied at a later time, if necessary.

e. Flammability limit data will be determined as a function of aerosol particle size, ignition sources, vapor pressure of liquid, aerosol-air mixture ratio, and aerosol-air-detonation product gas (three-component) mixture ratio. Construction of complete flammability limit curves (as we will do) will provide absolute information on the influence of aerosol concentration on ignition and combustion.

2. Experimental investigation: The experimental investigation of these variables will be accomplished as follows. Two pieces of equipment will be used, one a special combustion chamber, and the second a shock tube. The special combustion chamber will be a closed system with an expanding gas bag so that the pressure on the system remains constant. Aerosols will be generated with diesel fuel injectors. These fuel injectors have the advantage of (a) producing a predictable, comparatively uniform particle size, (b) generating the aerosol instantaneously; (c) allowing complete freedom in generation of any desired aerosol concentration; (d) allowing completely independent variation in advantages (a), (b), and (c); and (e) being cheap and commercially available.

Section III, Appendix I, describes in detail both the type of experiments to be performed and the information which will be obtained.

B. Phase II

Phase II is concerned with the use of additives to inhibit or prevent the flashing of aerosols. A series of additives will be evaluated by flash photolysis using a special material to represent the aerosol. This material will be similar to CW agents which are involved in the aerosol flashing problem.

Commercially available materials will be evaluated along with some agents previously observed to have had an effect on aerosols.

Two or more of the best additives will be evaluated on aerosols, using both the combustion chamber and the shock tube. The flammability limit curves in which the air-aerosol-product gas mixture ratios are evaluated will be reevaluated using these additives to determine their effects.

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