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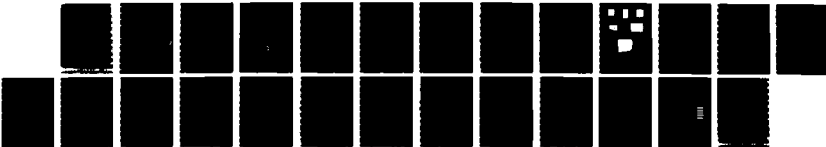
ATMOSPHERIC PRESSURE STUDIES OF LIQUID PROPELLANT DROPS
IN HOT FLOWS(U) ARMY BALLISTIC RESEARCH LAB ABERDEEN
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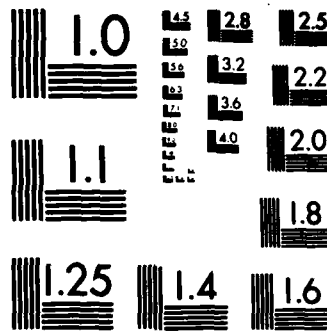
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TECHNICAL REPORT BRL-TR-2768

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ATMOSPHERIC PRESSURE STUDIES OF
LIQUID PROPELLANT DROPS IN HOT FLOWS

Richard A. Beyer

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19 ABSTRACT (Continue on reverse if necessary and identify by block number) Single drops of 13 M aqueous (HAN) ^{Chloroacetic acid Nitrate} and a liquid monopropellant (LP 1845) have been studied in hot flows from 700 to 950°C at ambient pressure. Measurements of drop diameter rate behavior show no effect of oxidizer presence and little temperature dependence. This report includes the first known observations of microexplosions of these liquid monopropellants. Drops are seen to explode from internal pressure after typically 25% of the volume has been consumed. These explosions do not appear to consume a substantial portion of the drop material, but they do result in the formation of many small drops, and hence, greatly increased surface areas. Arguments are presented that this effect is due to superheating of the water component.					
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I. INTRODUCTION

Although there is presently considerable activity in the pursuit of liquid propellant (LP) guns, little work has been done to characterize LP droplet and spray ignition and combustion under well-controlled conditions. More complete information of drop ignition behavior as a function of pressure, temperature, gas flow velocity, and history of the drop would allow new understanding of gas generation and heat release rates. Such advances could lead to enhanced performance or new design options.

Our previous studies in this area¹ have identified the free radicals that are abundant in the combustion of LPs, and have shown the feasibility of applying fluorescence imaging to LP spray combustion under conditions where droplet density is not excessive. However, these studies also brought out a definite need for an experimental design which has reproducible events so that many aspects of the process may be explored and techniques such as laser excited fluorescence may be applied.

Materials studied here have been 13 M aqueous hydroxylammonium nitrate (HAN) and LP 1845 (nominal composition, weight percent, HAN 63.2%, triethanolammonium nitrate 20.0%, and water 16.8%). The approach chosen was that which has been used successfully in research on hydrocarbon fuels. Single, reproducible droplets are made in the range from 50 to 300 micrometers diameter. These drops are then introduced into the hot gas flow, typically the post-flame gases of a laminar burner. Behavior can then be observed in microscopic detail as drops heat, ignite, and burn. The obvious difference between these and most earlier studies is that the present work is concerned with monopropellants and the role of the external oxidizer is no longer dominant. However, it is not unexpected that in many physical respects these drops will behave like multicomponent hydrocarbon fuels. These materials, particularly HAN, are known to decompose at temperatures ranging from approximately 120°C (standard DSC test),² through 190°C (drop suspended from a micro-thermocouple),³ to 230°C (drop freely floating in a fluid).³ Based on these numbers, and especially the lower values, which were the only ones available at the start of these studies, no difficulty was anticipated with achieving drop ignition. However, these studies are necessarily of a preliminary nature because of anticipated changes in ignition behavior as pressure is increased. Thus, a major goal is to develop the instrumentation and drop production capability in order to pursue a study of pressure effects with these same materials.

II. EXPERIMENTAL DESCRIPTION

Drops were formed for these studies using the technique commonly referred to as the "ink jet printer" method. This approach uses a piezoelectric ceramic cylinder which contains the liquid. One end is fed by a reservoir; the other has a small diameter orifice, approximately the diameter of the drops produced. The cylinder changes diameter and length upon application of a voltage pulse. This action sends a pressure pulse through the liquid and if conditions are correct one or more drops are ejected from the orifice. Drops can be produced on command from single drops up to kilohertz rates with excellent reproducibility.

Special considerations related to the corrosiveness, viscosity, and surface tension were necessary for adaptation to the production of LP drops. In particular, solders used to fasten an internal electrode to the ceramic were rapidly corroded; thus the LP itself was used to provide the ground connection. Care in design of the fittings to the ceramic eliminated trapped air bubbles which would interfere with correct functioning with any liquid.

Some preliminary observations were made with the drop maker and a simple burner. A very rich hydrogen/air flame on an upright 34 mm diameter sintered metal burner provided a small region of hot gas enclosed by the primary premixed flame and the conical diffusion flame of the leftover hydrogen with room air. The drops were injected upward at 45 degrees so that they passed over the burner and through this hot region. The corrosiveness of the drops on the sintered bronze was substantial and efforts were made to minimize contact.

After the above observation suggested that the LP drops would ignite and burn at atmospheric pressure, a freely falling drop experiment was designed and constructed for studies under more controlled and characterized environment. A schematic of this apparatus is shown in Figure 1. Drops were injected into the center of an inverted 58 mm diameter sintered bronze flat flame burner through a 7 mm diameter orifice. This injection tube was extended well below the burner surface to avoid interactions between the drop and the primary reaction zone of the flame. The hot gases were confined by a 350 mm long chimney. Quartz windows 50.8 mm wide and 250 mm long provided a view of the droplet heating region. Observations were made primarily with a microscope and camera, using a strobe light for backlighting. Drop sizes were obtained by measuring photographs taken through the microscope. This method resulted in typical uncertainties of less than five percent in diameter when good focus was maintained. For the studies reported here the flame was methane with either air or nitrous oxide.

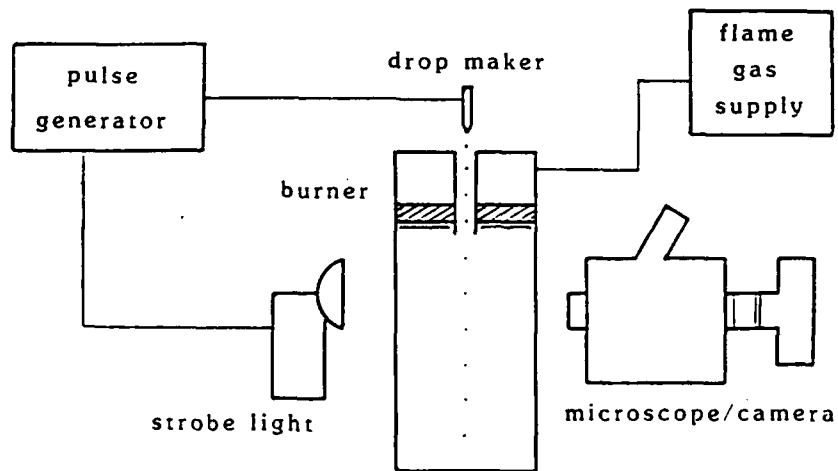


Figure 1. Schematic of Apparatus Used in Freely Falling Drop Observations

III. OBSERVATIONS

The preliminary observations with the injection of drops through the burnt gas region of the simple burner were both informative and encouraging. A drop formed in the present manner has an initial velocity on the order of 50 cm/sec. Thus the time in the hot gas region is not great and ignition might not necessarily take place at atmospheric pressure. However vigorous ignition was observed for drops up to $\sim 225 \mu\text{m}$ diameter. This ignition was characterized by bright, clearly visible emission. The most important characteristic of this ignition was that it occurred only in a primary reaction zone. Smaller drops ($\sim 100 \mu\text{m}$ diameter) ignited as they entered the hot region as they passed through the hydrogen-room air diffusion flame. Larger drops (up to $\sim 225 \mu\text{m}$ diameter) passed through the flame and ignited as they passed through the diffusion flame in leaving the hot region.

Following the success with the above simple configuration, the freely falling drop apparatus of Figure 1 was constructed. Observations with this apparatus were initially quite discouraging. In spite of the above mentioned values for LP and HAN decomposition under different conditions, and our moderate success with the simple configuration, virtually no ignition (light emission) was observed at first with the new design. The exception was when the drop impinged on a hot wall or window; in such cases ignition was prompt and vigorous. After some time was spent looking for ignition as characterized by substantial light output, careful observations were made of the drop diameters as a function of time in the flow. After the observation that the drops were clearly decreasing in size due to pyrolysis, it was found that emission from a weak flame in the drop path could be observed if an oxygen rich flow was used. As noted below, the presence of this flame did not affect the pyrolysis rate of the drops. Attempts were made to observe a flame in fuel rich conditions, where excess hydrogen should react with oxidizers from LP 1845 drop pyrolysis. No flame emission was observed under conditions where even a hydrogen-air flame should have been seen.

Typical drop size behavior as a function of time after pulsing the drop maker is shown in Figures 2 and 3. Because of limited view of the drops at the start of their fall, drops have already decreased in diameter prior to the measurements of these figures. Figure 2 compares the diameter data for 13 M HAN to that of LP 1845 below a methane-air flame in a region of approximately 800°C . Relative accuracy of drop diameter measurements are about $\pm 5\%$. In acquiring the data of these figures, gas mixtures were set to yield about 20% O_2 in the hot flow.

The calculated slopes from these plots are $-(1.2 \pm 0.2) \times 10^{-4} \text{ mm}^2/\text{msec}$ for the HAN and $-(1.6 \pm 0.3) \times 10^{-4} \text{ mm}^2/\text{msec}$ for the LP. Figure 3 shows similar behavior for $\sim 225 \mu\text{m}$ diameter drops in the 900°C flow of a methane-nitrous oxide flame. Although this larger drop survives longer in the hotter flow, the slope is comparable at $-(1.6 \pm 0.3) \times 10^{-4} \text{ mm}^2/\text{msec}$. Thus, within experimental accuracy, the behavior shown in these two figures is the same in terms of surface area loss rate.

Table 1 shows the results of two sets of measurements made to compare the effects of flow composition, particularly with respect to the presence of oxidizers. The numbers are recorded for the drop production and flame conditions as nearly the same as possible. For these observations, the time

and distance before microexplosion was maximized to get the greatest change in diameter. This condition was obtained by tuning the drop making parameters while observing the drop behavior in the flow. The 150 μm drops in the methane-air flame gases show that the presence of an oxidizing or reducing atmosphere does not strongly affect drop loss rate at one atmosphere. The second set of observations, with the 200 μm drops was intended to explore the effect of nitrogen oxides on the drop. These oxidizers are primarily nitric oxide and compose approximately one percent of the hot gases according to equilibrium calculations. However, from our previous flame studies⁵ it is more likely to be five percent or more. Again, within the experimental accuracy there is virtually no change in loss rate with the nitrous oxide.

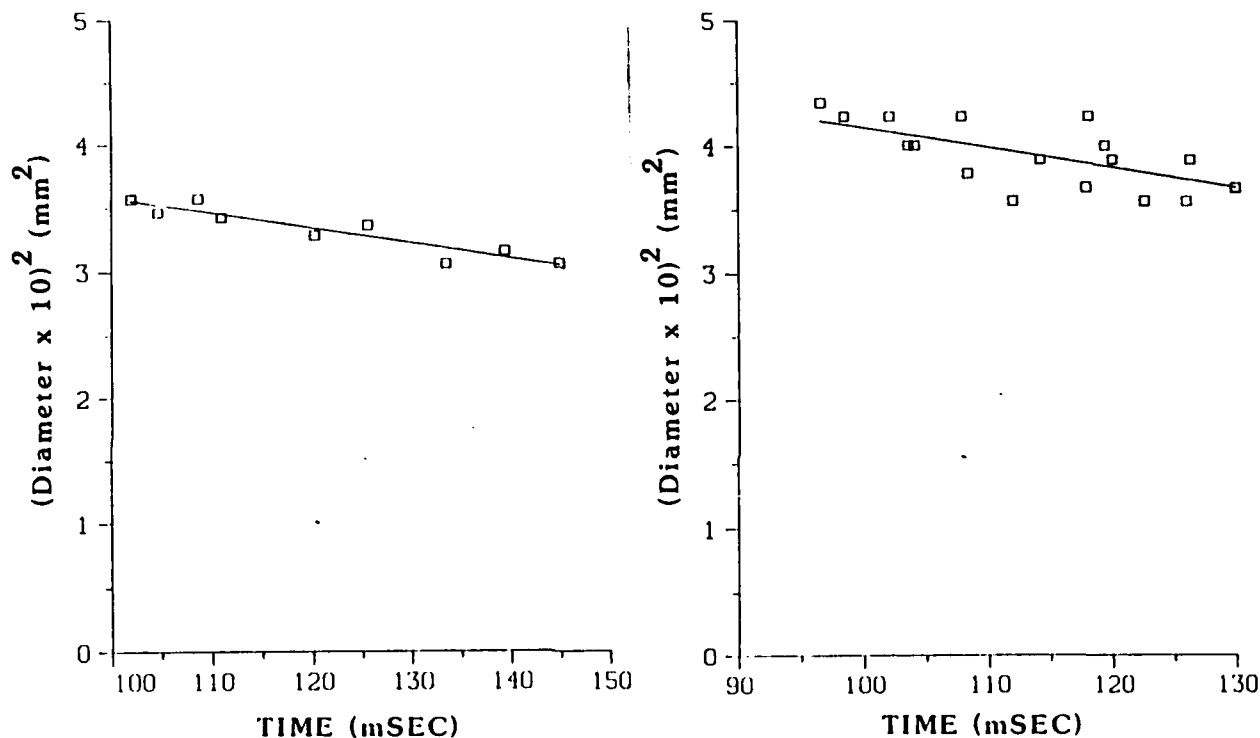


Figure 2. Drop Diameter Variation as a Function of Time After Drop Formation for (a) HAN and (b) LP 1845 Below a Methane-Air Flame at Approximately 800°C

As has been referred to above, after the drops undergo a period of heating and pyrolysis, they explode in a manner similar to that observed for other multicomponents drops in earlier studies.⁴ In appearance, the drops balloon up to as much as 4 times in diameter and burst. The explosions are extremely reproducible up to the burst point. The sequence is shown in Figure 4. When the drop bursts, it does so by splitting one side of the balloon, stretching the liquid into a line, and then forming a linear array of smaller drops which move in opposite directions from the original position. Because of the non-reproducibility from the time of burst point and the relatively

high velocity of the smaller drops, the distribution of sizes and total volume of liquid following the explosions has not been characterized. However, some of these post-explosion drops appear to be as large as one-fourth the diameter of the original drop. There are very small drops produced as well, as evidenced from the increased flame emission seen in the region of explosion. However, because of the substantial size of the larger residual drops, the entire original drop is not consumed immediately following explosion.

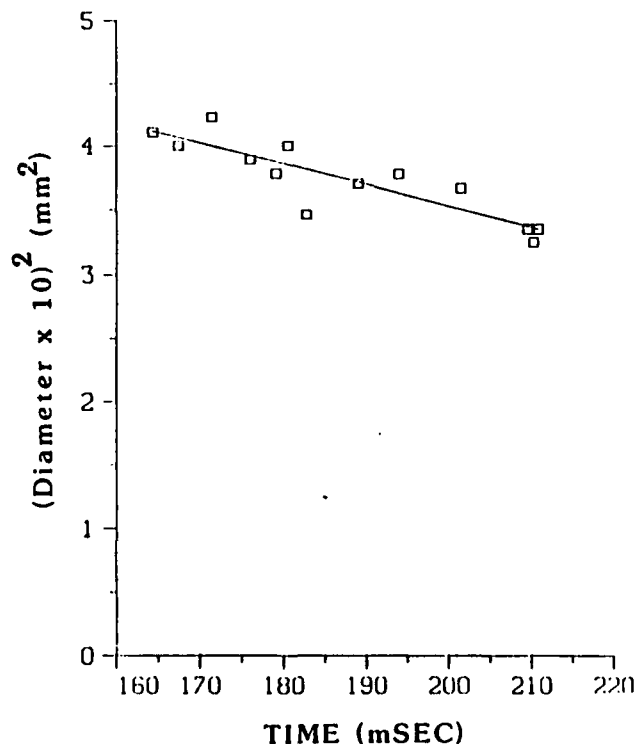


Figure 3. Drop Diameter Variation as a Function of Time After Formation for LP 1845 Below a Methane-Nitrous Oxide at 950°C

Table 1. Change in LP 1845 Drop Dimensions for Fixed Time (80 msec) and Temperature (850°C) as Hot Flow Composition is Varied

Flame	Initial Diameter (μm)	Percent Change	
		Diameter	Volume
CH ₄ /Air (lean)	150	11 ± 4	30 ± 4
CH ₄ /Air (rich)	150	12 ± 1	30 ± 4
CH ₄ /Air (lean)	200	8 ± 1	23 ± 4
CH ₄ /N ₂ O (lean)	200	7 ± 1	19 ± 2

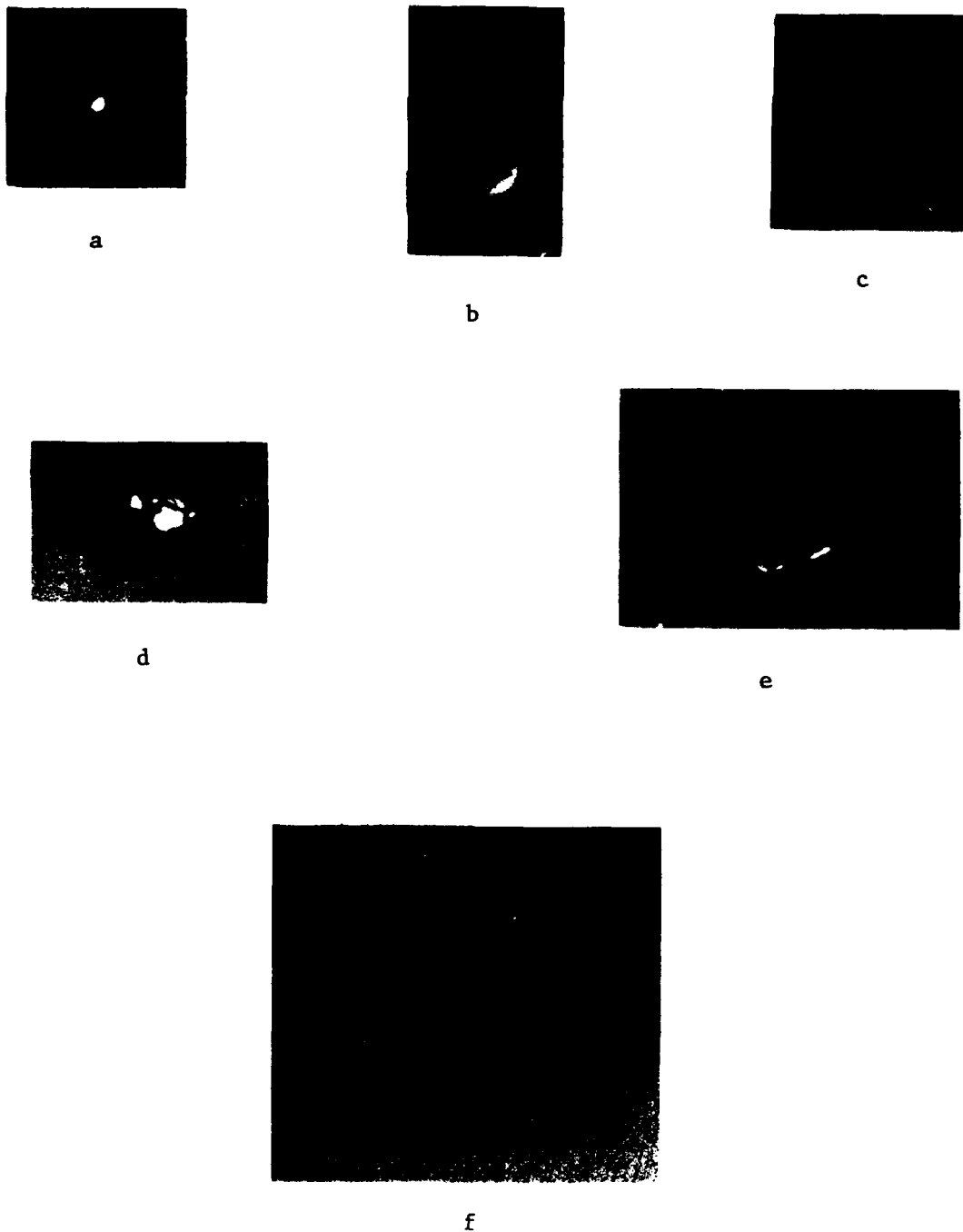


Figure 4. Drop Microexplosion Sequence for a 150 μm Diameter Drop of LP 1845. Shown are (a) the drop during pyrolysis, (b) a double exposure of ballooned drops, one near maximum diameter, (c) a balloon with the back opened, (d) one with the side opened, (e) an arc of liquid as surface tension pulls it together in one direction as momentum continues to stretch it in the other direction, and (f) the subsequent formation of an arc of drops moving apart.

IV. DISCUSSION

A. Decomposition Temperature of HAN and LP 1845

As was noted earlier, the decomposition temperature of HAN has been "known" to be ca. 120°C from DSC measurements. In the past, the assumption was made that LPs such as 1845 that are based on HAN will show similar decomposition characteristics; that is, the HAN will decompose as it would out of that solution. Concurrent with the present work, Decker, et al.,² have been more fully characterizing this decomposition value. However, it was clear once carefully controlled observations were under way that freely falling drops of neither HAN nor LP 1845 were in fact going to decompose noticeably or ignite under conditions where other liquids with higher ignition points were burning. This situation led to the micro-thermocouple and floating drop measurements of Law.³ There are two factors which may be important here. The first is that the heating rate of these materials is substantially faster than with conventional DSC experiments. It is well known that ignition of energetic materials is a time and temperature phenomenon; thus a direct comparison should probably be made only with care. It is also possible that the contact of a surface, even as a fine wire, changes the decomposition temperature significantly. It is easy to speculate on the mechanism for surface effects, especially with regard to the trapping of reactive species which might promote further reaction. The effect of the wire in the thermocouple tests suggests something different, perhaps providing a nucleation site for superheated water or a point where local concentrations of reactive species can build to a critical value. It is clear that this area requires further study if an understanding of controlling processes is sought.

B. Drop Pyrolysis Measurements

The measurements of drop diameter with time in the flow are clearly preliminary and probably should not be interpreted quantitatively. No effect on the presence of oxidizer in the flow or the appearance of a visible flame in the drop trajectory has been noted. There does appear to be a trend toward higher loss rate at the higher temperature of the nitrous-oxide flame with LP drops, but even that is not outside possible scatter in the data. However, these measurements do demonstrate that the drops are behaving in a classical pyrolysis fashion under these conditions. They show no behavior different from typical hydrocarbon fuels other than those properties that can be accounted for on the basis of viscosity and surface tension. Thus it will be extremely interesting to observe the changes in behavior as pressure is increased and monopropellant behavior becomes dominant.

C. Drop Explosions

The explosions of the drops as they pyrolyze is of considerable interest to understanding the events following the injection of an LP into a hot environment. Multicomponent drop explosions have been demonstrated and causes understood for several cases. There are three possible causes of these explosions. The first one has been demonstrated with miscible multicomponent drops where a concentration gradient builds because rapid surface evaporation of the most volatile component cannot be satisfied by the slower liquid diffusion rates.⁴ Following internal homogeneous nucleation of this species, it becomes superheated and interior pressure balloons the drop. The second

possible cause is interior liquid phase decomposition into gas phase products and subsequent pressure buildup.⁶ The third possibility would be some additional liquid phase reactions which liberate gas or a high vapor pressure liquid at the interior. This mechanism has not been reported in the literature and might in fact be difficult to distinguish from decomposition.

With the present measurements, there is little hope of determining whether the mechanism is physical, i.e., the first mentioned above, or from interior chemical activity. In a separate study,³ it has been found that the explosion temperature of a series of LPs prepared from 1845 by adding successively more water shows a convergence to the pure water explosion temperature as the LP gets very dilute. While the available data is not definitive, it is consistent with the LP explosions being driven by superheated water. The observation that small changes in drop formation parameters can turn the explosions on and off also strongly supports a physical mechanism.

The distinction between these two mechanisms becomes important because of possibly differing behavior as pressure increases. It has been shown that the physical one, superheating of water, occurs more rapidly with increasing pressure for miscible mixtures and water/oil emulsions. The explanation given is based on the pressure behavior of two parameters. The liquid boiling point and therefore the drop temperature can increase with pressure. On the other hand the limit of superheat, at which point liquid gasification occurs spontaneously, has little pressure dependence away from the critical point. Thus, with increasing pressure, the interior of the drop can more readily reach the superheat temperature and subsequently explode. If the explosions are dominated by chemical reactions, including decomposition, the pressure behavior may be different. In both cases, the rapid increase in burning rate of LP with pressure may make the drop size at explosion smaller. Because of the uniqueness and complexity of the LP drop situation, observations over at least a limited range of elevated pressures are critical to characterizing both the phenomenon and its importance in various gun designs.

V. FUTURE WORK

As mentioned above, this study was done primarily as a precursor to higher pressure studies where the LPs ignite as monopropellants. This work will be pursued in our laboratory at pressures up to about 1000 psi. In order of emphasis, studies of the drop burning and explosion phenomenology at elevated pressures will come first. Also of substantial interest are the changes in the heat release region and development of the gas phase flame around the drop as pressure is increased. The former will be studied by shadowgraph techniques; the latter by spectroscopic methods, especially laser excited two dimensional fluorescence imaging.

VI. SUMMARY

Freely falling liquid propellant drops have been studied in hot gas flows at ambient pressure. They have been found to pyrolyze partially and subsequently explode at flow temperatures from 700 to 950°C. Other observations strongly suggest that superheating of the water is more likely than decomposition of components as the explosion mechanism. Preliminary

measurements of the rate of drop diameter decrease show neither strong difference between 13 M HAN and LP 1845 under similar flow conditions nor strong temperature effects. The need for extending these observations to elevated pressure is clear.

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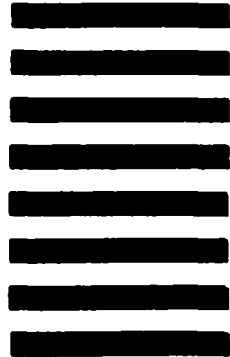
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