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Quarterly Technical Summary Report No. 10
September 1, 1964 to November 30, 1964

RESEARCH ON THE DEFLAGRATION OF
HIGH-ENERGY SOLID OXIDIZERS (U)

Contract No. AF 49(638)-1169

to
Air Force Office of Scientific Research
Washington 25, D. C.

from
Kinetics and Combustion Group
Atlantic Research Corporation
Alexandria, Virginia

January 19, 1965

MAY 20 1965

Chief Investigators: J. B. Levy
R. Friedman
Internal Consultant: G. von Elbe
Chemist: S. J. Adams
Chemist: C. Midkiff

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RESEARCH ON THE DEFLAGRATION OF HIGH-ENERGY SOLID OXIDIZERS (U)

I. ABSTRACT

Further measurements of the rate of deflagration of pressed strands of hydrazine diperchlorate at elevated pressures have been made. The data are erratic. The effect of copper chromite catalyst on the deflagration of hydrazine diperchlorate at atmospheric pressure has been examined at a higher catalyst content. Flame temperatures have been calculated for one, ten and one hundred atmospheres pressure and are fairly constant at 1600°K. Attempts at measurement of the flame temperature by means of fine Pt-Pt, 10% Rh thermocouples have been unsuccessful--apparently because of catalysis by the metals of condensed phase reactions. Exploratory experiments have been performed with ammonium perchlorate, hydrazine perchlorate and hydrazine diperchlorate on the deflagration of spheres of these oxidizers at atmospheric pressure in fuel gas streams.

II. INTRODUCTION

In this program we are studying the deflagration of a series of high energy oxidizers. In earlier work in this laboratory the self-deflagration of ammonium perchlorate has been studied in some detail (1). In the present program, we have examined the behavior of hydrazine perchlorate and have recently reported our results and conclusions on the self-deflagration of this oxidizer (2). Most recently we have begun the investigation of hydrazine diperchlorate and in our recent reports we have reported on the self-deflagration of this material from 300 psig to 1000 psig and on the effects of certain catalysts on the deflagration (3).

In the present report we describe further studies of the deflagration of hydrazine diperchlorate. The studies deal with the following aspects of the deflagration process: the self-deflagration of hydrazine diperchlorate in inert gas at elevated pressures; the deflagration of catalyzed hydrazine diperchlorate strands at one atmosphere; the flame temperature of hydrazine diperchlorate; the deflagration of hydrazine diperchlorate at ambient pressure in a stream of fuel gases.

III. PROGRESS DURING PRESENT PERIOD

A. The Self-Deflagration of Hydrazine Diperchlorate

In our last report data were presented for the self-deflagration of pressed strands of hydrazine diperchlorate for pressures from 300 psig to 1000 psig. The strands were cylindrical in shape with a diameter of 8 mm. It was pointed out that both the lower limit of deflagration, about 300 psig, and the general level of the deflagration rate were very similar to the corresponding parameters for ammonium perchlorate. A certain degree of scatter in the results for hydrazine diperchlorate was also noted. Because of the scatter in the data it was felt desirable to measure further deflagration rates and this has been done. The results are shown in Figure 1. The additional points, at 300 psig, 450 psig, 550 psig, and 735 psig have not reduced the scatter and they suggest that the poor reproducibility of the results is due to small differences in the composition. The new points are fairly self-consistent; however we intend to make further measurements.

B. The Deflagration of Hydrazine Diperchlorate-Copper Chromite Mixtures

Although pressed strands of pure hydrazine diperchlorate will not support deflagration below about 300 psig, we have found (3) that strands pressed from mixtures of hydrazine diperchlorate and copper chromite undergo smooth deflagration for copper chromite contents of 1%, 2% and 5% at one atmosphere; the rate increased linearly with catalyst content. We have extended these experiments to the composition containing 10% copper chromite. The data for this strand are tabulated below with our previous results for comparison.

TABLE I
The Deflagration of Hydrazine Diperchlorate-Copper
Chromite Strands at One Atmosphere

No.	Entry	Copper Chromite(weight %)	ρ (g/cc)	Linear Rate (cm sec ⁻¹)
1	193	0.5	2.07	did not deflagrate
2	192	1.0	2.09	0.081
3	189	2.0	2.11	0.12
4	187	5.0	2.11	0.25
5	237	10.0	2.16	0.24

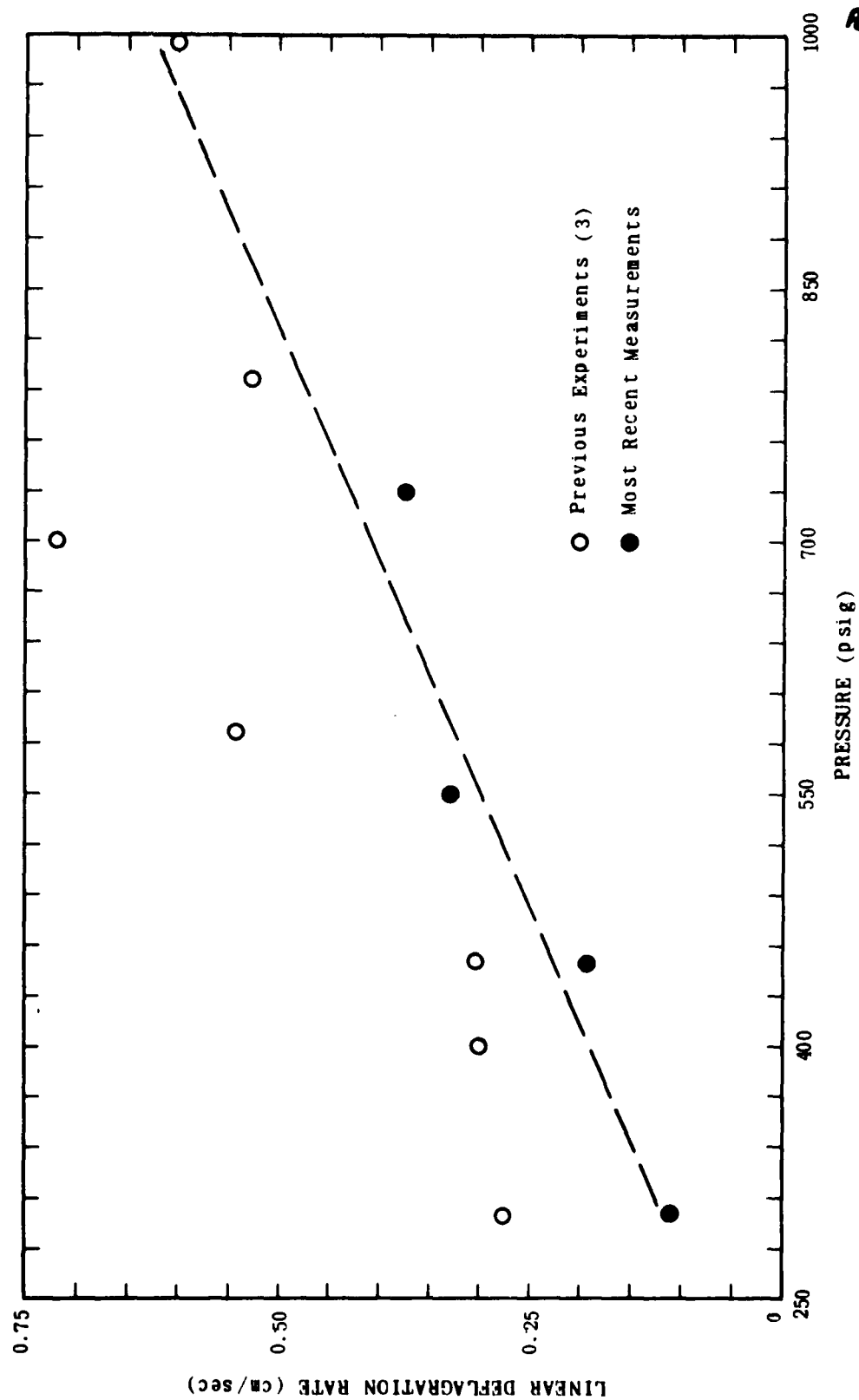


Figure 1. The Self-Deflagration of Pressed Hydrazine Dimerchlorate, ($\rho = 2.05 \pm 0.05$ g/cc).

The deflagration rate appears to have reached a plateau. This phenomenon probably arises as a result of a balance of the catalytic effect of the additive and its diluent effect--since it is not a fuel.

C. The Flame Temperature of Hydrazine Diperchlorate

In our last report a tentative figure of 1600°K was given for the flame temperature of hydrazine diperchlorate at one atmosphere. In the present period thermodynamic calculations have been performed for the state of the products of hydrazine diperchlorate self-deflagration at pressures of one, ten, and one hundred atmospheres. The calculations were performed on the IBA 7090 computer and assume constant-pressure adiabatic combustion to give equilibrium products. The results are shown in Table II. The flame temperatures are fairly constant at 1592.8°K, 1599.7°K, and 1604.0°K for one, ten, and one hundred atmospheres respectively.

We have performed a number of experiments in an effort to measure the flame temperature of hydrazine diperchlorate by means of fine (2-3 mil) Pt-Pt, 10% Rh thermocouples. The thermocouples have been inserted in holes drilled through the pressed strands. Thermocouple voltages were recorded by means of a Visicorder.

Experiments were first performed on ammonium perchlorate to check the procedure and results in agreement with our earlier work on this oxidizer were found (1).

Experiments with hydrazine diperchlorate to date have been unsuccessful in that the thermocouples have broken during the experiments. Examination of motion picture films of these experiments has yielded evidence that the thermocouple wire is catalyzing a subsurface reaction. Figure 2 is an enlargement of a frame in the motion picture record of an experiment at 400 psig. In this case the strand was pressed with the thermocouple in place. The glowing area around the head appears to be below the molten surface. Presumably the subsurface reaction results in destruction of the thermocouple. We are presently preparing ceramic-coated thermocouples (4) and will attempt measurements with these.

D. Deflagration Experiments in Fuel Gas Streams

At the Tenth Combustion Symposium, experiments were reported in

TABLE II
Hydrazine Diperchlorate Constant-Pressure Adiabatic
Combustion Initial Temperature 25°C

Pressure in Atmospheres	1	10	100
Flame Temperature (°K)	1592.8	1599.7	1604.0
Total Moles/100g.			
Species (Moles/100g)	3.440	3.437	3.436
H	0	0	0
O	2.802×10^{-5}	0.626×10^{-6}	0
N	0	0	0
Cl	2.167×10^{-2}	1.274×10^{-2}	7.361×10^{-3}
H ₂	0.858×10^{-5}	2.925×10^{-6}	0
H ₂ O	0.869	0.864	0.862
OH	1.223×10^{-3}	7.229×10^{-4}	4.193×10^{-4}
O ₂	1.280	1.282	1.284
H Cl	0.837	0.846	0.851
N ₂	0.427	0.427	0.427
NO	3.675×10^{-3}	3.788×10^{-3}	3.859×10^{-3}
NO ₂	1.879×10^{-5}	6.018×10^{-5}	1.918×10^{-4}
NH	0	0	
NH ₂	0	0	
NH ₃	0	0	

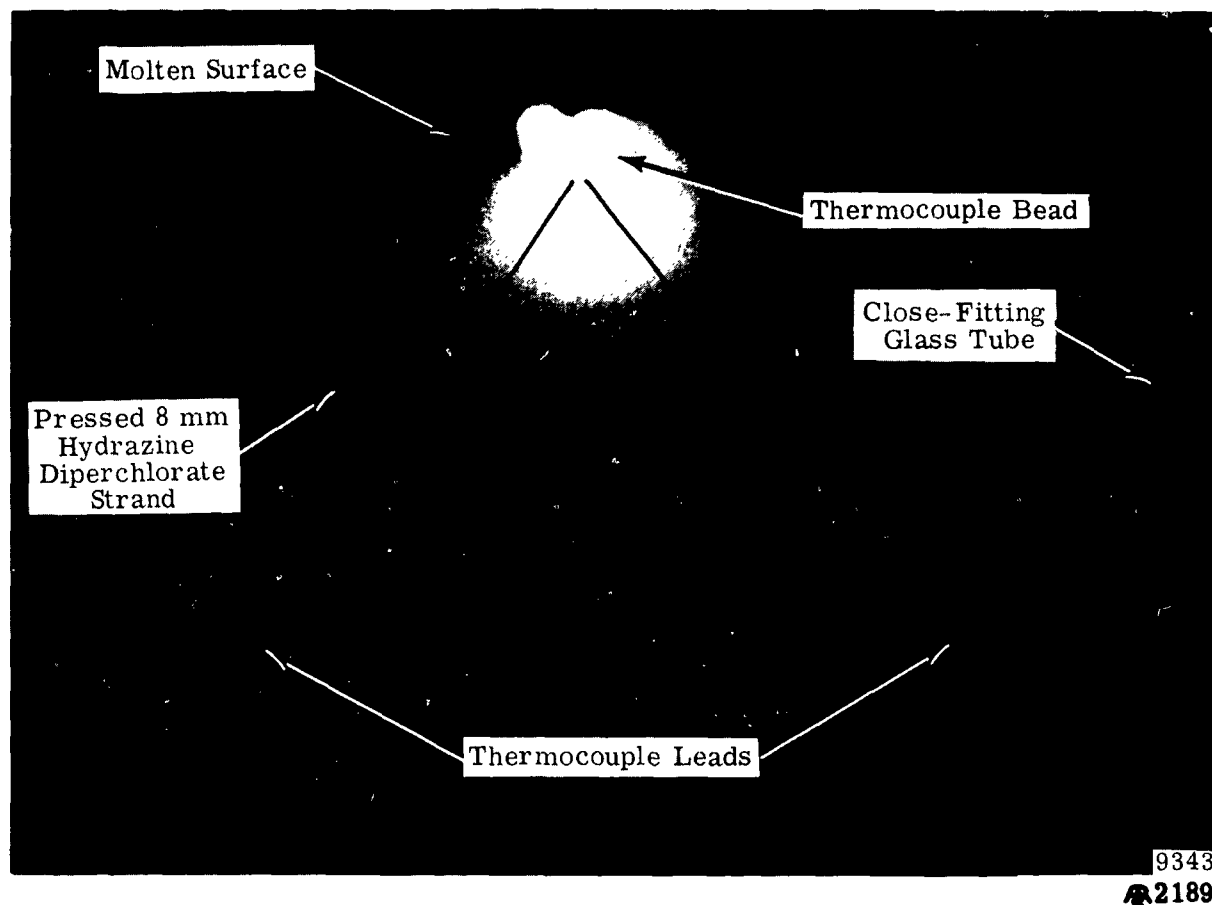


Figure 2. Deflagration of Pressed Hydrazine Diperchlorate With Pt-Pt, 10 Per Cent Rh Thermocouple Embedded, P = 400 psig.

which ammonium perchlorate spheres (of 6-7 mm diameter) at atmospheric pressure were supported on a wire grid located at the center of a rectangular duct, a fuel gas caused to flow through the duct, and the spheres ignited by a hot wire (5). The fuel gases used were propane, ammonia, hydrogen, and hydrogen diluted with nitrogen. It was found that ammonium perchlorate would undergo steady deflagration under these conditions while, if an inert gas replaced the fuel gas, deflagration did not occur. The data obtained obeyed various rate laws depending on the fuel.

In our studies to date with hydrazine diperchlorate, we have found that steady self-deflagration of pressed strands occurs at pressures above 300 psig, but that below this pressure deflagration does not occur. It seemed desirable to explore the application of the above technique to hydrazine diperchlorate. If meaningful results could be obtained it would be possible to extend the pressure range in which this material could be studied down to ambient pressure. In addition the form of the rate law so obtained should be informative as to the factors governing deflagration.

Accordingly we have carried out a number of experiments using this technique. The apparatus is shown in Figure 3. A Pyrex tube, 1.8 cm i.d. and 20 cm long, was connected to a fuel gas supply through copper tubing. The fuel flow rate was controlled by means of a critical flow orifice arrangement. A piece of stainless steel gauze was placed in the center of the tube and the pressed oxidizer placed on the gauze. Ignition was effected by an electrically-heated nichrome wire. The resultant events were recorded on motion picture film.

Experiments have been performed with ammonium perchlorate, hydrazine perchlorate and hydrazine diperchlorate.

1. Ammonium Perchlorate

Our interest in studying the deflagration of ammonium perchlorate by this technique was to allow a comparison of our results with those of Barrere and Nadaud (5) and to allow the comparison of ammonium perchlorate with hydrazine perchlorate and hydrazine diperchlorate. It was found that smooth deflagration could be attained in the flow rate range of 5.75-23.3 cm/sec. The limiting flow rates for deflagration were not investigated so that it may be that deflagration can occur outside the above limits.

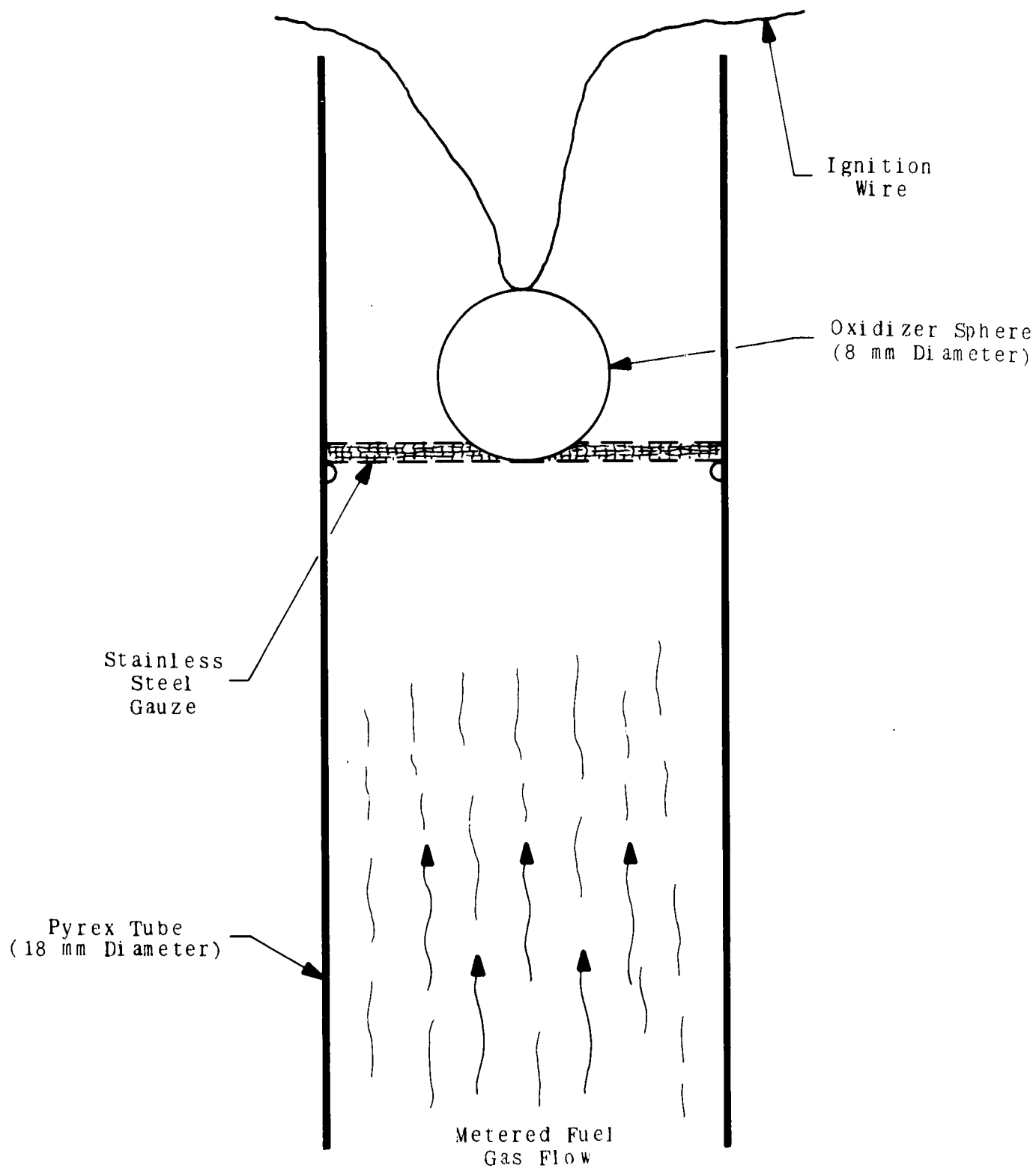


Figure 3. Apparatus for Deflagration in Fuel Gas Streams.

Subsequent experiments were performed on pressed 8 mm diameter spheres of ammonium perchlorate, prepared using a spherical mold. When the sphere was ignited it was possible to observe a first stage during which ignition spread over the sphere surface from the point at which the hot wire touched it. Thereafter the sphere deflagrated smoothly. However a major source of difficulty was encountered in the ammonium perchlorate experiments in that after about half of the sphere had been consumed, it would split into two or more irregularly-shaped fragments. The fragments assumed a spherical shape after sufficient further deflagration had occurred but were then quite small. This behavior occurred with all the fuel gases used, i.e., methane, propane and hydrogen.

The results for methane and propane have been plotted using the function $D^2 = D_0^2 - kt$ where D and D_0 are the horizontal diameters at time t and at zero time respectively, t is time in seconds, and k is a proportionality constant which is a measure of the deflagration rate. Barrere and Nadaud (5) found that their data obeyed this expression for propane and ammonia as fuels.

The type of data obtained in our experiments is illustrated by Figure 4 where our best results for ammonium perchlorate, hydrazine perchlorate, and hydrazine diperchlorate are shown. As Figure 4 shows, the data points are quite scattered but are roughly linear. The results obtained with ammonium perchlorate are tabulated in Table III below along with results for hydrazine perchlorate and hydrazine diperchlorate.

2. Hydrazine Perchlorate

Experiments were performed with hydrazine perchlorate spheres in streams of methane and propane. Here the spread of ignition over the sphere's surface was evidenced by the formation of a molten layer over the surface. The deflagration rate was many times faster than that for ammonium perchlorate. When the data were plotted in the same way as for ammonium perchlorate, slopes of the order of 5-10 times those obtained for ammonium perchlorate were found, see Figure 4. Deflagration of hydrazine perchlorate exhibited certain peculiarities. On the one hand it was observed that the hot ignition wire remained in contact with the hydrazine perchlorate sphere for periods as long as one minute with no perceptible effect other than melting of hydrazine perchlorate

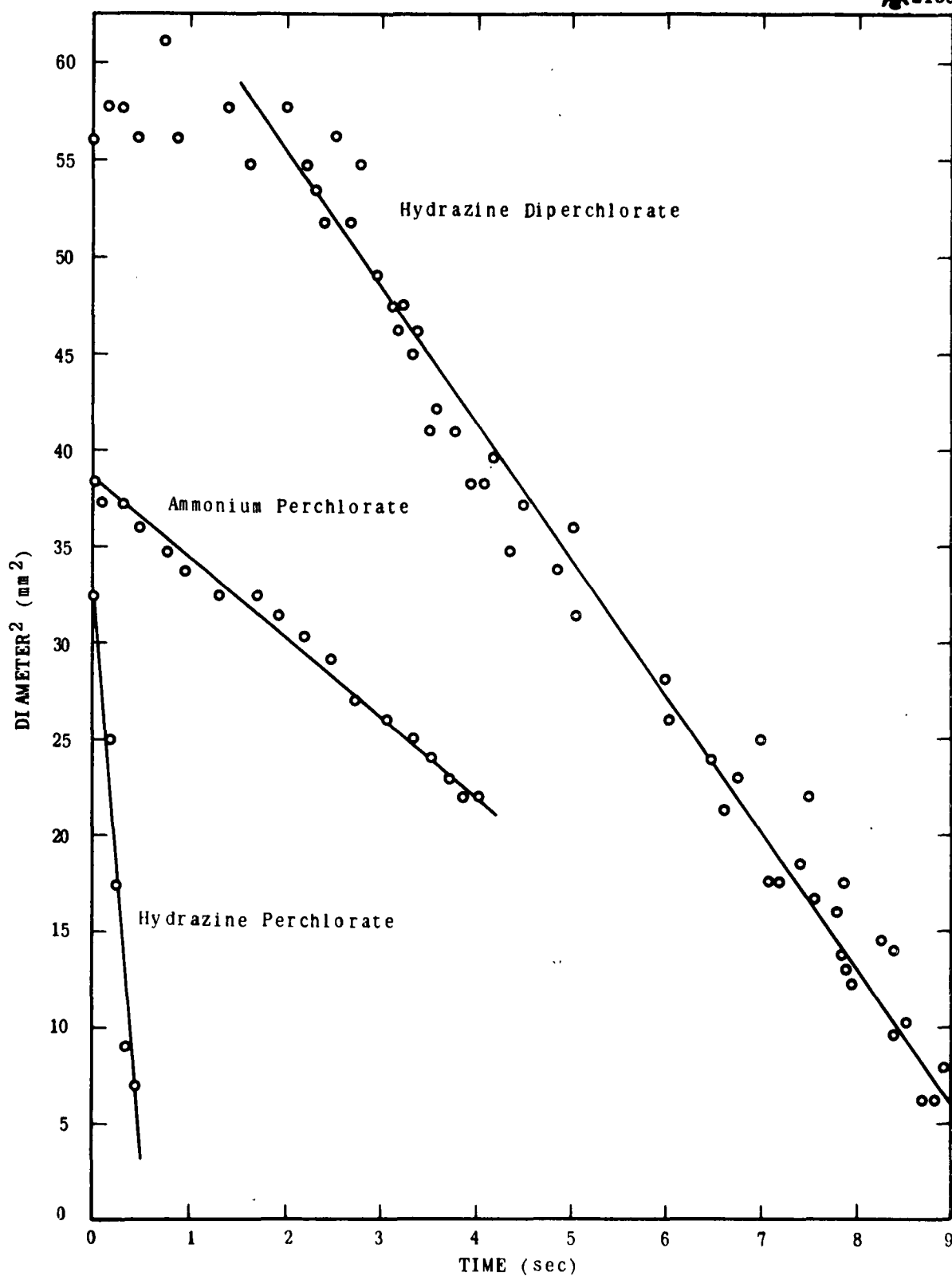


Figure 4. Deflagration Rates in a Methane Stream
(Flow Rate = 15.5 cm/sec).

at the point of contact. Ignition then occurred followed by very rapid deflagration. Another phenomenon that was noted with hydrazine perchlorate and was even more prominent with hydrazine diperchlorate was the occurrence of pulsing. The term pulsing is used to denote the following behavior: as deflagration proceeded the sphere appeared to expand slightly and then suddenly contract--as if gas were being released below the surface, acting to expand the surface layer and then escaping leaving a smaller sphere. The deflagration process was actually a series of such pulses. This resulted in very erratic plots of D^2 vs t .

3. Hydrazine Diperchlorate

Experiments with hydrazine diperchlorate have been performed with methane, propane and hydrogen fuel gases. Deflagration rates were of the same general magnitude as for ammonium perchlorate. Deflagration occurred with the presence of a molten surface layer and the pulsing phenomenon described above was very pronounced. The results are included in Table III.

TABLE III
Deflagration Rates in Fuel Gas Streams

Table No.	Entry	Oxidizer	Fuel Gas	Flow Rate (cm/sec)	Deflagration Rate k^a (cm ² sec ⁻¹)
1	209	AP ^b	CH ₄	15.5	0.040
2	227	AP	C ₃ H ₈	15.5	0.082
3	229	HP ^c	CH ₄	15.5	0.065
4	227	HP	C ₃ H ₈	15.5	0.040
5	222	HDP ^d	CH ₄	15.5	0.071
6	227	HDP	C ₃ H ₈	15.5	0.112
7	236	HDP	C ₃ H ₈	15.5	0.083
8	238	HDP	C ₃ H ₈	15.5	0.119

^a k is defined by $D^2 = D_0^2 - kt$

^b ammonium perchlorate

^c hydrazine perchlorate

^d hydrazine diperchlorate

4. Discussion of Deflagration Experiments in Fuel Streams

The experiments we have performed with this technique do not represent an intensive exploration of its possibilities. They do show the difficulties involved in obtaining data in this way.

With ammonium perchlorate our main difficulty has been that the spheres have invariably broken up in the course of an experiment. This has made it impossible to follow the deflagration for more than a fraction of its entire course. With hydrazine diperchlorate the principal difficulty is associated with the nature of the deflagration process. The pulsing phenomenon described above has resulted in rather erratic data. For hydrazine perchlorate too, the pulsing phenomenon is a complicating feature.

We have not observed a pulsing phenomenon for hydrazine diperchlorate in the strand deflagrations in inert gas at higher pressures. It may be that the pulsing phenomenon is associated with the deflagration process at low pressures but not at higher pressures. We feel the pulsing phenomenon is an indication of condensed phase gas release and we would like to know if such condensed phase behavior is less important at higher pressures. We therefore plan to extend this technique to see how the deflagration of hydrazine diperchlorate occurs at higher pressures, i.e., from ambient to 300 psia, where deflagration occurs in an inert atmosphere.

The results of Table III indicate that deflagration by this technique proceeds more rapidly in propane than in methane, but in view of the paucity of the data we do not feel this warrants discussion.

IV. PLANS FOR THE FUTURE

During the next period we will continue our investigations along the following lines:

1. Flame Temperature Measurements. We plan to measure flame temperatures using coated thermocouples at pressures (above 300 psig) where hydrazine diperchlorate undergoes reproducible self-deflagration.
2. We plan to make a series of deflagration rate measurements at elevated pressures making use of a single batch of hydrazine diperchlorate to see if consistent results can be found for a single batch of material.

3. We know of no reported study of the thermal decomposition of hydrazine diperchlorate. We plan to initiate experiments in this direction.
4. The fuel stream technique has not yielded very satisfactory data at ambient pressure but the pulsing phenomenon described earlier may be significant. We plan to investigate this technique at pressures above ambient to see how the deflagration proceeds under those conditions.

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