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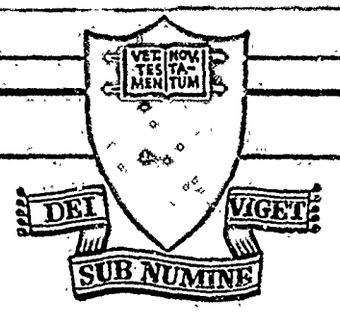

PRINCETON UNIVERSITY

DEPARTMENT OF AEROSPACE AND MECHANICAL SCIENCES

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Contract Nonr 1858(32)
Task No. (NR-092-516)
ARPA Order No. 23

SOLID PROPELLANT COMBUSTION

MECHANISM STUDIES (U)

Nineteenth Progress Report

For the Period 1 January 1965 to 31 March 1965

Aerospace and Mechanical Sciences Report No. 446-r

by

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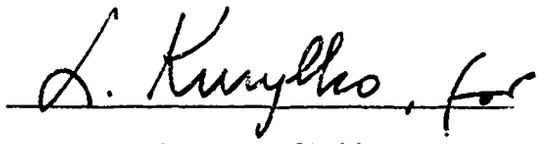

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June 1965

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ABSTRACT

In the combustion of composite solid rocket propellants based on ammonium perchlorate, the important rate determining processes have generally been presumed to occur within the gas phase flame. Recent results obtained in this laboratory suggest that condensed phase processes could also be important, particularly in low pressure combustion. The present research has been designed to study the nature of the processes involved in propellant gasification in the absence of the complicating effects of the gas phase flame.

The experimental method being employed in this study permits the measurement of propellant regression rates in the absence of the gas phase flame. An intense radiant flux from an arc-image furnace is substituted for the conductive heat flux generally associated with the flame. The flame itself is eliminated by working at a vacuum below the propellant combustion limit.

Two main questions as to the validity of this method are posed. Can variations in solid absorptivity and reflectivity with particle size cause specious effects of particle size on burning rate? Can the rapid variation of flux within the focal region of an arc-image furnace destroy the significance of the burning rates obtained with such a device? The conclusion is reached that both of these problems can be overcome and the method is valid. Preliminary tests on PBAA propellants subject to a radiant flux of 10 cal/cm^2sec have given burning rates of the order of 1/2 mm/sec with no evidence of a gas phase flame being present.

I. INTRODUCTION

At the present time, the combustion mechanism of composite propellants is not sufficiently well understood to allow one to predict the complete burning rate behavior of any particular propellant. However, in recent years, some very significant progress has been made toward this goal. Several theories have been proposed to explain the observed steady-state burning behavior of composite propellants (1). Some of these remain, at present, generally qualitative in nature; a few are sufficiently quantitative to allow critical comparisons with experimental data. Of these, however, only the Summerfield granular diffusion theory has met with significant success in predicting propellant burning rate behavior over an extensive range of some of the associated parameters (2). This report describes research currently in progress on some specific aspects of this theory.

Now, the variation of burning rate with pressure is one of the most important propellant combustion characteristics from the viewpoint of the rocket designer. It also constitutes one of the most readily tested predictions of any theory. The granular diffusion theory states that the variation of burning rate with pressure should have the form

$$\frac{1}{r} = \frac{a}{P} + \frac{b}{p^{1/3}}$$

This result is based on the assumption that the significant part of the energy release occurs in the gas phase. The rate of this energy release is, in turn, determined by two different mechanisms depending on the prevailing pressure. At high pressure, chemical reaction rates are very much faster than the inter-diffusion of the fuel and oxidizer species; therefore, diffusion is the controlling factor. At low pressure, the opposite situation is true,

therefore, chemical kinetics control the rate of energy release. The burning rate for any intermediate case is taken as a linear combination of the results for the two extreme cases. Thus, the first term in the equation is related to the reaction kinetics and the second term is related to the diffusion conditions; the value of a varies exponentially with flame temperature and b varies linearly with the average oxidizer particle size.

Burning rate measurements over an extensive range of pressure performed by Sutherland, Taback and Webb at Princeton showed good agreement with the theoretical equation for polystyrene propellants. The mixture ratio and oxidizer particle size were also varied and the resulting magnitude and variation of a and b were both found to be generally consistent with the theory. More recent measurements by Barrere, Boulard and Nadaud in France on various propellants over a narrower pressure range were in agreement with the granular diffusion theory. Some recent measurements by Yamazaki in Japan on polysulfide propellants also showed good agreement with the Summerfield equation.

However, some work done at Princeton by Bastress (3), Cole (4), and Most (5) at low pressures indicate that for polysulfide propellants at least, some anomalous behavior exists. Again, according to the granular diffusion theory, chemical kinetics is controlling at low pressures since diffusion is a very rapid process under such conditions. So, one would expect that at low pressure the effect of oxidizer particle size on burning rate would tend to vanish. This is due to the fact that within the framework of the theory, particle size only affects the diffusion conditions. Bastress made a fairly extensive study of particle size

effects on burning rate; and, in order to make these effects more concise, he used propellants containing narrow unimodal particle size distributions. His results indicate that the effect of oxidizer particle size on burning rate persists practically undiminished down to atmospheric pressure. Cole and Most have extended these results to subatmospheric pressures and have found that the particle size effects appear to persist all the way to the low pressure extinction limit of the propellant.

While Most was making these measurements he discovered a very unexpected phenomenon. For a particular polysulfide composition near its low pressure extinction limit, its burning mode suddenly changes to what appears to be a flameless combustion. A combustion wave continues to propagate into the propellant strand leaving a porous ash behind it, but there isn't any evidence of a visible flame during this propagation. Some preliminary tests indicate that the burning rate varies with particle size for this type of combustion.

So there are these two anomalies for polysulfide propellants: the persistence of oxidizer particle size effects at low pressures and the existence of what appears to be a flameless combustion. These facts are not necessarily in disagreement with the granular diffusion model. The particle size effects at low pressure could, for instance, still be due to a lack of complete diffusional mixing in the gas, but they also suggest the possibility that something more than just the separate pyrolysis of fuel and oxidizer may occur in the solid phase. For example, if the fuel and oxidizer were to interact to some degree in the solid, the rate of the resulting energy release could be proportional to the area of the particles or the perimeter of the particles on the surface. The

present research is designed to approach directly this whole question of what occurs in the solid phase during propellant combustion.

II. EXPERIMENTAL APPROACH

In any study of the solid phase combustion characteristics, the gas phase flame can only confuse the results. The first objective is, then, to eliminate the flame and replace it by a heat source that supplies the same amount of energy that the flame ordinarily would. This could be done, for instance, by pressing the propellant against a resistance-heated, porous plate or by sweeping the potentially reactive hot gases away with an inert gas jet. The method which is presently being used involves working at pressures below the normal extinction limit of the propellant. Under such conditions, the flame is not able to form because the rate of heat loss from the gases coming off the solid surface is greater than their rate of heat generation. Then the heat that would come from a flame is replaced by radiation from an arc image furnace. Now, the primary point of interest is resolving the question about particle size effects on burning rate at low pressure: do these effects originate in the solid phase or in the gas phase? According to the granular diffusion theory, the only reactions that occur in the solid phase are the separate pyrolyses of the fuel and the oxidizer. These reactions are assumed to proceed without mutual interaction and both can be described by an Arrenhius expression

$$r = Ae^{-E/RT_s}$$

but the net heat of reaction is positive because of the strong exothermicity of ammonium perchlorate decomposition. The oxidizer and binder vapors only interact in the flame above the propellant surface. If there is no

flame, the oxidizer particle size should not affect the burning rate. On the other hand, for a model in which solid phase reactions are significant, the particle size effects that Bastress and Most found should persist whether a flame exists or not.

Although it is relatively easy to prevent gas phase flame formation, the question of what heat input should replace it remains. At a given pressure, the burning rate could be forced to assume any value desired by adjusting the amount of radiant flux incident on the propellant surface. However, with such arbitrary burning rates there is no guarantee that the events which normally occur in the solid during ordinary combustion are not being drastically altered. To circumvent this problem, the tests are being conducted at pressures just below the flame extinction limit. The desired radiant flux is then that which gives the extrapolated burning rate for the fixed pressure and oxidizer particle size. Then with the pressure and radiant flux held constant, the particle size is varied to determine its effect, if any, on the burning rate.

III. VALIDITY OF EXPERIMENTAL APPROACH

Superficially these experiments appear to be fairly straightforward. However, the method of attack is quite unique and its validity needs further examination. Two questions are posed. First, is radiation an acceptable substitute for the usual conductive heat feedback from a flame? Second, is an arc image furnace a reliable source of this radiation? The remainder of this report will be devoted in large part to showing that except for one or two qualifications, both of these questions can be answered affirmatively.

The first question can be stated more specifically: it is

can variations in the reflectivity and absorptivity of the solid themselves cause the burning rate to vary with particle size? Figure 1 indicates the magnitude of the absorptivity for some ammonium perchlorate propellants. The variation of absorptivity with sample thickness is due to the fact that the arc light is essentially white rather than monochromatic. Figure 1 shows that the effective absorptivities are quite large and, in general, not strongly dependent in propellant composition. Figure 2 indicates the effect of oxidizer particle size on the absorptivity. While the data are somewhat scant, it appears that the change in absorptivity is less than 50% over the whole particle size range. It should be noted here that while these absorptivity values are large, the actual value that the radiation sees is probably even larger for two reasons. First, near the burning surface the fuel tends to char and blacken; second, at about 240°C the ammonium perchlorate goes through a crystal phase transition which renders the particles essentially opaque.

The question of how absorptivity affects burning rate can be approached analytically if the solid is taken to be homogeneous. The granular diffusion model is of interest in this respect since it does not allow particle size effects for any chemical reasons, but the question remains as to whether it allows particle size effects via changes in the gross absorptivity of the solid. The model is then a semi-infinite solid absorbing radiation in depth while it regresses at a constant rate; an exothermic reaction occurs on the surface only.

Then:
$$\frac{d^2T}{dx^2} + \dot{m}C_p \frac{dT}{dx} + \alpha I_o e^{-\alpha x} = 0$$

With boundary conditions:

$$x = 0 \qquad \dot{m}Q_s - \lambda \frac{dT}{dx} = \dot{m}C_g(T_s - T_o)$$

$$x = \qquad T = T_o$$

The solution is:

$$T = T_o + \frac{1}{\lambda\beta}\left\{\dot{m}[C_g(T_s-T_o)-Q_s] + \frac{\alpha I_o}{\alpha-\beta}\right\} e^{-\beta x} - \frac{I_o}{\lambda(\alpha-\beta)} e^{-\alpha x}$$

For the surface temperature

$$T_s = T_o + \frac{1}{\lambda\beta}\left\{\dot{m}[C_g(T_s-T_o)-Q_s] + \frac{\alpha I_o}{\alpha-\beta}\right\} - \frac{I_o}{\lambda(\alpha-\beta)}$$

Also

$$\dot{m} = r\rho_p = Ae^{-E/RT_s}$$

from the granular diffusion theory when no flame exists. If these last two equations are solved simultaneously using absorptivities of 100 cm^{-1} and 150 cm^{-1} along with typical values of the other parameters, it is found that this change in absorptivity causes only an approximately 3% change in the burning rate. On the basis of this analysis, then, it does not appear that changes in the gross absorptivity will have much effect on the burning rate. The probable qualitative reason for this fact is that the absorptivity is so large. As the limit of infinite absorptivity is approached, any finite variations in its value can have no effect since essentially all of the radiation is absorbed on the surface. This negative result then removes one area of uncertainty about the adverse effects of radiation.

It is much easier to deal with changes in the gross reflectivity of the solid due to the particle size variation. Once the reflectivity is known for each sample, all that needs to be done is to adjust the radiant flux so that the amount of radiation that is absorbed is the

same for all samples. A simple device for measuring propellant reflectivities is currently being constructed. Here again, one of the possible adverse effects of radiation should cause no problems.

Up to this point, the solid has been considered to be homogeneous. However, the solid is, in fact, heterogeneous and its degree of heterogeneity is affected by the oxidizer particle size. From a microscopic viewpoint, the radiation is incident on two different materials which have different values for both absorptivity and reflectivity. Therefore, the rate of energy input to the two materials will be different and their rate of thermal equilibration will be affected by particle size. Then, the question arises, can this cause any significant variation in the burning rate with particle size? This is an extremely difficult question to answer on a theoretical basis. It is nearly impossible to analyze the heat conduction in the solid under these conditions because the solid itself is so complex. At least one qualitative observation can be made here, though. That is, if these effects exist, they should only become significant when the oxidizer particle size is an appreciable fraction of the thickness of the thermal wave in the solid. One rather speculative point can also be raised. If the radiation imposed temperature difference between the fuel and oxidizer is stronger than a linear function of particle size, the effect on burning rate may only be significant for large average particle sizes. So there is some reason to believe that the absorptivity and reflectivity differences might only serve to eliminate average particle sizes greater than 100 to 150 microns from the experiment. But it is still possible that there may be some effect across the whole particle size

range. If this appears to be the case, the problem could be largely eliminated by modifying the propellants slightly to sharply reduce the differences in absorptivity and reflectivity between the fuel and oxidizer. Once more, the substitution of radiation for conduction has potentially adverse effects, but the problem appears to be surmountable.

The final point is related to the question of whether an arc image furnace is a suitable radiation source for the experiments. The problem is that in the focal region where the sample is placed, the radiation flux varies with distance. Figure 3 indicates the magnitude of this variation with the original optical system. On the basis of this data, the value initially anticipated was a 10 or 20% variation in 2 to 3 millimeters. Then the question arises as to whether such a variation in flux can cause wild changes in the burning rate during a given test. A rough idea of the effect of flux changes on burning rate can be obtained as follows. First, note that the characteristic time of the thermal processes in the solid is

$$\frac{1}{\alpha\beta K} = \frac{1}{\alpha r} \simeq \frac{1}{(100)(.05)} = 0.2 \text{ sec}$$

which is small compared to the rate of change of flux, the latter being of the order of 20% in 10 seconds, so the process is quasi-steady. Then all that needs to be calculated is $\frac{r}{I_o}$. For the granular diffusion model

$$r = Ae^{-E/RT_s} = Ae^{-E/R[T_o+Q_s/C_g+I_o/r\rho_p C_g]}$$

therefore

$$\frac{\partial r}{\partial I_o} = \frac{r\rho_p C_g}{\left\{\frac{R}{E}\frac{1}{r}\left[r\rho_p C_g(T_o+Q_s/C_g)+I_o\right]^2+\rho_p C_g\left[r\rho_p C_g(T_o+Q_s/C_g)+I_o\right]-r\rho_p^2 C_g^2(T_o+Q_s/C_g)\right\}}$$

If the following values are assumed

$$I_o = 20^{cal/cm^2 sec} \qquad r = 0.05^{cm/sec}$$

$$E = 20{,}000^{cal/mole}$$

as well as appropriate values for the other parameters, it is found that a 10% increase in flux gives a 9% increase in burning rate.

For a solid phase model in which surface reactions dominate the burning rate, the same expression holds but the activation energy must be changed. It can be estimated from known values of the temperature sensitivity of the burning rate

$$\left(\frac{\partial \ln r}{\partial T_o}\right)_p = \text{known const.} = \frac{-E}{R(T_o + Q_s/C_g)^2} = \frac{-E}{RT_s^2}$$

This gives a value of E of $3400^{cal/mole}$. Then a 10% increase in flux corresponds to a 5% increase in burning rate. It appears then that changes in the incident flux do not cause disproportionately large changes in the burning rate. However, the numbers used in these calculations are approximate at best so the possibility remains that the burning rate variation could be more serious. From an experimental viewpoint, a 20% variation in the burning rate during a test is pushing the limit of acceptability. To circumvent this problem, the optical system has been redesigned so as to provide the minimum possible flux variation short of devising a servo system to feed the sample into the arc focus continuously.

To sum up all of these points, then, it appears first, that any problems associated with substituting radiation for conduction can be solved, and second, the arc image furnace is an acceptable radiation source.

IV. DESCRIPTION OF THE APPARATUS

Figure 4 shows a picture of the apparatus that has been constructed to determine burning rates under the conditions described. First, the significance of the experiment hinges on the absence of a flame. Obviously, then, for each new propellant it is necessary to establish the fact that no flame is present. Two methods of doing this are being evaluated. The first is monitoring the space immediately above the propellant surface with a photocell to detect any flame luminosity. Particular care has to be taken here to prevent stray arc-light from giving false results. The second method involves scanning the space above the surface with a micro-thermocouple. The radiation also interferes in this case, so the point of interest is the slope of the thermocouple output as a function of distance rather than the absolute value of the output. These two methods should be sufficient to establish the non-existence of a flame.

The carbon arc used as the radiation source in these experiments is the same type normally used in motion picture theaters. With a 1500-1600 watt input it is capable of producing fluxes of the order of $120^{cal/cm^2 sec}$. The radiation is predominantly in the visible part of the spectrum; that which is not is largely absorbed by the various glass optical components. The sample exposure time is controlled by a pneumatically operated, double-leaf shutter; the opening and closing times are both of the order of 30 milli sec so the radiation pulse that the sample receives is essentially a square wave.

Figure (5) shows a diagram of the optical system. It comprises three mirrors; the two on the ends are 14" diameter elliptical mirrors,

the third mirror is 9" in diameter and spherical in configuration. This third mirror was introduced so as to greatly decrease the variation of flux with distance in the focal region where the sample is situated. This is accomplished by decreasing the angle of the converging radiation thereby increasing the depth of focus. There is an optimum diameter for such a mirror corresponding to a maximum heat flux. By means of the geometry of the system, the flux can be related to the convergence half-angle of the radiation

$$I_o = 27.0\ [-2\pi(\cos\theta - 1)] \left\{ 1 - \frac{\left(\frac{5\tan\alpha}{\tan\alpha+\tan\theta}\right)^2 (\tan\theta)^2}{\left[\left(35-\frac{5\tan\alpha}{\tan\alpha+\tan\theta}\right)\tan\phi\right]} \right\}$$

where α and ϕ are fixed angles and θ is the half-angle of convergence of the radiation; the number 27.0 is the flux per unit solid angle for the system. When the derivative of the flux with respect to θ is set equal to zero, one finds that the maximum flux is 25 $^{cal}/_{cm^2}$ sec and the corresponding diameter of the spherical mirror is that given above.

The combustion vessel which contains the propellant is provided with a nitrogen purge system to keep the window through which the radiation enters clean. Both the combustion vessel and the spherical mirror are mounted on compound heads which allow reproducible positioning within a few thousanths of an inch. This is well within the accuracy required for reproducible incident fluxes.

The problem of calibrating the flux level in the focal region of the arc is itself fairly complicated. Fortunately there are several designs available for radiation calorimeters. The device which has been constructed is modelled after the design of Beyer, et al. (6). It is a

transient type device consisting principally of a blackened disk on which the radiation is incident and a thermocouple to sense the temperature of the disk. It can be shown that when the disk dimensions are within certain limits the incident flux is given quite simply by an overall heat balance

$$I_o = \frac{mC}{\alpha A}\left(\frac{\Delta T}{\Delta t}\right)$$

where m is the mass of the disk, C its heat capacity, α its absorptivity, and A is the area of the aperture in front of the disk. The change in disk temperature with time, $\left(\frac{\Delta T}{\Delta t}\right)$, is obtained from the thermocouple output. The data in Figure 3 were obtained with such a device in the focal region of the second elliptical mirror.

The apparatus as a whole has just been completed so no systematic studies have been made as yet. However, preliminary tests have given what appears to be the type of combustion desired; with fluxes of the order of 10 $^{cal}/_{cm^2}$ sec, PBAA propellants exhibit burning rates of the order of 1/2 mm/sec with no evidence of a gas phase flame during the regression.

In summary, a new method and the associated apparatus for studying the phenomena which occur in the solid phase during composite propellant combustion have been described. It has been shown that the various problems which this method poses can be solved so that the points of real interest can be closely studied.

NOMENCLATURE

r - propellant burning rate, cm/sec

P - pressure, psi

E - activation energy, cal/mole

R - universal gas constant = 2.0 $\frac{\text{cal}}{\text{g-mole}^{\circ}\text{K}}$

T - temperature, $^{\circ}$K, $^{\circ}$C

$\dot{m}$ - mass burning rate, g/cm^{2}sec

C_p - propellant heat capacity, cal/g$^{\circ}$C

α - absorptivity, cm^{-1}

I_o - incident radiant flux, cal/cm^{2}sec

C_g - heat capacity of combustion gases, cal/g$^{\circ}$C

λ - thermal conductivity of propellant, cal/cm sec$^{\circ}$C

β - $\dot{m}C_p/\lambda$, cm^{-1}

ρ_p - propellant density, g/cm^3

K - thermal diffusivity of propellant, cm^2/sec

Q_s - heat release at solid surface, cal/g

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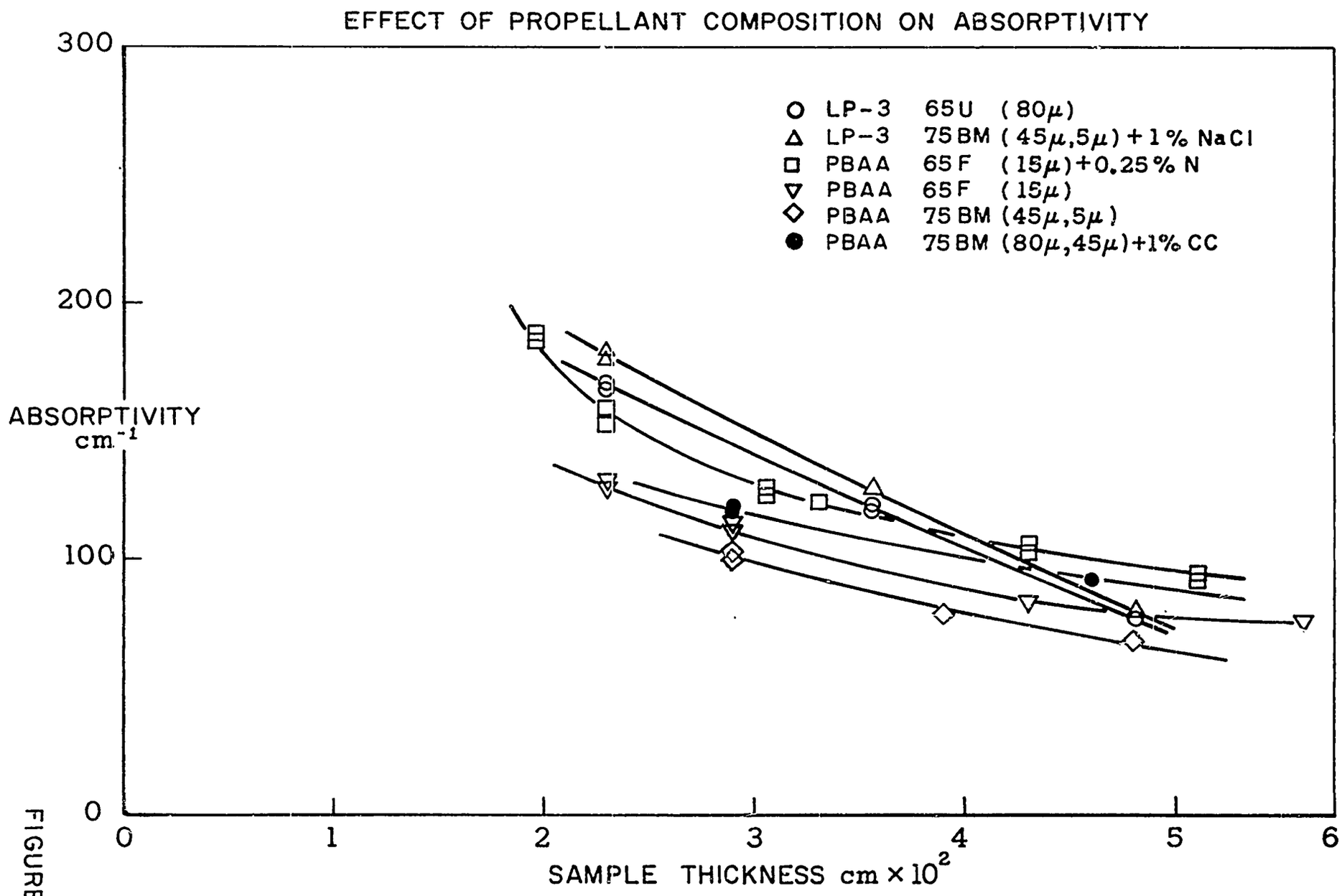

EFFECT OF PROPELLANT COMPOSITION ON ABSORPTIVITY
LP-3 65U (80μ)
LP-3 75BM (45μ,5μ) + 1% NaCl
PBAA 65F (15μ) + 0.25% N
PBAA 65F (15μ)
PBAA 75BM (45μ,5μ)
PBAA 75BM (80μ,45μ) + 1% CC
ABSORPTIVITY cm⁻¹
300
200
100
0
0
1
2
3
4
5
6
SAMPLE THICKNESS cm × 10²

FIGURE 1

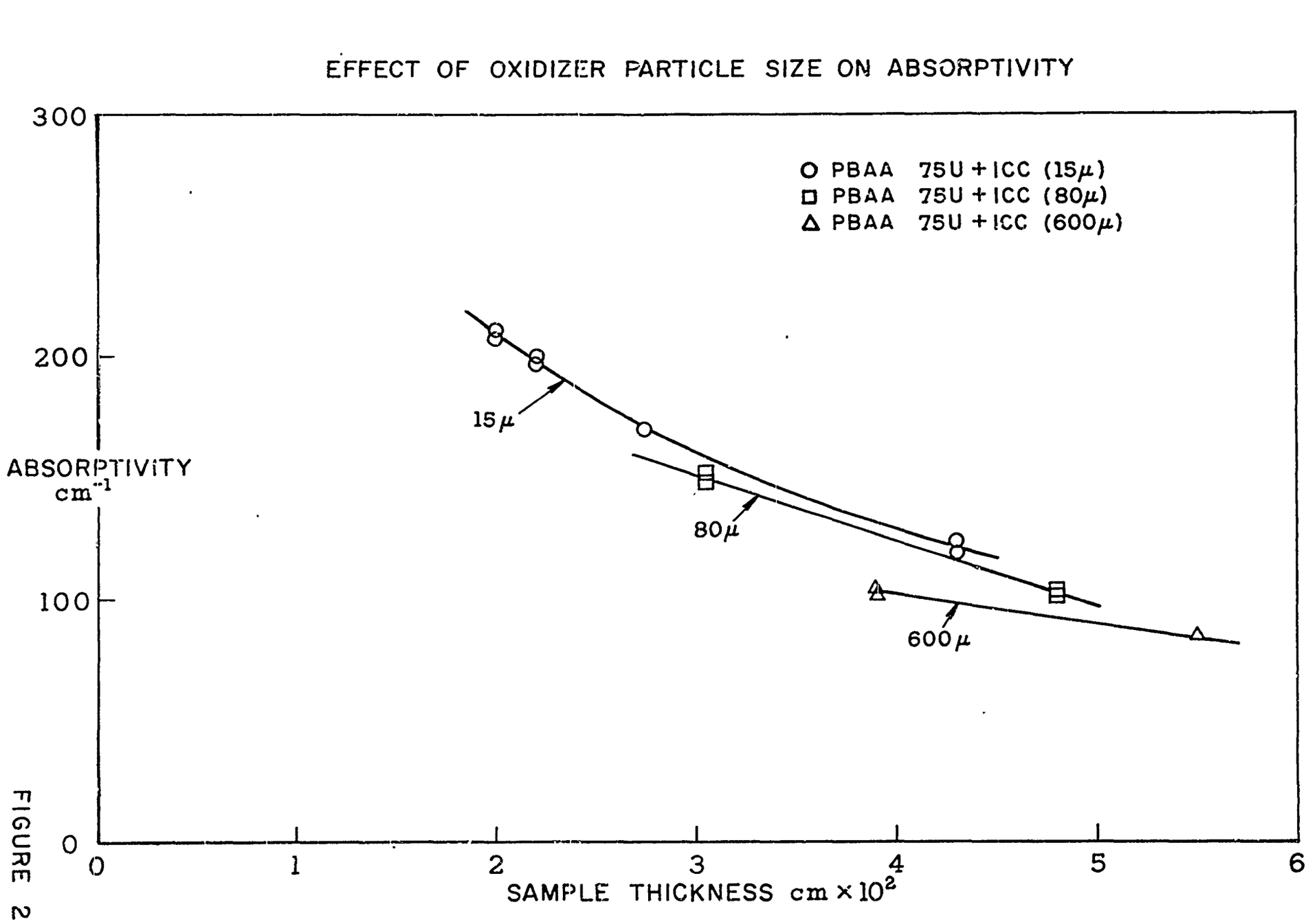

EFFECT OF OXIDIZER PARTICLE SIZE ON ABSORPTIVITY
O PBAA 75U + ICC (15μ)
□ PBAA 75U + ICC (80μ)
△ PBAA 75U + ICC (600μ)
15μ
80μ
600μ
ABSORPTIVITY cm⁻¹
300
200
100
0
0
1
2
3
4
5
6
SAMPLE THICKNESS cm × 10²

FIGURE 2

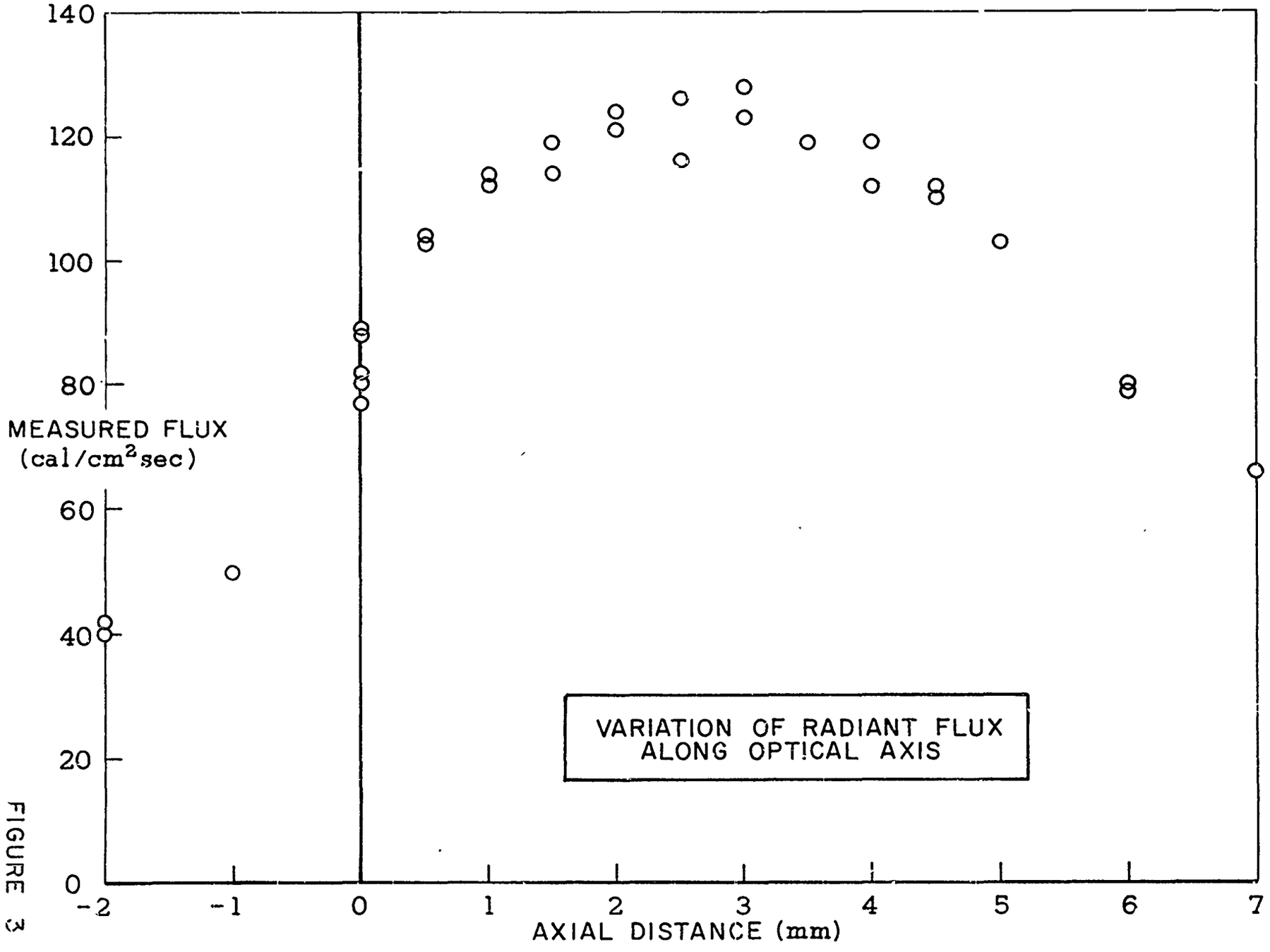
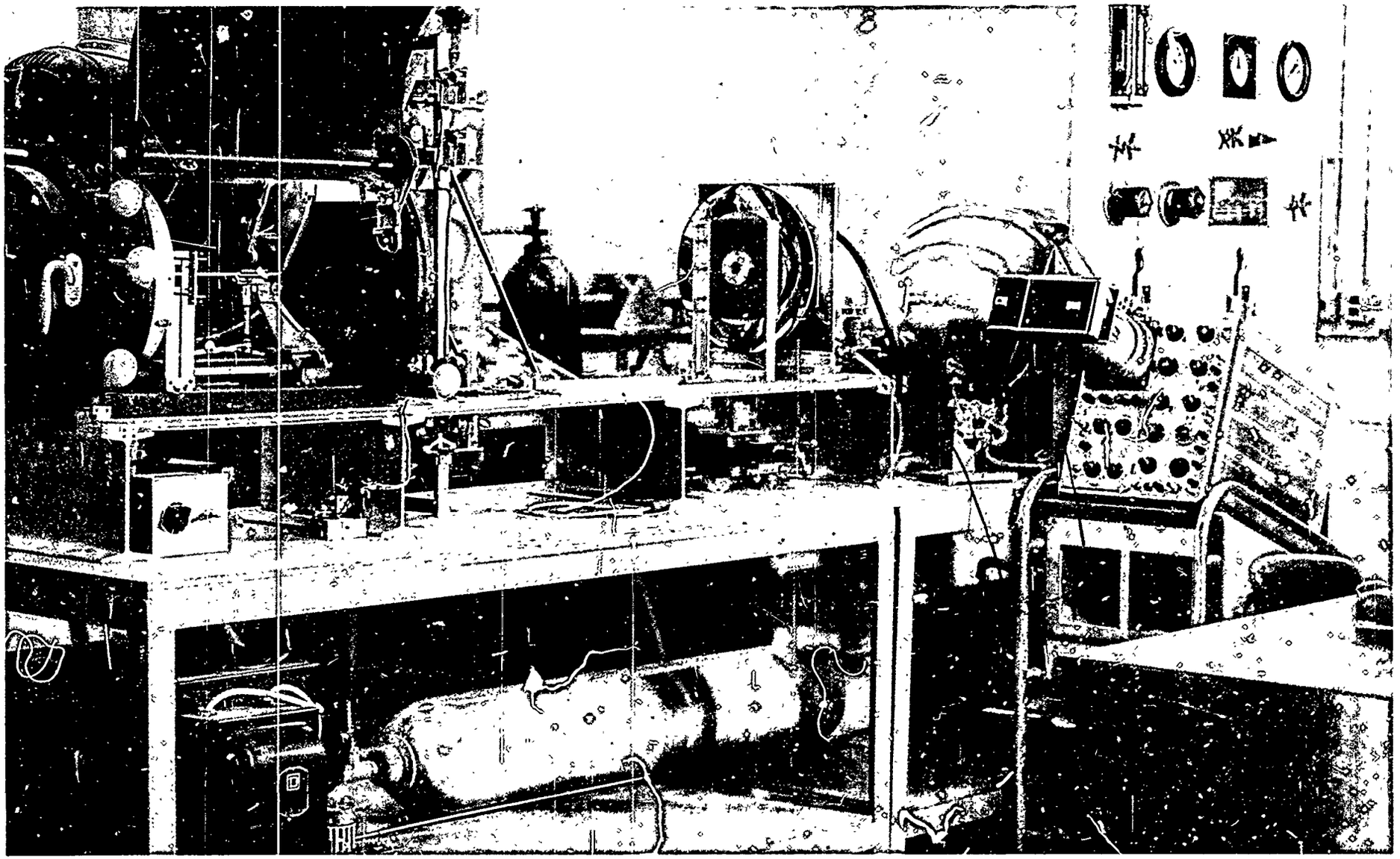

140
120
100
80
60
40
20
0
MEASURED FLUX
(cal/cm²sec)
-2
-1
0
1
2
3
4
5
6
7
AXIAL DISTANCE (mm)
VARIATION OF RADIANT FLUX
ALONG OPTICAL AXIS

FIGURE 3



APPARATUS FOR DETERMINING PROPELLANT BURNING RATES
UNDER FLAMELESS CONDITIONS

FIGURE 4

AP26-4003-65

ARC IMAGE FURNACE OPTICAL SYSTEM

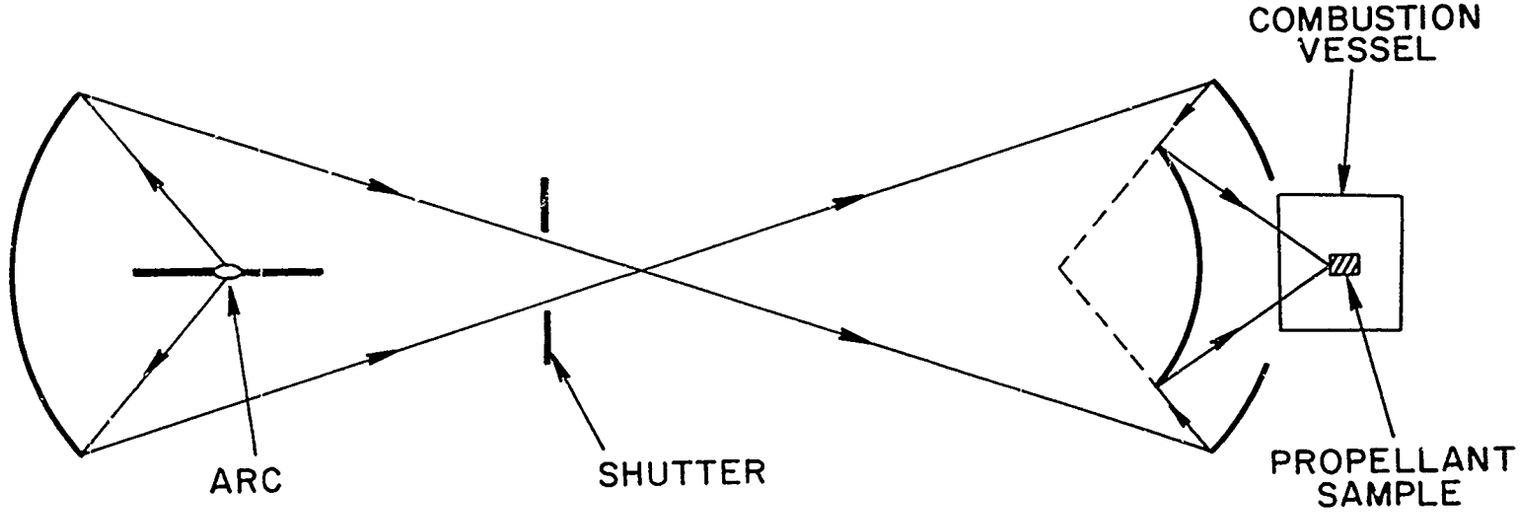


FIGURE 5

Unclassified
Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified.)

1. ORIGINATING ACTIVITY (Corporate author)	2a. REPORT SECURITY CLASSIFICATION
Princeton University Department of Aerospace and Mechanical Sciences	Unclassified
	2b. GROUP ----

3. REPORT TITLE

SOLID PROPELLANT COMBUSTION MECHANISM STUDIES (U).

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Technical Progress report for the period 1 January, 1965 to 31 March 1965

5. AUTHOR(S) (Last name, first name, initial)

Ohlemiller, Thomas J.
Wenograd, Joseph
Summerfield, Martin

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
June 1965	22	6

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
Contract Nonr 1858(32) b. PROJECT NO. C-932 c. ARPA Order No. 23 d. Task No. (NR-092-516)	446r 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) ----

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
-	Office of Naval Research Power Branch

13. ABSTRACT

In the combustion of composite solid rocket propellants based on ammonium perchlorate, the important rate determining processes have generally been presumed to occur within the gas phase flame. Recent results obtained in this laboratory suggest that condensed phase processes could also be important, particularly in low pressure combustion. The present research has been designed to study the nature of the processes involved in propellant gasification in the absence of the complicating effects of the gas phase flame.

The experimental method being employed in this study permits the measurement of propellant regression rates in the absence of the gas phase flame. An intense radiant flux from an arc-image furnace is substituted for the conductive heat flux generally associated with the flame. The flame itself is eliminated by working at a vacuum below the propellant combustion limit.

Two main questions as to the validity of this method are posed. Can variations in solid absorptivity and reflectivity with particle size cause specious effects of particle size on burning rate? Can the rapid variation of flux within the focal region of an arc-image furnace destroy the significance of the burning rates obtained with such a device? The conclusion is reached that both of these problems can be overcome and the method is valid. Preliminary tests on PBAA propellants subject to a radiant flux of 10 cal/cm^2 have given burning rates of the order of 1/2 mm/sec with no evidence of a gas phase flame being present.

DD FORM 1 JAN 64 1473

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