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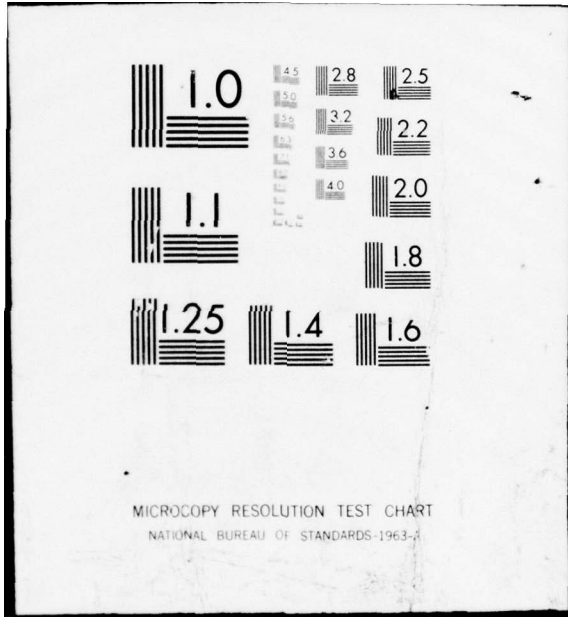
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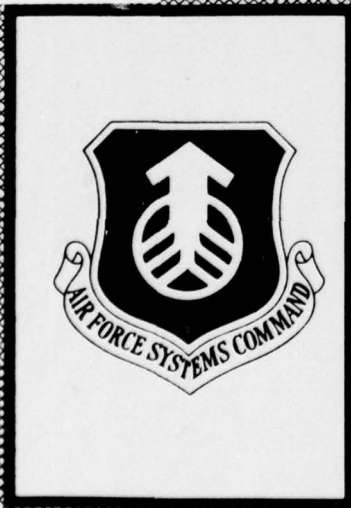


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FJSRL TECHNICAL REPORT - 77-0019  
AUGUST 1977

INVESTIGATION OF PLASTICIZER DIFFUSION  
WITHIN A CYLINDRICAL  
FUEL PELLET

ROBERT H. FOGLESONG  
J. PAUL JENDREK

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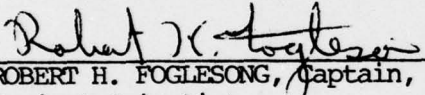
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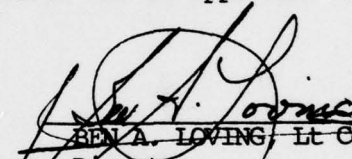
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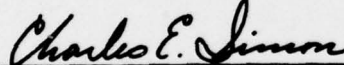
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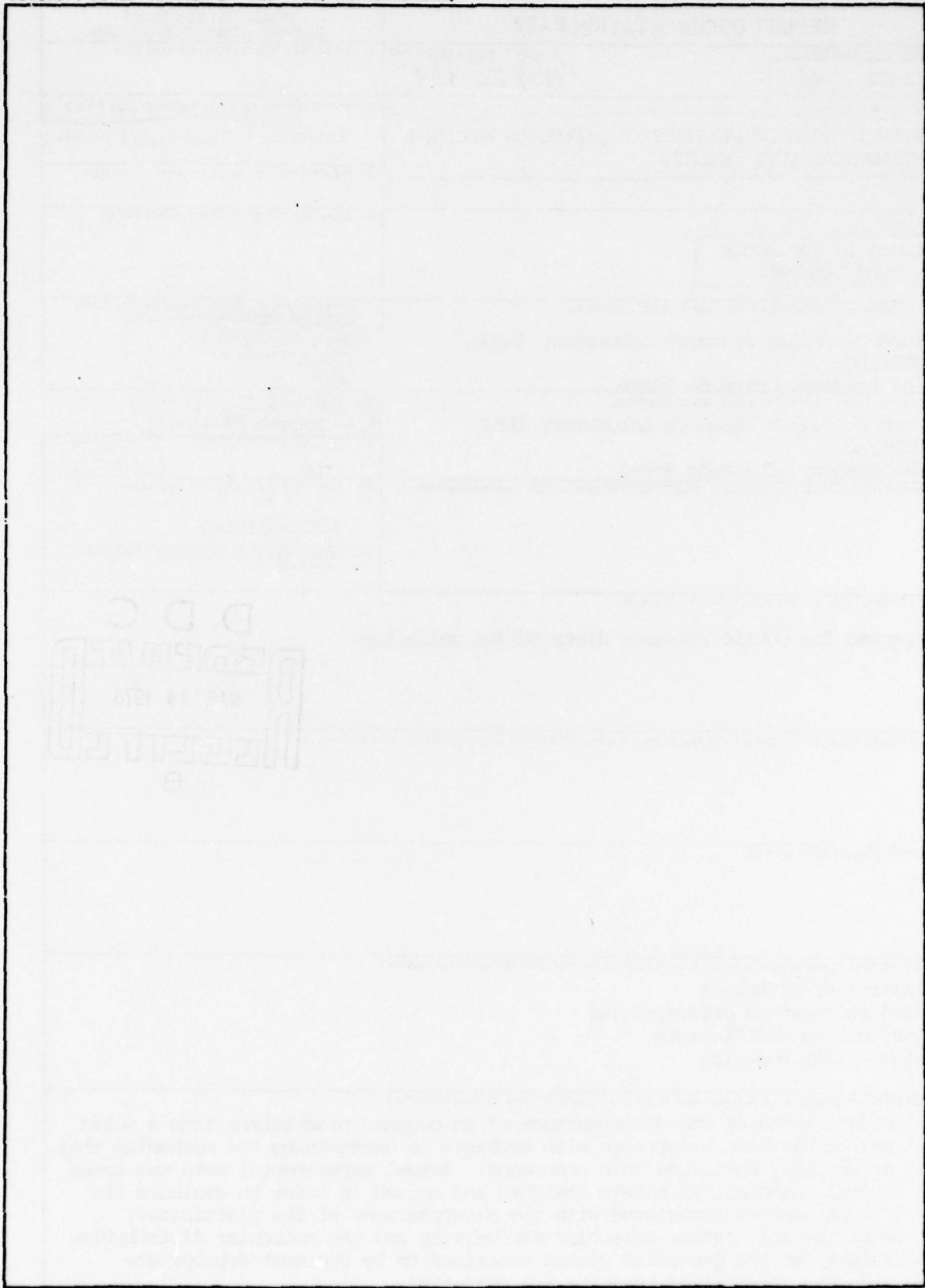
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WITHIN A CYLINDRICAL  
FUEL PELLET

Robert H. Foglesong  
J. Paul Jendrek

TECHNICAL REPORT SRL-TR-77-0019

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DIRECTOR OF CHEMICAL SCIENCES  
FRANK J. SEILER RESEARCH LABORATORY  
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## INTRODUCTION

The use of an organic plasticizer in solid rocket propellant binders, although necessary, has occasionally resulted in poor rocket performance. The primary reason for this fault was the loss of plasticizer from within the fuel pellet. The actual mechanism for this loss has been unknown. Thus, the problem of plasticizer disappearance within a fuel pellet was the topic of this research.

Initially, four desired results were envisioned that would essentially complete this project. These objectives were as follows:

1. Determination of factor(s) resulting in plasticizer disappearance (diffusion or kinetic controlled phenomena?)
2. Determination of physical and/or chemical parameters descriptive of the process involved in the migration/reaction of said plasticizer.
3. A mathematical model and solution for the concentration of plasticizer in a fuel pellet as a function of time and whatever special dependence was appropriate.
4. Use of the above results to retard or prevent the physical/chemical process from occurring.

While some of these results would undoubtedly require subjective evidence to support various conclusions and assumptions, it was highly desirable to obtain as many specific formulations and results as possible.

## DISCUSSION

### Fuel Pellet Construction/Characteristics

As it would be necessary to eventually have experimental data from an actual fuel pellet system it was decided to fabricate a pellet of materials similar to those utilized in the construction of the fuel pellet employed in the Minuteman Missile. The following considerations were appropriate when fabricating the experimental pellets:

1. The use of a solid cylindrical fuel pellet was appropriate for determination of data necessary to achieve the first two objectives of this project. The use of the actual Minuteman fuel pellet geometry would be necessary to verify the final mathematical formulation.

2. Special precautions were necessary to insure a proper medium was available to collect the appropriate data. The actual plasticizer that was employed in the Minuteman fuel pellet was selected for inclusion in the experimental pellet while the other ingredients were carefully selected to be as close a facsimile to the real fuel pellet as possible(1).

The experimental samples were constructed with the following composition:

Prepolymer (R45M)	15%
Curing Agent (DDI)	3%
Plasticizer (DOA)	6%
Filler (Ammonium Sulfate)	76%

These constituents were stirred until a homogeneous mixture was attained. Since the material was extremely viscous, it was necessary to use a vacuum apparatus to remove excess air bubbles in the mixture. The mixture was then cured at 60° C for 24 hours.

#### Objective Number One Consideration

Experimental data collected by Jendrek (1) using a chemical mix similar to that used in an actual fuel pellet were available. Initially, weight loss measurements were performed to determine which mechanism for plasticizer disappearance seemed appropriate for further study. At this point, three principal mechanisms had been theorized. These were as follows:

1. A diffusion controlled mechanism wherein only the physical process of plasticizer diffusion from within the fuel pellet to the surface of the fuel pellet occurs.
2. A reaction controlled mechanism wherein the plasticizer chemically degraded resulting in further degradation of the fuel pellet.
3. A combination of both diffusion and chemical degradation resulting in a net loss of plasticizer from both processes.

Thus, to proceed with the other objectives outlined earlier, it became necessary to determine which mechanism was appropriate for further study.

Mechanism number two, a reaction controlled phenomena was first eliminated for several reasons. It was thought that the possibility of chemical degradation was remote since dioctyl adipate is a relatively stable high molecular weight organic material. The low temperature range and relatively inactive ingredients used in the fabrication of the fuel pellets were not conducive to producing any type of chemical degradation. In addition, weight loss measurements indicated that material was disappearing from the experimental fuel pellets. Since a reaction controlled mechanism would not yield a gross weight loss unless the reaction products themselves diffused through the material, it was again implied that the reaction controlled mechanism was an unlikely candidate for further study.

Mechanism number three, a combination of diffusion and chemical degradation, was also eliminated because of weight loss considerations. This was due to the conclusion that a relatively large percentage of dioctyl adipate was disappearing. This could be explained only as a diffusion controlled phenomena and not a reaction controlled phenomena wherein no gross weight loss would be encountered.

Consequently, it was determined in a subjective fashion to pursue the diffusion controlled mechanism as a means to describe the phenomena occurring within the fuel pellet.

#### Objective Number Two Considerations

In order to eventually produce a useful correlation of the following form,

$$C_s = f(t, r, z) \quad (1)$$

three physical parameters describing the diffusional process appear in any attempt to mathematically model this phenomena. These parameters are  $D_I$  (the molecular diffusivity of the plasticizer in the solid fuel pellet),  $D_{II}$  (the molecular diffusivity of the plasticizer in the gaseous medium surrounding the open surfaces of the fuel pellet), and  $M$  (a Henry's Law type distribution coefficient correlating the concentration of plasticizer across the surface of the fuel pellet). As very little information was available on the three parameters noted, the use of mathematical modeling techniques to design experimental methods for determination of typical values for each parameter was accomplished.

The physical structure dictated by the experimental design and used to derive the solid state molecular diffusivities was a cylindrical pellet surrounded by a plastic sheath with one end coated to insure diffusion in only one axial direction. The pellet was kept in an inert nitrogen atmosphere and periodically weighed for gross weight

loss (1). These circumstances were forced by various boundary condition constraints imposed during the mathematical modeling of the diffusional phenomena.

The mathematical model and the techniques used to develop the desired correlation for the determination of  $D_I$  are contained in Appendix A (2). The correlation itself is as follows:

$$Y = \sum_{i=1}^{\infty} \frac{-2(-1)^i}{[(2i-1)/2]\pi} e^{-[\pi(2i-1)/2]^2 \tau} \cos\left(\frac{2i-1}{2}\pi \nu\right) \quad (2)$$

where

$$\tau = \frac{D_I t}{L^2}, \quad Y = \frac{C_s}{C_{s0}}, \quad \nu = \frac{z}{L} \quad (3)$$

(Consult the Nomenclature Table for definitions of various symbols)

Mathematical manipulation of Equation (2) yielded a similar expression with the average concentration of the plasticizer replacing the point concentration ( $C_s$ ).

The experimental data taken from the fuel pellets thus allowed determination of all physical parameters in Equations (2) and (3) except  $D_I$ .

While the previous design of experiment techniques centered on developing data and correlations that permitted the calculation of typical values of  $D_I$ , another set of experimental data and mathematical correlations were derived to permit the calculation of typical values of  $M$  and  $D_{II}$ .

The simplest experimental apparatus that permits fairly exact mathematical description and results in a correlation that permits calculation of the distribution coefficient ( $M$ ) was constructed by placing a fuel pellet of length  $L$  in a glass tube of the same diameter as the pellet. One end of the pellet was sealed to restrict diffusion. Again the other end was left open to permit plasticizer diffusion only in the axial direction. The glass tube was constructed to be long enough to assure no plasticizer would diffuse the entire length of the tube after leaving the open surface of the fuel pellet. As in the previous model, these physical circumstances were forced by various boundary condition constraints imposed during the mathematical modeling.

The actual mathematical model and techniques used to develop the desired correlation for determination of M are contained in Appendix B (3). The final correlation is:

$$\frac{C_{\text{avg}} - C_{\text{so}}}{-C_{\text{so}}M} = \frac{1}{M + \left(\frac{D_{\text{I}}}{D_{\text{II}}}\right)^{\frac{1}{2}}} \left(1 + \frac{1}{\sqrt{4D_{\text{I}}t}} + \frac{1}{L\sqrt{\pi}}\right) \quad (4)$$

By taking weight loss measurements and measuring various physical characteristics of the fuel pellets, it was possible to determine all terms in Equation (4) except M and  $D_{\text{II}}$ .  $D_{\text{II}}$  was calculated from an empirical equation developed by Hirschfelder, Bird and Spatz (4). Consequently, M was determined as the only unknown in Equation (4).

### RESULTS

To demonstrate the usefulness of Equation (2), values of  $D_{\text{I}}$  have been derived from the experimental data available. Table 1 shows a cross section of the values obtained. These values generally agree with published values of solid state molecular diffusivities (3). Values of an order of magnitude of  $10^{-7}$  m<sup>2</sup>/hr to  $10^{-9}$  m<sup>2</sup>/hr are commonly reported for solid systems similar to the system presently under study.

TABLE 1

Sample	Time (hr)	$D_{\text{I}}$ (m <sup>2</sup> /hr)
1	1660	$9.1 \times 10^{-8}$
2	1870	$9.7 \times 10^{-8}$
3	2037	$9.8 \times 10^{-8}$
4	2306	$9.6 \times 10^{-8}$

Values of the distribution coefficient (M) were calculated using Equation (4). These values and those of  $D_{\text{II}}$  calculated from the Hirschfelder, Bird and Spatz (4) correlation are shown in Table 2 (6).

TABLE 2

Sample	$D_{\text{II}}$ (m <sup>2</sup> /hr)	M x 10 <sup>9</sup>
1	.194	2.1
2	.194	3.4
3	.194	2.5
4	.194	3.0

As previously stated, the values presented in Tables 1 and 2 are presented only to demonstrate the usefulness of the correlations developed earlier.

In order to partially verify the conclusion deduced earlier wherein a diffusion controlled mathematical model would be the most appropriate as a description of the actual phenomena, Equation (2) and the average value of  $D_I$  were then used to predict gross weight loss from independent fuel pellet samples. Figures 1 and 2 indicated that a diffusion controlled model is adequate to approximate the phenomena that occurs within a fuel pellet (5).

#### FUTURE CONSIDERATIONS

While the work necessary to accomplish the first two objectives of this project are essentially complete, the work to complete the third objective is in the preliminary stages only. The most feasible model to describe an approximation of the actual fuel pellet geometry is briefly discussed in Appendix C. Further considerations of this model were postponed until completion of the first two objectives in order to determine whether the relative magnitudes of  $D_I$  and  $D_{II}$  would permit elimination of several terms as insignificant when these terms are compared to more significant terms. The following work is proposed to adequately verify the usefulness of any correlation derived from the model suggested in Appendix C.

1. Complete the mathematical solution of the proposed model in lieu of various simplifications that may be available from consideration of the relative sizes of  $D_I$  and  $D_{II}$ .
2. Construct several samples to approximate the geometry of an actual fuel pellet and take the appropriate weight loss data.
3. Construct several standard solid cylindrical fuel pellets of the exact same mix as was used to construct the pellet described in 2 above to be used with Equations (2) and (4) for the calculation of  $D_I$  and  $M$ .
4. Use the correlation developed in 1 above and the values of  $D_I$  and  $M$  calculated in 3 to compare to the actual weight loss curve found from 3 thus verifying the usefulness of said correlation to accurately predict plasticizer loss within an actual fuel pellet.

Objective number four can then be accomplished by subjectively evaluating the terms contained in the prediction correlation.

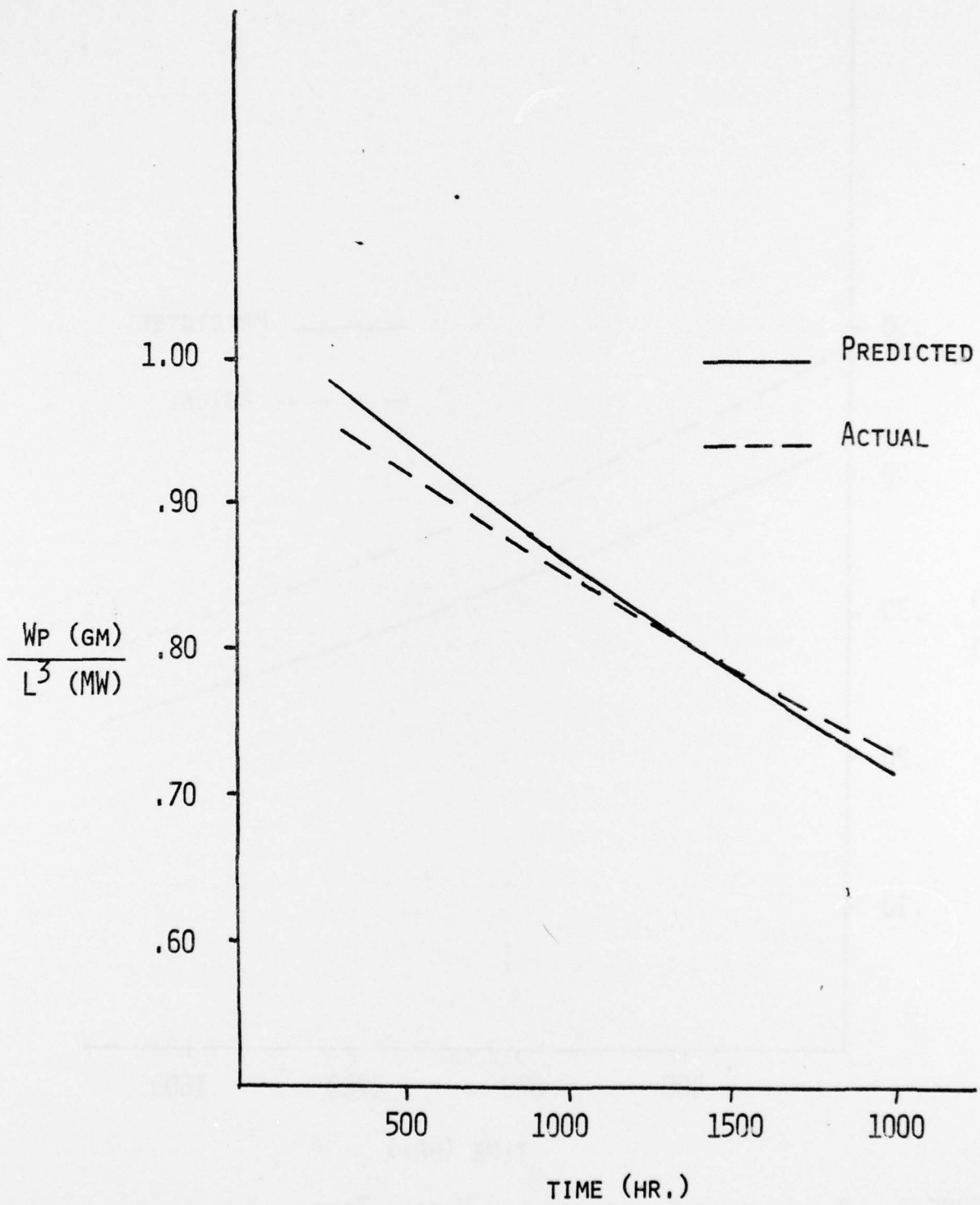


FIGURE 1. PLASTICIZER CONCENTRATION VERSUS TIME

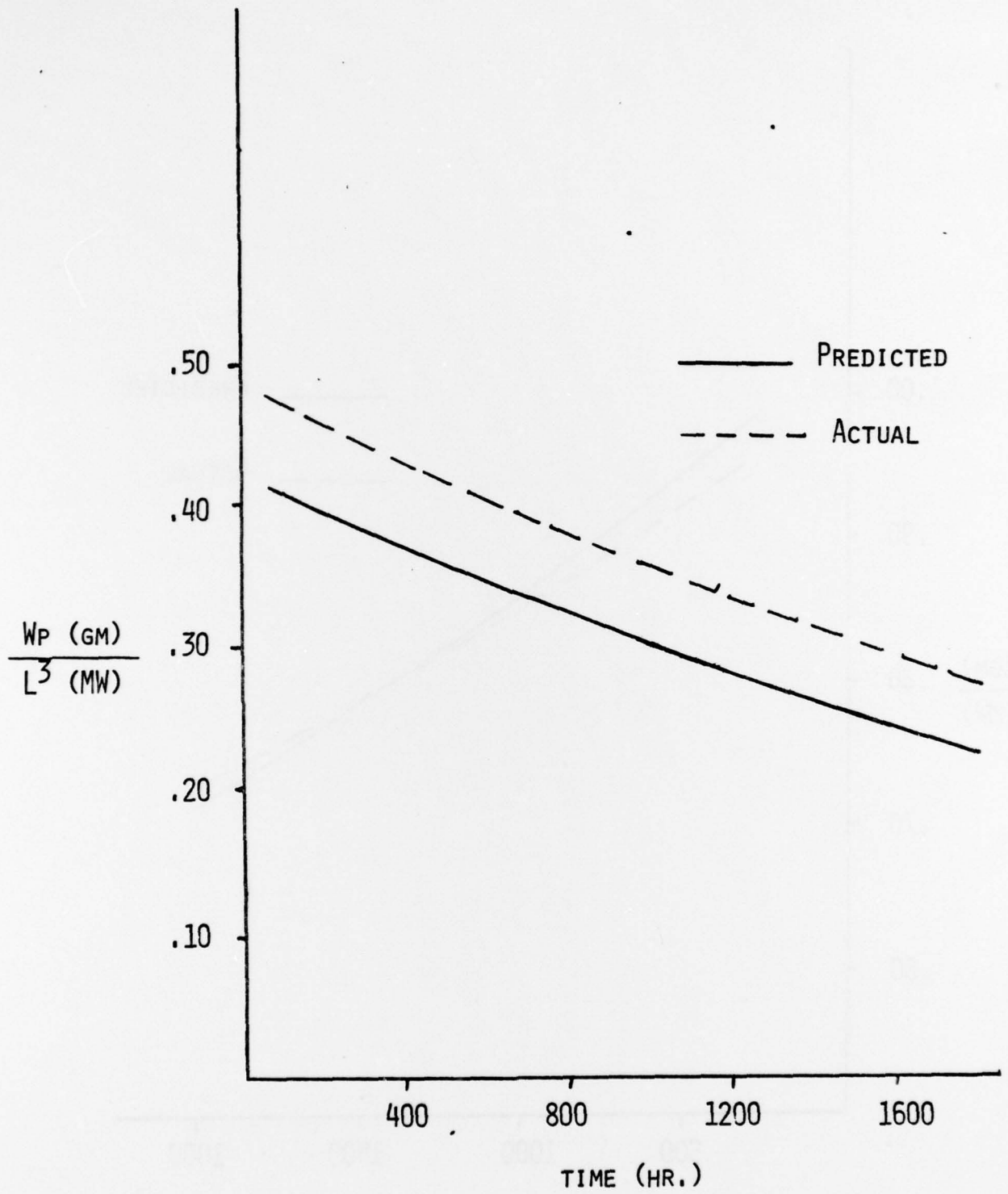


FIGURE 2. PLASTICIZER CONCENTRATION VERSUS TIME



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- (3) Bird, R. B., Stewart, W. E., and Lightfoot, E. N., Transport Phenomena, John Wiley and Sons, Inc., 1960, 624-625.
- (4) Hirschfelder, J. O., Bird, R. B., and Spotz, E. L., Revs., 44, 1949, 285-231.
- (5) Foglesong, Robert H., "Modeling and Prediction of Plasticizer Migration Within a Solid Cylindrical Fuel Pellet," Proceedings of the 6th Annual Pittsburgh Conference on Modeling and Simulation, 1975.
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## APPENDIX A

Derivation of a Correlation for Calculation of Solid State Molecular Diffusivities in a Fuel Pellet.

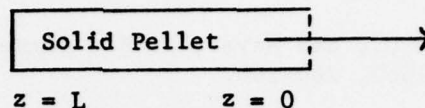


Figure 3

Assuming the concentration of the plasticizer in a solid cylindrical fuel pellet is not a function of radial direction, but will diffuse uniformly in the axial direction, Fick's 2nd Law of Diffusion yields:

$$\frac{\partial C_s}{\partial t} = D_I \frac{\partial^2 C_s}{\partial z^2} \quad (1A)$$

Rearranging Equation (1A) using the following dimensionless variables yields:

$$\frac{\partial Y}{\partial \tau} = \frac{\partial^2 Y}{\partial n^2} \quad (2A)$$

where

$$\tau = \frac{D_I t}{L^2}, \quad Y = \frac{C_s}{C_{s0}}, \quad n = \frac{z}{L} \quad (3A)$$

Applying separation of variables techniques to Equation (1A) results in the following expression:

$$Y = C_1 e^{-\alpha^2 \tau} (C_2 \sin \alpha n + C_3 \cos \alpha n) \quad (4A)$$

At  $z = 0$  ( $n = 0$ ), the flux of material is physically restricted to zero; therefore

$$\frac{\partial C_s}{\partial z} = 0 \quad \text{or} \quad \frac{\partial Y}{\partial n} = 0 \quad (5A)$$

Since the molecular diffusivity in the inert nitrogen atmosphere was several orders of magnitude greater than the molecular diffusivity in the solid phase, material can be carried away from the surface faster than it can be supplied from the solid core. Therefore, at  $z = L$ , the assumption was made that  $C_s$  equals zero. Consequently the following dimensionless boundary condition was applicable.

$$\text{at } n = 1, Y = 0 \quad (6A)$$

The initial condition for the time variable is as follows:

$$\tau = 0, Y = 1 \quad (7A)$$

Use of Equations (5A), (6A), and (7A) to evaluate the contents in Equation (4A) yielded the desired result.

$$Y = \sum_{i=1}^{\infty} \frac{-2(-1)^i}{[(2i-1)/2]\pi} e^{-[(2i-1)\pi/2]^2 \tau} \times \cos\left(\frac{2i-1}{2}\pi z\right) \quad (8A)$$

Solutions of this nature are generally stable after evaluating the first several terms of the expanded equation. Similar solutions appear in engineering literature for various physical geometries (2).

APPENDIX B

Derivation of a Correlation for Calculation of Molecular Distribution Coefficients

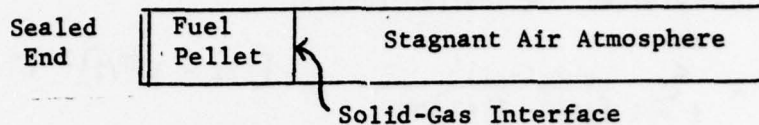


Figure 4

Since the fuel pellet was fabricated in a cylindrical glass shell, plasticizer diffusion was permitted only in the axial direction. Therefore, the concentration of plasticizer in the fuel pellet was not a function of radial distance. Applying Fick's 2nd Law of Diffusion to the physical system described above results in the following expression:

$$\frac{\partial C_s}{\partial t} = D_I \frac{\partial^2 C_s}{\partial z^2} \quad (1B)$$

The following initial boundary condition applies for the solid phase:

$$\text{at } t = 0, \quad C_s = C_{s0} \quad (2B)$$

The axial boundary conditions are:

$$\text{at } z = -\infty, \quad C_s = C_{s0} \quad (3B)$$

$$\text{at } z = 0, \quad D_I \frac{dC_s}{dz} = D_{II} \frac{dC_g}{dz} \quad (4B)$$

Similar equations can be written for the mass transfer phenomena on the gaseous side of the solid-gas interface. Again employing Fick's 2nd Law of Diffusion yields:

$$\frac{\partial C_g}{\partial t} = D_{II} \frac{\partial^2 C_g}{\partial z^2} \quad (5B)$$

The initial boundary condition for plasticizer diffusion in the stagnant gaseous region is as follows:

$$\text{at } t = 0, \quad C_g = 0 \quad (6B)$$

Axial boundary conditions are:

$$\text{at } z = +\infty, \quad C_g = 0 \quad (7B)$$

at  $z = 0$

$$D_I \frac{d C_s}{d z} = D_{II} \frac{d C_g}{d z} \quad (8B)$$

$$C_g = M C_s \quad (9B)$$

Applying the appropriate Laplace Transformation techniques to this series of equations results in the following expressions:

$$\frac{C_s - C_{s0}}{-C_{s0}M} = \frac{1 + \operatorname{erf} (z/\sqrt{4D_I t})}{M + (D_I/D_{II})^{\frac{1}{2}}} \quad (10B)$$

and

$$C_g = \frac{C_{s0} (1 - \operatorname{erf} (z/\sqrt{4D_I t}))}{1/M + (D_{II}/D_I)^{\frac{1}{2}}} \quad (11B)$$

These expressions are similar to those predicted in other studies (3) for a similar system.

Integration of Equation (11B) and application of the appropriate mathematical techniques results in the following correlation for the average concentration of plasticizer at any time:

$$\frac{C_{savg} - C_{s0}}{-C_{s0}M} = \frac{1}{M + (D_I/D_{II})^{\frac{1}{2}}} \left( 1 + 1/\sqrt{4D_I t} + 1/L\sqrt{\pi} \right) \quad (12B)$$

APPENDIX C

Proposed Multi-Region Model for Diffusion of a Plasticizer from a Fuel Pellet

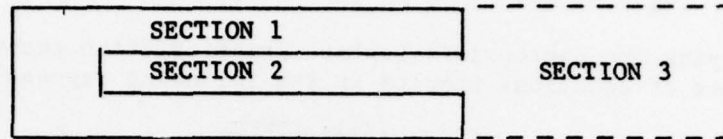


Figure 5

Equations of State for Each Section:

For Section 1

$$\frac{\partial^2 C_s}{\partial r^2} + \frac{1}{r} \frac{\partial C_s}{\partial r} + \frac{\partial^2 C_s}{\partial z^2} = D_I \frac{\partial C_s}{\partial t} \quad (1C)$$

For Section 2

$$\frac{\partial^2 C_g}{\partial r^2} + \frac{1}{r} \frac{\partial C_g}{\partial r} + \frac{\partial^2 C_g}{\partial z^2} = D_{II} \frac{\partial C_g}{\partial t} \quad (2C)$$

For Section 3

$$\frac{\partial^2 C_n}{\partial r^2} + \frac{1}{r} \frac{\partial C_n}{\partial r} + \frac{\partial^2 C_n}{\partial z^2} = D_{II} \frac{\partial C_n}{\partial t} \quad (3C)$$

Initial Boundary Conditions

at  $t = 0$

$$C_s = C_{s0} \quad (4C)$$

$$C_g = C_n = 0 \quad (5C)$$

At  $z = -L$

$$\frac{dC_g}{dz} = 0, \quad \frac{dC_s}{dz} = 0 \quad (6C)$$

At  $z = 0$

$$D_I \frac{dC_s}{dz} + D_{II} \frac{dC_g}{dz} = D_{II} \frac{dC_n}{dz} \quad (7C)$$

$$C_n = M C_s \quad (8C)$$

$$C_n = C_g \quad (9C)$$

Separation of Variables and Use of Various Simplification Techniques will yield:

$$\Theta_s = f(C_s) = K_s e^{-\beta^2 D_I t} \cos(Uz) \left( J_0(\lambda r) - \frac{J_1(\lambda R_2)}{Y_1(\lambda R_2)} Y_0(\lambda r) \right) \quad (10C)$$

$$\Theta_g = f(C_g) = K_g e^{-\beta^2 D_{II} t} \cos(U'z) J_0(\lambda' r) \quad (11C)$$

$$\Theta_n = f(C_n) = f(e^t, \cos z, J_0(r)) \quad (12C)$$

where

$$U, U', \beta, \lambda, \lambda' = f(M, D_I, D_{II}) \quad (13C)$$

## NOMENCLATURE

1.  $C_s$  - concentration of plasticizer in the fuel pellet
2.  $t$  - time
3.  $r$  - radial distance
4.  $z$  - axial distance
5.  $D_I$  - molecular diffusivity of the plasticizer material in the fuel pellet
6.  $D_{II}$  - molecular diffusivity of the plasticizer material in the stagnant gas atmosphere
7.  $M$  - distribution coefficient
8.  $i$  - dummy variable
9.  $Y$  -  $C_s/C_{s0}$
10.  $\tau$  -  $(D_I t)/L$
11.  $\eta$  -  $z/L$
12.  $L$  - length of the fuel pellet
13.  $C_{s0}$  - initial concentration of the plasticizer in the fuel pellet
14.  $C_{savg}$  - average concentration of the plasticizer in the fuel pellet
15.  $C_1, C_2, C_3, \alpha$  - separation constants
16.  $C_g$  - concentration of plasticizer in the stagnant gas atmosphere
17.  $C_n$  - concentration of plasticizer in Section 3
18.  $\theta_s$  -  $C_s/C_{s0}$
19.  $\theta_g$  -  $(C_g + C_{s0} M)/C_{s0} M$
20.  $\theta_n$  -  $(C_n + C_{s0} M)/C_{s0} M$
21.  $u, u', \beta, \lambda, \lambda', K_s, K_g$  - separation constants



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