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LATTICE DEFORMATION FEASIBILITY STUDY

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## FOREWORD

The work reported herein was sponsored by the Air Force Rocket Propulsion Laboratory, Research and Technology Division; Edwards Air Force Base, under Contract AF O4(611)-11548, Project No. 3148, Program Element No. 62405184. The program is monitored for the Air Force by Dr. Wm. J. Leahy, RPCS.

This report covers work performed during the first quarter of the program from 1 April 1966 through 30 June 1966. The investigations were performed by Aerojet-General Corporation personnel of the Propellant Research and Development Division, Sacramento, California, and the Engineering and Development Division, San Ramon, California. The principal investigators are Mr. J. P. Coughlin (of Sacramento), who is directly responsible for the Calorimetry and analysis portions of the program and overall program manager, and Mr. R. R. Tsukimura (of San Ramon), who is in charge of the sample irradiation phase of the program. Determination of optical-crystallographic properties will be performed by Mr. J. L. McGurk of Sacramento and Dr. D. W. Bainbridge (also of Sacramento) will investigate the changes in sample lattice parameters by specialized X-ray techniques.

This report contains no classified information.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

Dr. Wm. J. Leahy/RPCS

## ABSTRACT

During the first quarter of the program a majority of the preliminary steps required before the actual start of neutron irradiation were accomplished.

The Hazards Analysis Report was written and approved by the AGN Reactor Safeguards Committee. The pressure vessel design has been completed and hydrostatic testing of the main body showed a burst pressure higher than anticipated.

In preparation for energy content and analytical measurements, the heat of solution Calorimeter was re-assembled and the electrical measurements circuits checked out. Reagent grade ammonium perchlorate was purchased and screened to separate out crystals in the 500- to 1000-micron size range for the radiation and calorimetry work. A portion of this sample was recrystallized for X-ray and microscopic studies.

DTA and Bureau of Mines Impact tests have been run on the screened reagent sample-for comparison with similar measurements to be run with irradiated samples later in the program.

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## ABBREVIATIONS AND SYMBOLS

AGC	Aerojet-General Corporation
AGN	Aerojet-General Nucleonics, Engineering and Development Division of AGC, San Ramon, California
AGNIR	Aerojet-General Nucleonics Industrial Reactor
AP	Ammonium Perchlorate, $\text{NH}_4\text{ClO}_4$
"F" and "G" Rings	Positions in the AGNIR reactor pool in close proximity to the active core
HSMP	"High speed Mikropulverized", refers to a particular grind of AP of about 26 micron average particle diameter
MA	"Mikroatomized", refers to a grind of AP of about 12 micron average particle diameter
R	Roentgen Units of radiation
SSMP	"Slow speed Mikropulverized", refers to a grind of AP of about 120 micron average particle diameter
Type 6061-T6	An aluminum alloy composition particularly well suited for this work, containing minimum amounts of alloying agents or impurities which are capable of forming long life radioactive isotopes in a high neutron source.

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# I

## INTRODUCTION

The objective of this program is to determine the feasibility of increasing the energy content of solid materials by permanent non-equilibrium dislocations of lattice members.

The energy source chosen for this study is neutron irradiation and the material to be irradiated is ammonium perchlorate. Solution calorimetry will be utilized to determine the amount of energy storage.

The program is divided into two separate portions; with Phase I covering sample irradiation and Phase II covering calorimetry and analysis operations with the irradiated samples.

## II

### PHASE I - SAMPLE IRRADIATION

#### 1. INTRODUCTION

The Phase I operation is being conducted at the San Ramon Plant (Aerojet-General Nucleonics). The principal tasks involved are design and fabrication of pressure vessels, preparation and approval of the Hazards Analysis Report, and sample radiation and dosimetry.

#### 2. PRESSURE VESSEL DESIGN AND FABRICATION

During irradiation the individual  $\text{NH}_2\text{ClO}_4$  samples will be sealed in gas-tight aluminum pressure vessels which will permit passage of neutron radiation while retaining all gaseous decomposition products. The particular alloy to be used and the exact capsule design were chosen so as to minimize the hazard due to residual radiation of the container after exposure while at the same time imparting sufficient physical strength to retain all decomposition products in the event of complete sample decomposition.

The design utilizes 3/8" I.D. by 5/8" O.D. tubing of 6061-T6 aluminum alloy, as shown in Figure 1. Included in the design are a 1/4" O.D. filling tube and a combination end cap and puncture seal device. Additional details of the design may be found in the Hazards Analysis Report which is included as an appendix to this report.

An unexpected bonus was obtained during the hydrostatic testing of the pressure vessel components. These tests were performed to ensure the validity of calculations made for the working pressure of the vessel. The main body of the pressure vessel was expected to burst at 16,000 psi but did not actually burst until 25,000 psi. This increase in the burst pressure increases the design pressure of the vessel from 4040 to 6370 psia, permitting the use of a shorter primary vessel. This shorter primary vessel will now fit in the dummy-element, eliminating the necessity of modifying to accommodate the primary vessel.

The burst test for the primary pressure vessel model was not definitive, since the welds ruptured at ~12,000 psi. Examination of the welds showed the lack of full-penetration. Therefore, the weld-joints have been redesigned to obtain the necessary full-penetration.

Fabrication of the pressure vessel components, particularly the puncture seal, has been initiated. The puncture seal valve is also being fabricated.

# PRESSURE VESSEL FOR $\text{NH}_4\text{ClO}_4$ IRRADIATION

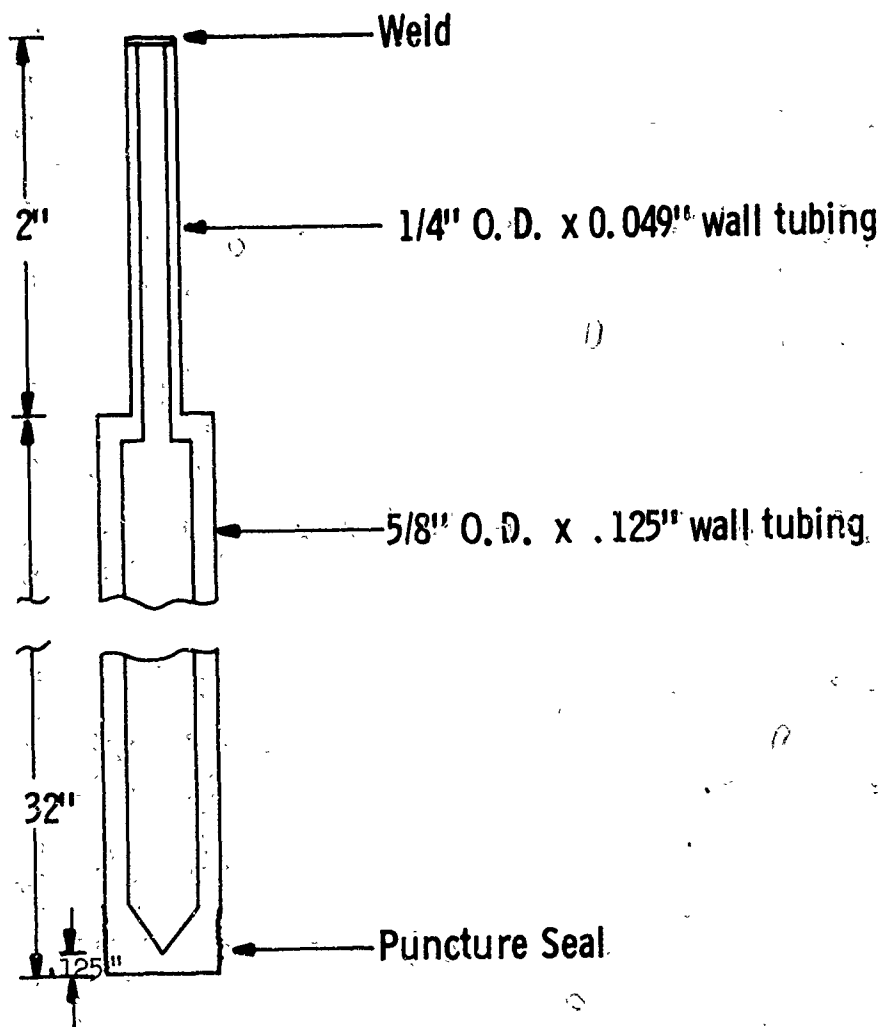


FIGURE 1

The secondary containment will be provided by the dummy-element. The working pressure of this capsule is only 620 psi, which is not sufficient to contain the radiolytic gases from the complete decomposition of 20g of  $\text{NH}_4\text{ClO}_4$ . Therefore, the secondary volume will be increased using tubing and an expansion volume. Plans for hydrostatic testing of the secondary container are complete.

### 3. HAZARDS ANALYSIS REPORT

The Hazards Analysis Report, which is a requirement of the Atomic Energy Commission, was completed and submitted to the AGR Reactor Safeguards Committee for review and subsequent approval. This document is reproduced for reference as the appendix to this report. Included in this safety analysis are provisions for hydrostatically testing the puncture seal, weldments and each pressure vessel to 6060 psia. Prior to pressure vessel fabrication, the 5/8" O.D. Tubing will be hydrostatically tested to its burst pressure to verify the use of 42,000 psi as the ultimate tensile strength for Type 6061-T6 aluminum tubing. One additional test, the thermal decomposition of  $\text{NH}_4\text{ClO}_4$  in a hydrostatically tested pressure vessel, will be performed to determine whether the vessel can contain the products (expected to be similar to those of radiolytic decomposition) and to evaluate the extent of corrosion caused by these same products.

The pressure vessel design is shown in Figure 1. A Whitey right-angle valve with an extended stem will be used to penetrate the pressure vessel at the puncture seal end for gas-sampling purposes. The irradiated  $\text{NH}_4\text{ClO}_4$  will be removed by cutting open the pressure vessel using a tube cutter. This operation will be performed in a glove-box containing an inert atmosphere to prevent any contamination of the irradiated  $\text{NH}_4\text{ClO}_4$ .

After reviewing the Hazards Analysis Report, the Reactor Safeguards Committee requested additional information and data regarding the explosive hazard of  $\text{NH}_4\text{ClO}_4$ .

In subsequent discussions regarding the explosive hazards of AP, the importance of crystal particle size and overall charge diameter was stressed. A review of available data showed that the critical charge diameter (charge diameter below which a detonation wave through the charge cannot be sustained) is a definite function of crystal particle size - increasing from 0.6 inch for a 12-micron particle size to 6 inches for a 200-micron particle size. Tables I and II, based on the work of Anderson and Pesante (1) and Cook (2,3), summarize the available data.

Differential Thermal Analysis tests were also cited in which it has been shown that the auto-ignition temperature of typical  $\text{NH}_4\text{ClO}_4$  lots used in solid propellant manufacture is about  $310^\circ\text{C}$  whereas the equivalent value for reagent grade material is  $380^\circ\text{C}$ .

Table I.

DONORS THAT WILL DETONATE AMMONIUM PERCHLORATE<sup>a</sup>

Acceptor-Ammonium Perchlorate						
Blend or Grind	Average Particle Diameter Microns	Charge Density gm/cc	Charge Diameter in.	Donor-Tetryl		
				Weight grams	Diameter in.	Length in.
MA	12	0.6	0.68	8.5	0.74	0.76
		0.8	0.68	8.5	0.74	0.76
		1.0	0.68	8.5	0.74	0.76
		1.25	2.50	46	1.40	1.25
		1.5	2.50	46	1.40	1.25
HSMP	26	0.6	1.25	46	1.40	1.25
		1.0	1.25	46	1.40	1.25
AF <sup>b</sup>	96	0.8-1.0	4.0	20	1.0	1.0
SSMP	120	0.8-1.0	4.0	20	1.0	1.0
HY <sup>c</sup>	140	0.9-1.1	2.0	80	2.0	1.0
Unground <sup>d</sup>		1.12-1.21	7.0	146 <sup>d</sup>	1-1/2	3

<sup>a</sup>From Reference (1,2), except for unground.

<sup>b</sup>70% SSMP (slow speed Mikropulverized) - 30% HSMP (high speed Mikropulverized).

<sup>c</sup>45% Unground - 35% SSMP - 20% MA (Mikroatomized).

<sup>d</sup>Reference (3). The donor, 146 gms of 50/50 pentolite, was the minimum booster required to detonate the AP. Sieve and chemical analyses are reported in the reference.

Table II

CRITICAL DIAMETERS<sup>a</sup>

Grind or Blend	Avg. Particle Diameter Microns	Approx. Pour Density, gm/cc	Estimated Critical Diameter, in.
MA	12	0.4-0.6	0.6
HSMP	26	0.5-0.6	0.8
AF	96	0.8-1.0	1.4
SSMP	120	0.8-1.0	1.4
HY	140	0.9-1.1	1.4
Unground	200	1.0-1.2	6 <sup>b</sup>

<sup>a</sup>From Reference (1,2), except for unground.

<sup>b</sup>Determined, Reference (3).

On the basis of these data, the  $\text{NH}_4\text{ClO}_4$  chosen for use in this study is reagent grade purity of a screen fraction size consisting of particles in the 500- to 1000-micron range (0.5 to 1 mm) with a charge diameter of 3/8 inch.

An informal discussion with personnel of the Atomic Energy Commission Reactor Safeguards Division confirmed that the judgement of the AGN Reactor Safeguards Committee as to the safety of irradiating ammonium perchlorate would be accepted without need of specific AEC approval.

Other points of discussion and changes and additions agreed to include:

- a. An automatic reactor shutdown if the primary pressure vessel wall temperature exceeds  $150^\circ\text{C}$  ( $250^\circ\text{F}$ ).
- b. A temperature profile of the total irradiation assembly from reactor pool to sample center.
- c. Corrosion calculations for HCl on aluminum.
- d. Outlining of experiment limitations.

Approval of the overall plan was granted after these additional data and information were summarized and presented to the committee as an "addendum" to the Hazards Analysis Report.

#### 4. SAMPLE RADIATION AND DOSIMETRY

The sample to be used has been purchased, (Matheson, Coleman and Bell, reagent crystals) and screened to separate out crystals in the 0.5- to 1-mm particle size range for study. In addition, a portion of the same sample was recrystallized for specialized microscopic and X-ray studies. Final plans for dosimetry, which will permit accurate recording of both neutron and gamma doses, have been completed.

Arrangements have been completed with the California State Bureau of Radiological Health to transfer the irradiated capsules (after suitable cooling-off period) to AGC-Sacramento, under the AGN License. The only stipulation under this arrangement is the requirement that AGN personnel deliver (to Sacramento), open and return the radioactive pressure vessel to San Ramon.

### III

#### PHASE II - CALORIMETRY AND ANALYSIS

##### 1. INTRODUCTION

Phase II of this program (being conducted at the Sacramento Plant) is devoted to determination of the changes in energy content, chemical analysis and physical properties brought about by irradiation of the sample. The principal tasks involved are: calorimetric determination of the energy content; chemical analysis of the irradiated sample, gaseous decomposition products and capsule corrosion products; X-ray studies and microscopic studies.

##### 2. CALORIMETRY

The calorimeter heater has been calibrated by passing a small constant current through the heater, in series with a 100-ohm standard resistor, and measuring the potential drop across each. The resultant calibration is 99.045 absolute ohms. The Eppley standard cell to be used has been calibrated, resulting in a standard EMF at 25°C of 1.01909 absolute volts.

The temperature sensitivity of the calorimeter resistance thermometer has been determined as approximately 1860 microvolts per degree centigrade for a thermometer current of 2 milliamps by comparison with a Parr calorimeter thermometer. This calibration is only approximate since in normal reduction of calorimetric data all temperature changes are expressed in microvolts and never converted to degrees.

The start of calorimetric measurements has been delayed because of the detection of a high resistance electrical short in the resistance thermometer measurement system. This defect caused erratic readings in the temperature measurement system and was probably caused by a slight leakage of moisture through a break in the tygon protective covering film.

This defect is currently being repaired and measurements are expected to begin during the coming month.

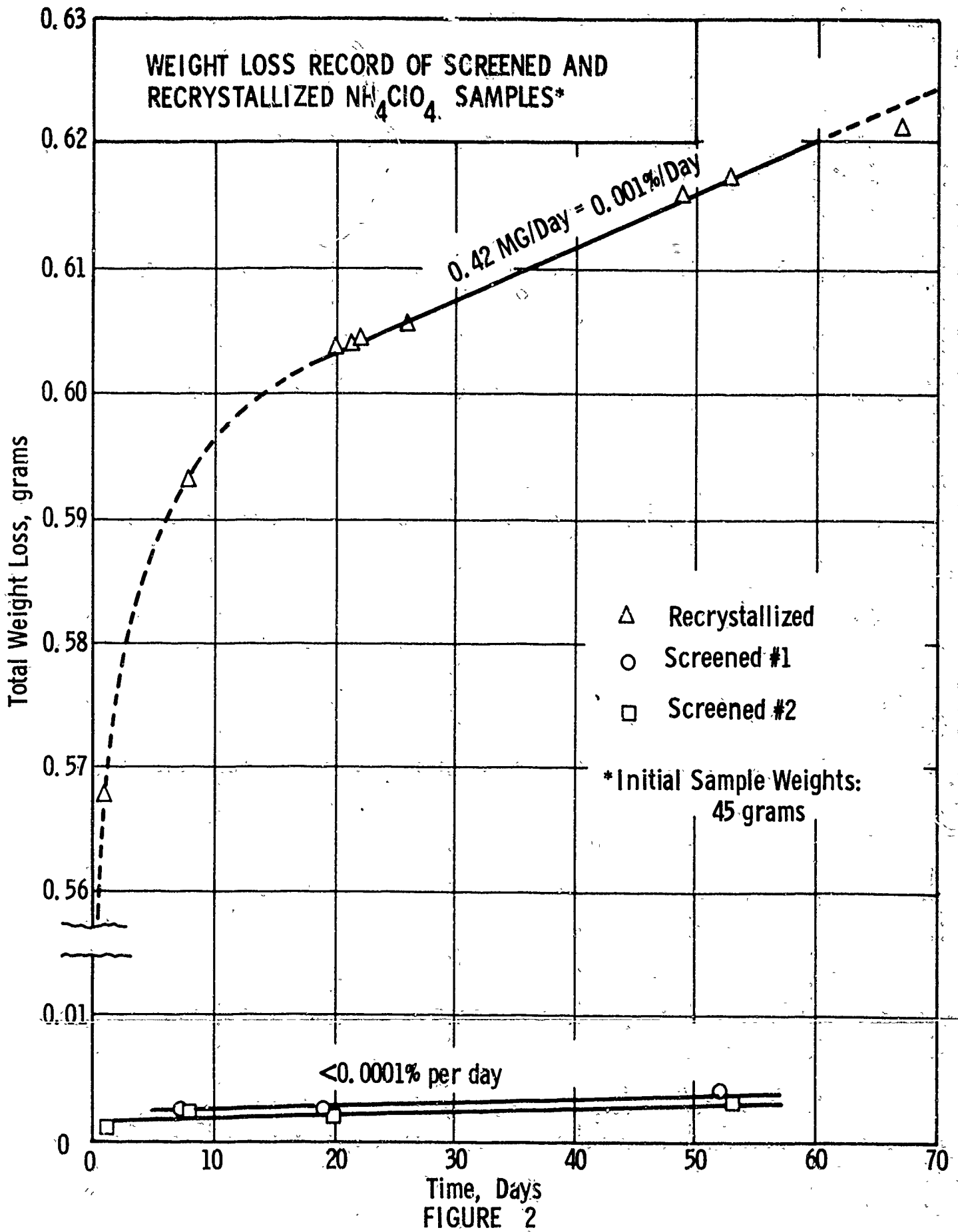
##### 3. ANALYSIS

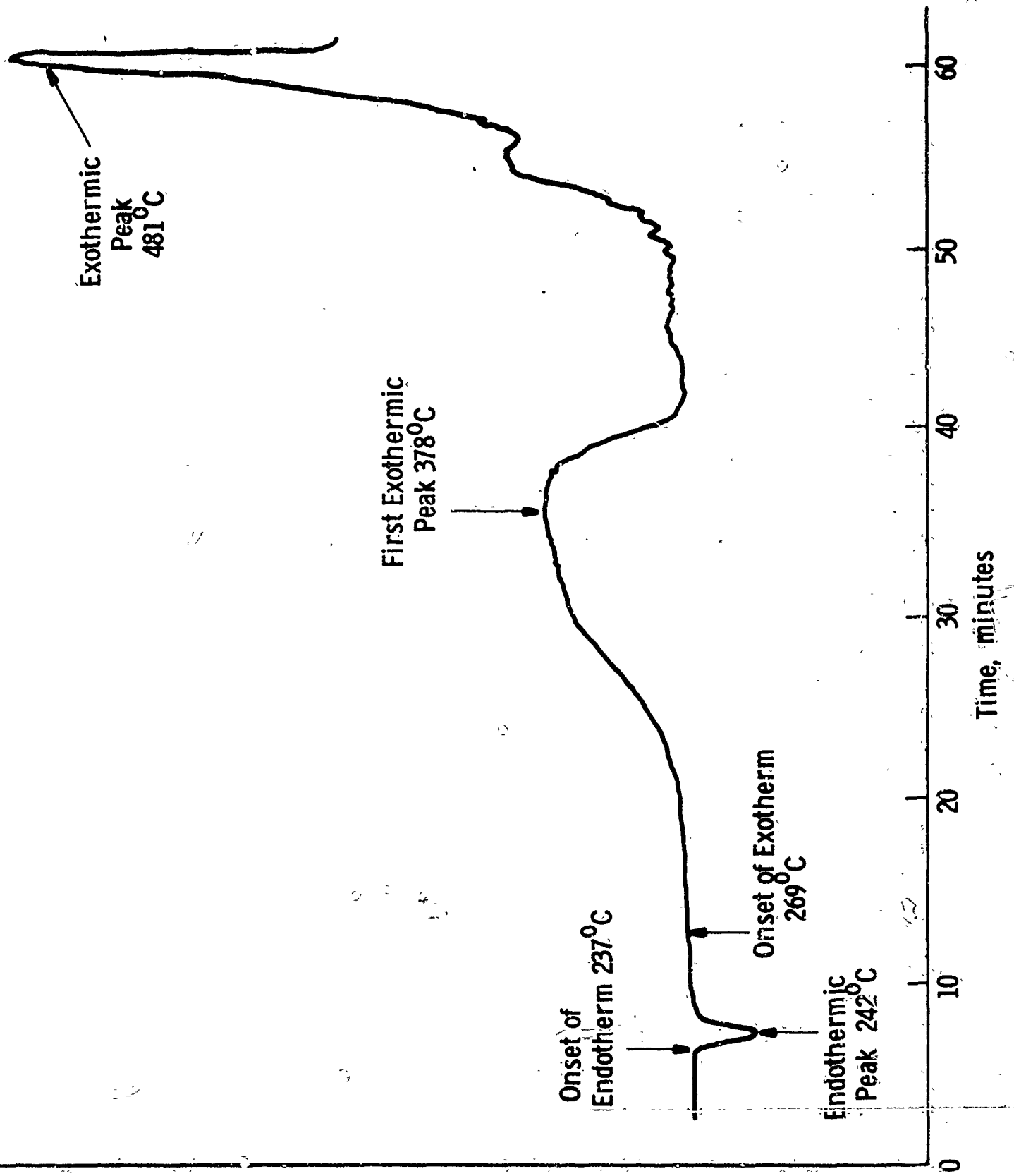
Preliminary microscopic analysis of the screened reagent grade ammonium perchlorate indicated that the sample (0.5- to 1.0-mm fraction) consisted principally of aggregates of smaller crystals ranging in size from about 200 to 400 microns.

Moisture analysis of both the screened reagent and specially recrystallized samples has been performed by periodic weighings of individual samples (40- to 45-gram size) stored over  $P_2O_5$  in a glass desiccator at atmospheric pressure. The screened reagent samples reached essentially constant weight after one week with a weight loss of about 2 to 2.5 mg or about 0.005%. Total weight losses after 53 days amounted to 0.006% and 0.008%, respectively, for two such samples.

The recrystallized sample, on the other hand, lost 0.57 grams the first day, a total of 0.6034 grams in the first 20 days and continued to lose at an approximately linear rate of 0.42 mg/day (0.001% per day) from the 20th to the 53rd day. These data are shown in Figure 2.

Differential Thermal Analysis (DTA) and Bureau of Mines Impact tests (Droptest) have been run on the screened and dried samples to be loaded into the irradiation pressure vessels. Both tests showed normal behavior for reagent grade ammonium perchlorate of this particle size range. The DTA (run at a heating rate of  $5^{\circ}C$ /minute with a 20-mg sample) showed the normal endotherm at  $241^{\circ}C$ , an exotherm peak at  $378^{\circ}C$  and a sharp exotherm (to complete decomposition) at  $481^{\circ}C$ , Figure 3. The 2-kg drop weight test using the bare anvil was negative at the maximum drop height of 100 cm. Addition of #180 grit increased the sensitivity to the measurable range, indicating a 50% point of 72 cm.





DTA PATTERN FOR REAGENT GRADE  
 $\text{NH}_4\text{ClO}_4$ ; 20 mg. SAMPLE;  $4.7^\circ\text{C}$  per MINUTE

FIGURE 3

## APPENDIX

### SAFETY ANALYSIS OF $\text{NH}_4\text{ClO}_4$ IRRADIATION IN AGNIR

#### 1. INTRODUCTION

The effect of fast neutrons on ammonium perchlorate ( $\text{NH}_4\text{ClO}_4$ ) will be evaluated by irradiating four samples of  $\text{NH}_4\text{ClO}_4$  in AGNIR. These fast-neutron interactions will create vacancy - interstitial pairs which are expected to increase the energy content of  $\text{NH}_4\text{ClO}_4$  above that of unirradiated  $\text{NH}_4\text{ClO}_4$ .

The irradiations will be performed in either the "F" or "G" rings of the AGNIR. The nominal fluxes for both fast and thermal neutrons is  $\approx 10^{12}$  neutrons/cm<sup>2</sup>-sec.

During irradiation the  $\text{NH}_4\text{ClO}_4$  will be contained in aluminum pressure vessels which will be contained in a modified dummy-element irradiation capsule (secondary containment). Two capsules, each containing approximately 10 grams of  $\text{NH}_4\text{ClO}_4$ , will be irradiated, simultaneously. The longest irradiation is scheduled for 600 hours, with other capsules scheduled for 40, 200 and 360 hour irradiations.

This appendix contains the results of the safety evaluation of various hazards, both nuclear and non-nuclear, associated with the irradiation of  $\text{NH}_4\text{ClO}_4$ .

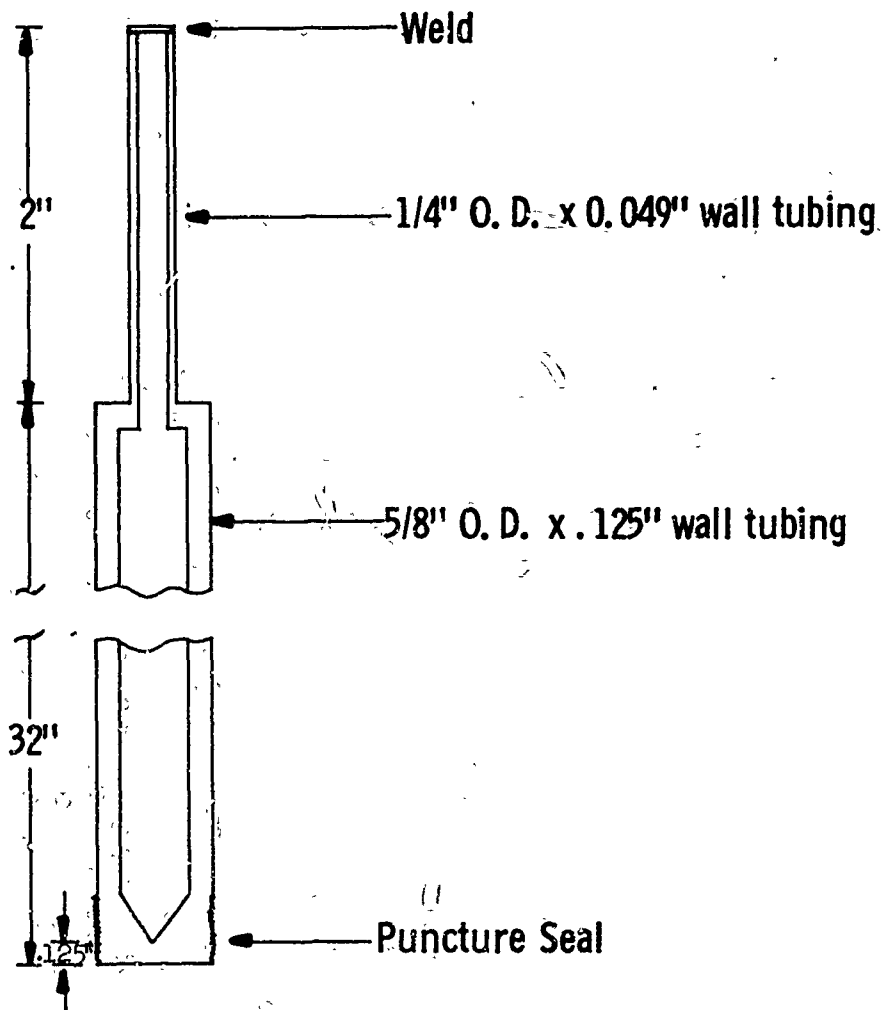
#### 2. EQUIPMENT AND MATERIALS

The pressure vessels will be fabricated from aluminum to keep the radiation dose rate from induced activities at a minimum. An iron-constantan thermocouple will be used to monitor the pressure vessel temperature during the irradiation.

##### a. Pressure Vessel

Type 6061 aluminum tubing and round stock will be used to fabricate the pressure vessels used for primary containment of the  $\text{NH}_4\text{ClO}_4$ . These pressure vessels will be fabricated in accordance with standards established by ASME for thin-walled pressure vessels. The design pressure for this pressure vessel is 4038 psia and the working pressure is 3029 psia. The main body of the pressure vessel will be 5/8 in. O.D. tubing with 0.125 in. walls by  $\approx 32$  in. long (see Figure 1). A 1/4 in. O.D. with .049 in. wall tube,  $\approx 2$  in. in length, will be welded at one end of the main body for sample loading access. This tubing will be welded shut after the loading operation. A puncture seal will be placed at the opposite end of the pressure vessel for post-irradiation gas sampling.

PRESSURE VESSEL FOR  $\text{NH}_4\text{ClO}_4$  IRRADIATION



APPENDIX - FIGURE 1

The complete pressure vessel, before loading of the sample, will be tempered to T6 and then hydrostatically tested to 6058 psia (2 times the working pressure) to ensure the integrity of the pressure vessel. Auxiliary tests include the hydrostatic testing of the 5/8 in. O.D. tubing to the burst pressure (expected to be >6060 psia) and the thermal decomposition of  $\text{NH}_4\text{ClO}_4$  in a fabricated and hydrostatically-tested pressure vessel.

The only instrumentation used will be an iron-constantan thermocouple attached to the pressure vessel used for the 600 hour irradiation. The signal from the thermocouple will be used to actuate an annunciator on the AGNIR control console when the capsule temperature is 150°C.

b. Secondary Container

The dummy-element irradiation capsule will be used for the secondary container for these  $\text{NH}_4\text{ClO}_4$  irradiations. The capsule is capable of containing 2 primary pressure vessels at any single moment (see Figure 2). A stainless steel bulkhead fitting will be used as the water-tight connector for passing the thermocouple into the secondary container.

c. Chemicals

Reagent grade  $\text{NH}_4\text{ClO}_4$ , purchased from Matheson, Coleman and Bell, will be used for the irradiation samples. The particle size of the  $\text{NH}_4\text{ClO}_4$  will be >500 $\mu$ , with additional larger particles, recrystallized from  $\text{H}_2\text{O}$ , added for X-ray analysis.

Approximately 10 grams of  $\text{NH}_4\text{ClO}_4$  will be irradiated in each pressure vessel for a total  $\text{NH}_4\text{ClO}_4$  loading of  $\approx$  20 grams during a single irradiation experiment.

Helium gas at 1 atm will be added over the  $\text{NH}_4\text{ClO}_4$  and in the secondary container to enhance the heat transfer within these containers.

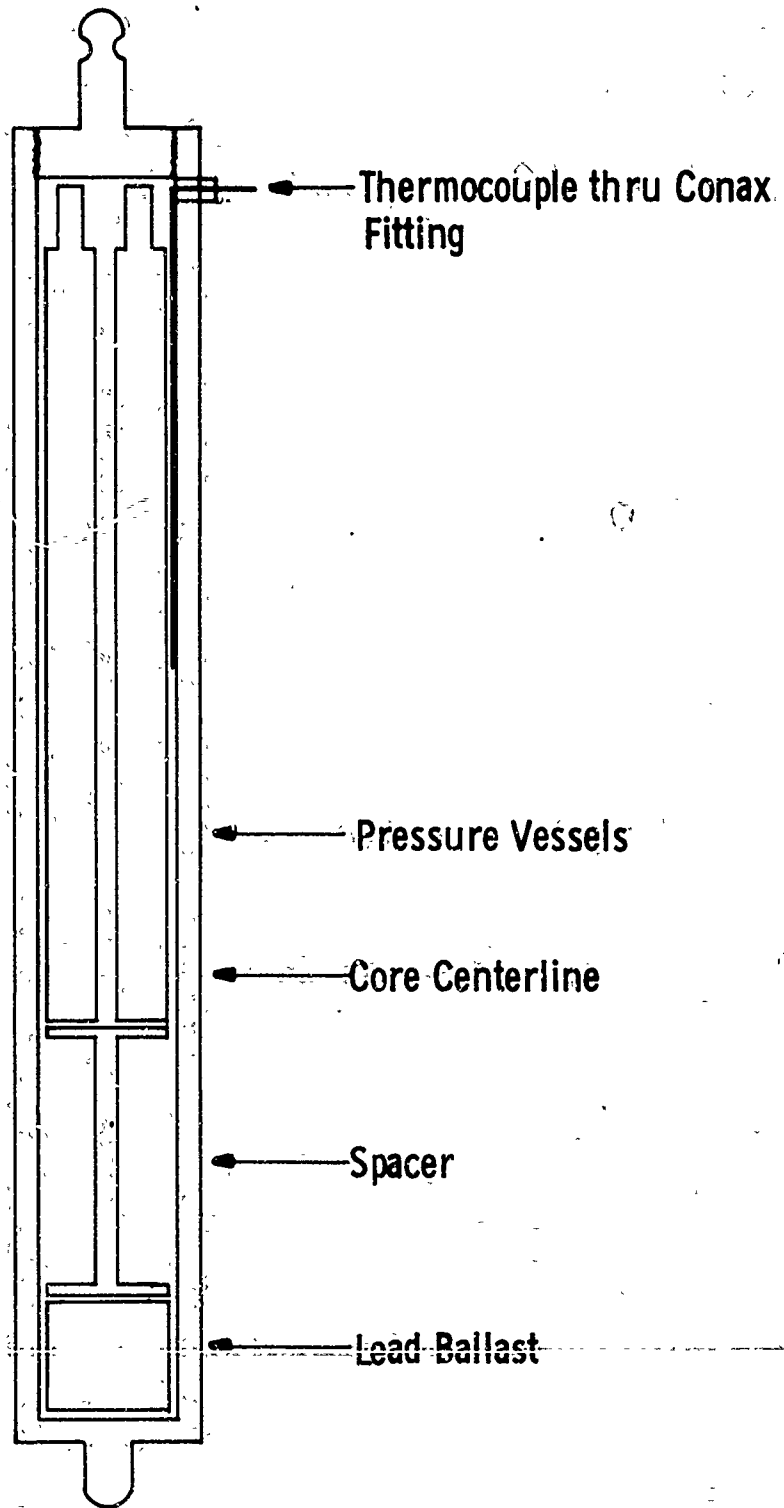
3. HEAT TRANSFER

Gamma heating will be the major source of heat generation in the pressure vessels. Based on the gamma heating rates calculated for the "F" ring, (4) the heat generation rate for a single pressure vessel will only be 2.36 Btu/hr-in. This heat, generated mainly in the walls of the pressure vessel will result in centerline temperatures of 70°C (157°F). This quantity of heat will not result in any excessive heating (within the pressure vessel) which might result in an exotherm or excessive pressure. Details of the heat transfer calculations are included in Paragraph 7.

4. SAFETY ANALYSIS

The major areas of concern when irradiating  $\text{NH}_4\text{ClO}_4$  are the pressure buildup caused by radiolysis of the compound and the possibility of detonation

**DUMMY-ELEMENT IRRADIATION CAPSULE CONTAINING  
2 PRESSURE VESSELS AND A THERMOCOUPLE**



**APPENDIX - FIGURE 2**

during irradiation. Both of these problems can be solved by appropriate engineering design. The reactivity effect caused by the irradiation capsule and the  $\text{NH}_4\text{ClO}_4$  is negative and negligible for this experiment.

a. Nuclear Hazards

The dose rates from neutron activation of two pressure vessels after irradiations of 1, 10 and 600 hours are shown in Figure 3. Although the dose rates at 1 foot are initially high, decay periods of one week or less will be sufficient to reduce the radiation field to a convenient, working level. The activation of the chlorine in  $\text{NH}_4\text{ClO}_4$  produces a radiation field of  $\approx 5$  R/hr at 1 foot at the end of the experiment. This dose rate is small compared to the dose rate from the irradiated pressure vessel and will diminish with a 37 minute half-life.

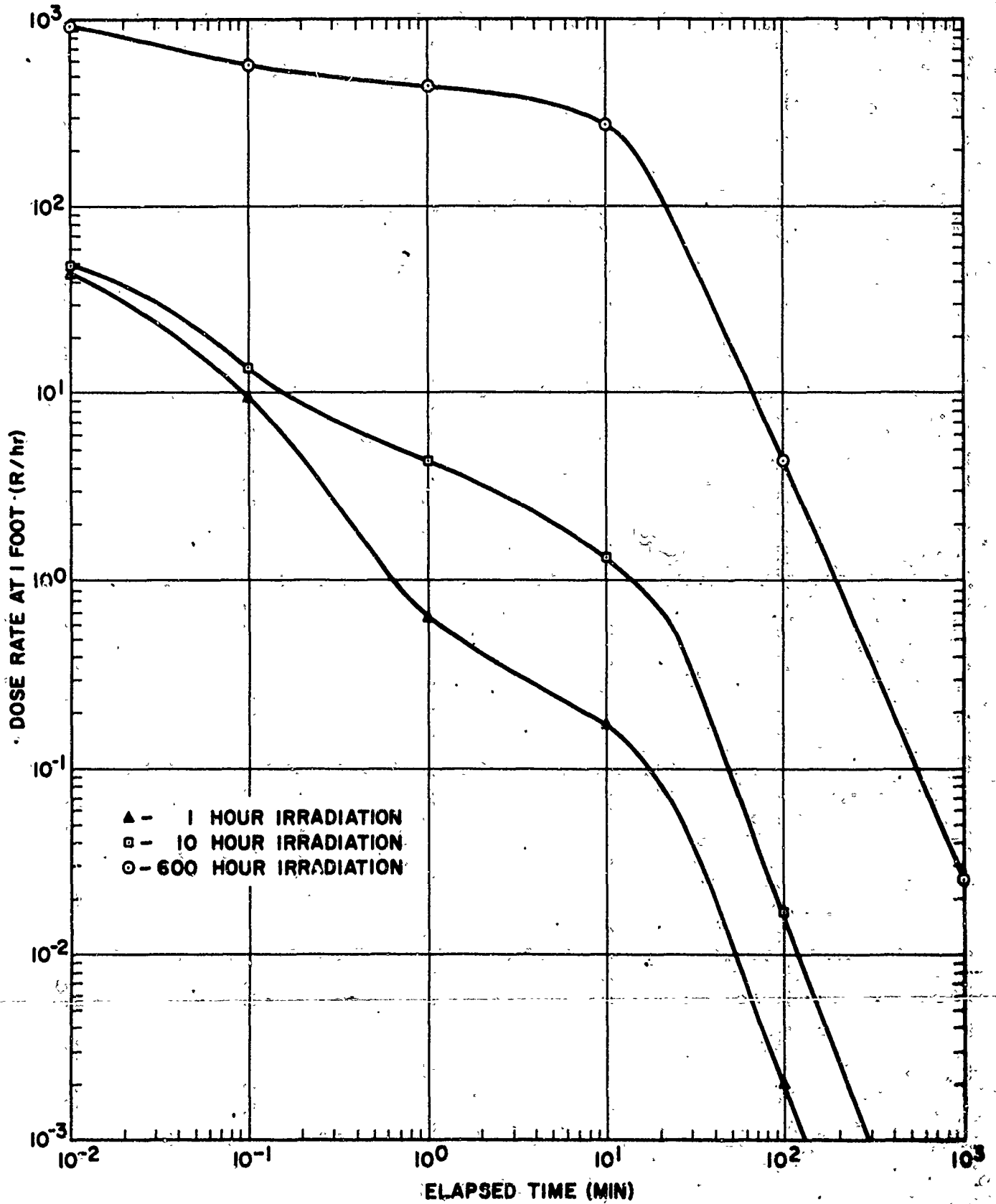
b. Non-Nuclear Hazards

The explosive properties of  $\text{NH}_4\text{ClO}_4$  are well known because of the extensive use of the compound in the formulation of solid propellants. Ammonium perchlorate is a low-grade explosive, listed as a Class 12 explosive according to Ordnance Standard 1724-C of the Ordnance Safety Manual (AMCR-385-225). The auto-ignition temperature for reagent grade  $\text{NH}_4\text{ClO}_4$  is  $380^\circ\text{C}$  ( $3100^\circ\text{C}$  for practical grade) with no exotherm occurring below  $240^\circ\text{C}$ . Of particular significance is the work performed by Andersen and Pesante on critical diameter as a function of particle size and tamped-density. (1) Their results showed that for 124 particle size and 0.6 g/cc density,  $\text{NH}_4\text{ClO}_4$  would not detonate (after use of No. 8 blasting caps to initiate the detonation) if the charge diameter were below 0.6 in. For an  $\text{NH}_4\text{ClO}_4$  density of 1.75 g/cc, no detonation occurred below 2.5 in. Since the pressure vessel internal diameter is 3/8" and the particle size is  $>500\mu$ , no detonation will occur during the proposed irradiation.

The radiolysis experiments of Freeman, et al, (5,8) and Odian, et al, (9,10) show that no detonation occurred during their experiments. Although the dose rates used by these experimenters are a factor of  $10^2$  lower than those calculated for this experiment, the effect of decomposition rate on pressure vessel integrity can be qualitatively evaluated by a thermal decomposition test. The heating rate will be selected to simulate the radiolytic decomposition rate at  $3 \times 10^7$  Rads/hr. This thermal decomposition test plus the examination of the pressure vessels after each irradiation should provide meaningful data about the pressure vessel used for the 600-hour irradiation. The fact that the total exposure dose for these experiments is also a factor of  $10^2$  higher than those of the other experimenters should not be significant since complete decomposition of  $\text{NH}_4\text{ClO}_4$  to the elements has been assumed for the purposes of this safety analysis.

The possibility of pressure vessel rupture caused by excessive pressure buildup by the non-condensable gases formed during the radiolysis of  $\text{NH}_4\text{ClO}_4$  is miniscule. The pressure vessels have been designed to

DOSE RATES FROM IRRADIATION OF ONE POUND OF ALUMINUM (TYPE 6061)



APPENDIX - FIGURE 3

accommodate the pressure at 70°C (157°F) caused by products formed by radiolytic decomposition of  $\text{NH}_4\text{ClO}_4$  to the elements. Although thermal decomposition of  $\text{NH}_4\text{ClO}_4$  results in 4.75 moles of product (including 1.5 moles of  $\text{H}_2\text{O}$ , the pressure vessels have been designed to contain 5 moles of product, the theoretical limit, for every mole of  $\text{NH}_4\text{ClO}_4$  irradiated. The effects of pressure vessel corrosion caused by the radiolysis products will also be evaluated by thermally decomposing  $\text{NH}_4\text{ClO}_4$  in a replica of the pressure vessel. Post-irradiation examination of the pressure vessel will be performed after 40, 200 and 400 hours of irradiation to determine the integrity of the pressure vessels against any corrosion.

#### 5. LIMITING CONDITIONS

The irradiations will be performed on an 8-hr/day basis in either the "F" or "G" rings of AGNIR. The temperature of one of the pressure vessels will be monitored at  $\approx 1$  hr. intervals during the length of the irradiation. The limiting pressure vessel temperature will be 200°C, which is below the 240°C transition temperature. The experiment will be discontinued if post-irradiation examination indicates excessive corrosion of the pressure vessels.

#### 6. DISCUSSION

Since  $\text{NH}_4\text{ClO}_4$  is widely used as the oxidizer in solid-propellant formulations, its explosive properties as a compound are readily overrated by the casual observer. The experimental results compiled by Andersen and Pesante on particle size-critical diameters and the extensive irradiation experiments performed by Freeman and Odian and their co-workers attest to the inherent safety of the irradiations discussed here. The pressure vessel diameter is less than the critical diameter of 1.52 cm (0.6 in.) required to sustain a detonation for even low-density  $\text{NH}_4\text{ClO}_4$ . The  $\text{NH}_4\text{ClO}_4$  particle size is an order of magnitude greater than the particle size (12 $\mu$ ) associated with the 1.52 cm critical diameter.

The temperature inside the pressure vessel (70°C) is well below the 240°C temperature below which no exotherms have ever been observed. By assuming complete radiolysis of  $\text{NH}_4\text{ClO}_4$  to the elements, the highest possible pressure is calculated. This pressure of 206 atm (3100 psia) at 70°C (157°F) is readily contained by both the primary and secondary containers. The minor problem of dose rates from induced activities is easily solved by allowing the appropriate time interval to elapse before handling.

The results of this safety analysis show that irradiation of  $\text{NH}_4\text{ClO}_4$  can be performed in AGNIR without any danger to the reactor itself, or the personnel associated with the experiment.

## 7. HEAT TRANSFER CALCULATIONS

The major source of heating during the AGNIR irradiation of  $\text{NH}_4\text{ClO}_4$  is from gamma radiation. The heating caused by the dissociation of the  $\text{NH}_4\text{ClO}_4$  ( $\Delta H = 665 \text{ cal/g}$ ) is only 0.7 Btu/hr (over a 40 hr irradiation) or  $\approx 1\%$  of the total heat generation rate.

For an aluminum tube, .625 in. O.D. x .125 in. walls, the heating rate per unit length is

$$\text{wt Al} = 16.387 \pi D \rho$$

$$= 8.7 \text{ g/in.}$$

where  $\rho = 2.7 \text{ g/cc}$

$$t = .125 \text{ in.}$$

$$D = .50 \text{ in.}$$

Since the  $\gamma$ -heating rate for the "F" ring is .079 watts/g (see EAD-268, J. K. Witthaus), the heating rate is

$$Q = (.079)(8.7)(3.41) = 2.4 \text{ Btu/hr-in.}$$

Assuming that the average distance between the pressure vessel and secondary capsule walls is .5r ( $L = .027 \text{ ft}$ ), the  $\Delta T$  is readily calculated as

$$\Delta T = \frac{QL}{kA} = \frac{(2.4)(.027)}{(.014)(.1)} = 47^\circ\text{F}$$

where  $k = .1 \text{ Btu/hr-ft-}^\circ\text{F}$  (for helium)

$$A = .014 \text{ ft}^2.$$

Therefore, the pressure vessel temperature is  $70^\circ\text{C}$  ( $157^\circ\text{F}$ ), well below the lowest exotherm temperature ( $240^\circ\text{C}$ ) for  $\text{NH}_4\text{ClO}_4$ . The gamma-heating in 10 grams of  $\text{NH}_4\text{ClO}_4$  is 3 Btu/hr,  $< 4\%$  of that for the pressure vessel.

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13. ABSTRACT			
<p>During the first quarter of the program a majority of the preliminary steps required before the actual start of neutron irradiation were accomplished.</p> <p>The Hazards Analysis Report was written and approved by the AGN Reactor Safeguards Committee. The pressure vessel design has been completed and hydrostatic testing of the main body showed a burst pressure higher than anticipated.</p> <p>In preparation for energy content and analytical measurements, the heat of solution Calorimeter was re-assembled and the electrical measurements circuits checked out. Reagent grade ammonium perchlorate was purchased and screened to separate out crystals in the 500 to 1000 micron size range for the radiation and calorimetry work. A portion of this sample was recrystallized for X-ray and microscopic studies.</p> <p>DTA and Bureau of Mines Impact tests have been run on the screened reagent sample for comparison with similar measurements to be run with irradiated samples later in the program.</p>			

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