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ADDENDUM NO. 4 TO REPORT NO. 2510 TO
AEC-NASA SPACE NUCLEAR PROPULSION OFFICE

FINAL TEST SPECIFICATIONS FOR PHASE I-C OF THE
COUNTERMEASURES RADIATION EFFECTS PROGRAM

VOL. I - TEXT AND UNCLASSIFIED APPENDIXES

Contract SNP-1

July 1965

NER VA Program

MASTER

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COUNTERMEASURES RADIATION EFFECTS PROGRAM

VOL. I, TEXT AND UNCLASSIFIED APPENDIXES

ROCKET ENGINE OPERATIONS - NUCLEAR

Contract SNP-1

July 1965

NERVA Program

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FOREWORD

This report is presented in two volumes. Volume I, containing the text and Appendixes A through D, is unclassified. Volume II, containing Appendix E, is classified "Confidential - Restricted Data."

ABSTRACT

The test specification presented herein describes in detail the irradiation tests to be performed at the General Electric Vallecitos Atomic Laboratory in fulfillment of Phase I-C of the Countermeasures Radiation Effects Program, REON Sub-Subtask 1.9.1. The volume of information required for this test specification dictates that it be issued as an addendum to REON Report 2510, Radiation Effects Testing, rather than as an insert for that report.

The experiments described in this document are the result of combined efforts by Aerojet-General's REON and Downey Ordnance Research Divisions, General Electric Company Vallecitos Atomic Laboratory, and the United States Army Picatinny Arsenal. This document is submitted in partial fulfillment of Contract SNP-1.

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I. INTRODUCTION

Aerojet-General Corporation's REON Division has initiated a program to investigate radiation effects on high explosives and propellants as they pertain to the NERVA Countermeasures System. This program has been designated as the Countermeasures Radiation Effects Program, Sub-Subtask 1.9.1.

This document is provided in two volumes to facilitate handling of the unclassified and classified portions. The unclassified volume will be used primarily by General Electric personnel to define operational procedures; the classified volume will be used primarily by the reactor safeguards personnel.

The specifications presented within this document are for the fourth portion of the Countermeasures Radiation Effects Program, Phase I-C. Included are descriptions of the experimental procedures to be used in Phase I-C to determine the most radiation-resistant high explosives and propellants. These procedures will involve gathering data on weight loss, gas release (continuous and point measurement), thermogravimetric analysis, explosion temperature, differential thermal analysis, and vacuum stability - all as a function of integrated radiation energy deposition. Then data will be obtained on mild detonating fuzes (MDF) made with booster and main charge type explosives, while selected cast and extrudable propellant strands will be burned to evaluate possible changes due to radiation.

A continuous monitoring of gas release and temperature distribution will be incorporated into Phase I-C to provide a better method of evaluating decomposition mechanisms, thus enabling a definition of maximum radiation stability (threshold).

Prior to irradiating the I-C samples, a mock irradiation capsule loaded with charcoal will be instrumented to allow a system "check-out" on the continuous temperature and pressure measurement apparatus. Also, this mock capsule will be irradiated for the anticipated maximum irradiation time of 5 hours to establish comparative temperature and pressure data.

Two new post-irradiation (thermogravimetric analysis and strand burner) tests will be utilized to evaluate the propellant formulations. The measure of the propellant's strand burning rate will demonstrate the functional characteristic of these materials and thus define destruct system integration requirements. These burning rates will be measured at 1000 psi and 70⁰F to provide data which can be compared to previous tests conducted at the United States Army Picatinny Arsenal.

This program is being performed under the joint direction of Aerojet-General Corporation (REON and Downey Ordnance Research Division), U.S. Army Picatinny Arsenal, and General Electric Co. (Irradiation Processing Operations). Samples will be irradiated in the GE Test Reactor at Vallecitos, California with subsequent tests performed jointly by Aerojet-General, General Electric, and Picatinny Arsenal personnel in the Radiological Materials Laboratory; GE personnel will be primarily responsible for radiological safety and monitoring.

Upon the completion of Phase I-C, a technical review of the data produced will be held at the General Electric Reactor and attended by each participating organization. Thereafter, copies of the conclusions reached

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by this group and of the raw test data will be compiled and forwarded to each organization for additional review.

II. DESCRIPTION OF IRRADIATION CAPSULES AND ASSOCIATED HARDWARE

Two types of irradiation capsules will be used in Phase I-C: instrumented and non-instrumented. The non-instrumented type are identical to the capsules previously used in Phases I, I-B and I-D, while the instrumented capsules have a modified top to allow continuous measurement of temperature and pressure.

A. NON-INSTRUMENTED CAPSULE

The basic non-instrumented capsule (Figure 1) developed by Aerojet's Downey Ordnance Research Division was designed in accordance with the following plan:

The physical containment capability must be such that the capsule will maintain its overall integrity when twice the amount of explosive anticipated for irradiation is detonated inside the capsule.

The gases produced by a detonation of twice the amount of explosive to be irradiated must be adequately contained after detonation and/or be released in a non-destructive manner, which for the General Electric Test Reactor (GETR) was established as no more than 10 cu cm/min.

The capsule dimensions must be such as to permit insertion into the Z-Trawl Cable Facility of the General Electric Test Reactor.

As illustrated in Figure 1, the principal features of the capsule design include:

A center cavity for insertion of a quartz tube which contains the explosive or propellant sample

A pattern of six cavities surrounding the center cavity to absorb a portion of the shock energy produced in the event the sample detonated

An expansion chamber (1.00-in. dia, 1.531-in. long) to reduce the gas pressure produced if detonation of the sample occurs

A continuous metal path from the center hole to the outside surface of the capsule, providing a transfer of heat which is generated by the radiation

A cap secured by 12 heat-treated bolts, torqued to 200 in.-lb

A blast cup, fabricated from "0" (soft) aluminum to expand and seal the expansion chamber in the event of detonation

A "Flexitallic" gasket, tested to permit no more than 10 cu cm/min leakage of gas from the capsule after detonation.

A view of the capsule and its internal components prior to assembly is provided in Figure 2 to illustrate arrangement and position of internal components as well as the principal features of the internal system. As noted, these features include:

A steel spring to raise the quartz tube upon opening the capsule

A quartz tube to contain the explosive during irradiation (except the two primary explosives which will be loaded into aluminum tubes)

A quartz-wool plug to prevent spillage of the explosive while manipulating and weighing the quartz tube

A ball-check-valve system such that gas generated from the explosive during irradiation is not re-introduced to the explosive

A retainer disk to hold the quartz tube in position and provide a base for the blast cup.

In addition to the internal components described above, flux wires and temperature indicators will be positioned in the explosive sample to provide data on integrated neutron (flux thermal and fast) and temperature (158 and 255°F) thresholds attained during irradiation. Assembly drawings illustrating these flux and temperature indicators are provided in Figures 3 and 4.

These indicators have also been made in different lengths (Figure 3 and 4) to facilitate visual identification during loading and thereafter in the X-ray negatives of the capsule assemblies.

B. INSTRUMENTED CAPSULE

The instrumented capsule (Figure 5) exhibits the same basic features as the non-instrumented capsule with the only modification being a new cap to accommodate the attachment of a 1/8-in.-dia aluminum tube; housed within the aluminum tube (Figure 5) are fibrous-glass-insulated 28-gage copper-constantan thermocouple wires to provide a continuous path from the capsule's interior to the third floor of the GETR where continuous measurements of temperature and pressure will be recorded.

A complete summation of design data and analytical calculations regarding the basic irradiation capsule has previously been reported in Reference 1 (pages 11 through 44 and Appendixes A through E) and Reference 2 (Appendix A). Additional qualification testing was accomplished on the instrumented capsule assembly to demonstrate its capability of meeting the requirements set forth by General Electric. Results of these tests are included in the Hazards Analysis portion (Section V) of this report.

C. PRESSURE AND TEMPERATURE MEASUREMENT APPARATUS

The gas pressure measurement apparatus is used in conjunction with the instrumented irradiation capsule to permit a continuous measurement of pressure during irradiation. The apparatus itself consists primarily of a panel (Figure 6), the design of which includes a Swagelok capsule coupling, needle valves, safety heads, vacuum gage and tank, pressure gage, and exhaust filter. Figure 7 illustrates the basic panel configuration and denotes the capsule connection position. The Swagelok coupling connects to the filter (Commercial Product No. 634-1-F2B) of the instrumented capsule assembly shown in Figure 5.

Prior to irradiation, the instrumented capsule assembly consisting of 40 ft of aluminum tubing (1/8 in. OD), a junction block, a bellows valve, and a filter, will be connected to the gas pressure measurement apparatus and pressure-checked for leakage with 75 psig of helium. Thereafter, the system

will be vented to atmospheric pressure by opening and closing the vent valve (location B on Figure 7). Then the bellows valve will be opened and the capsule vented to zero pressure, if necessary. The capsule will then be lowered into the Z-trail cable assembly and irradiated. During irradiation, joints in the piping system connected to the capsule will be monitored for leaks. After irradiation and retrieval, the entire system will be evacuated via the needle valve into the vent tank, and the bellows valve closed. The tubing will then be disconnected from the Swagelok coupling on the panel and the aluminum tube cut as close to the capsule as radiation permits. The tubing will be cut while the capsule is immersed in the GETR storage canal, and the end capped with a Swagelok cap to prevent water from entering the capsule. That portion of the tubing system which is severed is considered expendable.

A glove box (shown in Figure 8) having an open bottom will be used while cutting the capsule tubing. The bottom will be immersed in the reactor canal water, thus providing containment for any radioactive gas released, in addition to providing protection for the operator. The tubing leaving the capsule should be bent to block the beam of radiation from the capsule in the canal water.

Evacuation of the entire system before disconnection and severing of the capsule is done to prevent escape of radioactive gases from the system. In addition, the vent tank is large enough (30 liters) to contain all the gases produced when 1.8 g of explosive burns within the capsule, and still remain at 1/30 atmosphere. Unless a sample burns, or a leak occurs, it will not be necessary to re-evacuate the vent tank. The activity of the vent tank will be monitored periodically, and it will be pumped out, if necessary, to keep the activity to an acceptable level.

In the event of a detonation (the worst case), the seal ring and blast cup will seal the capsule. Should they malfunction, the rupture disk to the vent tank will open, relieving the pressure produced. Also, the tubing will contain the pressure released, even without venting. The filter in the

line will keep contamination out of the panel system and allow the tubing to be removed and new capsules to be attached without releasing contamination.

The temperature of the samples will be detected by the welded copper-constantan thermocouple imbedded in the powdered portion of the sample. The thermocouple wire will be of a small diameter to permit the lead wire to fit inside the aluminum tubing and also to assure rapid response to temperature changes.

The measured thermocouple resistance is 80 ohms, and insulation resistance is over 2000 megohms. These values will be checked just before irradiation to verify that the thermocouples have not been damaged in transit. Small-diameter copper-constantan thermocouple wire was chosen so as to reduce neutron activation, produce a high voltage output, and allow use of a copper-wired millivolt recorder for both temperature and pressure.

The wiring schematic for the thermocouple is shown in Figure 9. The use of an oven set at $150 \pm 0.2^{\circ}\text{F}$ for the cold junction compensation will permit increased sensitivity in the range of interest. This type of thermocouple has been used successfully in irradiation tests on similar materials;^{*} thus no problems are anticipated due to the presence of radiation.

^{*} ASD-TR-64-91, Part II, 2, pp 181-193, Dec 1964.

III. REACTOR EXPOSURE REQUIREMENTS

A. GENERAL

Phase I-C is designed to utilize general nuclear fission radiation consisting primarily of thermal and epi-thermal neutrons and gamma rays. There will be no attempt to select the type of radiation received by putting materials in the capsule to moderate or capture neutrons, or to absorb gamma energy. Naturally, the configuration of the irradiation capsule will influence the amount and types of radiation received, but no attempt has been made to control the radiation distribution. The lead sheath of the MDF samples will attenuate the radiation; however, since the samples will be used in this form, the effects of radiation on their performance should be representative of the configuration in the destruct system.

B. RADIATION DOSAGE DESIRED

Based on the anticipated NERVA environment (References 3 and 4), it is desirable to irradiate all samples at the maximum flux position in the GETR Z-trail cable facility. Previous irradiations in this facility have shown the peak fluxes present with the irradiation capsule to be approximately 1.4×10^{13} nv fast neutrons ($E > 0.18$ mev), 9×10^{13} nv thermal neutrons, and 4.2×10^8 R/hr gamma.

An irradiation schedule (Table 1) was prepared utilizing (1) the test results of previous phases, and (2) the following criteria:

The mock irradiation capsule will be irradiated late in cycle 66 (6-14-65 to 7-18-65) to allow adequate time for pre-irradiation equipment calibration.

Evaluation of samples will occur late in cycle 67 (7-19-65 to 8-22-65) so as to provide a more uniform exposure of the samples irradiated. (It is during the initial 14 days of the cycle that the greatest temperature and flux variations are experienced with irradiation capsule location or relocation.)

The maximum number of irradiated samples which can be analyzed in one day is four. However samples which, as a result of activation, require additional decay time may be irradiated with the samples to be analyzed immediately so as to utilize as much of the daily irradiation cycle as is practicable.

Previous phases experienced minor delays due to reactor scrams; therefore, cycle 67 irradiations are scheduled to be completed on 8-12-65, leaving approximately 7 days as a safety factor.

All five propellant samples will be irradiated and analyzed in cycle 67 to allow adequate time for choosing the best two samples for irradiation in cycle 68.

Cycle 68 (8-23-65 to 9-26-65) irradiations are to be completed 9-17-65, thereby allowing a safety factor of 5 days for reactor scrams.

1. Explosive Samples

As specified in Table 1, the maximum integrated flux and dosage on the explosives is expected to produce a 5 to 10% weight loss with a resultant pressure of approximately 40 psi. The temperature is expected to be between 100 and 150°C. Since rapid decomposition may initiate rapid temperature and pressure increases, it is planned to remove these irradiation capsules from the Z-trial cable if their pressure exceeds 40 psi or their temperature goes above 200°C. Representatives of Aerojet-General Corporation and Picatinny Arsenal will be present during the irradiations; modifications to the temperature and pressure limits may be incorporated at that time, dependent upon analysis of the first instrumented capsule.

After the instrumented capsule has been irradiated and analyzed, the remaining non-instrumented capsules will be irradiated at lower integrated fluxes and dosages which are estimated to be between 2 and 5 hours. The desired integrated fast neutron flux ($E > 0.18$ mev) would then be as follows:

<u>Irradiation Time (hours)</u>	<u>Approximate Integrated Fast Neutron Flux</u>
5	2.5×10^{17}
4	2.0×10^{17}
3	1.5×10^{17}
2	1.0×10^{17}

<u>Irradiation Time (hours)</u>	<u>Approximate Integrated Gamma Dose</u>
5	2.1×10^9 R
4	1.7×10^9 R
3	1.3×10^9 R
2	8.4×10^8 R

2. Propellant Samples

Non-instrumented capsules of each propellant will be irradiated for 2 hours, thus experiencing an integrated fast neutron ($E > 0.18$ mev) flux of 1.0×10^{17} and an integrated gamma dose of 8.4×10^8 R. This procedure is in response to a special request of Picatinny Arsenal AED personnel to enable a comparison of these materials with the results of the best candidate from Phase I-B. Thereafter, a joint decision will be made as to whether or not the second non-instrumented capsule should be irradiated to a higher or lower integrated fast neutron flux. Consequently, the exact irradiation time will be determined at the reactor site.

It should also be mentioned that after the two best propellants are selected from cycle 67, four additional capsules of these materials will again be loaded at Aerojet's Downey facility for irradiation in cycle 68. A total of two irradiation capsules (one instrumented, one non-instrumented) will be loaded with each of these materials and irradiated as described below.

Both non-instrumented capsules will be exposed to the maximum tolerable integrated fast neutron and gamma dosage experienced in cycle 67. These samples will be analyzed after approximately 3 months delay to determine if aging has a significant effect on the propellant properties.

Both instrumented capsules will be irradiated until approximately 5 to 10% weight loss occurs, based on pressure measurements.

3. Flux Monitors

a. Neutrons

Total neutron dosage will be determined by employing flux wires approximately 0.50 in. long, sealed in quartz tubing (2 mm OD, 1 mm ID). Aerojet Drawing No. 515265, illustrating the design of these flux wires, is provided as Figure 3. Aluminum cobalt wires will be used for thermal flux measurements, while nickel wires will be used for fast flux measurements. These wires have been supplied to Aerojet by the General Electric Company's NEPS department.

Each capsule for Phase I-C will contain one of each type of flux wire. The exact position of these wires will depend upon whether the capsule contains granular explosive (as in Phases I and I-B), or propellant strands.

These flux wires will be forwarded to the General Electric Company Chemistry Laboratory for counting after removal from the irradiation capsule. Counting may require as much as 2 weeks for completion, dependent upon the induced activity of these wires.

b. Gamma

The General Electric physics personnel will calculate and properly designate the position of maximum dose rate. Previous phases which employed an ionization chamber provided positions of maximum dose rate identical with those calculated by the physics personnel. Consequently, for Phase I-C, the calculated values are considered entirely adequate.

IV. OPERATIONAL PROCEDURES

To facilitate handling of the irradiation capsule and guarantee thorough data acquisition, the Program Manager at the General Electric Company Vallecitos Atomic Laboratory will issue explosive capsule data sheets prior to performing any of the irradiations. These data sheets have been previously provided in Table 1 through 10 of Addendum No. 3 to Report No. 2510.* Two additional post-irradiation tests will be performed during this phase: the thermal gravimetric analysis (TGA) and the propellant strand burner. The data sheets required to accommodate these tests are provided as Tables 2 and 3. These data sheets will be employed in the General Electric Test Reactor and Radiological Materials Laboratory (Hot Cell and Explosive Test Facility).

A. PRE-IRRADIATION

Prior to the irradiating of Phase I-C materials, a mock instrumented irradiation capsule loaded with charcoal will be irradiated to verify temperature and pressure measurements over a 5-hour run (2.5×10^{17} NVT, $E > 0.18$ mev) at peak flux (1.4×10^{13} , $E > 0.18$ mev). Five hours at peak flux is at present the best estimate of the upper threshold limit on most of the explosive samples; consequently, 2 to 5 hours of irradiation should adequately indicate each materials limit in terms of radiation stability by a temperature rise and pressure increase. The mock irradiation procedure will be carried out as described below.

1. Pre-Irradiation Checkout

- a. Put ice and distilled water in the thermos containing the thermocouple junction, and turn on the mixing motor.
- b. Turn on the recorder power supply; leave chart motor off.
- c. Carefully unroll aluminum tubing attached to capsule. Do not twist tube in relation to capsule or use it to support the weight of the capsule.

* Final Test Specification for Transient Nuclear Test (KIWI-TNT) of the Countermeasures Radiation Effects Program, Contract SNP-1, Aerojet Report No. 2510, December 1964.

- d. Locate pressure panel next to Z-trail cable opening and connect helium supply to panel.
- e. Connect capsule to pressure panel. Check to see that valves B, D, and E are closed. Make sure valve C is open. Check thermocouple resistance (it should be 80 ohms, and the thermocouple-to-tubing resistance should be over 1 megohm). Then connect thermocouple wires to the terminal strip on the panel.
- f. Slowly open valve A until gage reads 70 psi. Check capsule connection for leaks using a "Snoop" leak detector. Gross leaks will be shown by a drop in pressure. Correct leaking connections as necessary by tightening them in small increments and then re-checking. If fittings are tightened too much, the tubing will be crushed and weakened. Be careful not to twist or bend tubing connections!
- g. When it has been determined that there are no leaks in the pressure measurement system or the capsule hook-up to the panel, open valve B to bleed off the pressure. Close valve B.
- h. Place the capsule body in a bucket of water so that the capsule connection fitting is submerged. Open the capsule valve and then open valve A slowly to bleed helium into the capsule at a maximum rate of 15 psi/min until the gage reads 70 psi. Check for possible drop in pressure. (Note: It is important that the capsule be pressurized or depressurized at the specified rate since the orifices leading into the capsule are small and large differential pressures on either side of the blast cup can damage the T/C wires due to movement of cup.) A few seconds wait might be needed to allow the

pressure to come to equilibrium within the capsule. Any leaks around the capsule connection fitting should be readily visible in the water. Check for leaks around the capsule valve stem, T/C lead-through, etc., using the "Snoop" detector. Tighten connections if necessary.

- i. After leaks are eliminated, turn on recorder chart and check operation and pressure zero adjustment. Slowly bleed pressure down to atmospheric through valve B (maximum, 15 psi/min). Record both chart reading and pressure gage at 10 psi intervals.

2. Irradiation Procedure

- a. When recorder is working properly, slowly lower capsule into Z-trail cable tube. Be careful not to twist or bend tubing connections! Use cable - not tubing - to support weight.
- b. Note the time that the capsule reaches the peak flux position and enter this on the recorder chart.
- c. An initial temperature rise to about 90°C should occur within a few minutes, accompanied by a small increase in pressure due to heating of the gas in the capsule.
- d. Record pool water temperature, reactor power level, and capsule location. The center of capsule should be at the peak flux position. Record flux profile as obtained from physics calculations.
- e. Check pressure gage vs recorder readings and mark chart with gage readings and time periodically.
- f. Check thermocouple temperature with a potentiometer box and compare with chart record a few times; mark potentiometer reading on chart at time it is made. Use copper-constantan thermocouple calibration curve.

- g. Assuming recorder is working correctly, only a few manual readings need be made to check it. If the recorder malfunctions, manual recordings of pressure gage and potentiometer should be made at half-hour intervals.
- h. Irradiate for 5 hours at peak flux of GETR, then partially withdraw capsule to a zero flux position to cool. Use cable - not tubing - to pull up capsule.

3. Post-Irradiation Procedures

- a. Pressurize capsule to 50 psi and check for leaks as indicated by a drop in pressure for 5 min. Open valve B exhaust and bleed pressure down to zero psig, then close valve to capsule. Change pressure slowly to prevent sealing capsule.
- b. Disconnect capsule tube between filter and panel, and disconnect thermocouple wires from terminal strip on panel.
- c. Position transfer cask over Z-trail cable opening, with tubing and cable passing through cask.
- d. Withdraw capsule into transfer cask using cable.
- e. Transfer capsule to storage pool from cask.
- f. After capsule has cooled approximately 8 hours, withdraw as much tubing as possible, based on its activity. Cut off tubing with a tubing cutter, and thermocouple wires with a diagonal cutter. Cap end of tubing with a Swagelok cap. Cut cable with a cable cutter.
- g. Place capsule in transfer cask and move to RML. Check to be sure that cask will also hold three instrumented capsules.
- h. Open capsule by cutting off top in lathe.

- i. Cut thermocouple wires and remove cap of capsule.
- j. Remove contents of capsule using lucite tube assembly, and transfer vial to basement laboratory.
- k. Remove flux wires and send to laboratory for counting. Remove temperature monitors and observe their condition (melted or not melted). Save for possible further testing in boiling water.

B. IRRADIATION PROCEDURES FOR NON-INSTRUMENTED CAPSULE

- 1. Attach the Aerojet-supplied steel cable to the irradiation capsule. Check to make certain the cable is firmly attached.
- 2. Carefully lower the capsule into the position of maximum flux in the Z-trail cable facility. This position will be supplied by GE physics personnel prior to irradiation of each capsule.
- 3. As soon as the capsule reaches maximum flux position, the reactor technician will record the following information on the capsule data sheet:
 - a. Time that capsule reaches irradiation depth
 - b. Irradiation depth
 - c. Power level of reactor
 - d. Average rod bank
 - e. Pool water temperature inlet and outlet
 - f. Date of reactor startup
- 4. When the irradiation time has elapsed, remove the capsule from the maximum flux position and allow it to remain below water until reduction of the radiation level permits it to be pulled to the surface.

5. The reactor technician will record the following information at this time:
 - a. Time that capsule leaves irradiation depth.
 - b. Total irradiation time
 - c. Capsule activity, R/hr
 - d. Any unexpected events
6. The capsule is then pulled into a special transfer cask designed for Z-trail cable use and removed from the trail cable opening.
7. As soon as possible after irradiating the first capsule, attach the lowering cable to the second irradiation capsule and carefully position this capsule in the maximum flux location.
8. While the second capsule is being irradiated, the first capsule will be transferred to the reactor canal and stored until all capsules have been irradiated.
9. The reactor technician will record the canal storage position.

C. IRRADIATION PROCEDURES FOR INSTRUMENTED CAPSULE

1. Check resistance of thermocouple and insulation resistance. Attach thermocouple wires and pressure connection from capsule assembly to pressure measurement panel.
2. Check to see that steel cable and tubing fittings are properly connected and tight. Turn on recorder.
3. Pressurize panel and connection as far as valve with helium at 75 psi. Then shut off helium valve and be sure pressure does not decrease over a 5 min period. Check connections with a soap solution and tighten if necessary to make leak-tight joints. Check recorder against indicator.

5. The reactor technician will record the following information at this time:
 - a. Time that capsule leaves irradiation depth.
 - b. Total irradiation time
 - c. Capsule activity, R/hr
 - d. Any unexpected events
6. The capsule is then pulled into a special transfer cask designed for Z-trail cable use and removed from the trail cable opening.
7. As soon as possible after irradiating the first capsule, attach the lowering cable to the second irradiation capsule and carefully position this capsule in the maximum flux location.
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1. Check resistance of thermocouple and insulation resistance. Attach thermocouple wires and pressure connection from capsule assembly to pressure measurement panel.
2. Check to see that steel cable and tubing fittings are properly connected and tight. Turn on recorder.
3. Pressurize panel and connection as far as valve with helium at 75 psi. Then shut off helium valve and be sure pressure does not decrease over a 5 min period. Check connections with a soap solution and tighten if necessary to make leak-tight joints. Check recorder against indicator.

4. Bleed off pressure through filter to vent line; close vent line.
5. Open valve to capsule and check initial pressure.
6. Carefully lower capsule into location of peak flux in Z-trail cable facility.
7. Record initial temperature and pressure, plus the same data as for non-instrumented capsules.
8. Watch temperature and pressure recordings; stop irradiation if pressure exceeds 40 psi, or temperature exceeds 200°C.
9. Discontinue radiation after 5 hours (if not stopped sooner) by withdrawing capsule from irradiation zone and recording data as before.
10. Open valve to vent tank to reduce pressure to below atmospheric. Close valves and disconnect tubing and thermocouples from panel.
11. Transfer to reactor canal as before.
12. After samples have cooled in canal, withdraw as much tubing as possible, based on its radioactivity. Cut off tubing with a tubing cutter and wires with a wire cutter, and cap off cut end with a Swagelok cap.

D. POST-IRRADIATION PROCEDURES

The anticipated daily irradiation and post-irradiation analysis cycle is provided in Table 4.

1. Transfer of Irradiated Capsules From The Test Reactor To The Radiological Materials Laboratory (Hot Cell)

The procedures described below are to be followed in transferring capsules from the test reactor to the Radiological Materials Laboratory.

Remove the irradiated capsules from their storage positions in the reactor canal zone and place them into one of the GEVAL series 300 casks or equivalent. Lower the GEVAL cask from the reactor third floor level to the

first floor level. Carefully transfer the GEVAL cask to the RML building. Handling operations before, during, and after cask transfer should be such that impact or vibratory movements are not applied to the capsules or their containers. No other precautions are deemed necessary. Remove the three capsules from the transfer bucket in the hot cell. Check capsule identification and record.

2. RML (Hot Cell) Operation

The sequence of operations regarding the RML (Hot Cell) includes measuring gas release (non-instrumented capsules only), determining capsule void volume (non-instrumented capsules), and disassembling each capsule.

a. Gas Release Measurement (Puncture System, Figures 10 and 11)

- (1) Wipe off outer surface of capsule to remove any grit or other materials which interfere with sealing.
- (2) Wipe sealing and positioning surfaces of puncture block so that they are free from foreign materials.
- (3) Insert capsule in lower puncture block so that bottom of capsule is against rear stop and so that two bolt heads are symmetrical with centering wedge.
- (4) Position in place and clamp upper puncture block.
- (5) Close valves 3 and 4.
- (6) Open valves 1, 2, and 5.
- (7) Using N_2 bottle (out-of-cell) pressure system to 90 psi, record time and pressure after closing valve No. 5.
- (8) Return N_2 regulator setting to 0.

- (9) When no pressure drop has been detected for 3 min, open valve 4. If pressure drops, check for leaks with soap solution. Tighten packing on drill stem slowly if necessary. Do not over-tighten.
- (10) Close valves 2 and 4.
- (11) Open valves 3 and 5.
- (12) Pressurize system with N_2 to approximately 1400 mm pressure on gage G_2 .
- (13) Close valve 5; record time and pressure reading of gage G_2 .
- (14) Return N_2 regulator setting to 0.
- (15) When no pressure drop has been detected for 3 min, open valve 4. This completes leak testing.
- (16) Be sure valves 1, 2, 3, and 4 are open. Be sure drill is contacting capsule. Close valves 1, 2, 3, and 4. Take depth reading where drill contacts capsule.
- (17) Drill capsule to pre-set drill stop and return drill to starting position.
- (18) Slowly open valve No. 1 and watch pressure indication on gage G_1 . If pressure exceeds 33 psi, shut valve No. 1 and proceed as described in step No. 19. If pressure is less than 33 psi when equilibrium is reached, proceed as described in step No. 21.
- (19) If gage G_1 indicated greater than 33 psi when shut off, open valve No. 2.

- (20) Slowly open valve No. 1 and watch the indicated pressure on gage G_1 . If indicated pressure is greater than 30 psi, open valve No. 1 fully and record pressure indicated on gage G_1 . Proceed to step 22. If indicated pressure is less than 30 psi, proceed with step 21 below.
- (21) Open valve No. 3; after 1 min, record pressure, the valves open, and capsule number.
- (22) Open valves No. 4, 2, and 3 in the order stated.

b. Void Volume Determination

- (1) Close valves No. 2 and 4.
- (2) Pressurize system with N_2 until a pressure of approximately 1400 mm is indicated on gage G_2 . Close valve No. 3 and record pressure.
- (3) Return N_2 regulator setting to 0.
- (4) Open valve No. 4.
- (5) Close valves 4 and 2.
- (6) Open valve No. 3; after 1 min, record indicated pressure on gage G_2 .
- (7) Open valves 2 and 4.
- (8) Unclamp capsule and remove same from puncture head.
- (9) Close valves No. 2 and 3 and blow out chips from puncture head with N_2 flow from out-of-cell.

c. Precautions

The following precautions should be taken:

- (1) Do not tilt the capsule downward after drilling.

- (2) Be sure N_2 regulator setting is returned to 0 after each use so that gage G_2 is not over-pressured.
- (3) Wherever possible, read gage G_2 which is the most accurate.

d. Capsule Disassembly

Each irradiated capsule will be transferred to the remote controlled lathe station within the RML (hot cell) and opened by machining the upper expansion plenum from the main capsule body. Also, as back-up, the remote-controlled tools (electric drill bolt remover, etc.) will be supplied in the event the lathe operation malfunctions. The principal disadvantage of the latter procedure is the extra time required, thereby increasing the time between irradiation and post-irradiation analysis.

The machining of the instrumented capsule will require special attention to prevent accidental spillage of the explosive sample since the thermocouple wires are embedded in the center of the quartz container; consequently, the operator must carefully remove the machined-off top and cut the thermocouple wire before completely removing the capsule top. The remaining disassembly procedures are as follows:

- (1) Place a special lucite vial into the open top of the capsule and over the quartz container. Enclose the vial and capsule with the vial holding jig. Secure the jig to the capsule.
- (2) Remove the assembly from the lathe and tilt the top end of the capsule about 30° below the horizontal. "Pour" the quartz container into the plastic vial. Use care -- breakage of a quartz container would cause loss of data and considerable delay during subsequent operations.

- (3) Place the jig assembly on its base and remove the empty irradiation capsule. Plug the plastic vial with a cork or rubber stopper. Remove the vial and place it in a transfer cylinder. Secure the lid of the cylinder and move it from the immediate work area. Record the identity of the quartz vial and transfer cylinder.
- (4) Repeat the operations on the remaining irradiation capsules.

3. Transfer of Samples From RML Hot Cell to Explosive Test Facility

- a. Place the transfer cylinders on the floor of the interlock.
- b. Remove one unit at a time from the lock.
- c. Debag each cylinder of plastic and transfer by hand to a decontamination room hood.
- d. Wipe down each cylinder with a wet swab and remove the cylinder's top.
- e. Wipe down the lucite vial, remove the cork, and deposit the explosive contents on clean swabs within the hood area.
- f. The explosive samples are now ready for testing and analysis by members of the Aerojet-Picatinny team in the testing facility illustrated in Figure 12.

4. Explosives Test Facility

The sequence of operations to be performed within this facility will include the following:

- a. Weight loss measurement
- b. Sample removal

- c. Recovery of flux and temperature monitors
- d. DTA analysis
- e. Vacuum stability analysis
- f. Melting point determination
- g. Explosion temperature analysis
- h. Plate dent and detonation velocity determination
- i. TGA analysis
- j. Strant burner analysis

Table 5 summarizes the number and type of tests to be performed on each sample while Table 6 delineates the post-irradiation analysis schedule. It should be noted that reactor scrams or minor malfunctions may necessitate delays. These delays, however, will be evaluated and mutually agreed to by the Aerojet-Picatinny-General Electric team.

Detailed descriptions and appropriate discussion have previously been provided on operations (a) through (h) on pages 23 through 34 of Reference 2, and on pages 34 through 47 of Reference 5. Consequently, the procedural discussion here will be limited to the newly incorporated tests, TGA, and strand burner.

a. Strand Burner

The strand burner assembly illustrated in Figure 13 has been incorporated in Phase I-C to measure the effect of radiation on the burning rate of the propellants selected. In previous phases, it was necessary to allow the propellant strands to radioactively decay until they could be shipped to Picatinny Arsenal for analysis; under present conditions they can be analyzed at the reactor site within a period of 5 to 6 days as compared with 4 to 6 months in the past.

As noted in Figure 13, the strand burner assembly consists of an ARC Model SRB-212H strand burner test cell (item 1) which is

connected to a Heise pressure gage (item 2), control box (item 5), and a miniature forged needle valve (item 4). Pressure of 1000 psi is supplied by a cylinder of compressed nitrogen (item 7) and ignition by the control box (item 8) in accordance with the electrical schematic of Figure 14.

The strand burner test cell will be located within a detonation velocity chamber to eliminate contamination and safety hazards.

b. Strand Burner Operating Procedure

The basic operation of the strand burner will employ the following procedures:

- (1) Prepare sample by wiring the ignition hole with a piece of 22 gage Nichrome wire, and the "start" and "stop" holes with 22 gage lead fuse wire. Record distance between "start" and "stop" holes.
- (2) Coat the end of the strand around the ignition wire with a paste of T-28 propellant dissolved in acetone (AGC and AGQ only).
- (3) After T-28 has dried, grease the strand with Dow Corning silicone stopcock grease. Inspect strands to be sure all surfaces are coated with grease.
- (4) Wire the prepared sample into bomb head. (Wires should be wrapped around terminals at least four times.)
- (5) Place bomb head into bomb and screw clockwise until hand-tight; then back off 1/8 turn.
- (6) Plug electrical connector into bomb head.
- (7) Close inner confinement box door, and close hood doors. Proceed to control panel and check electrical continuity with lights on control unit; rewire if necessary.

- (8) Open nitrogen tank valve to control panel and raise pressure to 1000 psi using needle valve on control panel. Close valves.
- (9) Ignite sample by activating firing switch on timer unit. Record temperature.
- (10) Record burning time (from automatic clock).
- (11) Upon completion of burning, open exhaust valve to discharge gases.
- (12) To purge the bomb, open storage tank valve, and let gas flow through bomb to vent. Close valves.
- (13) Open hood and box door, disconnect electrical connector, unscrew bomb head, and thoroughly clean.
- (14) In the event sample does not ignite, disconnect electrical connection, remove sample, and retain for examination.
- (15) Grease bomb seal and threads with silicon grease after each cleaning.

c. Thermogravimetric Analysis (TGA)

The TGA apparatus to be used is an attachment for the DuPont 900 DTA apparatus. Basically, it involves heating a small sample (0.01 g) slowly while suspended on the beam of a recording balance, as illustrated in Figure 15. The loss of weight vs temperature data will be used to determine the temperature stability of the explosives and propellants, and is particularly useful for materials where volatile liquid products are evolved. Isothermal decomposition rates will also be evaluated and decomposition rate curves obtained which are useful in determining the type of decomposition occurring. In many explosives, the decomposition reaction is first order, and a plot of log wt loss vs time results in a straight line. When several of these are obtained at different temperatures, it may be possible to determine the activation energy and Arrhenius constant.

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A thorough technical description and account of the method of operation of the TGA is provided in Appendix A.

V. HAZARDS AND SAFETY REVIEW

Radiological safety monitoring shall be performed in accordance with standard General Electric procedures. General Electric shall have the responsibility for providing film badges, dosimeters, radiation monitoring gages, etc., to all non-General Electric personnel connected with this program at Vallecitos. In addition, General Electric will provide complete radioactive safety monitoring of all testing procedures (including ventilation) and assigned personnel. All non-General Electric personnel shall follow General Electric instructions regarding safety.

The hazards associated with the handling and testing of the explosives and propellants have previously been discussed in the data packages for the Phase I, I-B and I-D tests (References 1, 2, and 5, respectively). Only a brief description of each condition is provided to recapitulate those previously reported.

General loading instructions for irradiation capsule assembly are to be found in Appendix B.

The ability of the capsule to contain the detonation of an explosive depends upon proper assembly. Therefore, all critical assembly steps will be witnessed by a quality control inspector. A typical certification of acceptance is shown in Appendix C. As a double check, X-rays will be showing the internal assembly taken of all loaded capsules; these X-rays will be examined by Aerojet personnel and then forwarded to GE for review.

A. GAS RELEASE

Gas release measurement on non-instrumented capsules will be performed with the GE-supplied equipment wherein all of the pressure will be released prior to opening the capsule. Since the gas pressure measurement equipment will have a stop spacer on the drill bit, the possibility of drilling in to the explosive sample is eliminated. A revised packing gland supplied by Aerojet will be used which can be tightened as required to prevent leakage.

Details of this gland are shown in Figure 11. Previously, this seal was provided by O-rings which had to be replaced if they leaked. This was very time-consuming, since the head had to be decontaminated and removed from the hot cell when replacing O-rings. If the new seals leak, they can be replaced directly in the hot cell.

B. WEIGHT LOSS AND SAMPLE REMOVAL

Standard explosive handling procedures shall be adhered to when weighing the quartz vials on the analytical balance.

The most electrostatic and friction-sensitive materials to be tested are AGY and AGK. These formulations will be loaded into aluminum tubes (Figure 16) to prevent static electricity buildups. Particular care should be exercised when handling this material to assure that grounding straps are used and that loading and unloading operations are performed behind plastic shields. Removal of the quartz wool plug and sample from the quartz or aluminum tube should be done behind a plastic shield using long handled tools. While removing the sample, keep the tube in a vise and avoid pointing the end toward exposed areas. Then place the sample in a plastic or aluminum container for storage.

C. DIFFERENTIAL THERMO ANALYSIS

Differential thermo analysis involves heating a 10 mg sample until it burns or explodes; therefore, small explosion occurs at the end of each test. During the test the sample is inside a capillary tube which is located in an aluminum block heater; the heater will adequately contain any explosive fragments in all but the vertical direction. The heater block will be placed inside a heavy glass bell jar designed to contain any and all detonations of this size (Du Pont Series 900). Loading of the capillary tubes will be performed inside the hood, thus eliminating possible hazards.

D. VACUUM STABILITY

Vacuum stability operations involve use of a 200 mg sample which may explode during the test; therefore, it is located in a hood, and has

adequate shielding to contain the fragments. For quantities of 200 mg, a plastic shield is adequate. During previous tests a number of samples burned harmlessly in this apparatus, so no future hazards are expected.

E. DIMENSIONAL CHANGE

Dimensional change testing involves taking the diameter of each strand of propellant with a micrometer, plus weighing each strand. The dimensions of the strands should be taken with the pellet behind a plastic shield; standard operating procedures will govern the weighing of the pellets.

F. PLATE DENT AND DETONATION VELOCITY

This test will be done only on MDF during this phase. Although the explosive hazard is slight, the radioactivity of the lead sheath will require careful handling and cleanup to keep exposures and contamination to a minimum.

The MDF will be allowed to cool 30 days before testing. Two sizes of MDF will be tested (the largest is 1/8 in. in diameter, and contains 15 grains/foot, of explosive). The neutron activation analysis in Figure 17 shows the radiation expected from this sample vs time elapsed after a 5-hour irradiation.

The same procedure will be used for MDF detonation velocity testing as in Phase I-D. (A detailed description of the test can be found in Reference 5.) The high level of radioactivity of the lead sheaths will require that the detonation velocity apparatus be handled and cleaned carefully to avoid contamination. Data on similar samples tested in Phase I-B are shown in Table 7.

G. THERMO GRAVIMETRIC ANALYSIS

The Du Pont 950 TGA will heat a 10 mg sample inside a quartz tube in an oven. Although this sample may burn, gas will be vented to the hood and will escape harmlessly. In addition, the sample will be confined inside a tube furnace.

A detailed description of the apparatus and procedure is given in Appendix A. The measuring head and furnace will be located inside the hood to prevent spread of contamination during loading or unloading, or in case of a leak in the system.

H. STRAND BURNER

The strand burner test will be made in a standard bomb with a 1.3 g sample at 1000 psi. The bomb was designed for use with samples up to 100 g at pressures up to 5000 psi. It has been tested at pressures up to 7500 psi (Appendix D); therefore no safety hazard is involved in the pressure to be used. Radioactive gasses and solids will be vented and purged to the exhaust system; the apparatus is adequately enclosed in the hood to control contamination during loading. Even though the propellants being tested are not electrostatically sensitive, grounding on an aluminum foil will be used to prevent any possibility of ignition.

I. INSTRUMENTED CAPSULE

The radiation capsule cap was modified to permit measurement of internal capsule pressure and temperature as shown in Figure 5. Modifications included:

1. A 1/8-in.-dia aluminum tube with two glass-fiber insulated thermocouple wires inside it was attached to the capsule with a high-pressure tubing fitting, as shown in Figure 18.

2. A pressure measuring and recording system was attached to the exit end of the tubing. In this apparatus, a rupture disk and evacuated tank are provided to harmlessly reduce the pressure in the system if sudden burning or detonation occurs. Excess pressure (over 50 psi) from slow decomposition will be vented off manually using the vent valve.

3. Although the external system is expected to withstand pressure from a detonation, a steel seal ring has been inserted over the blast

cup. This arrangement will provide additional safety by preventing a high-pressure gas pulse from leaving the capsule in case of a detonation.

Eight proof tests (detonating twice the expected amount of explosive) were made to demonstrate the safety of the modified system. Figure 19 and 20 show post-firing views. Six successful tests fired by AGC, plus two additional tests, were witnessed by GE safety representatives. In addition, a static pressure test on the assembly was made which showed that the weak link in the system is the tubing, which burst at 8000 psig.

A summary of the eight tests is given in Table 8.

In the first test, the effectiveness of the ring sealing system was checked with no attempt to prevent eventual gas release. Gas release volume was measured in the assembly shown in Figure 21. Putty was put in the hole in the top of the capsule where the tubing will be connected. The sealing system was judged adequate since this material was not dislodged by the gas flow. The total gas volume of 2125 cu cm agrees with that expected from 3.6 g of explosive.

The second test had a pipe fitting closure attached, and only 25 cu cm of gas escaped during the first 5 min. The remainder of the 200 cu cm was released gradually over a 16-hour period. The seal ring was hardened to provide a better sealing action. The third test was the same setup as the second, and in both cases the leak rate was less than 10 cu cm/min.

Test No. 4 was the first to have tubing connected directly to the capsule. A Swagelok fitting was used; although it leaked, the rate was acceptably slow. The maximum pressure of 183 psi provided experimental data from which a maximum pressure of 92 psi for a 1.8 g sample can be predicted.

Tests No. 5 and 6 were witnessed by GE personnel. In Test No. 5, the Swagelok fitting leaked too fast because the nut was loosened by the shock-wave. In Test No. 6, the fitting nut was clamped, and leakage was acceptable.

Tests No. 7 and 8 were conducted using a stronger type of tubing fitting made by Imperial. A static pressure test made with this fitting on the capsule, held until the tubing burst at 8000 psi. A clamp was also used to hold the fitting nuts securely in these tests. Test No. 7 had a high enough

leak rate around the seal ring to cause the rupture disk to blow in 4 min. In Test No. 8 the seal ring reduced the leakage to a rate where the pressure never built up enough to blow the rupture disk.

In conclusion, all tests successfully limited gas release to a rate which would not damage the reactor Z-trail cable. By using a hardened steel seal ring in a narrow groove as shown in Figures 18 and 22, a clamped high pressure tubing fitting as shown in Figures 18 and 23, the gas release rate can be kept below 10 cu cm/min. The small gas leakage from the tubing fitting is caused by the deformation of the tubing when the explosive shock wave passes through it, and would not be produced except by a high order detonation. In reactor tests, the pressure could be manually released within a minute, thereby stopping release of radioactive gases. Capsule bulging was less than 0.014 in. in all cases, further proving the basic capsule's ability to contain a detonation.

The neutron activation analysis shown in Table 9 was made to determine the radioactivity of the gases generated. Most of this activity would be trapped in the cooling water as the gas bubbled through it. In addition, in the remote chance that gas starts to be released, it can be stopped immediately by simply opening the valve to the evacuated vent tank.

Since these tests were made with double the amount of explosive to be used in the reactor, and were detonated rather than deflagrated, the capsule should provide no hazard to the reactor.

If gas is released from a capsule during radiation, it may release radioactivity into the reactor area. As soon as any indication of this occurs, such as a decrease in capsule pressure, or a rise in the background activity at the mouth of the Z-trail cable, the capsule pressure will be reduced to atmospheric pressure by opening valve D to the evacuated vent tank. Most of the activity of the gas will be absorbed in the cooling water as it bubbles through it. However, the maximum activity of the gas would correspond to the gas produced if AGC propellant burned at the end of irradiation; this is shown per gram of gas in Table 9. A gram of gas occupies 726 cu cm; therefore the

maximum rate of activity release per minute would be 0.014 times the amount shown for 10 cu cm/min leakage.

The neutron activation analyses shown in Figures 17, 24, and 25 were made on the two most active materials. Reported data show that a delay of at least a day is desirable before analysis of AGC, AGE, AGS, and AGZ, and 20 days for MDF samples AGI, AGT, AGU, and AGM. The schedule in Tables 1 and 5 provides for this.

The rate of radioactive decay of other materials is expected to be faster than the rates calculated for AGE and AGM. Therefore these data can be used as "worst" cases in determining the hazards involved. It should be noted that the activity reported is at a distance of 3 ft, and is therefore approximately the whole body dose rate of operators analyzing samples. Finger rings and film badges will be worn during analysis, and each sample will be monitored to determine handling limitations prior to analysis.

REFERENCES

1. Addendum to Report No. 2510, Final Test Specifications for Phase I of the Countermeasures Radiation Effects Program, Contract SNP-1, December 1963 (Unclassified).
2. Addendum No. 2 to Report No. 2510, Final Test Specification for Phase I-B of the Countermeasures Radiation Effects Program (U), Contract SNP-1, September 1964 (Confidential).
3. Final Report for Phase I-B of the Countermeasures Radiation Effects Program, Contract SNP-1, April 1965 (CRD).
4. Reactivity Effects on the Proposed SNPO-C Countermeasure Explosive Projectile Destruct System, WANL-TME-1147, Appendix B, 15 May 1965.
5. Addendum No. 3 to Report No. 2510, Final Test Specifications for Transient Nuclear Test (KIWI-TNT) of the Countermeasures Radiation Effects Program, Contract SNP-1, December 1964 (Unclassified).

TABLE 1

PHASE I-C IRRADIATION SCHEDULE

<u>Date</u>	<u>Cycle No.</u>	<u>Material and Irradiation Time (hrs)</u>
August 2, 1965	67	V-10(4), I-7(2), E-7(2), Z-7(2)
August 3, 1965	67	S-10(5), T-7(4)
August 4, 1965	67	B-10(4), V-8(3), U-7(4)
August 5, 1965	67	W-10(5), M-7(4), C-7(2)
August 8, 1965	67	V-7(2), W-8(4), E-8(3), G-7(2), Q-7(2)
August 9, 1965	67	A-10(5), Z-8(3), C-8(3)
August 10, 1965	67	Y-10(4), G-8(3), Q-8(3)
August 11, 1965	67	A-8(4), H-10(5), W-7(3)
August 12, 1965	67	S-8(4), A-7(3), H-8(4)
September 12, 1965	68	Y-8(3), S-7(3), K-10(5)
September 13, 1965	68	N-10(5), T-8(5), \$-9(?)
September 14, 1965	68	Y-7(2), B-7(3), K-8(4), \$-10(?)
September 15, 1965	68	I-8(3), H-7(3), \$-10(?), \$-9(?)
September 16, 1965	68	N-7(4), K-7(3), M-8(5)
September 17, 1965	68	U-8(5)

Note: Each material will be preceded by the letters AG.

\$, ? - represent two best propellants to be selected during Cycle 67.

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TABLE 2

THERMAL GRAVIMETRIC ANALYSIS DATA SHEET

Capsule No.	Date	Time	Initial Sample Wt, g	Heating Rate °C/min	Temperature Wt Loss Starts, °C	Inflection Points		Remarks
						°C	g	

Table 2

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TABLE 3

STRAND BURNER DATA SHEET

Capsule No.	Date	Time	Gage Length, in.	Pressure, psig		Temp, °F	Burning Time sec	Burning Rate, in./sec	Log Mean Average Pressure, psig	Remarks
				Start	End					

Table 3

TABLE 4

PHASE I-C DAILY OPERATIONAL CYCLE

<u>Approximate Time</u>	<u>Operations</u>
4:00 pm (First Day) - 6: am (Second Day)	Irradiate up to four capsule
7:00 - 8:00 am (Second Day)	Transfer capsules to RML
8:00 - 10:00 am (Second Day)	Measure gas release (if required) and open capsules
10:00 am (Second Day)	Transfer capsules to post- irradiation examination facility
10:00 am - 5:00 pm (Second Day)	Analyze irradiated samples in RML laboratory
4:00 pm (Second Day)	Start irradiation of capsules

TABLE 5

PHASE I-C TEST SCHEDULE

<u>No. of Capsules</u>	<u>Mock</u>	<u>AGA</u>	<u>AGB</u>	<u>AGC</u>	<u>AGE</u>	<u>AGG</u>	<u>AGH</u>	<u>AGI</u>	<u>AGK</u>	<u>AGM</u>	<u>AGN</u>	<u>AGQ</u>	<u>AGS</u>	<u>AGT</u>	<u>AGU</u>	<u>AGV</u>	<u>AGW</u>	<u>AGY</u>	<u>AGZ</u>
Non-Instrumented	0	2	1	2	3	2	2	2	2	2	1	2	2	2	2	2	2	2	3
Instrumented	1	1	1	0	1	0	1	0	1	0	1	0	1	0	0	1	1	1	1
For Post-Irradia- tion Tests																			
Gas Release		2	1	2	3	2	2		2		1	2	2			2	2	2	3
Weight Loss		2	1	2	3	2	2		2		1	2	2			2	2	2	3
Vac. Stab.		3	2	2	4	2	3		3		2	2	3			3	3	3	4
DTA		3	2	2	4	2	3		3		2	2	3			3	3	3	4
TGA		3	2	2	4	2	3		3		2	2	3			3	3	3	4
Explo. Temp.		3	2	2	4	2	3		3		2	2	3			3	3	3	4
M.P.									3		1					3		3	
Plate Dent								2		2				2	2				
Detonation																			
Velocity								2		2				2	2				
Strand Burner				2	3	2						2							3

Table 5

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TABLE 6

PHASE I-C POST-IRRADIATION ANALYSIS SCHEDULE

<u>Date</u>	<u>Samples</u> *			
3 Aug	V-10	C-0	I-0	(Strand burner check on IB 7170)
4 Aug	Z-0	U-0	T-0	B-0
5 Aug	B-10	V-8	K-0	G-0
6 Aug	W-10	Q-0	M-0	S-10
9 Aug	V-7	E-7	Z-7	W-8
10 Aug	G-7	Q-7	C-7	A-10
11 Aug	Y-10	Z-8	G-8	Q-8
12 Aug	H-10	C-8	A-8	W-7
13 Aug	S-8	A-7	H-8	E-8
13 Sep	Y-8	I-7	T-7	K-10
14 Sep	N-10	M-7	U-7	S-7
15 Sep	Y-7	B-7	K-8	-
16 Sep	H-7	\$-10	N-0	-
17 Sep	N-7	K-7	\$-10	-
18 Oct	I-8	T-8	U-8	M-8
17 Dec	\$-9	\$-9	(Strand burner tests only)	

* All sample numbers have AG as a prefix

\$ Stands for one of the propellants to be chosen for irradiations in Cycle 68.

TABLE 7

INDUCED ACTIVITY IN MDF SAMPLES

<u>Sample No.</u>	<u>Irradiation Time, hours</u>	<u>Time from End of Irradiation, days</u>	<u>Activity at Surface (β/γ), mr/hour</u>	
AGT-1	2	9	-	2500
AGT-2	3	9	-	2400
AGM-1	0.5	24	2000	600

TABLE 8

INSTRUMENTED CAPSULE PROOF TESTS

No.	Date	Diameter of Capsule (in.)			Gas Released		Remarks
		Before	After	Increase	Total Vol, cu cm	Release Rate, cu cm/min	
1	19 Apr 65	2.130	2.130	0	2125	700	Gas released slowly; putty around vent hole still in place after test; tubing not connected.
		2.129	2.135	0.006			
		2.128	2.130	0.002			
2	28 Apr 65	2.123	2.124	0.001	200	2	Gas released slowly; maximum pressure 10 psi; pipe fitting leaked.
		2.124	2.129	0.005			
		2.124	2.129	0.005			
3	4 May 65	2.122	2.123	0.001	2000	1	Gas released slowly; maximum pressure 10 psi; pipe fitting leaked after 3 min
		2.122	2.136	0.014			
		2.122	2.125	0.003			
4	20 May 65	2.124	2.124	0	2000	10	Gas released slowly; maximum pressure 183 psi; rupture disk not used.
		2.123	2.134	0.011			
		2.122	2.125	0.003			
5	21 May 65	2.124	2.124	0	1165	130	Gas released slowly; fitting leaked, no pressure measured
		2.124	2.133	0.009			
		2.124	2.125	0.001			
6	21 May 65	2.124	2.124	0	25	6	Gas released slowly; maximum pressure 90 psi when rupture disk blew; fitting clamped and did not leak
		2.124	2.135	0.011			
		2.123	2.124	0.001			
7	2 Jun 65	2.124	2.124	0	15	6	Rupture disk blew at 90 psi in 4 min.
		2.124	2.134	0.010			
		2.124	2.127	0.003			
8	25 Jun 65	2.123	2.123	0	1140	8	Slow leakage; peak pressure 25 psig
		2.123	2.137	0.014			
		2.123	2.125	0.002			

TABLE 9MAXIMUM RADIOACTIVITY OF SMOKE AND GAS
FROM AGE PROPELLANT

<u>Maximum Activity,*</u> <u>R/hr at 3 ft (per gram of propellant)</u>	<u>Irradiation Time (hr)</u>
5.518×10^{-1}	1
7.359×10^{-1}	2
7.991×10^{-1}	3
8.227×10^{-1}	4
8.330×10^{-1}	5
8.389×10^{-1}	6

* Activity at the end of the irradiation time.

RN-S-0235

Figure 2

Internal Components of Non-Instrumented Capsule

Figure 3

Neutron Flux Indicator Assembly

4

3

2

1

		REVISIONS		
SYM	ZONE	DESCRIPTION	DATE	APPROVED
		SEE RE. DNG DCN DATE 12-13-63	12/13/63	E. L. [initials]
A		SEE 'A' CHG DCN	10/13/64	E. L. [initials]
B		SEE 'B' CHG DCN	11/5/65	E. L. [initials]

NOTES:

1. DISTILLED WATER LEVEL SUFFICIENT TO COVER -5 OR -7 WIRE.

2. MATL: QUARTZ TUBE, 2 MM O.D. X 1 MM I.D.

3. APPROVED SOURCE: GENERAL ELECTRIC CORP WILLOUGHBY, OHIO. ALL SOURCES MUST COMPLY WITH PHYSICAL AND FUNCTIONAL REQUIREMENTS OF MANUFACTURERS ITEM INDICATED.

4. CERTIFICATION OF COMMERCIAL PRODUCTS SHALL INCLUDE MATERIALS, STRENGTH AND DIMENSIONS.

5. MARK CONTAINER IN ACCORDANCE WITH ASD 5215N.

6. CLASSIFICATION OF CHARACTERISTICS PER MIL-W-9411 DENOTED BY [C] CRITICAL, [M] MAJOR AND NO SYMBOL = MINOR.

[C] 7. TUBE ENDS TO BE 100% SEALED.

DASH NO.	A
-5	38
-7	.50

-3	1	1	516863-1
-1	1	1	516863-1
-3	1	1	515267-13
-1	1	1	515267-13

AR	AR		COML PROD	DISTILLED WATER					
AR	AR		COML PROD	QUARTZ TUBE	2/3				
1			-7	TEMPERATURE SENSITIVE WIRE .025 DIA, 255°F MELTING POINT	CERRO CORP NEWARK, N.J.				
	1		-5	TEMPERATURE SENSITIVE WIRE .025 DIA, 158°F MELTING POINT	CERRO CORP NEWARK, N.J.				
-3	-1	SYM	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL	SPECIFICATION	UNIT WT.	ZONE ITEM NO.

CONTINUED IN ZONE 1/B

			-3	1	1	513646-19		UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES ON DECIMALS ANGULAR	DRAWN J. CONDON	DATE 12-13-63		AEROJET-GENERAL CORPORATION DOWNEY, CALIFORNIA	
			-1	1	1	513646-19		XX ± .03	CHECK [initials]	12/13/63			
			-3	1	1	515267-5		.XXX ± .010 ± —	DESIGN [initials]	12/13/63			
			-1	1	1	515267-5		DO NOT SCALE DRAWING	STRESS [initials]	12/13/63			
			-3	1	1	515267-3		TREATMENT	WEIGHTS [initials]	12/13/63	TEMPERATURE INDICATOR ASSEMBLY		
			-1	1	1	515267-3		FINISH	PROD. [initials]	12/13/63			
			-3	1	1	515267-1			DESIGN ACTIVITY APPD [initials]	12-13-63			
			-1	1	1	515267-1			CUSTOMER [initials]	12-16-63			
ON	THRU	PART DASH NO.	NEXT DASH NO.	FINAL QTY REQD PER ASSY	NEXT ASSY	USED ON	APPLICATION	SIMILAR TO	ACT WT	CALC WT	CODE IDENT NO. 13309	DWG SIZE C	DWG NO. 515266
EFFECTIVE SERIAL NO.		USAGE DATA		DRAWING LEVEL		1					SCALE 4/1	RELEASE DATE	SHEET

06-067-004 (REV 4-63)

4

3

2

515266

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Figure 4

Temperature Indicator Assembly

Figure 5

Instrumented Capsule Assembly

Figure 6

Pressure Measurement Panel

Quantity	Description
1	1 Pressure Gauge, Heise 0-100 psi, 8-1/2" dial, .2 psi per division, 1/5 subdivided, with remote 5K potentiometer, 316 s.s. tube.
2	1 Vacuum Gauge, Marshalltown No. 22B, 2' dial size.
3	1 Vacuum Tank, 2000 cu. in., 12" x 24", s.s.
4	5 Needle Valve, Whitey No. OKS 2-316, 1/8" size, s.s. w/swagelok fittings.
5	1 Safety Head, screw type, Black, Sivalls & Bryson Assy No. SA-9; s.s. - 100 psia bursting pressure
6	1 Filter, Nupro "F" Series Inlet Filler, Cat. No. SS-2F-7, 5-10 microns, s.s.

Figure 7

Pressure Measurement Panel Diagram

NOTES:

- 1) MAKE FROM .060-.125" 6061-T6 AL SHEET, HELIARC JOINTS
- 2) USE NEOPRENE GASKETS AT GLOVE PORTS + PLEXIGLASS WINDOW
- 3) EXACT POSITION OF C-CLAMP TO BE DETERMINED DURING FABRICATION.

Figure 8

Glove Box Assembly

Figure 9

Temperature and Pressure Measurement Schematic

No.	Req'd	Description
1	1 ea	Valve, 1/4" size, stainless steel
2	1 ea	" " " " "
3	1 ea	Valve, 1/4" size, stainless steel
4	1 ea	" " " " "
5	1 ea	Valve, 1/4" size, stainless steel
6	1 ea	Check Valve
G ₁	1 ea	Pressure Gage; 0-100 psig, stainless steel
G ₂	1 ea	Pressure Gage; 0-15 psig, stainless steel, graduations at 2mm Hg intervals, 0.2% of full scale accuracy; Wallace & Tiernan FA 145
V _E	1 ea	Expansion Tank; stainless steel, Volume 159.5 cc
F	1 ea	Filter, Stainless Steel

Figure 10

Capsule Puncture System Schematic

Figure 11

Puncture Tool and Pressure Head Seal Assembly

Figure 12

RML Explosive Testing Facility

Item	Quantity	Description
1	1	Strand Burner Test Cell; Hastalloy "B", Atlantic Research Corporation Model SRB-212H
2	1	Pressure Gauge; Heise 0-3000 psi, 8-1/2" dia; 316 S.S. Tube; with peak load indicator.
3	1	Cylinder Control Needle Valve with 0-4000 psi Gauge and Quick Coupler, Matheson No. 52A-580 (Spec)
4	2	Miniature Forged Needle Valve; S.S.; 1/4" size; Matheson Model No. G12TP-4T
5	1	Control Box; with 6 v. transformer, Firing & Safety Switch, Fuze and Safety Light
6	1	Electric Timer
7	1	N ₂ Cylinder, High Pressure, 3500 psig; Mod. 580
8	AR	Tubing, 1/4" O.D. x .035" wall; Type 304 S.S. with Tylok Compression Type Fittings.

Figure 13

Strand Burner Assembly

Figure 14

Strand Burner Control Schematic

Figure 15

Figure 17

Dose Rate vs Time After Shutdown as a Function of Operation Time (T_o)
from Neutron Activation of 1 Gram of AGM Mild Detonating Fuze

[illegible]

Figure 16

Tube Assembly Fixed Plug

Figure 18

Instrumented Capsule Cross Section

Figure 19

Capsule Closure After Detonation

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Figure 20

Cross Section of Instrumented Capsule Seal After Detonation

Figure 21

Capsule Qualification Apparatus

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Figure 22

Instrumented Capsule Components

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Figure 23

Instrumented Capsule Assembly

Figure 24

Dose Rate vs Time After Shutdown as a Function of Operating Time (T_o)
 from Neutron Activation of 1 Gram of AGE Propellant

Figure 25

Dose Rate Versus Time for AGE Propellant

APPENDIX A

THERMOGRAVIMETRIC ANALYSIS PROCEDURES

I. INTRODUCTION

The Du Pont 900 series of instruments for thermal analysis has been designed as modular attachments to a basic instrument console which is the 900 Differential Thermal Analyzer. Thus the basic instrument functions of the 900 dia are utilized, with the attachments, to provide a capability of measuring a variety of thermal properties of materials. This manual describes the construction and operating techniques of one such attachment - the Du Pont 950 Thermogravimetric Analyzer (TGA).

The 950 TGA is a semi-micro balance designed to measure the weight of a material as a function of a linearly increasing temperature. The instrument is capable of attaining temperatures up to 1200°C with any linear heating rate between 0.5 and 30°C/min. It can also be utilized to study the weight change of a material as a function of time at some specified temperature and atmosphere. In this isothermal mode of operation, the time axis scale can be varied from 0.1 to 20 min/in.

Several critical design criteria have been incorporated into this attachment to ensure flexibility of application as well as superior performance. These criteria include the following features:

- A. A small sample size to minimize heat transfer problems
- B. An accurate sample temperature-sensing device to allow a high programmed heating rate
- C. Accurate and precise weighing

- D. Rapid rebalancing of weighing system together with a relatively temperature-insensitive weighing mechanism

The 950 TGA operates on a null-balancing principle. The balance beam is maintained in the reference position by an optically actuated servo loop which employs a light source focused on twin photo-voltaic cells through a slit aperture signal flag located on the balance beam. The beam is balanced through a taut band electric meter movement. The beam is constructed in two pieces.

The hot section of the beam is a replaceable quartz rod. The furnace tube enclosing the hot end of the beam and the Pyrex envelope enclosing the cold section are sealed to the balance housing by O-rings, thus allowing the sample to be examined in a vacuum. Purge gas flow rates up to 2.5 liters/min have been used with no resultant base line instability due to gas turbulence. The operational characteristics of the TGA unit are shown in Table A-1.

II. DESCRIPTION OF THE ANALYZER

A. SYSTEM DESCRIPTION

The 950 TGA can conveniently be considered as consisting of three units: furnace assembly; balance assembly; and cabinet assembly containing the balance operating controls and associated electronic circuits.

The TGA system is shown schematically in Figure A-1. Components enclosed by dashed line area A are located in the 950 module. Components enclosed by dashed line area B are located in the 900 console.

The balance movement is a taut-band D'Arsonval type galvanometer movement. The balance arm is attached directly to this movement. When the balance arm is deflected from its reference position, the photocell amplifier adjusts the current in the movement coil until the balance arm returns to the null position. This current, after amplification, determines the position of the pen on the Y-axis of the X-Y plotter. The furnace is programmed by the temperature programmer-controller of the 900 Differential Thermal Analyzer. The temperature of material being weighed is determined with a thermocouple which is located close to the sample. The temperature is recorded on the X-axis of the X-Y plotter.

B. FURNACE

The external, plug-in, 500-watt Kanthal wire-wound resistance furnace has a low thermal mass. It can be heated and cooled rapidly, and has a temperature range from ambient to 1200°C. A Chromel-Alumel control thermocouple, located in the furnace wall, monitors furnace temperature. The furnace can be heated at a rate continuously variable from 0.5°C to 30°C per minute. For very rapid work, with one analysis immediately following another, two furnaces can be used alternately.

C. BALANCE ASSEMBLY

The balance assembly, mounted on horizontal support rods, has an envelope at each end. The large glass envelope encloses the control end of the balance. The other envelope, called the furnace tube, encloses the furnace end of the balance. Two furnace tubes are supplied: Pyrex, for operation to 500°C; and quartz, for operation to 1200°C.

In operation, the material to be analyzed is placed in a sample pan suspended from the end of the quartz rod. In most cases, the sample thermocouple is positioned inside the sample pan close to the sample.

When the sample and thermocouple are in place, the furnace tube is placed over the furnace end of the balance. The furnace tube is held in place by the furnace tube retainer and sealed with silicon (red) and Teflon (white) O-rings. The assembly is then moved along the horizontal support rods until the furnace tube is in the furnace. There is a stop on one of the support rods that is set to place the sample pan at the center of the furnace. This stop is properly positioned at the factory and is approximately 1 in. from the edge of the furnace. Purge gas is admitted through the hose connection at the rear of the balance housing. A vacuum source may be connected to the end of the furnace tube.

D. CONTROLS

Operational controls for the 950 are located on the 900 console as well as on the 950 cabinet. The controls on the DTA console will be

will be designated 900 controls, and those on the TGA as 950 controls.
(Figure A-2 shows the 950 control panel.)

1. 950 Controls

Temperature/Time Base:	Two-position toggle switch for either temperature or time operation on X-axis.
Fan:	"On-off" toggle switch. The fan helps protect the balance mechanism from damage due to an excessive heat flow.
Recorder Zero:	Shorts out electrical signal from the 950 and permits recorder to be zeroed.
Balance Zero:	Allows 10 mg of electrical tare to zero the balance.
100% Span:	Permits recording of sample weight directly in terms of wt% full scale.
Suppression:	Maximum suppression is 110.00 mg, stepped and continuously variable. Provides amplification of small weight changes by suppressing part of the total weight.
Time Constant:	0, 1, 2, 5, 10, 20 sec recorder response time. A time constant of 1 sec is recommended for most applications. Under adverse vibration conditions, it may be desirable to use longer time constants.

2. 900 Controls

Power Switch:	A three-position rotary switch. "Off" - all circuits de-energized; "stand by" - all circuits in both the 900 and the 950 energized except the recorder servo motors; "record" - all circuits energized.
T Zero Shift:	Nine-position rotary switch. Determines position of zero on X-axis.
T Scale:	Five-position rotary switch. Determines magnitude of temperature scale.

ΔT Zero Shift:	Continuously variable potentiometer. Determines vertical pen position on chart. (+) moves pen up on chart; (-) moves pen down on chart.
ΔT Scale:	Seven-position rotary switch. Determines magnitude of scale on Y-axis. Scale setting ($^{\circ}\text{C}/\text{inch}$) times 2 equals mg/in .
Reset Button:	Resets program to temperature selected as starting temperature.
Power Light:	Light indicates system energized by 110 volts.
Heater Voltage:	Indicates voltage to furnace.
Starting Temperature:	Determines starting temperature of program.
Program Mode:	A six-position rotary switch. Only three are used with the 950; "heater off" - heater is de-energized; "heat" - heater energized (temperature of furnace increases linearly at selected rate); "isothermal" - the furnace is maintained at constant temperature.
Temperature Rate:	Determines rate at which the 900 programmer-controller programs furnace temperature.

E. THERMOCOUPLE CONNECTOR STRIP

The thermocouple connector strip, located at the rear of the 950 module, is illustrated in Figure A-3. The Chromel-Alumel cold junction thermocouples are installed in positions 2 and 3, and 4 and 5, as shown (red leads go to positions 2 and 4). The couple junctions are then inserted into an ice-water bath.

F. SAMPLE PANS

Sample pans are suspended directly from the end of the quartz rod. Both aluminum and platinum pans are provided. Since aluminum melts near 600°C , platinum pans are recommended for use above 500°C .

Tweezers are provided for handling the pans. A hot flame can be used for cleaning platinum pans. Aluminum pans, however, should not be heated above 500°C and may require replacement if cleaning is unsuccessful. Additional pans may be ordered from Du Pont or fabricated by the user.

G. COUNTERBALANCE AND CALIBRATION WEIGHTS

Riders are provided for counterbalancing the sample pans as indicated below.

1	5 mg
2	10 mg
2	20 mg
1	50 mg
1	100 mg
1	200 mg

A Class M weight (50 mg) is provided for beam calibration.

TABLE A-1

DU PONT 950 THERMOGRAVIMETRIC ANALYZER

OPERATIONAL CHARACTERISTICS

Capacity:	300 mg (including boat)
Weight ranges:	1, 2, 5, 10, 20, 50, 100 mg full scale (5 in.)
Suppression:	110.00 mg (stepped and continuously variable)
Suppression accuracy:	± 0.02 mg
Sensitivity:	0.2 mg/in. (maximum)
Accuracy:	$\pm 0.2\%$ of full scale
Noise band:	0.008 mg (zero time constant) < 0.002 mg (1 sec time constant); (unaffected below gas flow rates of 2.5 liters/min)
X-axis time base (for isothermal operation):	0.5% linearity variable, 1 to 200 min full scale
Time constant:	0, 1, 2, 5, 10, 20 sec

Figure A-1

System Description

Figure A-2

Figure A-3

Thermocouple Connector Strip

APPENDIX B

LOADING INSTRUCTIONS FOR IRRADIATION CAPSULE ASSEMBLY

I. GENERAL INSTRUCTIONS

- A. Record temperature and humidity in glove box at time the explosives, propellants, propellant strands, pellets, or MDG cords are loaded into their respective tube assemblies.
- B. Record barometric pressure and temperature (as obtained downstairs) at 8:00 am and 12:30 pm every loading day.
- C. Scales must be "checked for zero" each time a weighing is to take place. Make sure no heavy objects are placed (or removed) from the table or bench on which the scale is sitting once the scale has been "zeroed."
- D. Allow scales to come to complete rest before taking reading.
- E. Ultrasonic-clean all temperature and flux indicators in acetone; do not touch with bare hands after cleaning.
(Note: Clean one type at a time to avoid mixups.)
- F. Ultrasonic-clean the following parts: quartz tube assys, retainer disk, and blast cups.
- G. Keep powdered explosives and propellants clean and free from contamination. Replace container lids immediately after removal of material.
- H. All powdered explosives shall be dried to "constant weight" or handled in accordance with standard drying procedures before being loaded into the sample tubes.

- I. Use a rubber stopper to cap off tube assemblies when they are removed from the glove box for weighing.
- J. Make sure glove box is properly flushed with helium before loading or assembly starts.
- K. It is imperative that the proper flux and temperature indicators be used in each tube assembly. Maintain identities by opening and removing indicators from one container at a time. Indicators can be identified visually on the basis of the measurements given below.

<u>Drawing Number</u>	<u>Description</u>	<u>Length of Vial in.</u>	<u>Length of Wire in.</u>
515265-1	Flux Indicator (Al-Co)	0.75	0.44
515265-3	Flux Indicator (Ni-Co)	0.75	0.56
515266-1	Temperature Indicator (158°F)	1.12*	0.38
515266-3	Temperature Indicator (255°F)	1.12*	0.50

* Partially filled with water.

- L. Although nesting position of flux and temperature indicators in the tube assemblies is optional, every effort should be made to maintain the sequence and approximate position shown on the tube assembly drawing.

II. ASSEMBLY, LOADING, AND TESTING INSTRUCTIONS FOR INSTRUMENTED CAPSULE ASSEMBLY (P/N 518247-1, -3, AND -5)

A. ASSEMBLY OF T/C WIRE AND PRESSURE LEAD

- 1. Cut off 40 ft of 1/8-in.-OD aluminum tubing, slip tube support (P/N 518253) over one end and one tube connector nut, and place sleeve over each end. Flare tube ends using 37° Imperial flare tool.

- *2. Inspect flared ends (no cracks or thinned-out flanges allowed).
3. Push music wire (0.024 in. dia) through tube, make hook on emerging end, fasten free end of T/C wires, and pull T/C wire through tubing. Cut T/C wires allowing approximately 1-1/2 ft extending out on each end of tubing.
4. Assemble valve (Hoke R 308 AK), filter (Hoke 634 1 F2B), 1/8 in. brass tee, reducing adapter (Cajon 6-RA-2), and male part of tube connector (Imperial 848 FB) in accordance with the blue print. Use Teflon tape in joints.
5. Check to see that valve is closed and the joints are tight.
6. Push T/C wire through tube connector, straight run of tee, and reducing adapter.
7. Remove outer insulation as required and then remove individual T/C wire insulation for approximately 5-1/2 in.
8. Thread insulating tubing ("spaghetti") over bare end of T/C wires and fill seal assembly with potting compound (EPD Corp No. TC-413).
- *9. Thread bare T/C wires through lead through tubes in seal assembly (P/N 518249) and solder T/C wires in place as indicated on the blueprint.
10. Hold seal assembly stationary and turn adapter-tee assembly onto seal assembly.
11. Assemble male part of steel tube connector (Imperial 848-FS) to cap (P/N 518213).
12. Thread tail of T/C wires through ring (P/N 513645), cap (P/N 518213) w/connector, seal (R4-25B), protection disk (P/N 518246), and seal ring (P/N 518210).

* Procedure completion to be certified by Quality Control.

13. Peel off outer insulation on T/C wire up to base of cup. Coat ends of individual T/C wires with Eastman 910 cement (approximately 1/2 in.).
14. Thread T/C wires through holes in cup (P/N 518155) and push individual T/C wires into groove in cup. Place small amount of water gloss on bottom of protection disk and press down on top of cup. Allow to dry.
15. Thread T/C wires through center hole in retainer disk (P/N 513643).
16. Secure assembly with masking tape and send assembly to Engineering Laboratory for welding of the T/C junction and calibration.

B. LOADING OF TUBE ASSEMBLY AND ASSEMBLY OF CAPSULE

1. Verify that tubes (P/N 515264-3) will fit freely in cavity of body (P/N 513585-7) in which they will be used.
- *2. Mark tube on side with identification symbol and S/N, using felt pen.
- *3. Mark body (513585-7) with identification symbol and serial number; letters on side of body must be at least 1-in. high and letters on bottom must be at least 1/4-in. high. Use vibratool and felt pen.
- *4. Weight out approximately 1.8 g of test material (explosive or propellant) on analytical balance. Record total weight explosive and pan to +0.0001 g.
- *5. Weigh and record the weight of all paraphernalia (such as funnel, glass disk, Kimwipes, etc.), including the pan containing the test sample to be used in loading the tube assembly.

* Procedure completion to be certified by Quality Control.

6. Load tube assembly approximately half full as shown on the blueprint, and insert T/C junction into this depth with the junction approximately in the center of the tube.
- *7. Continue to load tube assembly with test material and indicators and plug off with quartz wool as indicated on the blueprint.
- *8. Insert tube into capsule assembly and follow with the remaining capsule hardware strung on the T/C, making sure that each component is properly assembled.
9. Insert two cap-screws to secure cap.
- *10. Weigh all items used in loading the tube assembly. The difference between this weight and the original weight with the test material determines the exact amount of test material in the tube. Note: Net amount of test material in tube shall be 1.8 ± 0.05 g.
11. Place capsule body in vise; insert remaining bolts, clamping bracket (P/N 518252), and locking bracket (P/N 518250).
- *12. Tighten 12 bolts to 180 in.-lb; tighten across.
- *13. Tighten 12 bolts to 200 in.-lb; tighten around.
14. Attach clamps (P/N 518251 -1 and -2) with screws and nuts in accordance with the blueprint.
15. Check T/C wires for continuity and ascertain that they are not shorted to capsule assembly.

C. LEAK TESTING OF CAPSULE ASSEMBLY

1. Attach capsule assembly to pressure-measuring apparatus, open valve, and submerge capsule assembly in water.

* Procedure completion to be certified by Quality Control.

- *2. Slowly bleed helium into capsule to a maximum pressure of 100 psig. Check for leaks, and correct if necessary.
 - 3. Slowly release pressure down to atmospheric pressure; close and seal valve.
 - *D. CALIBRATE CAPSULE INTERNAL VOLUME AGAINST KNOWN VOLUME AND RECORD DATA
 - *E. X-RAY CAPSULE BODY TO CONFIRM THAT ALL PARTS ARE PROPERLY ASSEMBLED
 - *F. ATTACH 40 FT OF CABLE AND CLAMP IN ACCORDANCE WITH THE BLUEPRINT; THEN VERIFY THAT ASSEMBLY IS COMPLETE
 - G. ATTACH INSPECTION TAG
 - H. PACKAGE CAPSULE ASSEMBLY IN ACCORDANCE WITH APPLICABLE INTERSTATE COMMERCE COMMISSION (ICC) REGULATIONS
- III. LOADING, ASSEMBLY, AND TESTING INSTRUCTIONS FOR NON-INSTRUMENTED CAPSULE ASSEMBLY (P/N 513646-21, -23, AND -25)
- A. LOADING OF TUBE ASSEMBLIES (P/N 515267-3, -13, -15)
 - 1. Assemble component parts and material required for the applicable tube assemblies in accordance with Drawing No. 515267 as follows:

<u>For Capsule Assembly</u>	<u>Tube Assemblies Required</u>
513646-21	515267-3
513646-23	515267-13
513646-25	515267-15
 - 2. Verify that tubes (515264-3) will fit freely in cavity of body (513585-7) in which they will be used.
 - 3. Mark tube (515264-3) on side with identification symbol and S/N, using felt pen.

* Procedure completion to be certified by Quality Control.

4. Prepare test material (MDF cord, propellant strands, powdered explosive, and propellant) and component tube assembly hardware as follows:
 - a. Explosive-Powder-Loaded Capsule Assembly (P/N 513646-21)
 - * (1) Jointly weigh the component parts of tube assembly 515267-3 (excluding the explosive) to the nearest 0.0001 g, using an analytical balance.
 - (2) Weigh out approximately 1.8 g (or sufficient amount to obtain a total of 1.8 ± 0.05 g net weight of explosives in test tube after loading) of pre-dried explosives on an analytical balance. (When loading black powder sample, the 1.8 g maximum will consist of two pellets (1.2g total) and 0.6 g powder.)
 - (3) Place component parts and explosive inside glove box and flush thoroughly with helium.
 - (4) Load explosive, flux indicators, and temperature indicators into tube in accordance with Drawing 515267-3 and general loading instructions. Remove capped tube from glove box.
 - * (5) Weigh tube assembly (515267-3) on analytical balance to the nearest 0.0001 g. Place in glove box.
 - b. MDF Loaded Capsule Assembly (P/N 513646-23)
 - * (1) Space limitations determine the number of MDF sticks to be loaded in each tube assembly. MDF sticks shall be cut to different lengths (e.g., 5.00, 4.90, 4.80, etc.).

* Procedure completion to be certified by Quality Control as having met Quality Control specifications.

- * (2) Record the length of each piece to the nearest 0.010 in.; weigh each piece on analytical balance and record to the nearest 0.0001 g.
 - (3) Place component parts, MDF material, and aluminum powder in glove box and flush thoroughly with helium.
 - (4) Load MDF cord, aluminum powder, and indicators in tube in accordance with Drawing 515267-13. Remove capped tube from glove box.
 - * (5) Weigh assembled tube assembly (515267-13) on analytical balance to the nearest 0.0001 g. Place in glove box.
- c. Propellant-Loaded Capsule Assembly P/N 513646-25
- * (1) Jointly weigh the component parts of tube assembly 515267-15 (excluding the propellant strands and/or propellant powder) to the nearest 0.0001 g, using an analytical balance.
 - * (2) Cut one propellant strand so as not to exceed 1.3 g in weight or 5.00 in. in length. Using a vernier recorder, measure the length to the nearest 0.010 in. (This strand should have holes pre-drilled.)
 - * (3) Weigh propellant strand on analytical balance and record to the nearest 0.0001 g.
 - (4) Weigh out enough powdered propellant on analytical balance to provide a total weight of 1.8 $\pm$ 0.05 g when combined with the strand in paragraph c,3.
 - (5) Place component parts, propellant strands, and propellant powder in glove box, and flush thoroughly with helium.

* Procedure completion to be certified by Quality Control as having met Quality Control specifications.

- (6) Load propellant strand, propellant powder, and flux and temperature indicators in tube in accordance with Drawing 515267-15. Remove capped tube from glove box.
- (7) Weigh assembled tube assembly (515267-15) on analytical balance to the nearest 0.0001 g. Place in glove box.

B. ASSEMBLY AND TESTING OF CAPSULE ASSEMBLIES (P/N 513646-21, -23, -25)

1. Assembly component parts required for the applicable dash-number capsule assembly in accordance with Drawing 513646.
2. Mark body (513585-7) with identification symbol and serial number; use minimum 1-in.-high letters on side of body and minimum 1/4-in.-high letters on bottom; vibratool and felt pen are to be used in this operation.
3. Assemble irradiation capsule assembly in accordance with Drawing 513646.
 - a. Place component parts inside loading chamber.
 - b. Flush loading chamber with helium.
 - *c. Assemble parts in the following sequence:
 - (1) Insert compression spring.
 - (2) Place tube assembly in position.
 - (3) Insert retainer disk (with screw and nut).
 - (4) Insert blank cup.
 - (5) Put gasket in place.
 - (6) Place cap in position; position steel washer.
 - (7) Insert two bolts.

* Procedure completion to be certified by Quality Control as having met Quality Control specifications.

- d. Seal Irradiation Capsule:
 - (1) Tighten two bolts until they are finger tight.
 - (2) Remove capsule from chamber and place in vise.
 - (3) Insert remaining 10 bolts and bracket.
 - *(4) Tighten 12 bolts to 180-in.-lb; tighten across.
 - *(5) Tighten 12 bolts to 200 in.-lb; tighten around.
 - e. Weigh assembled capsule on scale to an accuracy of 0.1 g.
4. Perform leak check
- a. Place capsule in vessel filled with water.
 - b. Draw vacuum over surface of liquid for 5 to 10 min.
 - c. Release vacuum and leave capsule in water for 30 min.
 - *d. Remove capsule from water, dry, and weigh capsule. If weight gain exceeds 9 g, reload capsule.
5. X-ray capsule to confirm that all parts are properly assembled.
6. Affix inspection tag.
7. Package irradiation capsule in accordance with applicable Interstate Commerce Commission regulations.

* Procedure completion to be certified by Quality Control as having met Quality Control specifications.

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APPENDIX C

INSPECTION INSTRUCTIONS FOR CAPSULES

Aerjet-General

B P NO <u>513646</u>		INSPECTION INSTRUCTION	PAGE <u>1</u> PART <u>513646-21</u>	
CHG LTR <u>D</u> EOS			PLANNING OPER NO	
PROJECT MODEL REON			PART NAME IRRADIATION CAPSULE ASSY	
LOT SIZE	UNIT SERIAL/ LOT NO		ISSUE DATE <u>5/24/65</u>	REV DATE REV NO REVISED BY

FORM AGC 912 33 01

CHAR ITEM NO	REF CAT	INSP OPER (CHAR LOC	CHARACTERISTIC	TYPE OF GAGE OR INSPECTION EQUIPMENT	SEQ NO	RECORD VALUE	INSP FREQ AGL	INSP STATUS
1			Record Shop Order No. _____					
			Verify evidence of previous AGC inspection and acceptance of component parts:					
			513584-1 Cap (1)	S/O				
			513585-7 Body (1)	S/O				
			513642-1 Cup (1)	S/O				
			513643-1 Retainer Disk (1)	S/O				
			513645-1 Ring (1)	S/O				
			515262-1 Bracket (1)	S/O				
			515267-3 Tube Assy (1)	S/O				
			515268-3 Spring (1)	P/O				
			AN515-4-8 Screw (1)	Certs				
			MS35649-41 Nut (1)	Certs				
			.25-20UNC2A SCK HD Cap Srew Steel (12)	*Certs				
			Com'l Prod. R4-25B Seal (1)	**Certs				
2			Com'l Prod. 7.5 MM Dia. Glass Ball Perforated (1)	**Certs				
			Record calibration dates of all gages and tools to be used on attached recording sheet.	Visual			100%	
3		G/N 5	Assembled per instructions given on Work Release and on Assembly Loading and Testing Instructions.	Witness/ Visual			100%	

PREPARED BY

R. K. Zickler
 REMARKS *Certifications must conform to G/N
 #'s 1,3 and 4. **Certifications must conform
 To G/N #'s 1 and 4

APPROVED BY

*A. L. Zickler*RECORD VALUES
LOADED THUS

Aerjet-General

B P NO <u>513646</u>		INSPECTION INSTRUCTION	PAGE <u>2</u>		PART NO <u>513646-21</u>	
CHG LTR <u>D</u> EOS			OF <u>4</u>		PLANNING OPER NO	
PROJECT MODEL REON					PART NAME IRRADIATION CAPSULE ASSY	
LOT SIZE	UNIT SERIAL/ LOT NO		ISSUE DATE <u>5/24/65</u>	REV DATE	REV NO	REVISED BY

FORM AGC 912 33 01

CHAR ITEM NO	REF CAT	INSP OPER CHAR LOC	CHARACTERISTIC	TYPE OF GAGE OR INSPECTION EQUIPMENT	SEQ NO	RECORD VALUE	INSP FREQ ACL	INSP STATUS
4			<u>FOLLOWING APPLIES TO 515267-3 ASSY ONLY</u>					
			Verify for previous AGC Inspection acceptance by means of IPS, P.O. or Certs for detail parts used in this assembly. NOTE: Certs must conform to G/N #3	Visual				
			515264-3 Tube (1)	IPS				
			515265-1 Flux Indicator (1)	IPS				
			515265-3 Flux Indicator (1)	IPS				
			515266-1 Temperature Indicator (158°) (1)	IPS				
			515266-3 Temperature Indicator (255°) (1)	IPS				
			Material Explosive 2 (AR)	P.O.				
			Com'l Prod. Quartz Wool (AR)	Certs				
5		G/N 6	Verify free movement, prior to loading explosives, of tube in cavity of the body 513585-7. Identify tube with S/N using felt pen. Identify body with S/N using felt pen and vibro tool.	Manual/ Visual			100%	
6			Witness weighing of detail parts, excluding explosives, and record on attached recording sheet.	Analytical Scale			100%	
7			Verify that loading of explosives complies with loading instructions and Dwg. 515267-3	Witness			100%	
8			Witness weighing of 515267-3 assembly after loading of explosives and record on attached recording sheet.	Analytical Scale			100%	
9			.38 Min. Dim.	Scale/ Visual			100%	

PREPARED BY

R. R. Robinson

REMARKS

APPROVED BY

*M. G. L. L. L. L.*** RECORD VALUES
CODED THUS *

*Aerjet-General***INSPECTION
INSTRUCTION**

B/P NO <u>513646-</u>	
CHG LTR <u>D</u>	EO <u>8</u>
PROJECT MODEL REON	
LOT SIZE	UNIT SERIAL/ LOT NO

FORM AGC 912 33 01

PAGE <u>3</u>	PART <u>513646-21</u>		
OF <u>4</u>	PLANNING OPER NO		
PART NAME IRRADIATION CAPSULE ASSY.			
ISSUE DATE <u>5/24/65</u>	REV DATE	REV NO	REVISED BY

CHAR ITEM NO	REF AT	INSP OPER CHAR LOC	CHARACTERISTIC	TYPE OF GAGE OR INSPECTION EQUIPMENT	SEQ NO	RECORD VALUE	INSP FREQ ACL	INSP STATUS
10			Record actual amount and type of explosive used on attached recording sheet.	Substrate findings of Char. 6 from 8			100%	
11			Container identified per ASD 5215N	Visual			100%	
12			<u>FOLLOWING APPLIES TO 513646-21 FINAL ASSY</u> Body 513585-7 identified with S/N on side and bottom with felt pen and vibro tool.	Visual			100%	
13			Capsule assembled per B/P, Assembly, Loading and Testing Instructions.	Witness/ Visual			100%	
14		G/N 6	Spring and seal to conform to G/N #6	Depth Gage			100%	
15		G/N 7	Cap screws torqued to 170-200 inch lbs.	Torque Wrench			100%	
16		G/N 8	2.156 max. envelope Dia. for noted length.	Micro.			100%	
17			Witness and record weight of assembled capsuled, prior to leak test, on attached recording sheet.	Manual/ Visual			100%	
18			Witness and record weight of assembled capsule, after leak test, on attached recording sheet.	Manual/ Visual			100%	
19			Verify that capsules have not gained more than 8 grams. <u>NOTE:</u> Capsules that gain more than 8 grams are to be reloaded.	Substrate findings of Char 17 from 18			100%	
20			Component parts have been assembled properly.. (To be determined after assemblies have been X-Rayed.)	Review X-Rays			100%	
21			Tagged with P/N per ASD 5215M	Visual				
22			Record Lot No. in appropriate block					
INSPECTION COMPLETE				SIGN DATE				

PART NO

PLANNING OPER NO

PREPARED BY

R. K. Peterson

REMARKS

APPROVED BY

*H. R. Shier*RECORD VALUES
CODED THUS *

RN-S-0235

APPENDIX D

CERTIFICATION OF STRAND BURNER PRESSURE TEST

[illegible]