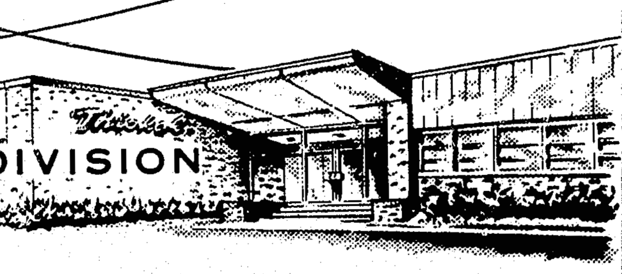
Thiokol CHEMICAL CORPORATION

PROPELLANT EXPULSION BLADDER FOR THE SATURN V/S-IVB

Report RMD 5125-Q5

Report Period: 1 November 1968 through 31 January 1969

Contract No. NAS8-21149



● REACTION MOTORS DIVISION
DENVILLE, NEW JERSEY

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Control No.
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FOREWORD

This report was prepared by Thiokol Chemical Corporation, Reaction Motors Division, Denville, New Jersey under Contract No. NAS8-21149, "Propellant Expulsion Bladder for the Saturn V/S-IVB" for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. Mr. J. Schell is the project engineer.

This is the fifth quarterly report and covers the work conducted during the period 1 November 1968 through 31 January 1969 on RMD Project 5125.

The personnel of Thiokol Chemical Corporation, Reaction Motors Division assigned to the project were Mr. R. L. Heilman (Project Leader) and Mr. D. Sinclair under the direction of Mr. N. Levine and Dr. C. Grelecki.

ABSTRACT

Development of a rubber-to-gold adhesive system was satisfactorily completed following evaluation of systems using state-of-the-art adhesives, CNR liquid polymer and NR copolymer. A second six inch mandrel and four RCS size mandrels were delivered to NASA/MSFC for goldplating. Brush coating has been selected as the preferable CNR coating technique. The first six inch mandrel has been goldplated and delivered for bladder fabrication.

CONTENTS

ILLUSTRATIONS

TABLES

I. SUMMARY

Difficulty experienced in goldplating the six inch diameter mandrel prompted the preparation and delivery of a back-up six inch mandrel for plating. All four RCS size mandrels were also delivered for goldplating at NASA/MSFC. Successful plating of the first six inch mandrel was accomplished and shipped to RMD near the end of the report period.

Development of an improved adhesive system has been completed following extensive evaluation of systems utilizing state-of-the-art adhesives, CNR liquid polymer and NR copolymer. Resistance to delamination throughout seven days exposure to N_2O_4 has been demonstrated to be repeatable.

Development of a CNR coating technique has continued with the conclusion that bladder fabrication will be initiated using a brush coating procedure.

The program schedule will require an extension in time to permit bladder fabrication and testing following receipt of plated mandrels.

II. INTRODUCTION

Expulsion bladders are the most reliable and efficient device for metering propellants into a rocket engine thrust chamber under zero gravity conditions. Elastomers are ideally suited for this application, however, until recently their use was limited due to the unavailability of materials with the requisite resistance to propellants such as nitrogen tetroxide. The development of carboxy nitroso rubber (CNR) by Thiokol-RMD was a major milestone in this area. This material, which is resistant to N_2O_4, was characterized for expulsion bladder application in a company funded program and a subsequent USAF-AFML/NASA-MSFC jointly funded program. In the latter, CNR expulsion bladders closely approximating the Saturn V/S-IVB RCS configuration were successfully fabricated and tested.

The desirability of an impermeable bladder for the Saturn V/S-IVB RCS application prompted the study of metal foil/CNR laminates in the aforementioned program. Since all polymeric materials are permeable to liquids and gases, the use of a flexible metallic liner is necessary to prevent permeation. Thiokol-RMD demonstrated the suitability of electroformed gold/CNR laminates for zero permeability propellant storage although results were limited by poor adhesion between CNR and gold.

The objective of this program is the fabrication and demonstration testing of composite electroformed gold/CNR full scale Saturn V/S-IVB RCS configuration bladders. In the execution of this goal, Thiokol-RMD will:

1. Characterize the material properties of CNR, electroformed gold and laminates thereof, for bladders for the Saturn V/S-IVB-LEM mission.

2. Classify bladder failure modes.

3. Determine design criteria for both vertically and horizontally oriented bladders by testing full scale bladders.

4. Determine bladder safety margins, as possible within the scope of the testing program.

5. Demonstrate full scale bladder characteristics when tested using N_2O_4.

Work on this program was stopped from mid-May 1968 until mid-August 1968. During this time the program status and scope was reviewed and revised. Reference A provides details of the revised program plan.

III. TECHNICAL DISCUSSION

A. Back-up Adhesive Study

1. Candidate Screening

As an initial screening procedure, a series of lap shear tests were conducted to evaluate a variety of surface preparations and primers alone and in conjunction with CNR liquid polymer. Samples for these tests included bonded strips of gold foil, fiberglass reinforced gold foil, goldplated aluminum and goldplated stainless steel. Promising test results were achieved with tin plate and with Y2300 Silane.

A series of peel test samples were also prepared, bonding cured CNR to gold foil and gold plated aluminum and stainless steel, using candidate adhesive systems. Peel strengths of 7.0 to 9.5 pounds per linear inch (pli) were achieved using Monsanto Gelvas 260 and 263 and the flexibilized epoxies, Epon 871 and 872. Details of procedures and test results of the above tests were covered in RMD reports 5125-ML-11 and 5125-ML-12.

A series of sample coupons were then cut from 0.005 thick gold sheet, 0.5 inch wide for peel tests, and 2 inches (diam.) round for delamination tests. All coupons were roughened with 120 grit emery paper, washed with acetone and hot air-dried prior to applying the primer. Peel and delamination test samples were made utilizing each of the promising candidates selected above. These samples were coated with CNR using a single component, pressure-feed, air spray gun. Details of the primers, the CNR formulations and the test results are given in Table I. The CNR film produced on these samples had a spongy texture similar to that previously produced on this program. This will be discussed later under "Coating Technique." None of the results were encouraging except that tin plate did exhibit the expected good compatibility with N_2O_4. Follow-up strip and disc samples were made applying tin plate to roughened gold foil and CNR coating as shown in Table I, except this time by brush coating instead of by spraying, to a film thickness of 0.012 inch. Peel strength on this sample was 0.55 pli. The delamination test was continued for seven days, after which no delamination had occurred. This disc sample is shown on Figures 1 and 2. The delamination test fixture is shown on Figure 3.

All peel samples discussed in this report were pulled using an Instron tensile testing machine with a crosshead speed of 2 inches per minute.

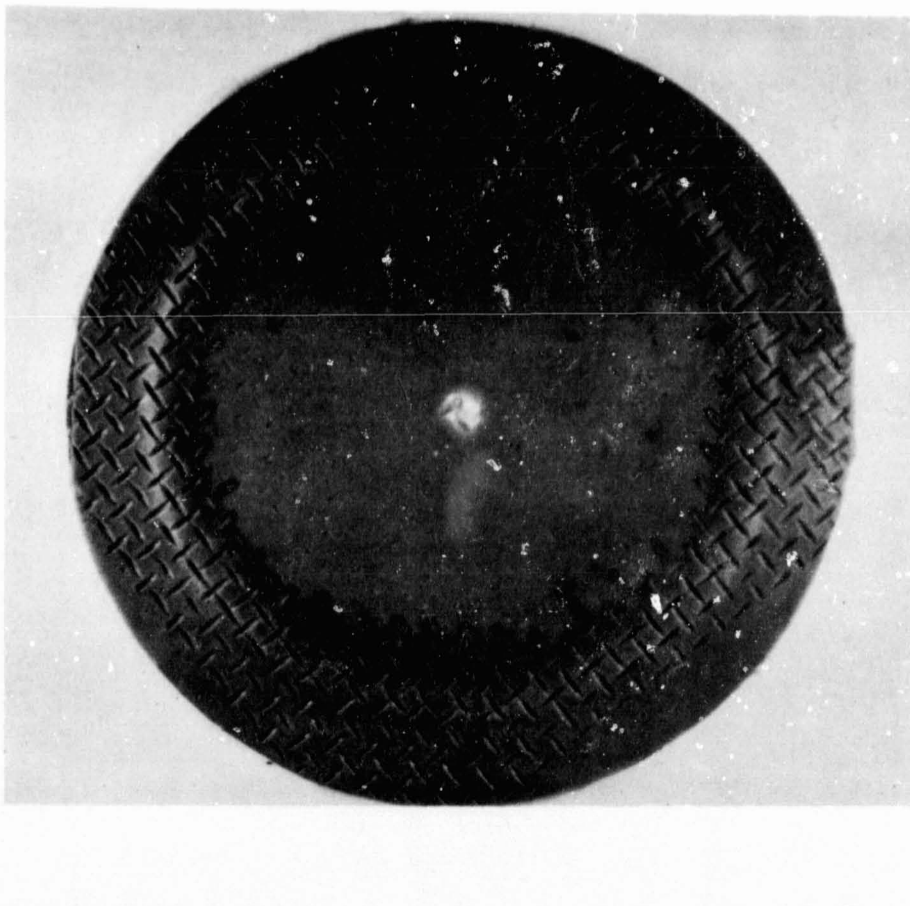

Figure 1. Tin plated Delamination Sample After 7 Days Exposure to N_2O_4 (mag. 1.7X)

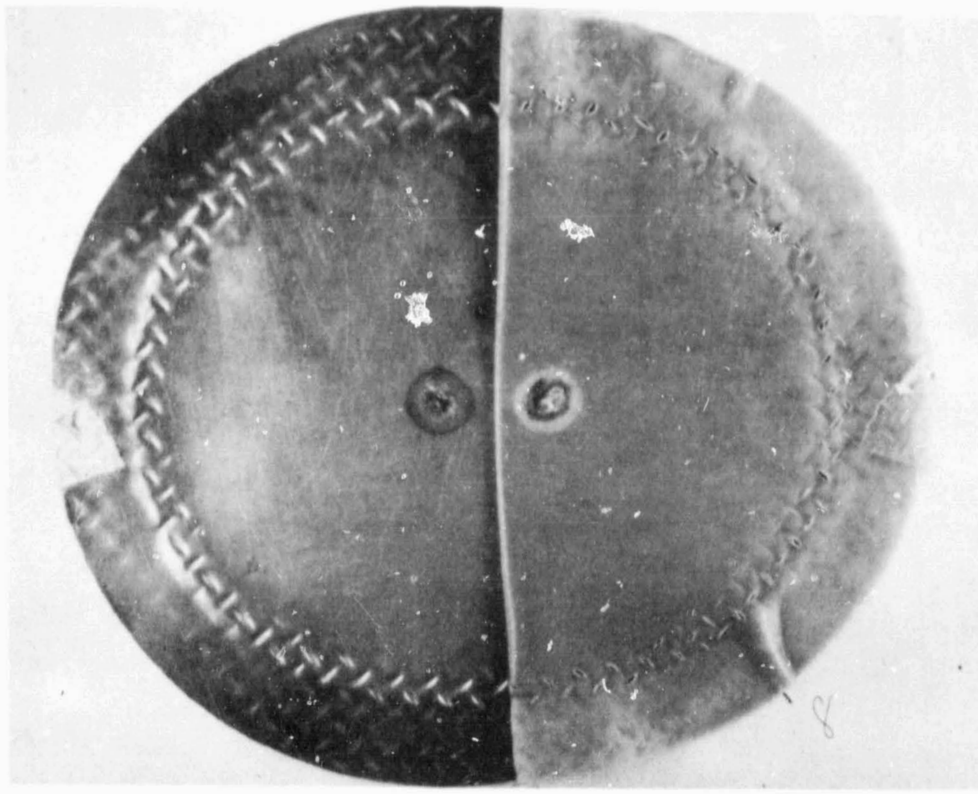

Figure 2. Tin plated delamination Sample After 7 Days Exposure to N_2O_4, Peeled Back to Expose Pinhole (Mag. 1.7X)

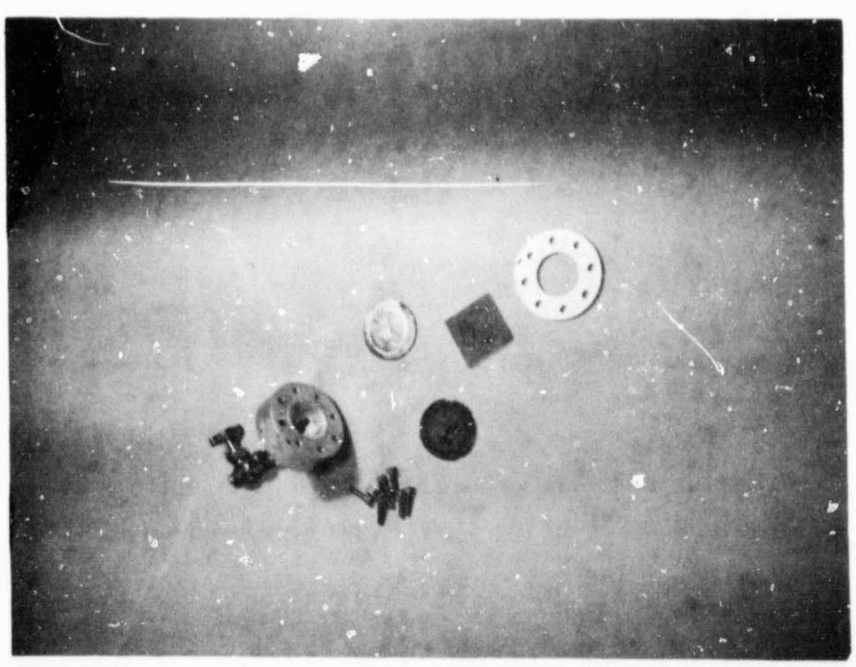

Figure 3. Delamination Test Fixture

TABLE I

GOLD/CNR ADHESION TESTS - PEEL AND DELAMINATION

Primer[1]	Peel[2] Sample No.	Disc No.[3]	CNR Film Thickness (in.)	Average Peel Strength (pli)	Delamination Test (N_2O_4) Results
A. Tin plate (0.0008" thk.) surface not roughened	1		0.023	1.0	-
	2		0.028	0.6	-
	3		0.024	0.8	-
		1-D	0.023	-	After 48 hrs. 1/4" diam. delaminated at pinhole
B. Y 2300 Silane; 1 thin wipe coat; Cured 15 min. at 300°F	4		0.028	0.75	-
	5		0.027	0.80	-
	6		0.028	0.75	-
		4-D	0.020	-	Partially delaminated during CNR cure; after 48 hrs. 1/8" diam. delaminated at pinhole
C. MPS 260 Gelva solution; w/50% MEK (bv); 2 brush coats each cured 20 min. @ 200°F	7		0.028	4.0	-
	8		0.033	3.5	-
	9		0.028	3.0	-
		7-D	0.022	-	After 65 hrs. fully delaminated (Reattached as it dried)
D. MPS 260 Gelva solution; w/50% MEK (bv); 1 brush coat cured 20 min. at 200°F; MPS 263 Gelva solution w/25% MEK (bv); 1 brush coat cured 20 min. @ 200°F	10		0.028	2.0	-
	11		0.037	1.8	-
	12		0.038	2.0	-
		12-D	0.023	-	After 24 hrs. fully delaminated (Reattached as it dried)

TABLE I (CONT'D)

Primer[1]	Peel[2] Sample No.	Disc No.[3]	CNR Film Thickness (in.)	Average Peel Strength (pli)	Delamination Test (N_2O_4) Results
E. Gelva solution same as "D" for	13		0.037	1.4	-
one coat; added one brush coat	14		0.035	1.5	-
of CNR 105 solution, cured 60	15		0.028	1.6	-
min. @ room temperature		13-D	0.023	-	After 24 hrs. fully delaminated (Reattached as it dried)
F. Epon 871, 50 pbw	16		0.022	8.0	-
Epon 828, 50 pbw	17		0.023	8.0	-
Catalyst Z, 15 pbw; one thin	18		0.021	rubber tore at 9.0	-
coat applied w/blade, cured 30 min. @ 300°F		16-D	0.017	-	After 113 hrs., over 50% delam.
G. Epon solution same as "F" for	19		0.018	10.2	-
one coat; added one brush coat	20		0.024	9.0	-
of CNR 105 solution, cured 15	21		0.024	8.8	-
min. @ 200°F		19-D	0.018	-	After 48 hrs., fully delaminated
H. Epon solution same as "F" for	22		0.030	6.0	-
one coat; applied a second coat of	23		0.038	5.5	-
same solution and sprinkled on	24		0.031	4.5	-
ground (40 mesh) cured CNR; cured 45 min. @ 300°F		22-D	0.026	-	After 48 hrs., fully delaminated
I. Epon 872, 140 pbw, w/25% (bv)	25		0.016	4.5	-
Xylene Catalyst Z, 7.5 pbw	26		0.013	no test	-
one thin coat applied w/blade,	27		0.013	4.5	-
cured 50 min. at 300°F		25-D	0.027	-	After 48 hrs., fully delaminated

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TABLE I (CONCLUDED)

(1) All primed samples were sprayed together with CNR solutions as follows, using a single component type, pressure-feed air spray gun:

First coat: CNR solution 102 (RMD Dwg. 318501-102) with 2 PHR curative solution*
Subsequent Coats: CNR solution 105 (RMD Dwg. 318501-105) with 5 PHR curative solution*

CNR film cure:
1 hr. - air dried
2 hr. @ 225°F
1 hr. @ 250°F

*Curative solution is chromium trifluoroacetate (CTA) dissolved in a 90% solution of tetrahydrofuran (THF)

(2) Peel samples are gold foil (0.005 thk.) strips 0.5 inch wide with 1 inch long peel area.

(3) Delamination samples are gold foil discs 2 inches in diameter. The area exposed to N_2O_4 is 1.375 inches in diameter. A hole approximately 1/16 inch diameter in gold foil permitted N_2O_4 contact with coating.

The foregoing tests conclusively showed that good compatibility of the primer with N_2O_4 is a more critical requirement than high peel strength. Subsequent material evaluations were therefore initiated from a condition of known good compatibility.

A series of peel test samples were prepared utilizing CNR Solutions 102 and 105 (dash numbers to RMD Dwg. 318501 - see Ref. B) as described in Table II. All coating was applied by brushing with 10 min. drying time between coats. Curing was done in a circulating air oven.

The results given in Table II suggested that tin plate does not enhance peel strength and it was therefore eliminated as a primer agent.

A set of peel test samples were prepared with a variety of primers as detailed in Table III. All but the first primer were basically CNR 105 coating solution (RMD Drg. 318501-105). All CNR coating was done by brushing, applying the coats at ten minute intervals. Curing consisted of an overnight air dry, followed by oven curing 2 hours at 225^{0}F and 1 hour at 250^{0}F. Preparation of gold foil strips consisted of roughening with 120 grit emery, Acetone cleaning and air drying.

The CNR film using primer "C" in Table III produced the highest peel strength and was therefore selected as the leading candidate. Two delamination disc samples were prepared using the same procedure as for Samples No. 4 and 5 in Table II and were put into test. After seven days exposure to N_2O_4 the first disc exhibited no delamination . This sample is shown in Figure 4. Note that there is a slight lightening of color directly behind the pinhole but no detrimental characteristics. At the close of this report period, the second disc had completed the fourth day of testing with similar results.

TABLE II

GOLD/CNR ADHESION (PEEL) TESTS

	Sample Description	Sample No.	CNR Film Thickness (in.)	Peel Strength (pli)
A.	Gold sheet (.005): 1 coat 102 solution; 37 coats 105 solution; 5 phr CTA curative; overnight air dry; 2 hrs. @ 225°F; 1 hr. @ 250°F	1 2 3	0.011 0.012 0.011	0.70 0.68 not tested
B.	Tin plated gold sheet: 1 coat 102 solution; 37 coats 105 solution; 5 phr CTA curative; same cure as "A"	4 5 6	0.010 0.012 0.010	0.49 0.55 not tested
C.	Matte finish gold electroplate, tin plated: 1 coat 102 solution; 37 coats 105 solution; 5 phr CTA curative; same cure as "A"	7 8 9	0.010 0.010 0.011	0.70 0.62 not tested
D.	Gold Sheet: 6 coats 102 solution; 30 coats 105 solution; 2.5 phr CTA curative; air dry over weekend; 2 hrs. @ 225°F; 1 hr. @ 250°F	10 11	0.012 0.012	0.96 0.96
E.	Gold sheet; 30 coats 105 solution; 2.5 phr CTA curative; same cure as "D"	12 13	0.012 0.011	1.26 0.98
F.	Tin plated gold sheet; coated same as "E"	14 15	0.011 0.011	0.82 0.85
G.	Tin plated gold sheet; 3 coats 105 solution blended with 20 phr copolymer solution; 30 coats 105 solution; 2.5 phr CTA curative; same cure as "D"	16 17	0.010 0.009	1.26 1.10

TABLE III

GOLD/CNR ADHESION (PEEL) TESTS

	Primer	Sample No.	CNR Film Thickness (in.)	Peel Strength (pli)
A.	Y2300 Silane 10% solution in denatured alcohol; one brush coat; cured 15 min. @ 300°F	1	0.014	0.92
B.	CNR 105 solution, w/80 phr (solids) Freon E-9, curative - 3.6 phr CTA; 3 brush coats	2 3	0.015 0.013	0.96 1.00
C.	CNR 105 solution, w/80 phr (solids) Nitroso copolymer gum, curative - 3.6 phr CTA; 3 brush coats	4 5	0.017 0.016	1.20 1.50
D.	CNR 105 solution, w/80 phr (solids) CNR liquid polymer; 3 brush coats	6 7	0.018 0.015	0.70 0.54

Note: CNR film was built up with 38 brush coats of CNR 105 solution at 10 minute intervals; curative, 2.5 phr CTA.

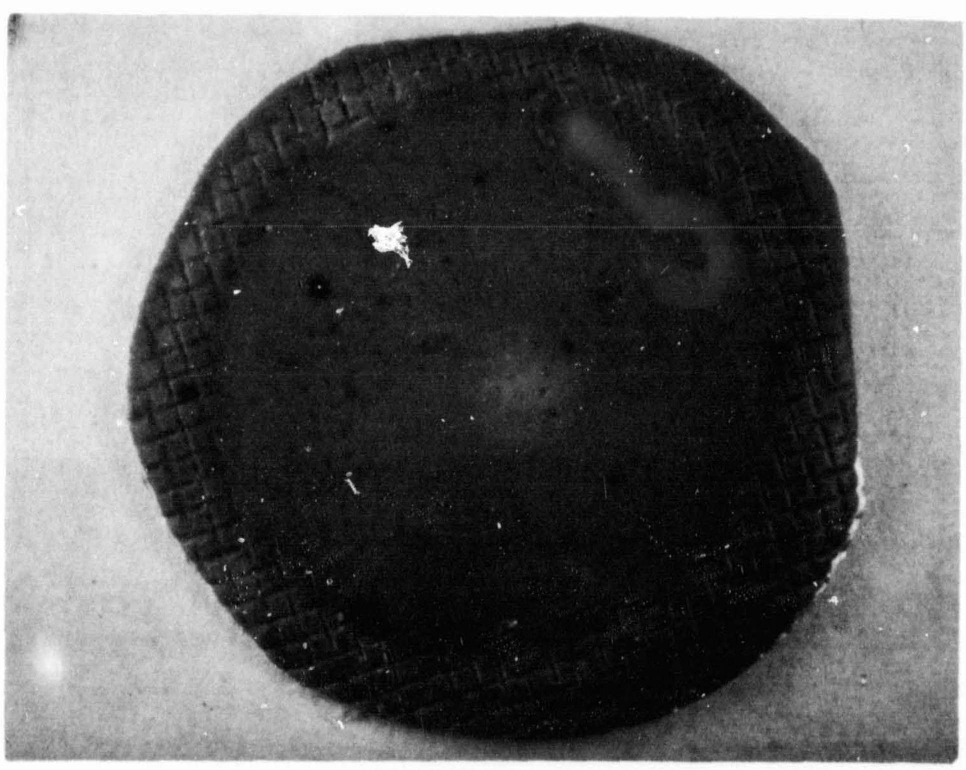


Figure 4. Delamination Sample After 7 Days Exposure to N_2O_4 (Primer: 105/copolymer)(mag. 1.8X)

Because it remains uncured, the Nitroso copolymer serves as a tackifier and is capable of being more readily deformed and elongated than cured CNR. It was postulated that this type of material would bond to both gold and CNR and serve as a link between the two interfaces.

To evaluate the effect of varying the amount of copolymer used, a set of peel test samples were prepared with copolymer varied from 40 to 100 phr. Details and peel strengths are given in Table IV. It is of interest that all samples except No. 1 and No. 2 peeled in an intermittent fashion, cycling between cohesive and adhesive failure. Cohesive failure occurred at the interface between primer coats and straight CNR. The optimum quantity of copolymer appears to be 80 phr.

TABLE IV

GOLD/CNR ADHESION (PEEL) TESTS

	Primer	Sample No.	CNR Film Thickness (in.)	Peel Strength (pli)
A.	CNR 105 solution, w/40 phr (solids) Nitroso copolymer gum; 3.6 Phr CTA curative; 6 brush coats	1	0.014	1.45
		2	0.016	1.53
B.	Same as "A" except w/80 phr copolymer	3	0.017	2.40
		4	0.014	2.60
C.	Same as "A" except w/100 phr copolymer	5	0.016	1.92
		6	0.018	1.55
D.	CNR 102 solution, w/80 phr copolymer; 3.6 phr CTA curative; 6 brush coats	7	0.016	2.60
		8	0.018	2.70

Note: CNR film was built up with 36 coats of CNR 105 solution; 2.5 phr CTA curative; overnight air dry; oven cure 2 hrs. @ 225°F; 1 hr. @ 250°F

2. Candidate Chosen

The adhesive system selected to be used for fabricating the first six inch bladder will therefore be the CNR 105 solution with nitroso copolymer gum added. A summary of the formulations of primer and coatings is given in Table V.

TABLE V

PRIMER AND COATING SOLUTIONS FOR 6" BLADDER A

Ingredients		Phr
Primer: CNR Coating Solution 105 (RMD Dwg. 318501-105) (6 coats)		
CNR	8.34 pbw	100
Hi Sil 233	1.66 pbw	
TriSolvent Blend	90.00 pbw	
Nitroso Copolymer Gum Solution		
Nitroso Copolymer Gum	10.00 pbw	80
TriSolvent Blend	90.00 pbw	
Chromium Trifluoroacetate (CTA) Solution		
CTA	10.00 pbw	3.6
Tetrahydrofuran (THF)	90.00 pbw	
Coating: CNR Coating Solution 105 (see above)		
CTA Solution (see above)		
CTA		2.5

B. Mandrel Dissolving Solution Development

This effort was completed as reported in RMD Report 5125-Q4. The caustic solution selected is Oakite No. 130 at a concentration of 0.5 lb/gallon of water.

C. CNR Coating Technique Evaluation

An attempt was made to develop spray coating technique for CNR solutions by practicing with neoprene as an inexpensive simulant. Severe cobwebbing was experienced and could not be overcome readily. It was concluded that neoprene had spraying characteristics too adverse to be considered as a practicable practice medium. Fabricating neoprene bladders was therefore dropped from the program.

The initial set of test samples, discussed in Section III. A. 1 were made by spraying CNR solutions. A pressure-feed gun was used to spray the premixed (coating solution and curative) solution. The quantity to be sprayed did not warrant the added complication of the two- component spray gun. Results were unsatisfactory: Cobwebbing was a serious problem; film buildup was very difficult to control and lumps could not be avoided; solvent entrapment was a problem with the result that the cured film had a spongy texture.

Vendors of airless spray equipment were consulted to evaluate whether their equipment would eliminate any of the above problems. The conclusions were that airless spraying would be worth considering for production quantity work. Actual demonstration would be required prior to assessing the improvement over air spraying. Since this was not economically feasible on this program, airless spraying was dropped from consideration.

Brush coating of CNR solution has been used by RMD for some time on a variety of conformal coating applications. It was also used briefly on a bladder program for USAF (see Reference C). Although there are disadvantages to brush coating, the obvious advantages listed below make it an attractive method for this program:

- Film thickness control
- Less material waste
- Ease in accomodating changing solution viscosity
- Convenient to provide drying time between layers

This method was therefore selected as the coating technique to be used for bladder fabrication.

To evaluate the optimum drying time between coats of CNR solution, a test was conducted where coating solution was applied to a hot aluminum plate and to a room temperature plate with the elapsed time between applications varied from 0.5 minutes to 13 minutes. The resulting films were cured, visually examined under 30X magnification for evidence of bubbles and tensile tested. All hot plate samples contained entrained bubbles. All room temperature samples were of superior quality, with no evidence of entrained solvent bubbles for 4 minutes (and up) elapsed time between coats. The tensile test results are summarized on Figure 5. They too show the advantage of applying CNR solution to a room temperature mandrel. Subsequently, all coating work will be air dried approximately 10 minutes between layers.

D. Fabrication and Tooling

Development of mandrel goldplating technique was performed at NASA/MSFC during this period. Some difficulty was experienced in plating the six inch diameter mandrel so a back-up unit was prepared and shipped by RMD to MSFC. The four RCS size mandrels were polished and shipped to MSFC for plating. The surface finish on these units ranged from 16 to 125 rms (estimated). Also, there were localized surface irregularities and some shallow dents. All were carefully examined prior to delivery and assessed to be acceptable conditions for the application; i.e., they would not preclude the achievement of a continuous, leak-free layer of electro-deposited gold.

The first plated six inch mandrel was received at RMD near the end of the period. It was inspected visually and by X-ray and appeared suitable for use in fabricating the first bladder.

IV. PROGRAM STATUS

The operating schedule, Figure 6, attached to the back of this report, has been updated to illustrate program status in terms of technical accomplishments and also manhours expended as of January 31, 1969. The program schedule will require an extension in time to permit bladder fabrication and testing following receipt of the plated mandrels.

V. ANTICIPATED WORK

Fabricate and test the first six inch bladder.

Prepare bladder test plans.

Design handling fixture for RCS bladder.

Legend:

* ⊙ CNR solution applied to vertical hot aluminum plate (120-160°F); 1 coat 102 solution; 16 coats 105 solution; final thk 0.005 avg.

* △ Chilled 105 solution applied to vertical aluminum plate at room temperature - 1/102; 16/105; final thk 0.005 avg.

** ⊡ Later data - 33 coats 105 solution applied at room temperature; final thk 0.010.

* Cure: 1 hr. @ room temp., 2 hr. @ 225°F, 1 hr. @ 250°F

** Cure: 16 hr. @ room temp., 2 hr. @ 225°F, 1 hr. @ 250°F

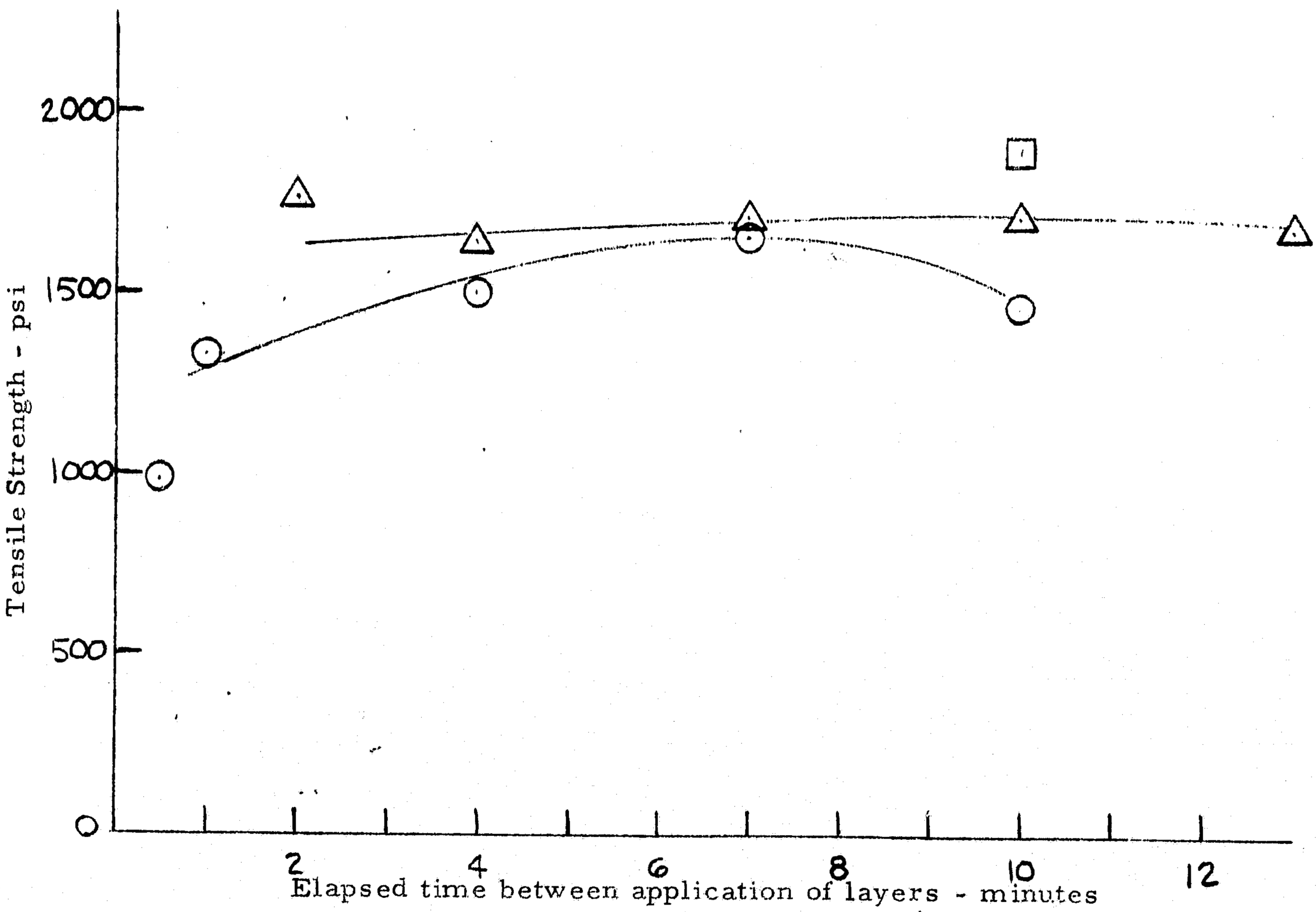


Figure 5. Tensile Strength vs CNR Coating Drying Time

VI. REFERENCES

Reference A - Letter Defense Contract Administration Services District, Newark, N. J., Attention Mr. W. Calabrese from Thiokol-RMD dated 7/15/68 (NASA/MSFC 6401A).

Reference B - Thiokol-RMD Drawing 318501 "Coating Solutions."

Reference C - Technical Report AFML-TR-68-158 prepared by Thiokol-RMD under USAF Contract No. AF33(615)-5311, Project 7381. "Nitroso Rubber Expulsion Bladders for Nitrogen Tetroxide Application"

FIGURE 6

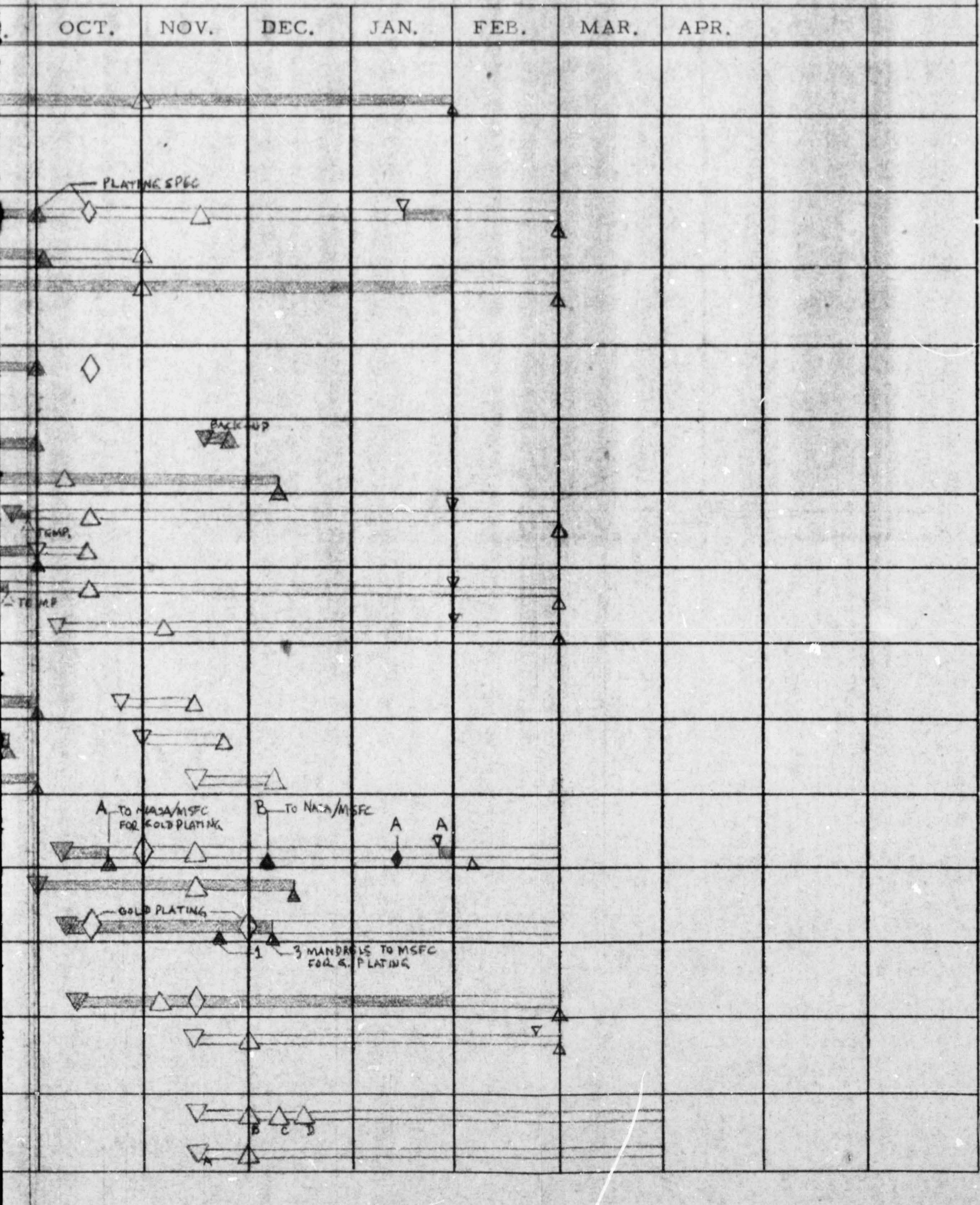
◇ NASA-MSFC Action/Approval

OPERATING SCHEDULE	LEGEND: BASIC SCHED. / REV. START / REV. COMPL. / ACT. START & COMPL.				
PHASE I			1968 JULY	AUG.	SEPT.
TASK I - MATERIALS AND FABRICATION					
A. Back up Adhesive Study - Liq. Polymer/Epoxy Test Sample Fabrication and Test					
B. Physical Properties - Complete					
C. Specifications - Product and Process					
D. Mandrel Dissolving Solution Dev.					
E. CNR Coating Technique Evaluation					
TASK II - BLADDER DESIGN AND TOOLING					
A. Dwgs. - RCS Bladder; Mandrel					
B. Mandrels:					
1. 6 Inch - Rwk. to Accom. Standpipe (1 pc)					
2. RCS - Inspect (6 pcs) and Rwk. (5 pcs)					
C. Mandrel Dissolving Tooling Rwk.					
D. Rotating Fixture (for Spray. Op.) Design and Fab.					
E. Bladder Handling Fixture - Design and Fab. (3 pcs)					
F. Bladder Port Trimming Tools - Leak Test Fixture - Design and Fab.					
G. Plastic Tank - Procure Small Parts - Fab. Wood Stand					
H. Vertical Twist Rig Rework					
I. Horizontal Slosh Rig Rework					
TASK III - BLADDER FABRICATION					
A. 6 Inch - Using Liq. Polymer Adh. (1 pc) PLUS BACK-UP					
B. RCS - Neoprene; Partial Size (6 Pcs)					
C. RCS - Gold Plate /CNR (4 pcs)					
TASK IV - BLADDER TESTING					
A. Test Plan - RCS Bladders					
B. 6 Inch - Permeation and Flexure (1 pc)					
C. RCS - Gold Plate/CNR (Design Compliance Tests)					
1. Non-Destr. Test (3 pcs).					
2. Destr. Tests (1 Pc.)					

RMD RE 105-2

PROJECT	PHASE	TASK	OPER.	PAGE	DATE	COMPONENT
5125			ALL	1 of 2	1/31/69	

1969



OPERATING SCHEDULE

LEGEND: BASIC SCHED. — REV. COMPL. — REV. START — ACT. START & COMPL.

			1968 JULY	AUG.	SEPT.
TASK IV - BLADDER TESTING (Cont'd.)					
D. Functional Tests - In plastic Tank with Water - 1 RCS Bladder					
1. Fill, twist and Expulsion Tests - Vertical Attitude					
2. Slosh Tests - Horizontal Attitude					
E. Functional Tests with N_2O_4 - In RCS Tank					
TASK V - DESIGN REVIEW AT RMD					
TASK VI - REPORTS					
(Q-Quarterly; M-Monthly; F-Final)					M
Planned Manhours	MONTHLY Δ — CUMULATIVE —			41	31 4
Actual Manhours					9/30

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PROJECT	PHASE	TASK	OPER.	PAGE	DATE	COMPONENT
5125			ALL	2 of 2	1/31/69	

1969