21 May 1969

[redacted]

UNCLASSIFIED

UMC DOCUMENT # PDS-552

COPY # FIVE OF SEVEN COPIES

UNIDYNAMICS PHOENIX

A DIVISION OF UMC INDUSTRIES, INC.

PHOENIX, ARIZONA

SC-CR-69-3182

PROGRESS REPORT #3

ANALYTICAL STUDY

FOR A

LOCATION AID DEVICE (U)

RS 3415 75431

[redacted]

[redacted]

Prepared By: W. J. Higgins
Senior Engineer

Approved By: C. E. Simpson
Project Manager

[redacted]

SANDIA SYSTEMATIC DECLASSIFICATION REVIEW
DOWNGRADING OR DECLASSIFICATION STAMP

CLASSIFICATION CHANGED TO: U AUTHORITY: W. C. Layne

Emelda Selph 7/14/97
PERSON CHANGING MARKING & DATE

RECORD ID: 97SN2301

Carmela Dilley 7/22/97
PERSON VERIFYING MARKING & DATE

DATED: 6/30/97

UMC INDUSTRIES, INC

[redacted]

UNCLASSIFIED

UNCLASSIFIED

## SUMMARY

(U) This progress report covers the following:

I. Growth and force of polyacrylamide using an ethylene glycol/water solution as reported in progress report 2.

II. Retesting of wet chemical delays data that was reported in progress report #2.

III. New dry chemical delay.

IV. Delay testing summary.

V. Felt metal treatment

VI. Manufacture of polyacrylamide

VII. Geometry and salinity effect on growth rate of polyacrylamide in sea water.

VIII. Ejection of capsule test.

IX. Sympathetic detonation study.

X. Firing of primer under water.

Appendix A Drawings

Appendix B Research reports on manufacture of polyacrylamide.

UNCLASSIFIED

I. Polyacrylamide Testing with Ethylene Glycol/Water Solution

(C) Growth Test - 10 Units

UNCLASSIFIED

Per the work plan generated with Sandia and UMC on 6 December 1967, ten units were fabricated using the acrylamide design per SK1567. (See Figures 1 & 2) Five of these units were tested at +35°F and five units were tested at 77°F in order to determine the temperature coefficient of these units over the expected operational range. Each piston was set to actuate upon .4" travel and the delay solution was 1/3 ethylene glycol and 2/3 water. (Ref. Progress Report #2)

Results:

| +77°F | +35°F |
|---|---|
| 6,962 | 29,782 |
| 10,688 | 20,983 |
| 12,072 | 35,845 |
| 21,244 | 47,878 |
| $\bar{X}$ = 12,740 | $\bar{X}$ = 36,122 |

$$\frac{\bar{X}\ 35^{\circ}F}{\bar{X}\ 77^{\circ}} = 2.83$$

Two of the test units delay times were not included since one in the 77°F temperature group went off sympathetically at 21,244 minutes and one unit in the +35°F group failed to go due to the ball sticking in the release mechanism.

(C) Retest - 10 Units

A second group of acrylamide delays were assembled per SK1567, however, felt metal was used as a feeder element since it was observed that the polyacrylamide had a non-uniform swelling at the wicking interface.

The delay element travel was .3".

UNCLASSIFIED

(U)

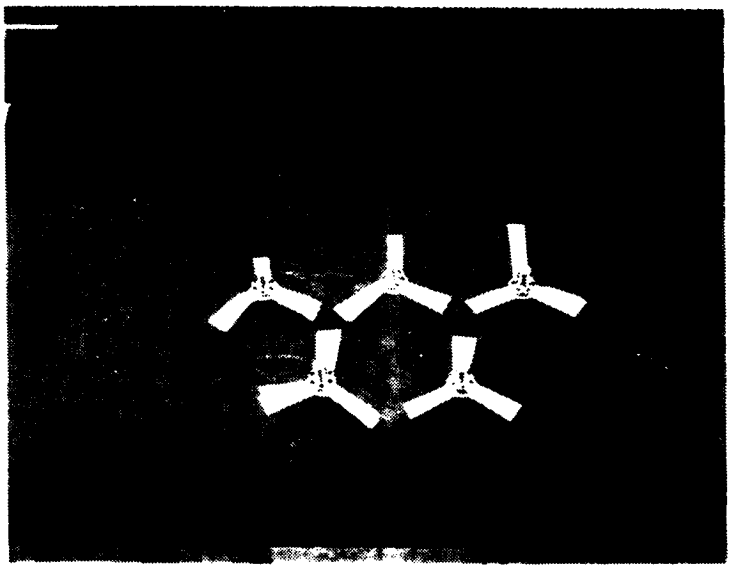


Figure 1

(U)

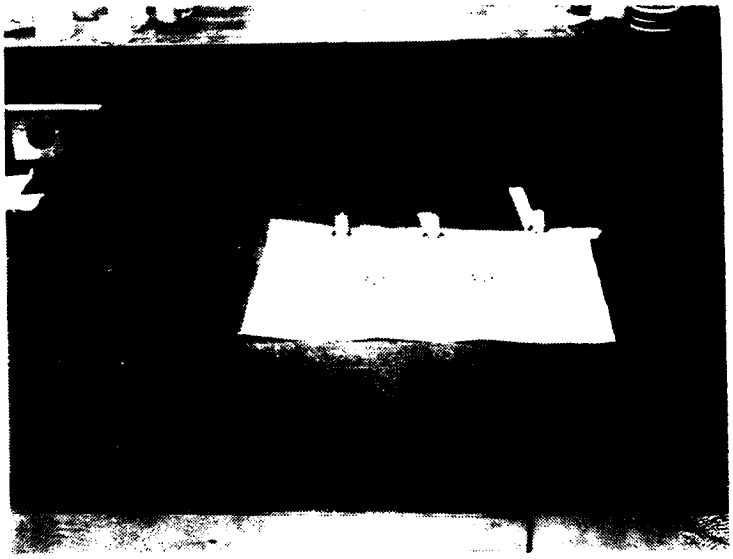


Figure 2

Polyacrylamide Testing

(U) Solution used to activate was 1/3 ethylene glycol and 2/3 water (three cc's). (See Progress Report #2) UNCLASSIFIED

Lot #5 SK1556 Rev. O.I. used in 1 - 5 77°F flat.

Lot #4 SK1556 Rev. O.I. used in 6 - 10 35°F champhered.

Results:

| +77°F | +35°F |
|---|---|
| 1,099 | 5,824 |
| 1,524 | 8,073 |
| 1,664 | 8,947 |
| 1,665 | 9,225 |
| 2,151 | 11,245 |
| $\bar{X}$ = 1,620 | $\bar{X}$ = 8,663 |

$$\frac{\bar{X}\ 77^{\circ}F}{\bar{X}\ 35^{\circ}F} = 5.63$$

(C) These results have an indicated temperature ratio of 5.63, however, some of the apparent temperature effect may be due to the use of champered acrylamide at 35°F producing a slower delay.

(C) Force Test using ethylene glycol/water solution

Polyacrylamide pressure test with chatillion pressure gage

Standard test specimen - .140 dia x .360 lg

Solution - 1/3 ethylene glycol and 2/3 distilled water

Temperature - 72°F

Test #1

(U) 5# preload

| Hour Time | Pounds Force |
|---|---|
| 0 | 0 |
| ½ | 1½ |
| 1½ | 3 1/8 |
| 2 | 3 5/8 |
| 5 | 4 3/8 |
| 6 | 4 3/4 |
| 7 [redacted] | 5 |
| 23 [redacted] | 7 ½ |
| 24 [redacted] | 7 ½ |

UNCLASSIFIED

UNCLASSIFIED

Test #2

(C) 2# preload -

| Elapsed Time | Pounds Force |
|---|---|
| 5 min | 1/4 |
| 10 min | 1/2 |
| 15 min | 1 |
| 25 min | 2 |
| 1 1/2 hr | 5 |
| 2 hr | 5 1/4 |
| 3 hr | 6 |
| 3 1/2 hr | 6 1/8 |
| 3 3/4 hr | 6 1/4 |
| 4 hrs | 6 1/2 |
| 5 | 7 |
| 7 | 7 1/2 |
| 22 | 8 3/4 |
| 24 | 8 3/4 (stopped growing) |

Test #3

(C)

| Elapsed Time | Pounds Force |
|---|---|
| 12 min | 1/4 |
| 14 min | 1/2 |
| 15 min | 3/4 |
| 15 3/4 min | 1 |
| 17 min | 1 1/4 |
| 18 min | 1 1/2 |
| 22 min | 2 |
| 33 min | 3 |
| 38 min | 3 1/4 |
| 48 min | 3 3/4 |
| 1 hr | 4 1/4 |
| 1 1/4 hr | 4 3/4 |
| 2 hr | 5 7/8 |
| 2 1/4 hr | 6 1/8 |
| 3 hr | 6 7/8 |
| 4 hr | 7 3/8 |
| 4 1/2 hr | 7 5/8 |
| 5 3/4 hr | 7 7/8 |
| 22 hr | 12 1/4 |

Stopped test unit leaking.

UNCLASSIFIED

UNCLASSIFIED

Test #4
(repeat of test #3)

(C)

| | |
|---|---|
| 45 min | 2 3/4 |
| 1 1/2 hr | 4 1/4 |
| 2 1/2 hr | 5 5/8 |
| 4 hr | 6 3/4 |
| 6 1/2 hr | 7 7/8 |
| 7 1/4 hr | 8 1/8 |
| 23 hr | 11 3/4 |
| 29 hr | 11 3/4 |
| 4 days | 16 3/4 |
| 6 days | 17 |

Stopped test unit leaking.

UNCLASSIFIED

UNCLASSIFIED

## II. Wet Chemical Delays

(C) Two groups of chemical delays were assembled per SK1648 and tested to verify the results reported in progress report #2. The first test group used solution X1 and the second group used solution X2. These mixtures were defined in progress report #2. The estimated times for +35°F results were 3.2 and 8.8 days respectively. This agrees very well with the 3.38 days and 5.66 days actually observed. (See Progress Report #2)

Wet Chemical Delay Test Results:

(C)

| | Time Minutes 77°F | Time Minutes 35°F | | |
|---|---|---|---|---|
| X-1 | 801 | 4281 | $\bar{X}$ 35°F | |
| | 835 | 4365 | | = 5.15 |
| | 945 | 5171 | | |
| | 1011 | 5226 | $\bar{X}$ 77°F | |
| | 1130 | 5311 | | |
| | $\bar{X}$ = 944 min | $\bar{X}$ = 4870 min | | |
| | .65 days | 3.4 days | | |
| X-2 | 1317 | 7117 | $\bar{X}$ 35°F | |
| | 1357 | 7726 | | |
| | 1875 | 8151 | | = 4.49 |
| | 2719 | 8275 | | |
| | | 9495 | $\bar{X}$ 77°F | |
| | $\bar{X}$ = 1817 min | $\bar{X}$ = 8152 min | | |
| | 1.26 days | 5.66 days | | |

UNCLASSIFIED

## III. New Dry Chemical Delays

(C) During the program it became apparent that carrying fluid along with the system required a sealed vial of actuating fluid and was very costly in terms of space and weight. Since a ready supply of actuating fluid was available (sea water), methods of utilizing this fluid were investigated and the invention by W. Higgins and R. Wood of the dry chemical delay resulted. This delay is shown in SK-1850. The major element is a pellet of $CuSO_4$ crystals and NaCl crystals mixed and consolidated into a solid form. The pin restricting wire is fed through a hole drilled into this pellet. Addition of water to the NaCl/$CuSO_4$ pellet cuts the restraining wire in a fixed delay time. This was reviewed by Sandia and received separate funding. As seen in drawing SK-1850, a felt metal wick is used to feed water to the pellet since the $CuSO_4$/NaCl does not effect pure nickel only the music wire is attacked by the solution.

### Preliminary Dry Chemical Delay Testing

(C) Our first attempts to test the dry chemical delay were as follows:

1. One percent NaCl used with CuSO4 crystals (20,000 psi on pellet). The delay operated for 12 days before firing using distilled water.
2. Fifty percent NaCl used with $CuSO_4$ crystals (20,000 psi loading pressure of pellet). One delay was at 35°F and fired in 51.6 minutes the other delay was at 80°F and fired in 27.9 minutes. Both used distilled water.

UNCLASSIFIED

[redacted]

(U) 3. 100 mg $CuSO_4$ and 700 mg NaCl pressed at 10,000 psi.

Salt water used to activate (all wicks dry)

| Amb | 35°F |
|---|---|
| 136.3 (pellet cracked) | 753.8 |
| | 951.5 |

4. 100 mg $CuSO_4$ and 700 mg NaCl pressed at 30,000 psi.

All wicks dry and salt water used to activate

| Amb | 35°F |
|---|---|
| 121.1 (pellet cracked) | 756.7 |
| 107.5 | 452.5 |
| 72.0 | 851.2 |

5. 500 mg $CuSO_4$ and 500 mg NaCl pressed at 30,000 psi.

All wicks dry and salt water to activate (reused wick)

| Amb | 35°F |
|---|---|
| 92.4 | 1060.0 |
| 47.5 | 366.6 |
| 48.8 | |

The above results showed the feasibility of this delay.

Testing

Various wire diameters

(C) Pellets for this test were a 50-50 mix pressed at 30,000 psi. During manufactur of pellets, the NaCl was dried after screening (60 mesh) and before mixing with $CuSO_4$.

$CuSO_4$ was not dried.

Wires were wiped clean with TCE prior to assembly.



[redacted]

UNCLASSIFIED

(C) One cc sea water used in each delay.

Music wire used in all units.

Units were tested using SK-1850 configuration less the output charge.

| .018 Dia | S/N | Tension Spring # | Delay Time Minutes |
|---|---|---|---|
| Ambient | 1 | 9 | 117.3 |
| | 2 | 8 | 56.7 |
| | 3 | 8.5 | 72.5 |
| | 4 | 9 | 55.7 |
| | 5 | 7.5 | 40.0 |
| 35°F | 6 | 7 | 94.2 |
| | 7 | 7 | 80.8 |
| **.031 Dia** | | | |
| Ambient | 1 | 7 | 228.3 |
| | 2 | 7.5 | 286.5 |
| | 3 | 7 | 304.5 |
| | 4 | 7 | 271.5 |
| | 5 | 7 | 133.0 |
| 35°F | 6 | 7 | 195.1 |
| | 7 | 7 | 1010.7 |
| **.035 Dia** | | | |
| Ambient | 1 | 7.5 | 252.6 |
| | 2 | 7 | 200.9 |
| | 3 | 8 | 249.2 |
| | 4 | 7 | 190.9 |
| | 5 | 7.5 | 180.6 |
| **.055 Dia** | | | |
| Ambient | 6 | 9 | 684.6 |
| | 7 | 8 | 703.7 |
| | 8 | 8 | 573.6 |
| | 9 | 8 | 780.4 |
| | 10 | 8 | 603.9 |
| **.063 Dia** | | | |
| Ambient | 11 | 8 | 594.8 |
| | 12 | 7 | 823.1 |

UNCLASSIFIED

[redacted]

(C) Varying Pellet-Density and Percent CuSO4 using .018 Diameter Music Wire.

UNCLASSIFIED

| 20/80 mix (20% $CuSO_4$) | Delay Time Min. | Delay time Min. |
|---|---|---|
| Loading pressure | 20,000 psi | 30,000 psi |
| Ambient | 196.4 | 302.0 |
| | 162.2 | 176.0 |
| | 147.9 | 147.0 |
| | 128.9 | |
| | 121.2 | |
| 35°F | 414.4 | 309.0 |
| | 375.4 | 309.0 |
| | 341.6 | 223.0 |
| 50/50 mix | | |
| Ambient | 95.3 | 58.1 |
| | 31.2 | 52.0 |
| | 31.1 | 43.0 |
| | 28.7 | 37.0 |
| | 26.4 | 31.8 |
| Retest at ambient @ 30,000 psi only | | 33.3 |
| | | 32.2 |
| | | 28.2 |
| | | 28.1 |
| | | 27.7 |
| 35°F | 79.0 | 62.4 |
| | 65.9 | 49.8 |
| | 51.5 | |
| Retest at ambient @ 30,000 psi only | | 50.2 |
| | | 43.6 |
| | | 39.5 |

UNCLASSIFIED

[redacted]

(U) Conclusions:

1. The dry chemical delay is a feasible system.
2. The indications are that the release system should be improved, wire is sticking.
3. This system lends itself to sea water actuation.
4. This delay has good reliability in that it always fires.

UNCLASSIFIED

(C) IV. Delay Testing Summary

In comparing the $\frac{\bar{x} @ 35^{o}F}{\bar{x} @ 77^{o}F}$ ratios of the testing using polyacrylamide, x-1 and x-2 wet chemical, and dry chemical delays is as follows:

| $\frac{\bar{x} @ 35^{o}F}{\bar{x} @ 77^{o}F}$ | Description |
|---|---|
| 1.55 | Dry Chem - 50/50 mix @ 30k |
| 1.76 | Dry Chem - 20/80 mix @ 30k |
| 2.12 | Dry Chem - 50/50 mix @ 20k |
| 2.55 | Dry Chem - 20/80 mix @ 20k |
| 2.83 to 5.63 | Acrylamide |
| 4.49 | Wet Chemical - X-2 |
| 5.15 | Wet Chemical - X-1 |

(U) More work needs to be expended as to the reasons why the dry chemical delay is so much better than the wet chemical delay with respect to temperature differential.

UNCLASSIFIED

(U) V. Felt Metal

It was found that although the felt metal (product of Huyack Metals) worked as a solution feeder its rate of feed varied due to surface tension. To eliminate this a preconditioning treatment was developed as follows:

One half hour soak in mixture of 1/3 ethylene glycol and 2/3 water distilled.

Allow felt metal to drain on absorbent paper for 1/2 hour.

Then dry for 4 hours in 90°C oven.

Store in a dry box.

UNCLASSIFIED

## VI. Polyacrylamide Manufacture

(U) After evaluating 24 batches (figure 3, 4, 5, 6 & 7) of polyacrylamide (see test 1 & 2 on following pages), it was decided that the following concentrates are to be used:

50% monomer

100 rad dose

5,000 total dose

Ice bath

1¼" O.D. glass tubing - 1.115 I.D.

2' 0" length

50 lb. batches

(U) The above concentrations were selected from the standpoint of both polyacrylamide growth and consistency after being subjected to water for 24 hours.

(U) Also, this concentrate was selected because of its relative ease and repeatability of manufacture.

(U) See Appendix B for Unidynamics Research reports on the manufacture and testing of polyacrylamide.

(C) Indications are that a high rad total dose of polymerization will yield polyacrylamide with a low growth rate. For example in Test #1, Batches IX, XVII, and XVIII all had very low growth rates and all were polymerized with a total dose of 20,000 rads.

UNCLASSIFIED

UNCLASSIFIED

(U)

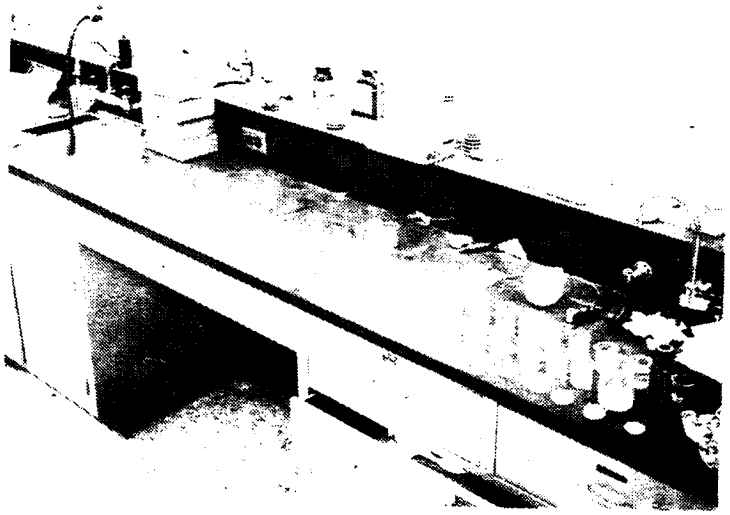


Figure 3

(U)

Figure 4

UNCLASSIFIED

(U)

Figure 5

UNCLASSIFIED



(U)

Figure 6

(U)

Figure 7

UNCLASSIFIED

Polyacrylamide Growth Testing - 24 Batches

Test #1 - Distilled Water

(C)

Conditions: Ambient temperature, polyacrylamide cut into cubic configuration and immersed into small beakers with measured amount of water (see Fig. 3, 4, 5, 6, & 7).

| Batch No. | Start Size Inches | Start Volume In.Cu. | 24 Hours Size Inches | 24 Hours Volume In.Cu. | 24 Hours Volume Incr.* | 96 Hours Size Inches | 96 Hours Volume In.Cu. | 96 Hours Volume Incr.* |
|---|---|---|---|---|---|---|---|---|
| I | .200x.200x.115 | .0046 | 1.125x1.0 x.5625 | .632 | 13.7k | 1.28 x1.0 x.625 | .800 | 19.9k |
| II | .175x.165x.130 | .0038 | 1.06 x .875x.625 | .580 | 15.2k | | | |
| III | .200x.200x.125 | .005 | 1.125x1.1875x.625 | .834 | 16.7k | | | |
| IV | .210x.175x.130 | .0048 | 1.125x1.0 x.625 | .703 | 14.6k | | | |
| V | .185x.170x.135 | .0042 | .9375x.875x .625 | .513 | 12.2k | 1.125x1.0 x .750 | .844 | 20.1k |
| VI | .175x.165x.120 | .0035 | .9375x .875x .5625 | .461 | 13.2k | 1.125x .9375x .625 | .660 | 18.85 |
| VII | .200x.180x.140 | .005 | 1.06 x1.0 x .750 | .795 | 15.9k | | | |
| VIII | .190x.175x.125 | .004 | 1.06 x1.06 x .5625 | .632 | 15.8k | | | |
| IX | .170x.160x.125 | .0034 | .875x .6875x .5 | .300 | 8.8k | | | |
| X | | | | | | | | |
| XI | .210x.190x.150 | .006 | 1.250x1.1875x.8125 | 1.210 | 20.1k | 1.375x1.219x .9375 | 1.571 | 26.2k |
| XII | .200x.205x.125 | .005 | 1.250x1.1875x.625 | .928 | 18.6k | | | |
| XIII | .200x.190x.125 | .0048 | 1.1875x1.0 x .59 | .700 | 14.6k | | | |
| XIV | .210x.155x.120 | .0039 | 1.094x1.0 x .625 | .684 | 17.5k | 1.125x1.0 x .656 | .738 | 18.9k |
| XV | .220x.190x.110 | .0046 | 1.250x1.0 x .594 | .743 | 16.2k | | | |
| XVI | .200x.160x.130 | .0042 | 1.1875x.875x.656 | .682 | 16.2k | | | |
| XVII | .215x.190x.115 | .0047 | .9275x .875x .5 | .410 | 8.7k | | | |
| XVIII | .190x.185x.125 | .0044 | .875x.875x.5625 | .430 | 9.8k | | | |
| XIX | .200x.175x.130 | .0045 | 1.125x1.0 x .625 | .703 | 15.6k | 1.1825x1.0 x .6875 | .816 | 18.1k |
| XX | .140x.140x.120 | .0023 | 1.0 x .875x .5625 | .492 | 21.4k | | | |
| XXI | .210x.210x.125 | .0055 | 1.0 x .969 x .5 | .485 | 8.8k | | | |
| XXII | .200x.190x.115 | .0044 | 1.06 x1.06 x .625 | .702 | 15.9k | 1.25 x 1.1875x.750 | 1.113 | 25.3k |
| XXIII | .220x.760x.130 | .0046 | 1.0 x .625 x .5 | .313 | 6.8k | 1.094x .75 x .4375 | .359 | 7.8k |
| XXIV | .170x.160x.120 | .0033 | .875x .875x .625 | .479 | 14.5k | | | |
| 4 | .225x.210x.125 | .006 | 1.0 x 1.0 x .5 | .500 | 8.3k | | | |
| 5 | .250x.180x.125 | .0056 | 1.1875x.875x .5 | .520 | 9.3k | | | |

* From Original

Batches 4 & 5 are from previous work.

UNCLASSIFIED

UNCLASSIFIED

UNCLASSIFIED

(C)

Test #2 - Sea Water

Conditions: Same as Test #1

| Batch No. | Start Size Inches | Start Volume In.Cu. | 24 Hours Size Inches | 24 Hours Volume In.Cu. | 24 Hours Volume Incr. | 96 Hours Size Inches | 96 Hours Volume In.Cu. | 96 Hours Volume Incr. |
|---|---|---|---|---|---|---|---|---|
| I | .225x.210x.120 | .0057 | .4375x.4375x.250 | .0488 | 8.4 | .469x.4375x.281 | | |
| II | .200x.185x.130 | .0048 | .375x.406x.250 | .038 | 7.9 | | | |
| III | .210x.170x.125 | .0045 | .469x.375x.250 | .044 | 9.7 | | | |
| IV | .200x.185x.130 | .0048 | .4375x.4375x.281 | .054 | 11.2 | | | |
| V | .180x.170x.125 | .0038 | .375x.375x.250 | .035 | 9.2 | .469x.375x.281 | .049 | 12.8 |
| VI | .175x.165x.120 | .0035 | .4375x.375x.250 | .041 | 11.7 | .500x.500x.281 | .070 | 20.0 |
| VII | .205x.165x.140 | .0047 | .5 x.4375x.3125 | .068 | 14.4 | | | |
| VIII | .180x.170x.120 | .0037 | .375x.344x.281 | .036 | 9.7 | | | |
| IX | .180x.155x.120 | .0035 | .563x.375x.250 | .053 | 15.1 | | | |
| X | | | | | | | | |
| XI | .210x.175x.140 | .005 | .5625x.406x.3125 | .071 | 14.2 | | | |
| XIII | .190x.180x.125 | .0043 | .5 x.4375x.281 | .062 | 14.4 | | | |
| XIV | .220x.190x.130 | .0054 | .5 x .4375x.250 | .055 | 10.1 | | | |
| XV | .195x.180x.125 | .0044 | .5 x.375x.250 | .047 | 10.6 | .5 x.375x.406 | .076 | 17.2 |
| XVI | .195x.190x.130 | .0048 | .5 x.375x.250 | .047 | 9.7 | | | |
| XVII | .190x.160x.130 | .004 | .4375x.375x.1875 | .032 | 5.4 | | | |
| XVIII | .85x.180x.130 | .043 | .375x.375x.250 | .031 | 7.2 | | | |
| XIX | .175x.175x.135 | .041 | .375x.344x.281 | .036 | 8.7 | .4375x.375x.3125 | .051 | 12.4 |
| XX | .200x.170x.120 | .041 | .406x.344x.250 | .035 | 8.5 | | | |
| XXI | .75x.185x.125 | .040 | .4375x.4375x.250 | .048 | 12.0 | | | |
| XXII | .200x.190x.130 | .049 | .4375x.4375x.219 | .036 | 7.3 | .5 x.5 x.250 | .062 | 12.6 |
| XXIII | .200x.155x.130 | .040 | .4688x.375x.250 | .044 | 11.0 | .531x.406x.281 | .061 | 15.2 |
| XXIV | .150x.145x.125 | .027 | .375x.3125x.250 | .029 | 10.7 | | | |
| 4 | .220x.205x.125 | .056 | .5 x.4375x.250 | .055 | 9.8 | | | |
| 5 | .195x.180x.120 | .042 | .4375x.375x.219 | .036 | 8.5 | | | |

Batches 4 & 5 are from previous work.

UNCLASSIFIED

UNCLASSIFIED

VII. Geometry and Salinity Effect on Growth

(C) Variable Surface Area

Effect on growth by varying diameter with fixed length of .375 inches. All testing using sea water at ambient temperatures. (See figures 8, 9, & 10)

(C)

| Elapsed Hours | Diameter Size Inches .125 | .250 | .500 | .625 |
|---|---|---|---|---|
| 1 | .000 | .003 | .043 | .048 |
| 2 | .000 | .029 | .064 | .072 |
| 3 | .000 | .0485 | .082 | .090 |
| 4 | .001 | .0665 | .096 | .104 |
| 5 | .0155 | .0775 | .106 | .116 |
| 6 | .0295 | .0875 | .115 | .132 |
| 7 | .0400 | .0975 | - | - |
| 23 | .141 | .207 | .250 | .2625 |
| 24 | .1455 | .212 | .258 | .271 |
| 25 | .1515 | .2175 | .263 | .275 |
| 26 | .155 | .222 | .280 | .2815 |
| 27 | .161 | .2265 | .288 | .2885 |
| 28 | .166 | .2315 | .292 | .2955 |
| 29 | .171 | .2355 | .290 | .3015 |
| 30 | .176 | .241 | .294 | .302 |
| 31 | .181 | .2465 | .300 | .306 |
| 47 | .243 | .309 | .585 | .413 |
| 48 | .247 | .313 | .585 | .415 |
| | | | .585 | .417 |

(U) There is some indication that diameter size has and effect on growth rate as can be seen in the above. Possible much of the expansive force is used to overcome surface tension, in the smaller diameters.

Constant Surface Area

(U) Effect on growth with a constant diameter of .500". All testing using sea water.

UNCLASSIFIED

UNCLASSIFIED

(C)

| Elapsed Hours | Lenght, Inches .129" | .250" | .500" | .750" | 1.000" |
|---|---|---|---|---|---|
| 1 | .0495 | .0445 | .0490 | .0400 | .0265 |
| 2 | .077 | .068 | .080 | .073 | .052 |
| 3 | .093 | .087 | .101 | .094 | .0673 |
| 4 | .105 | .1025 | .122 | .105 | .0755 |
| 5 | .118 | .122 | .1285 | .1215 | .0865 |
| 6 | .136 | | | | |
| 25 | | .213 | .308 | .301 | .200 |
| 29 | | .260 | .337 | .330 | .210 |

(C) Length of test specimen seems to have little effect on growth for the .125" thru .750" lenghts, why the 1.000 lenght did not grow as fast needs further study; it is surmised that the 1.000 lenght would grow over a longer period of time.

Temperature and Salinity effect on Overall Growth

This test was performed using .200 dia x .130" lg specimens of polyacrylamide, all from batch 14 (specimens immersed in sea water).

| (C) alt Content | Ambient temp. Size After 5 hours | 35°F Size After 5 hours | Ambient temp. Size After 24 hours | 35°F Size After 24 hours |
|---|---|---|---|---|
| istilledWater | .875x.625 | very twisted approx 3/4dia | 1.000x.625 (maxgrowth) | .969x.594 |
| 1% | .5625x.500 | .532x.313 | .750x.438 | .656x.375 |
| 2% | .500x.3438 | .438x.313 | .625x.375 | .562x.344 |
| 3% | .4688x.2812 | .468x.282 | .563x.344 | .562x.344 |
| ea Water | .438x.281 | .406x.250 | .462x.312 | .469x.312 |
| 4% | .4375x.3125 | .438x.282 | .563x.344 | .531x.344 |
| 5% | .4688x.3125 | .375x.250 | .563x.344 | .500x.312 |
| 15% | .375x.2812 | .375x.250 | .500x.312 | .500x.312 |
| 30% | .375x.250 | .375x.219 | .500x.312 | .438x.281 |

Indications are that up to five hours the growth at ambient and 35°F is very much the same but at 24 hours the growth seems to be considerably greater at ambient at least up to 3% of NaCl. It should be noted that at 15% there was very little change even at 24 hours between ambient and 35°F.

UNCLASSIFIED

UNCLASSIFIED

(U)

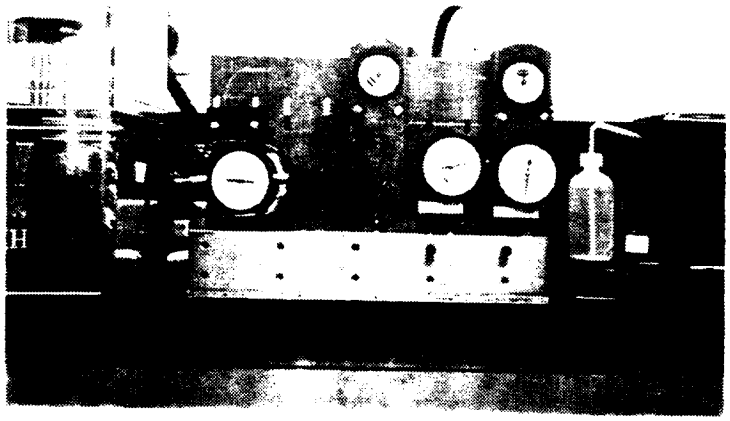


Figure 8

UNCLASSIFIED

(U)

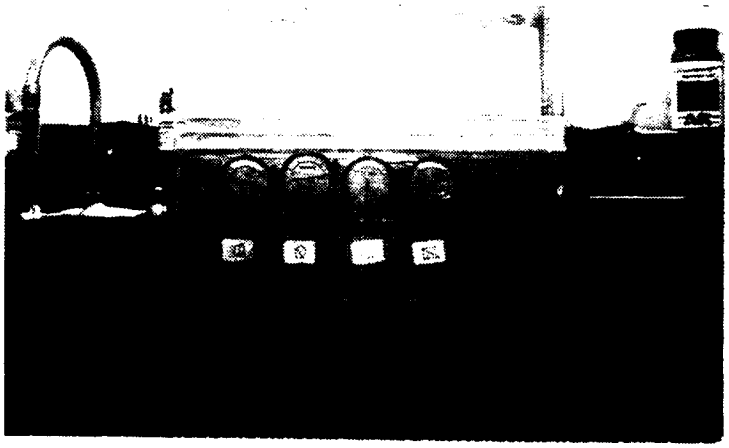


Figure 9

TEST #4

[redacted]

(C)

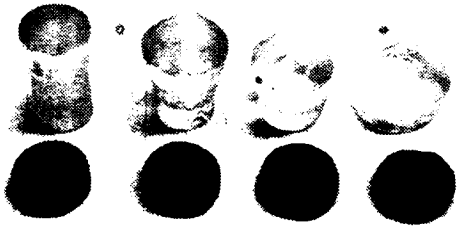


Figure 10

UNCLASSIFIED

[redacted]

[redacted]



UNC[illegible]

## VIII. Delay System Ejection Tests

(S) Three test capsules were assembled to test the ejection delay design. Capsules which were 1.7" long by 1.32" diameter were to be ejected into high pressure water using a 2.9 gram IMR4350 propellant charge. Complete design details are included in progress report #2. The test chamber and cartridge are shown in 20-1330, SK1550, and SK1588. All testing was done at 8,000 psi.

Cartridge #1:

(S) This unit leaked before reaching 8,000 psi and test was stopped.

Cartridge #2:

(S) This unit fired, ejected and the delay burned for 8 seconds then set-off the output charge. Complete success.

Cartridge #3:

(S) This unit fired and ejected but the delay column did not start. This test showed that it is possible with the present design to eject at 8,000 psi. The delay column ignition, we think, is solved by buttering both sides of the screen at the base of the delay column with an azide mix to allow for quicker ignition of the delay column. Further testing needs to be done to prove out the reliability of this system.

UNCLASSIFIED

[redacted]

UNCLASSIFIED

## IX. Sympathetic Detonation Study

### Three Unit Assembly SK-1649

(S) As reported in report #2 a three unit test was to be performed using 5 grams charge per drawing SK-1649 (see report #2). See figures 11 and 12 for assembly photos. Figures 13 and 14 were photos taken after the center charge was exploded in a 55 gallon barrel of water. The charge assembly was suspended into the center of the water with resulting damage to three charges and barrel. The adjacent charges did not fire but the resulting adjacent damage was excessive.

(S) After consultation with Sandia, it was decided to filament wrap the outer case with epoxy impregnated fiber glass (as supplied by Ferro Corporation), and the filament winding was done at UMC. Figure 15 shows the comparison of identical output charges with the unit on the right having .100 thick fiberglass wrapped on the exterior. The 5 gram charge was well contained and prevented excessive damage to adjacent charges.

(S) It is recommended that filament wrapping be used on future explosive charge designs.

### Egg-Crate Design

(S) A test package was fabricated per drawing Sk-1677 and test fired underwater at UMC (see figure 16, 17, 18, and 19).

UNCLASSIFIED

ASSIFIED

[redacted]



(S)

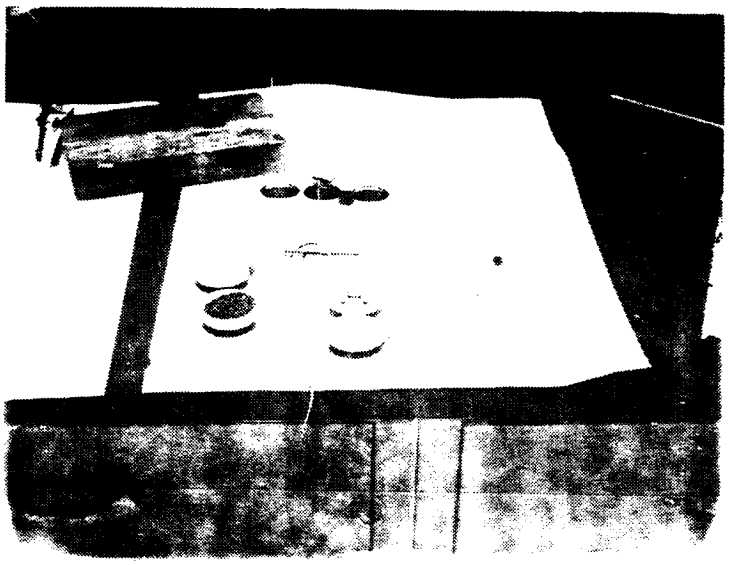


Figure 11

(S)

Figure 12

ASSIFIED

[redacted]

[redacted]

(S)

Figure 13

(S)

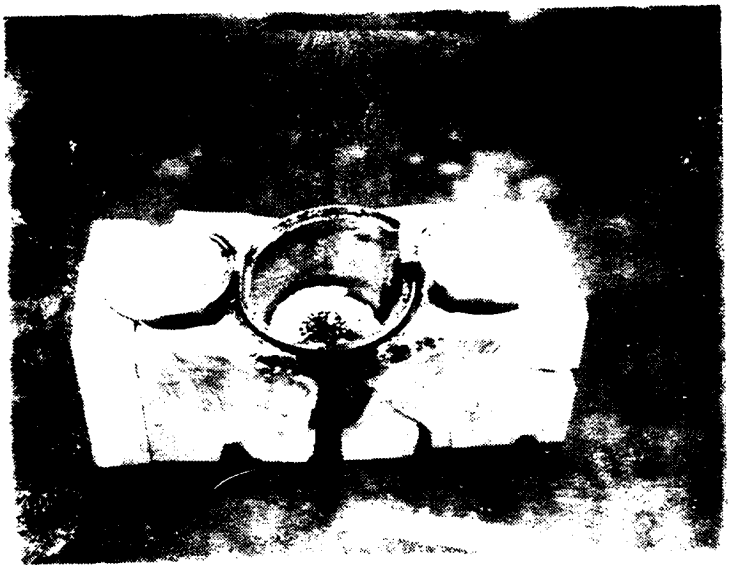


Figure 14



[redacted]

[redacted]

(C)

Figure 15



[redacted]

(S)

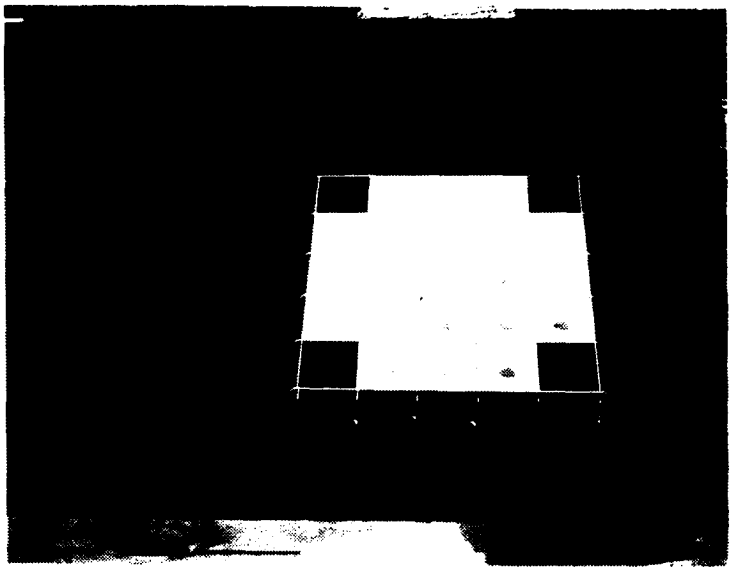


Figure 16

(S)

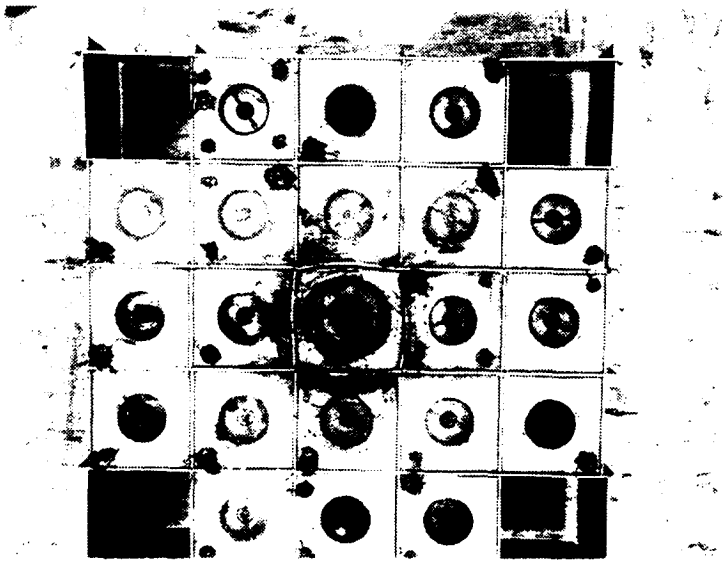


Figure 17

UNCLASSIFIED

[redacted]



(S)

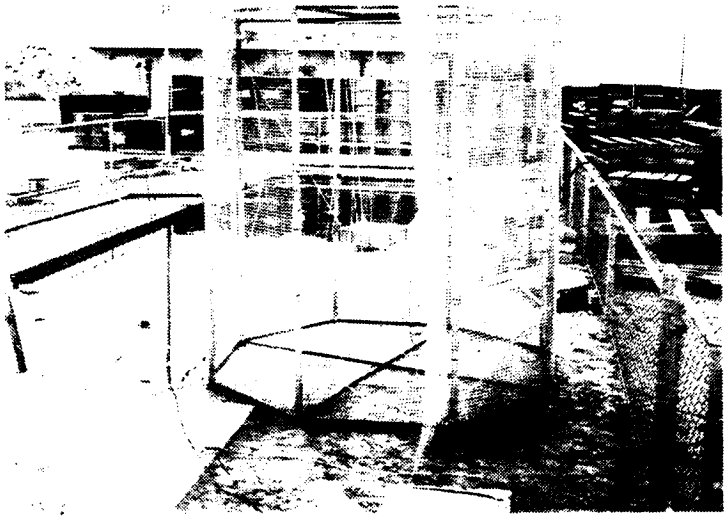


Figure 18

(S)

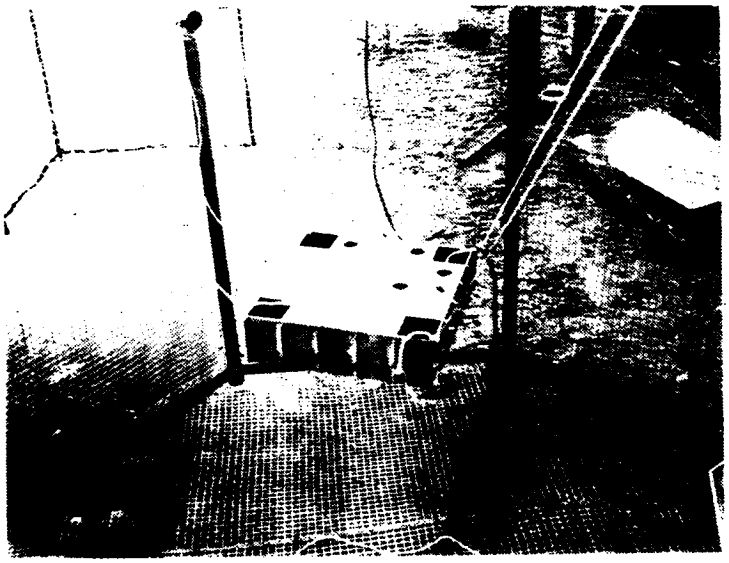


Figure 19

UNCLASSIFIED

[redacted]

UNCLASSIFIED

(S) The package contained one five gram charge in the center module of the same design as previously discussed above using the filament wrap (see SK-1649).

Sympathetic Firing of Primers

(S) After discussions with Sandia, it was decided that 2 gram charges would be sufficient for the output so a redesign was started and a package per SK-1822 using small pistol primers was fabricated. After firing the center charge, the adjacent charges fired and all three charges fired (the adjacent charges sympathetically) (see figures 20 and 21). It was suspected that the primers fired so the test was repeated per SK-1822 test #2 which tested a small pistol and a magnum primer. After firing the center charge, the small pistol primer fired but not the magnum primer (see letter of 12 June 1968 from R. Wood to P. Langdon).

(S) Package #3 (SK-1823) was fabricated after it was decided that the conial charge should give the required output. The unit was tested and the package was destroyed as shown in figure 21. It was then decided to redesign and make the center section a solid aluminum block bored out for the three charges as shown in SK-1824 using a filament wrap around the charge. A much improved design was realized as can be seen in figure 22.

(S) A further improvement was realized, and will be reported in the next report, by replacing the syntactic foam immediately around the charge with maraging steel (Almar 362 see report #2).

UNCL

UNCLASSIFIED

(S)

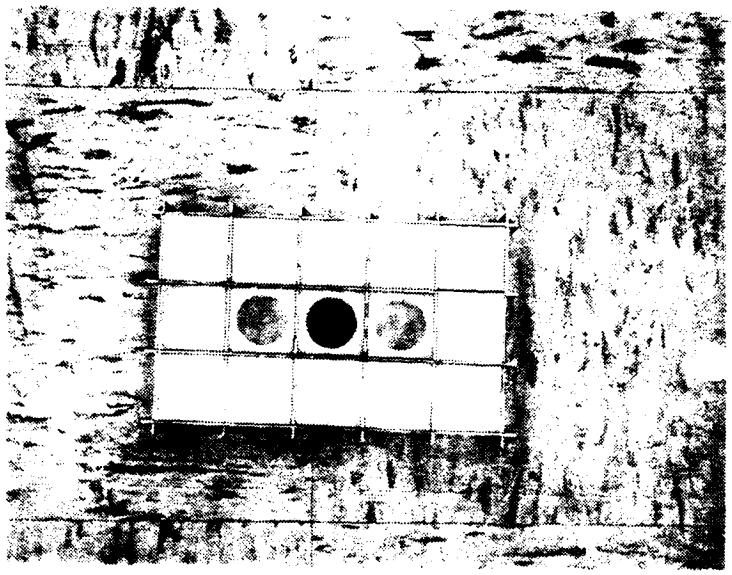


Figure 20

(S)

Figure 21

UNCLASSIFIED

UNCLASSIFIED

(S)

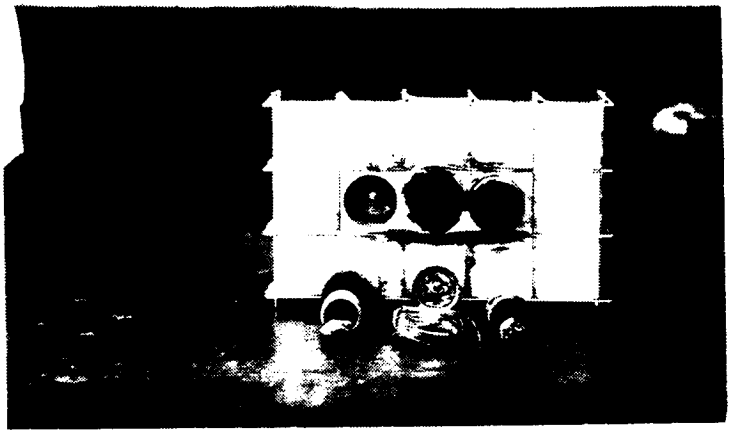


Figure 22

UNCLASSIFIED

UNCLASSIFIED

(C) On future designs it is recommeded that we use the magnum primers, maraging steel immediately around the charge, and an aluminum body to hold the modules.

UNCLASSIFIED

UNCLASSIFIED

## X. Firing of Primers Underwater

(C)

Since the environment of the system is basically water, it was discussed that if we could fire the primer in a water environment we could eliminate costly seals.

On both the polyacrylamide and the chemical delays, successful tests were performed to show that the feasibility existed for a streamlined firing pin with proper force that could fire a primer in a completely flooded system with reliable results. The new firing pin system will be discussed in the next report.

UNCLASSIFIED

[redacted]

UNCLASSIFIED

APPENDIX A

[redacted] UNCLASSIFIED

(This page is Unclassified)

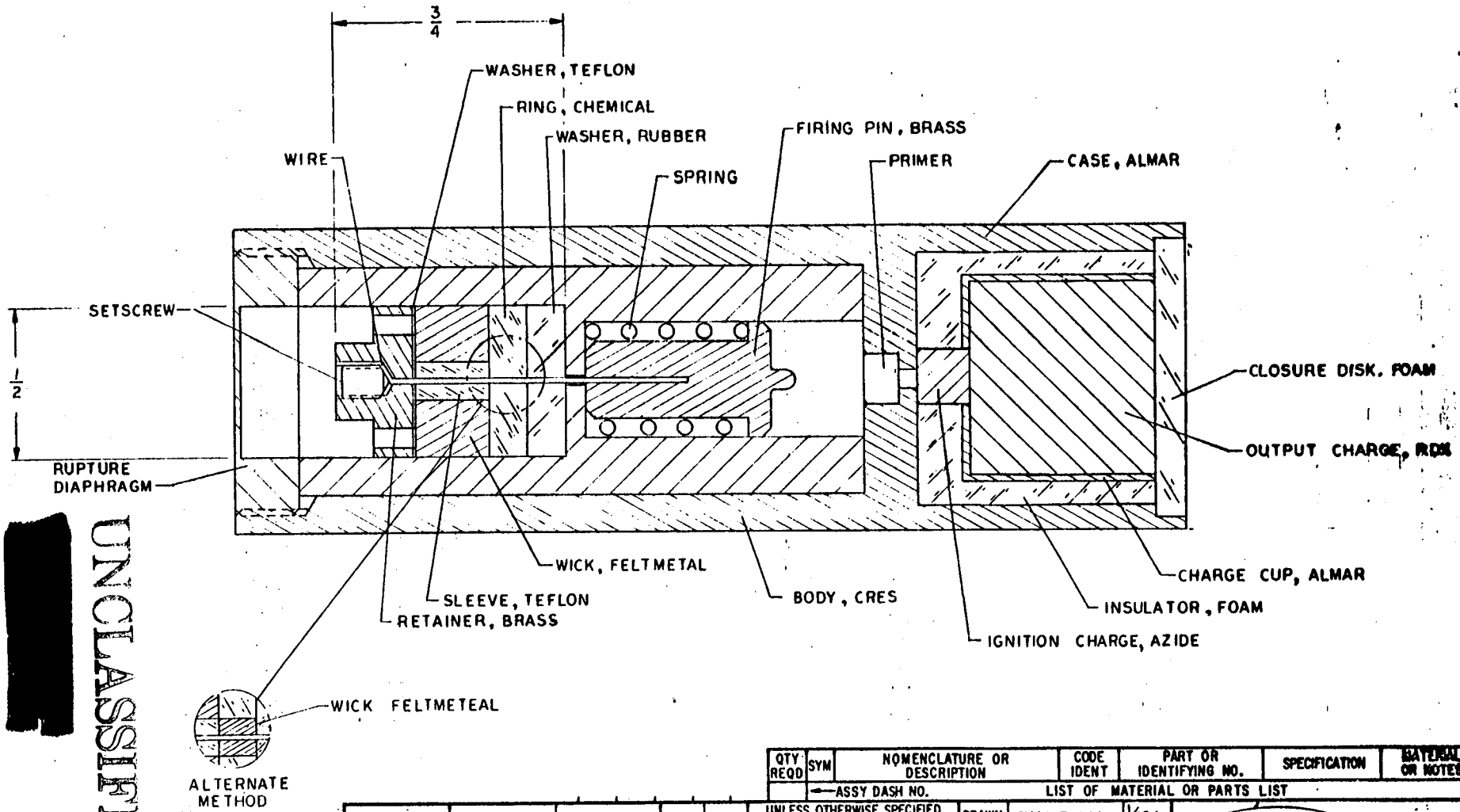
[redacted]

| LTR | DESCRIPTION | REVISED BY | DATE | APPROVED |
|---|---|---|---|---|

UNC DOCUMENT # PDS-127
COPY # 1 OF 1 COPIES

UNCLASSIFIED

[redacted]




[redacted]

UNCLASSIFIED

[redacted]

| QTY REQD | SYM | NOMENCLATURE OR DESCRIPTION | CODE IDENT | PART OR IDENTIFYING NO. | SPECIFICATION | MATERIAL OR NOTES | UNIT WT | ZONE | ITEM NO. |
|---|---|---|---|---|---|---|---|---|---|
| | | ← ASSY DASH NO. | | LIST OF MATERIAL OR PARTS LIST | | | | | |

| PART NUMBER | NEXT ASSY | USED ON | NEXT ASSY | FINAL ASSY |
|---|---|---|---|---|
| | APPLICATION | | QTY REQD | |

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES
TOLERANCES ON
FRACTIONS ±1/64 | DECIMALS .XX=±.01 .XXX=±.005 | ANGLES ±2°
INTERPRET DWG IN ACCORDANCE WITH APPLICABLE STANDARDS PRESCRIBED BY MIL-D-70327

SYMBOLS
B=BULK MATL X=XPL MATL
V=VENDOR ITEM—SEE SOURCE CONTROL OR SPECIFICATION CONTROL DRAWING

| | | |
|---|---|---|
| DRAWN | CHAMPION | 1/19/68 |
| CHECK | | |
| | JMY | 1/21/68 |

UNIDYNAMICS PHOENIX DIVISION
UNIVERSAL MATCH CORPORATION
PHOENIX, ARIZONA

DELAY, DRY CHEMICAL

| SIZE | CODE IDENT NO. | |
|---|---|---|
| C | 12079 | SK-1850 |
| SCALE 4/1 | WEIGHT | SHEET 1 OF 1 |

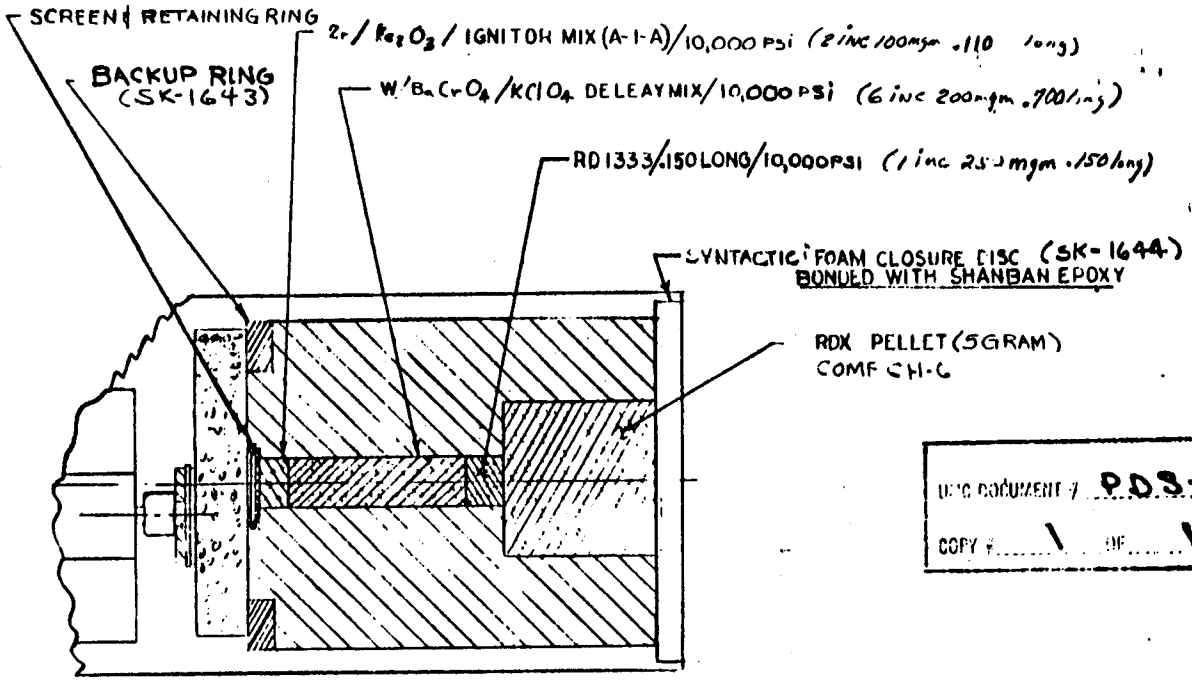
4 [redacted] DO NOT SCALE THIS DRAWING RELEASE DATE:

5 4 3 2 1

INTERPRET DWG IN ACCORDANCE WITH STANDARDS
PRESCRIBED BY MIL-D-

[redacted]

| REVISIONS | | | | |
|---|---|---|---|---|
| ZONE | LTR | DESCRIPTION | DATE | APPROVED |
| D2 | A | ADDED INCREASING DEFINITION | 26 Oct 67 | [illegible] |




UNCLASSIFIED

UNC DOCUMENT # PDS-121
COPY # 1 OF 1 COPIES

[redacted]

[redacted]

[redacted]

[redacted]

| PART NUMBER | NEXT ASSY | USED ON | NEXT ASSY | FINAL ASSY |
|---|---|---|---|---|
| | APPLICATION | | QTY REQD | |

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES ON FRACTIONS DECIMALS ANGLES ± 1/64 .XX=±.01 .XXX=±.005 ± 2°

MATERIAL:

DRAWN R. Wood DATE 16 NOV 67
CHECK

UNIDYNAMICS PHOENIX DIVISION UNIVERSAL MATCH CORPORATION P.O. BOX 2990 · PHOENIX ARIZONA

EJECTION CAPSULE

| SIZE | CODE IDENT NO. | DWG NO. |
|---|---|---|
| B | 12079 | SK - 1588 |

SCALE 2/1 WEIGHT SHEET OF

[redacted]

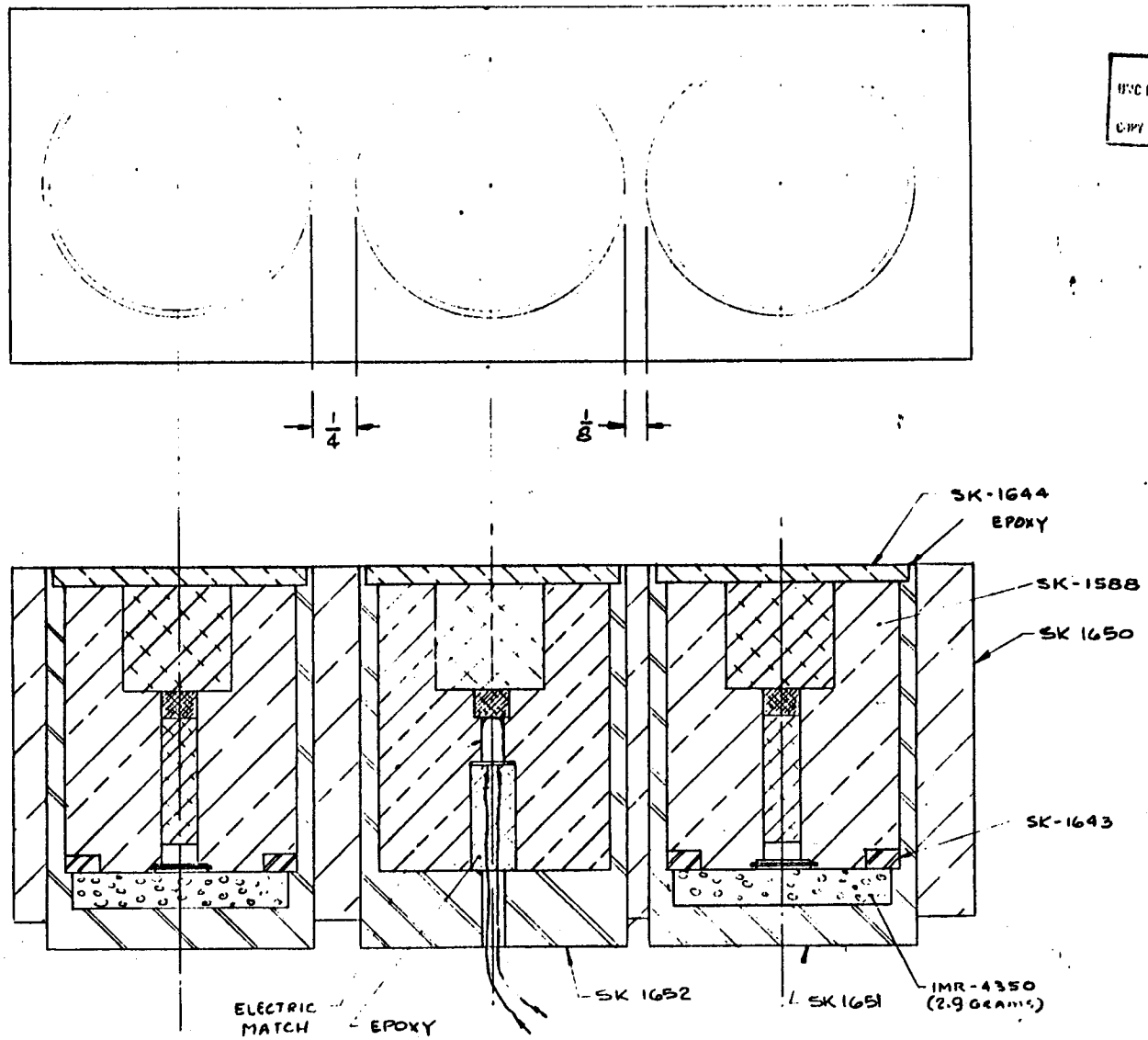
DO NOT SCALE THIS DRAWING

RELEASE DATE =

| REVISIONS | | | | |
|---|---|---|---|---|
| LTR | DESCRIPTION | REVISED BY | DATE | APPROVE |

[redacted]

USC DOCUMENT P DS-128
COPY 1 OF 1 COPIES




UNCLASSIFIED

UNCLASSIFIED

[redacted]

[redacted]

[redacted]

| PART NUMBER | NEXT ASSY | USED ON | NEXT ASSY | FINAL ASSY |
|---|---|---|---|---|
| | APPLICATION | | QTY REQD | |

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES
TOLERANCES ON

| FRACTIONS | DECIMALS | ANGLES |
|---|---|---|
| ± 1/64 | .XX = ±.01<br>.XXX = ±.005 | ± 2° |

INTERPRET DWG IN ACCORDANCE WITH APPLICABLE STANDARDS PRESCRIBED BY MIL-D-70327

| | | |
|---|---|---|
| DRAWN | [illegible] | 11-17-67 |
| CHECK | | |

UNIDYNAMICS PHOENIX DIVISION
UNIVERSAL MATCH CORPORATION
PHOENIX, ARIZONA

TEST CONFIGURATION
DETONATION STUDY

| SIZE | CODE IDENT NO. | |
|---|---|---|
| C | 12079 | SK - 1649 |
| SCALE 2/1 | WEIGHT | SHEET 1 OF 1 |

[redacted]

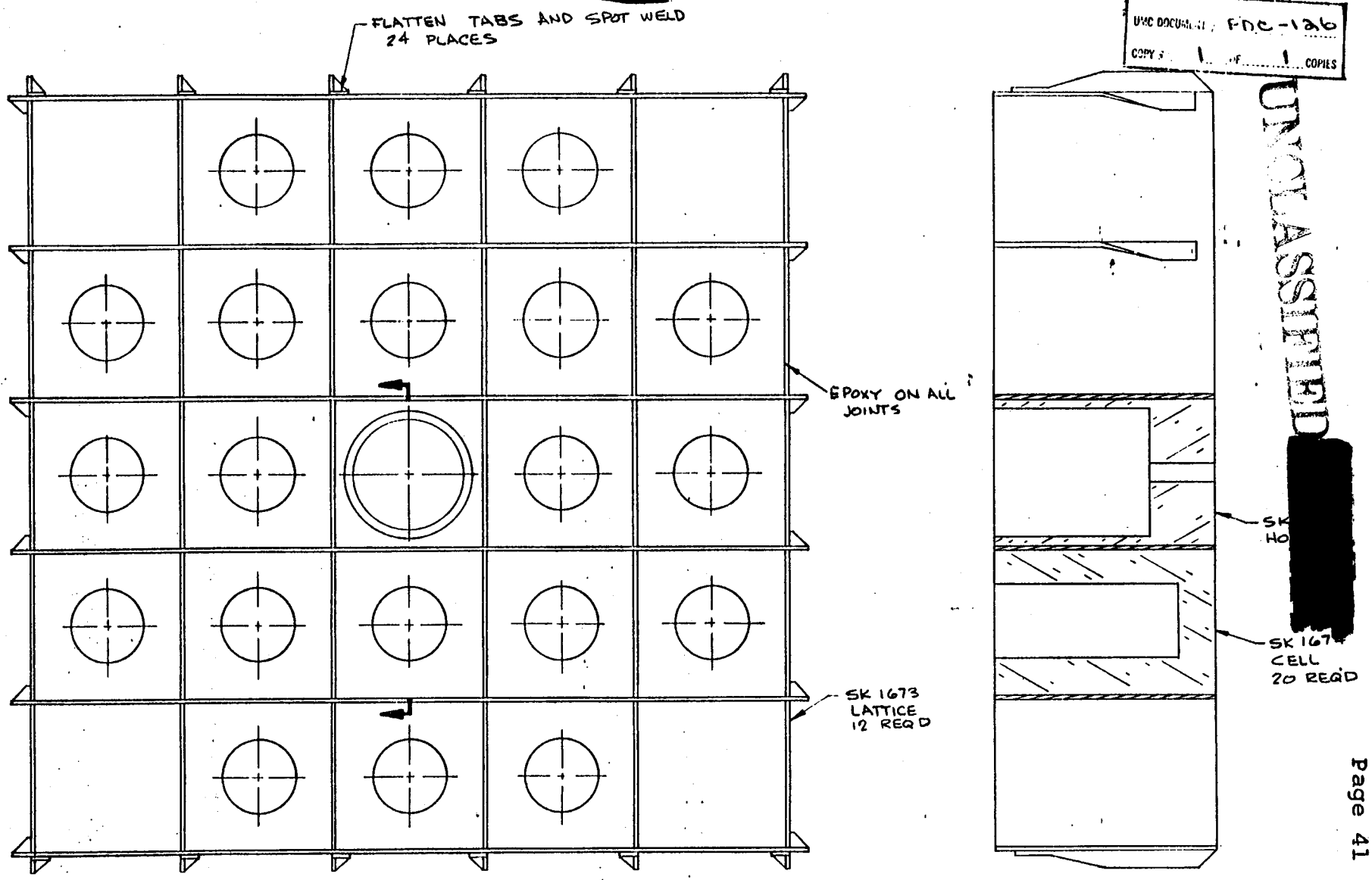
DO NOT SCALE THIS DRAWING

RELEASE DATE:

[redacted]

| LTR | DESCRIPTION | REVISED BY | DATE | APPROVED |
|---|---|---|---|---|

UNC DOCUMENT FDC-126
COPY 1 OF 1 COPIES




UNCLASSIFIED

[redacted]

UNCLASSIFIED

[redacted]

| PART NUMBER | NEXT ASSY | USED ON | NEXT ASSY | FINAL ASSY |
|---|---|---|---|---|
| | APPLICATION | | QTY REQD | |

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES
TOLERANCES ON
FRACTIONS ±1/64 | DECIMALS .XX=±.01 .XXX=±.005 | ANGLES ±2°
INTERPRET DWG IN ACCORDANCE WITH APPLICABLE STANDARDS PRESCRIBED BY MIL-D-70327

| | | |
|---|---|---|
| DRAWN | CHAMPION | 3/12/68 |
| CHECK | | |

UNIDYNAMICS PHOENIX DIVISION
UNIVERSAL MATCH CORPORATION
PHOENIX, ARIZONA

TEST CONFIGURATION FOR CHRISTMAS TREE EFFECT

| SIZE | CODE IDENT NO. | |
|---|---|---|
| C | 12079 | SK 1677 |
| SCALE | WEIGHT | SHEET OF |

DO NOT SCALE THIS DRAWING

RELEASE DATE:

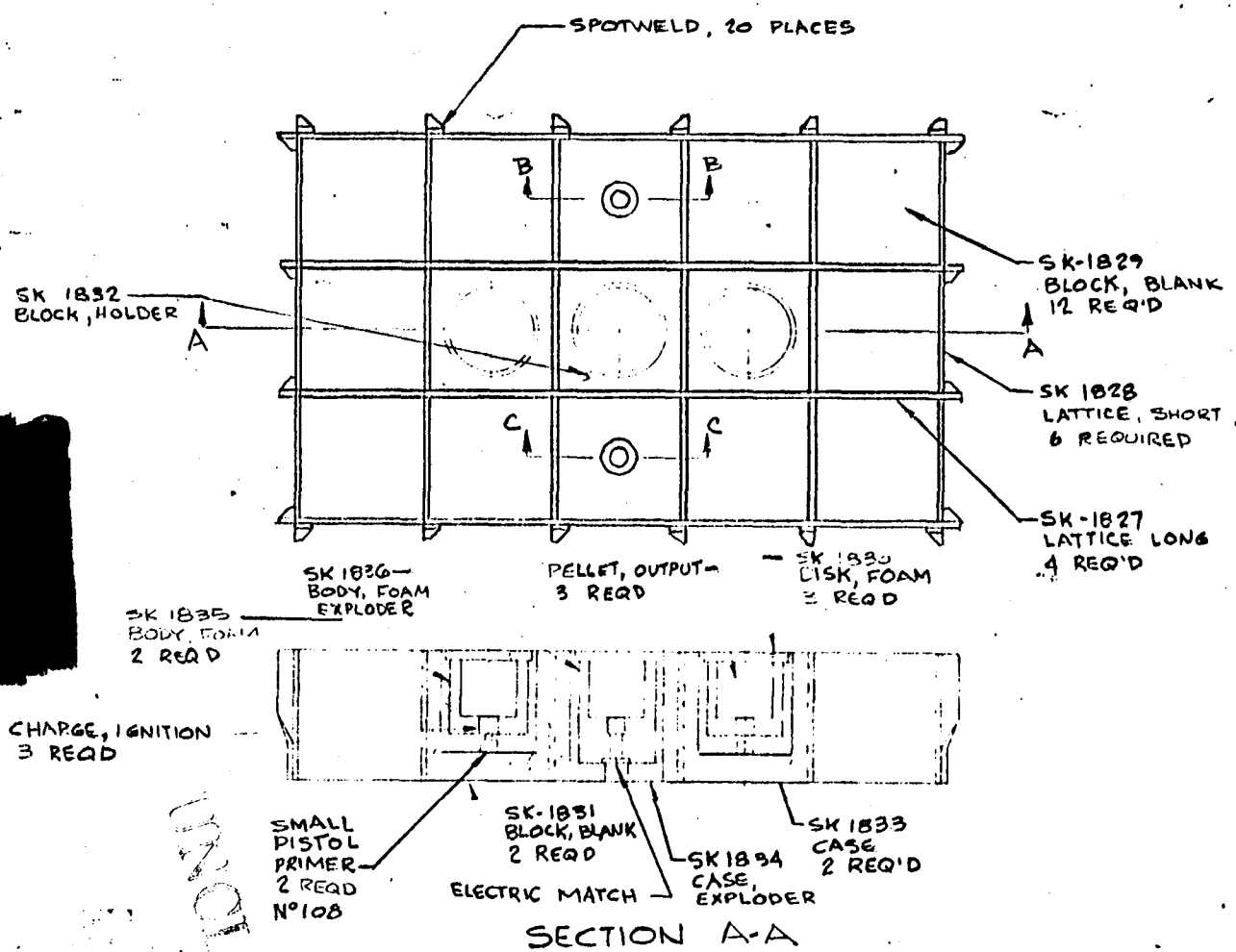
[redacted]

| LTR | DESCRIPTION | DRAWN BY | DATE | APPROVED |
|---|---|---|---|---|

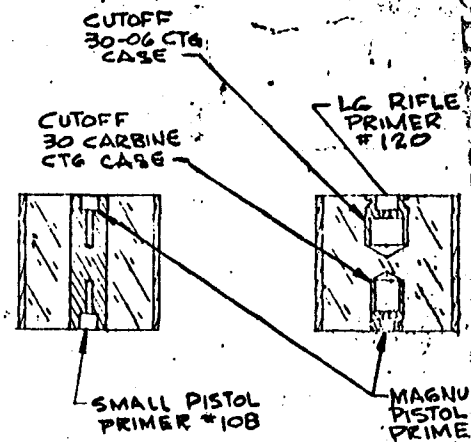
UMC DOCUMENT # PDS-119

COPY 1 OF 1 COPIES

UNCLASSIFIED




SECTION A-A




SECTION B-B

SECTION C-C

THESE SECTIONS ARE FOR N° 2 TEST ONLY

| PART NUMBER | NEXT ASSY | USED ON | NEXT ASSY | FINAL ASSY |
|---|---|---|---|---|
| | APPLICATION | | QTY REQD | |

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES
TOLERANCES ON

| FRACTIONS | DECIMALS | ANGLES |
|---|---|---|
| ± 1/64 | .XX = ±.01<br>.XXX = ±.005 | ± 2° |

INTERPRET DWG IN ACCORDANCE WITH APPLICABLE STANDARDS PRESCRIBED BY MIL-D-70327

| | | |
|---|---|---|
| DRAWN | CHAMPION | 5/14/68 |
| CHECK | | |

UNIDYNAMICS PHOENIX DIVISION
UNIVERSAL MATCH CORPORATION
PHOENIX, ARIZONA

DETONATION STUDY
TEST CONFIGURATION Nº 1 & 2

| SIZE | CODE IDENT NO. | |
|---|---|---|
| C | 12079 | SK 1822 |
| SCALE 1/1 | WEIGHT | SHEET 1 OF 1 |

DO NOT SCALE THIS DRAWING

RELEASE DATE

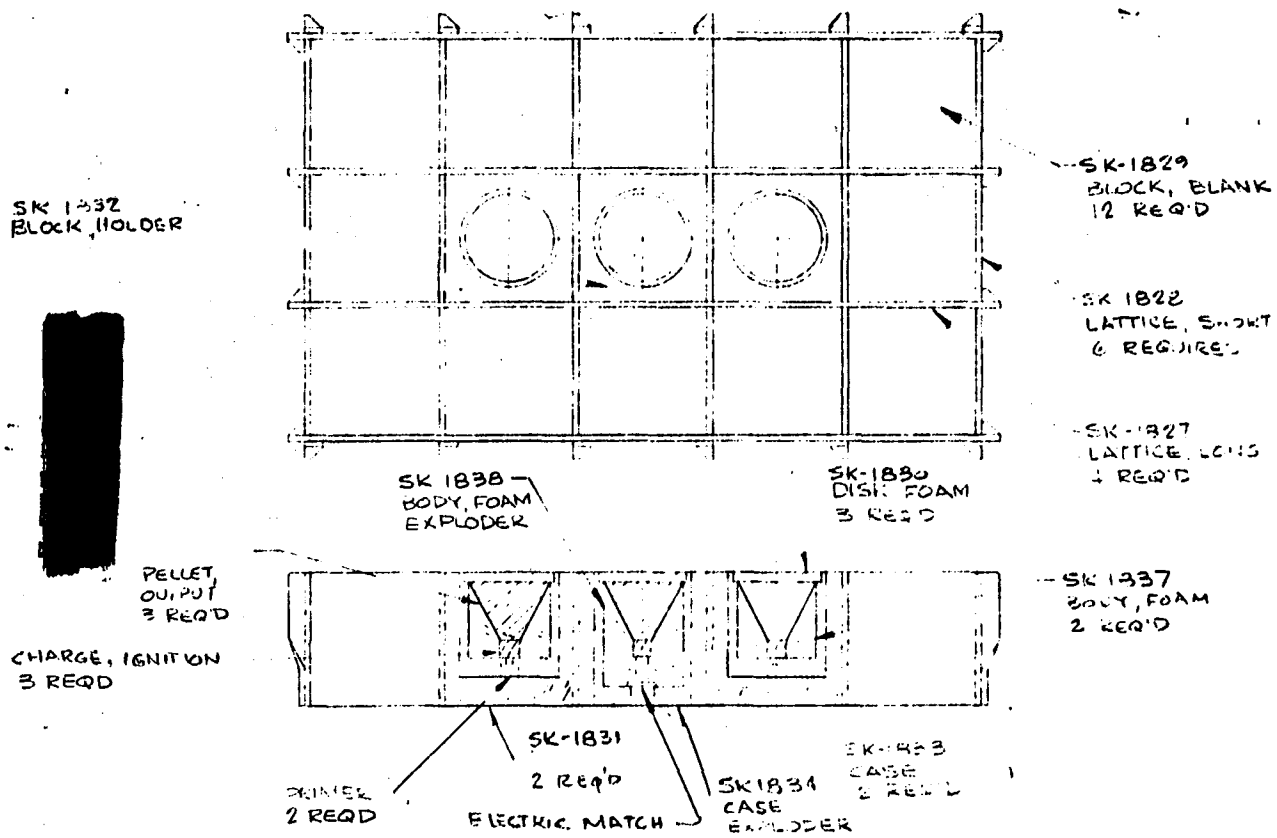
[redacted]

Page 47

| LTR | DESCRIPTION | REVISED BY | DATE | APPROVED |
|---|---|---|---|---|

UNC DOCUMENT PDS-117
COPY 1 OF 1 COPIES

[redacted]




[redacted]

UNCLASSIFIED

UNCLASSIFIED

[redacted]

[redacted]

| PART NUMBER | NEXT ASSY | USED ON | NEXT ASSY | FINAL ASSY |
|---|---|---|---|---|
| | APPLICATION | | QTY REQD | |

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES
TOLERANCES ON
FRACTIONS $\pm\frac{1}{64}$ | DECIMALS .XX = ±.01 .XXX = ±.005 | ANGLES ±2°
INTERPRET DWG IN ACCORDANCE WITH APPLICABLE STANDARDS PRESCRIBED BY MIL-D-70327

| | | |
|---|---|---|
| DRAWN | CHAMPION | 5/13/68 |
| CHECK | | |

UNIDYNAMICS PHOENIX DIVISION
UNIVERSAL MATCH CORPORATION
PHOENIX, ARIZONA

DETONATION STUDY
ASSY TEST Nº 3

| SIZE | CODE IDENT NO. | |
|---|---|---|
| C | 12079 | SK 1823 |
| SCALE 1/1 | WEIGHT | SHEET 1 OF 1 |

[redacted]

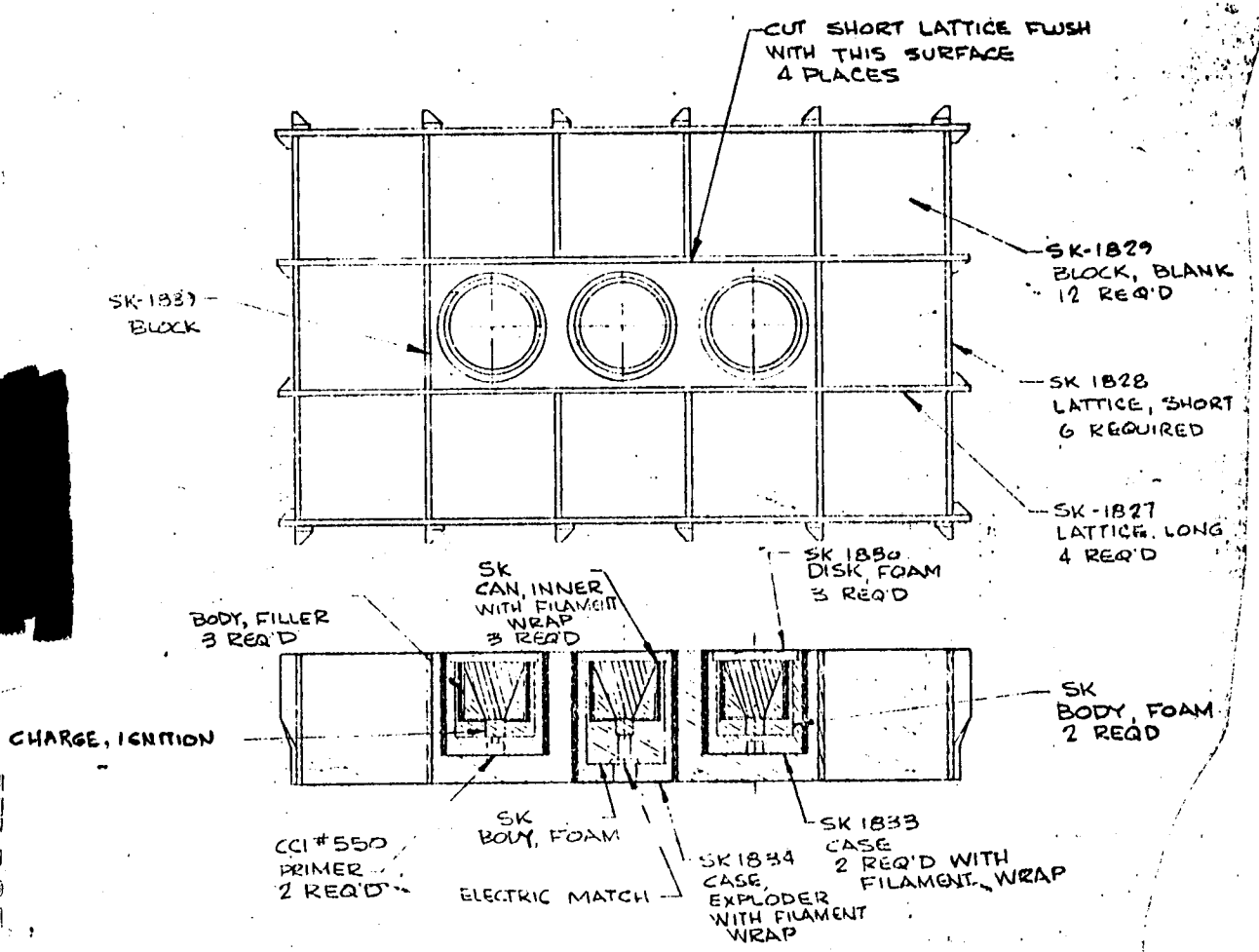
DO NOT SCALE THIS DRAWING

RELEASE DATE

[redacted]

| LTR | DESCRIPTION | REVISED BY | DATE | APPROVED |
|---|---|---|---|---|

UNIC DOCUMENT # PDS-118
COPY # 1 OF 1 COPIES




[redacted]

UNCLASSIFIED

[redacted]

Page 44

[redacted]

[redacted]

| PART NUMBER | NEXT ASSY | USED ON | NEXT ASSY | FINAL ASSY |
|---|---|---|---|---|
| | APPLICATION | | QTY REQD | |

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES
TOLERANCES ON
FRACTIONS $\pm\frac{1}{64}$ | DECIMALS .XX = ±.01 .XXX = ±.005 | ANGLES ±2°
INTERPRET DWG IN ACCORDANCE WITH APPLICABLE STANDARDS PRESCRIBED BY MIL-D-70327

| | | |
|---|---|---|
| DRAWN | CHAMPION | 5/13/68 |
| CHECK | | |

UNIDYNAMICS PHOENIX DIVISION
UNIVERSAL MATCH CORPORATION
PHOENIX, ARIZONA

DETONATION STUDY
ASSY TEST Nº 4

| SIZE | CODE IDENT NO. | |
|---|---|---|
| C | 12079 | SK-1824 |

SCALE 1/1 | WEIGHT | SHEET 1 OF 1

[redacted]

DO NOT SCALE THIS DRAWING

RELEASE DATE

APPENDIX B

UNCLASSIFIED

(U) PRELIMINARY INVESTIGATIONS OF GAMMA RADIATION INDUCED POLYACRYLAMIDE PERTINENT TO CONTRACT C77-736

Prepared by:

R. B. Heath

Reviewed by:

R. L. Curtin

UNCLASSIFIED

(U) As part of program C77-736, an investigation of radiation polymerized polyacrylamide was performed. Originally the plan called for the study of three aqueous concentrations of acrylamide monomer (40, 50, 60% w/w) each radiated at three dose rate levels (100, 50, 25 Rad/min) and each in turn exposed to total doses of 5K, 10K, 20K Rads. The polymer products thus formed were to be evaluated with regard to some of their chemical and physical properties before and after oven drying. The dried product was then to be released to engineering for "in usage" evaluation. With a few exceptions, this work has been completed as planned with some twenty-four samples of dried polymer submitted for engineering evaluation.

(U) The acrylamide used in these studies was from a single lot obtained from Eastman Organic Chemicals (Catalog #5521). Samples were prepared for irradiation by dissolving accurately weighed amounts of monomer in weighed quantities of freshly boiled distilled water. The water was freshly boiled to remove as much dissolved gas ($O_2$; $CO_2$, etc.) as possible. Since the dissolution of the monomer is endothermic, warm water or gentle warming to facilitate dissolution could be tolerated. The solutions were prepared in the needed quantities as shortly as possible before irradiation. The solution was then placed in the reaction vessel, usually a 10-16 inch length of 1.155 ID glass tubing sealed the bottom with a neoprene stopper. Nitrogen was then bubbled through this solution for approximately

(U) one hour to further remove dissolved oxygen. A stopper with a stop cock was placed in the tube and as much as possible of the remaining dissolved gases were removed by vacuum. The sample was then placed in the proper position in source chamber and irradiated. Removal of the oxygen is necessary since the polymerization will not take place in the presence of $O_2$ and induction times (time from beginning of irradiation until polymerization begins) are proportional to the amount of $O_2$ present in the solution and reaction vessel at the beginning of irradiation. When possible the polymerization was monitored by measuring the temperature changes with a thermocouple and millivolt recorder.

(U) Immediately upon removing the wet polymer rod from the reaction vessel it was sampled and analyzed for monomer content. Samples for the analysis were taken by cutting thin cross sectional discs of the material and further dividing these as finely as possible. Triplicate samples from each polymer rod weighing approximately 0.5 grams each were analyzed by a bromate-bromide titration. The remainder of the rod was tagged and dried to constant weight in an oven at 105-110°C.

(U) After drying, physical measurements were taken and a monomer analyses (in triplicate) on fine lathe turnings from selected samples were performed. Data compiled in these studies is summarized in Table 1.

(U) It is noted by the examination of the tabulated data that no completely satisfactory samples of polymer were produced using the 60% aqueous monomer solution within the range of conditions covered in these experiments. Samples made with this concentration were consistently more highly colored. On drying the polymer rods were badly distorted by uneven shrinkage. Analysis of the monomer content of the wet polymer rod revealed a higher occurrence of incomplete polymerization. Although further investigation might reveal the contrary, within the range of dose conditions covered by these experiments the practical concentration limit of the aqueous monomer apparently lies somewhere below 60%.

(U) It was also found that in irradiating the quantities used in these experiments no totally satisfactory material was produced at dose rates of 100 and 50 Rads without some temperature control. Those materials irradiated under ambient conditions were unsatisfactory mainly because of entrapped bubbles caused by high internal temperatures generated by the exothermic polymerization reaction. Marginally acceptable samples were produced with the 40 and 50% monomer solutions at dose rates of 25 Rad/min mainly because the maximum temperature during polymerization was fairly low.

(U) Reduced radiation dose rate did not seem to be the answer to the problem. The existence of a lower limit for dose rate

(U) seemed to be indicated by the results of irradiating 60% solutions at 20 Rad/min and total dose of 20K Rad. With these solutions, 25 Rad/min was sufficient to cause bubble formation while at 20 Rads/min polymerization was incomplete and non-uniform even at total doses of 20K Rads.

(U) Several good samples of polymer were produced at dose rates of 100 Rads by controlling the temperature rise during irradiation. Two methods were tried, intermittant radiation and an ice-water bath. In the case of the intermittant dose the radiation of the polymer was interrupted when the temperature reached approximately 70°C. When the sample had cooled to 50°C, the radiation was resumed. On the fifth radiation interval the temperature failed to reach the 72°C temperature and the radiation proceeded normally. The polymer thus formed had good color and consistency with no bubbles present. However, the presence of minute flakes of material dispersed throughout the material was noted. These flakes appeared to be small crystal like particles, but could possibly be small fractures caused by the alternate cooling and heating of the material.

(U) Samples of irradiated material cooled by an ice bath during polymerization were, with the exception of the one sample made from a 60% monomer solution, satisfactory. The samples were clear and of good color. They were virtually free of bubbles and were fairly uniform in appearance on drying.

(U) Time, temperature curves recorded during the irradiation of most samples indicated that polymerization was virtually complete at the 5000 Rad total dose level. However, chemical analysis of monomer content of the wet polymer rod indicated that this dose level was perhaps marginal. In instances where the monomer content of the wet polymer rod was significant, the monomer content after drying to constant weight at 105+10°C was appreciably reduced by heat induced polymerization of the residual monomer. In samples adjudged "good" in the evaluation, the monomer content of the dried rod was virtually nil. The effects, if any, of varying amounts of this heat induced polymer on the chemical and physical properties of the dried rod was not studied, but perhaps the possibility should be noted.

(U) The method for determining residual monomer content was developed by some modification of the method used by Collinson, Dainton & McNaughton.[1] See notebook pages N60633-N60643 for details of this work.

(U) Evaluation of the polymer samples on the basis of the residual monomer in the wet rod shows that the most consistent low results were obtained by irradiation of the 50% monomer solution. With the exception of one sample (No. XIII) which was irradiated

---

1. Trans. of the Faraday Society, Vol. 53, April 1957, pages 476-488. The Polymerization of Acrylamide in Aqueous Solution. Part 1 the X and $\gamma$ Initiated Reaction.

(U) in a horizontal position, no results of over 2.0% monomer were obtained. On drying at 105-110°C, however, the monomer content was insignificant in all cases.

(U) Density measurements of the dried polymer were performed by liquid displacement methods (weight/vol. liquid displaced). Accuracy was somewhat limited due to entrapped bubbles, etc., but with a few exceptions the densities of the samples were fairly consistent. The significance of these differences with regard to final usage cannot be determined until performance data of the material in the test fixture is available.

(U) No information on the comparative molecular weights of samples was collected. Because of inherent chemical and physical nature of the polymer none of the commonly used M.W. determination methods based on viscosity, osmotic pressure, effects on solutions, etc., were applicable. It was therefore concluded that the expenditure of the time and money required to develop a suitable method would exceed that available on this program and was at this time unfeasible.

(U) Based on the data currently compiled, general conclusions as to conditions for producing the best polymer are as follows:

1. Diameter of reaction vessel - 1.15" I.D.
2. Concentration limit of aqueous solution less than 60% with best results at 40-50%.
3. Temperature control with ice bath with sufficient quantity of ice to maintain 0°C temperature of bath throughout polymerization period.

(U) 4. Minimum dose rate of 25 Rad/min. with 50 to 100 Rad/min. preferred.

5. Degassing ($O_2$) of the monomer by purging with nitrogen followed by exposing the solution to vacuum prior to irradiation.

6. Total dose slightly more than 5000 Rad to assure as near complete polymerization as possible.

7. Drying temperature 105-110°C.

UNCLASSIFIED

TABLE 1

| IRRADIATION CONDITIONS | | | | | | | VISUAL EVALUATION OF WET POLYMER ROD | | | TEST EVALUATION OF POLYMER | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample Identification | % Monomer | Dose Rate Rad/min | Total Dose | Induction Time (min) | Temp. at Start | Max. Temp Est. °C | No. Entrapped Bubbles | Color≠ | Uniformity | Amount of Useable Matl | Dia. of Wet Rod | Avg. Dia Dried Rod | Ratio D/W | % Monomer in Wet Rod | % Monomer in Dried Rod | Density g/cc | Test Position |
| I | 40 | 25 | 5000 | 10 | Amb. | 56 | Shrinkage Bubbles | 2 | Poor | 50% | 1.155 | .863 | .750 | 1.7 | N.A. | 1.26 | 1 |
| VIII | 40 | 25 | 20000 | -- | Amb. | -- | " | 2 | Poor | 50% | 1.155 | .904 | .782 | 1.4 | N.A. | 1.27 | 1 |
| VI | 40 | 50 | 5000 | 9 | Amb. | 74 | " | 2 | Poor | 50% | 1.155 | .885 | .765 | 4.5 | 0.48 | 1.24 | 3 |
| XV* | 40 | 50 | 10000 | -- | Amb. | -- | " | 2 | Poor | 50% | 1.155 | .842 | .730 | 3.0 | N.A. | -- | 12 |
| XVI | 40 | 50 | 10000 | -- | -- | -- | " | 2 | Poor | 50% | 1.155 | .835 | .725 | 0.1 | N.A. | -- | 9 |
| XXII | 40 | 100 | 5000 | -- | Ice bath | -- | Very few | 1 | Good | 90%+ | 1.155 | .850 | .735 | 6.0 | 0.1 | 1.36 | 4 |
| VII | 40 | 100 | 10000 | -- | Amb. | -- | Shrinkage | 2 | Poor | 50% | 1.155 | .902 | .78 | 1.2 | 0.45 | 1.25 | 4 |
| II | 50 | 25 | 5000 | -- | Amb. | -- | 1 large bubble | 2 | Inclusion matter | ? | 1.153 | .925 | .80 | 2.0 | N.A. | . | 1 |
| III | 50 | 25 | 10000 | 67 | Amb. | 92 | Many & Shrinkage | 2 | Poor | 50% | 1.155 | .916 | .792 | 1.5 | 0.25 | 1.15 | 2 |
| IX | 50 | 25 | 20000 | -- | Amb. | -- | Shrinkage | 2 | Fair | 50% | 1.155 | .880 | .78 | 1.9 | <0.1 | 1.41 | 2 |
| V | 50 | 50 | 5000 | 7.0 | Amb. | 92 | " | 2 | Poor | 50% | -- | -- | -- | 1.5 | N.A. | 1.3 | 1 |
| XIII* | 50 | 50 | 10000 | -- | Amb. | -- | " | 2 | Poor | 50% | 1.155 | .918 | .795 | 7.0 | 1.6 | 1.27 | 11-H |
| XIV | 50 | 50 | 10000 | -- | Amb. | -- | " | 2 | Poor | 50% | 1.155 | .922 | .80 | 1.6 | N.A. | 1.45 | 8 |
| XIX** | 50 | 100 | 5000 | 20 | Amb | 74 | None | 1 | Good*** | 100% | 1.155 | .990 | .795 | 1.2 | <0.1 | 1.28 | 4 |
| XX | 50 | 100 | 5000 | 30 | Ice bath | 56 | Very few | 1 | Good | 90% | 1.155 | .900 | .795 | 0.88 | N.A. | 1.28 | 4 |
| XXIV | 50 | 100 | 5000 | 17 | Ice bath | 30 | Very few | 1 | Good | 90% | 1.155 | .900 | .795 | 0.14 | N.A. | 1.19 | 4 |
| XXI | 50 | 100 | 10000 | -- | Ice bath | -- | Very few | 1 | Good | 90% | 1.155 | .925 | .800 | 0.11 | <0.1 | 1.28 | 4 |
| XVII* | 60 | 20 | 20000 | -- | Amb. | -- | Many | 3 | Very poor | None | large dia. | -- | -- | 8.5 | N.A. | -- | 14 |
| XVIII | 60 | 20 | 20000 | -- | Amb. | -- | Some | 3 | " | None | 1.155 | .970 | .84 | 13.3 | 1.1 | 1.44 | 13 |
| IV | 60 | 25 | 5000 | 46 | Amb. | 66 | Some + shrinkage | 3 | " | None | 1.155 | .900 | .78 | 4.8 | 0.18 | 1.16 | 1 |
| X | 60 | 25 | 20000 | -- | Amb. | -- | Shrinkage | 3 | Poor | 50% | 1.155 | -- | -- | 0.7 | N.A. | N.A. | 5 |
| XI* | 60 | 50 | 10000 | -- | Amb. | -- | " | 3 | Poor | 50% | 1.155 | .965 | .835 | 2.0 | N.A. | 1.38 | 10 |
| XII | 60 | 50 | 10000 | -- | Amb. | -- | " | 3 | Poor | 50% | 1.155 | .956 | .83 | 0.86 | 0.3 | 1.22 | 7 |
| XXIII | 60 | 100 | 5000 | 12.5 | Ice bath | 30 | Much Shrinkage | 1 | Poor | 50% | 1.155 | 1.00+.05 | .86 | 4.6 | 0.14 | 1.50 | 4 |

* Tube radiated on Horizonal isodose Line

** Intermittent dose to Control Temperature

*** No bubbles, but contained tiny flake of material which appeared crystaline.

≠ Color notations: 1. Clear & colorless 2. Slight coloration or cloudiness 3. Amber color (approx. that of beer) or cloudy

UNCLASSIFIED

UNCLASSIFIED



RECOMMENDED PROCEDURE FOR PREPARING

RADIATION POLYMERIZED POLYACRYLAMIDE

AUGUST 1968

(U) I. MATERIALS AND EQUIPMENT REQUIRED:

1. acrylamide monomer
2. distilled water
3. glass tubing (1¼" I.D. and 6-8 mm)
4. nitrogen tank and regulator
5. ice
6. ice bath container 3" x length required (2000 ml graduated cylinder)
7. neoprene stoppers to fit 1¼" tubing
8. vacuum pump
9. thermocouples and complimentary temperature recorder
10. $Co^{60}$ source capable of producing 100 Rad/min fairly uniform dose in an 18" - 20" reaction vessel

UNCLASSIFIED

## II. GENERAL INFORMATION

(U) Radiation polymerization of acrylamide must be carried out in an oxygen free system since $O_2$ is an excellent inhibitor of free-radical reactions of this type. A small quantity of oxygen may be tolerated since its main effect will be merely to lengthen the induction time (time from beginning of irradiation to initiation of polymerization) for periods proportional to the amount present.

(U) All radiation absorbed during the induction period is ineffectual with regard to the polymerization reactions. It therefore cannot be accurately included in the total dose if the induction period is significant in regard to total time irradiated. Close control of the oxygen content and resulting induction time of the irradiated material must be maintained if uniformity between lots of polymer produced is desired.

## III. PROCEDURE

(U) 1. Prepare a 50% w/w solution of acrylamide in freshly boiled distilled water. Prepare fresh solution daily in amounts required for day's production. Note: Desolution is slow and may be aided by agitating the mixture with bubbling dry $N_2$ and gentle warming ($<40^{o}C$) to compensate for the endotherm. Do not stir or shake the solution in air as this may redissolve undesired $O_2$ and $CO_2$.

2. Prepare a reaction vessel by tightly sealing one end of a 1¼" O.D. flint glass tube with a neoprene stopper. Tape stopper in place with strong tape. Fill the sealed tube with water and leak test by drawing a vacuum on the tube. If any leak occurs as evidenced by bubbles entering around the bottom stopper, the tube cannot be used.

3. Empty and dry the glass tube.

4. Fill tube with acrylamide solution. Pouring the solution into a $N_2$ flushed tube is recommended.

5. Bubble nitrogen for 45 min. to 1 hr. through the solution as vigorously as possible without blowing the solution from the tube. The tube should be filled to within 2" of the top when bubbling is completed.

6. Quickly remove the $N_2$ tube and evacuate the remaining dissolved gases by applying a vacuum to the solution for 15-30 minutes.

(U) 7. Quickly and under a stream of nitrogen, if possible, replace the vacuum apparatus stopper with one which has a "rod" thermocouple inserted through its center.

8. Center the air tight reaction vessel in the ice bath container and pack ice as tightly as possible around the reaction vessel. Fill remaining space with ice water to the height of the acrylamide level.

9. Connect thermocouple to temperature recorder (reaction is monitored by polymerization exotherm).

10. *Irradiate sample at a dose level of 100 Rad/min for 50 min. If polymerization is not initiated, as indicated by a rise in temperature, within five minutes, deoxygenation of the solution was incomplete.

11. Remove wet polymer rod from glass tube, sample for analysis**, tag and dry to constant weight at 105-110°C.

12. When dry, analyze for monomer content if required.

* - Dose level is determined by Fricke dosimetry of a solution irradiated in a similar reaction apparatus.

** - See Recommended Procedure for Sampling and Analysis of Radiation Polymerized Polyacrylamide

# RECOMMENDED PROCEDURE FOR THE SAMPLING AND ANALYSIS OF RADIATION POLYMERIZED POLYACRYLAMIDE

AUGUST 1968

## EQUIPMENT AND MATERIALS REQUIRED

(U)

1. Burette (50 ml) and stand
2. Iodine titration flasks (500 ml funnel mouthed with stoppers)
3. Analytical balance
4. Pipettes (10 ml)
5. Potassium bromate ($KBrO_3$) Reagent grade
6. Potassium bromide (KBr) Reagent grade
7. Sodium thiosulfate ($Na_2S_2O_3$-Reagent grade $\cdot$ $5H_2O$)
8. Chloroform (may be omitted if solution is not stored for prolonged period)
9. Soluble starch
10. Sulfuric acid ($H_2SO_4$ Reagent Grade)
11. Potassium iodine Reagent grade

## PREPARATION OF REQUIRED ANALYTICAL SOLUTIONS

(U)

1. Potassium bromate 0.25N standard solution -

Weigh accurately 6.960 g of dried $KBrO_3$ into a 1000 ml volumetric flask. Add excess potassium bromide KBr (30-36 g), dissolve and make to one liter. Dried $KBrO_3$ at 120°C overnight treat as primary standard.

(U) 2. Sodium thiosulfate 0.1N -

Dissolve 25.0 grams reagent grade and 1cc chloroform ($HCCl_3$) and make to one liter.

3. Starch indicator solution - standard preparation.

4. Sulfuric acid 6.0 N -

1 part $H_2SO_4$ concentrated reagent grade in 5 parts distilled water.

5. Potassium iodine 20% w/v

Dissolve 20 grams KI in distilled water and make to 100 ml volume.

(U) SAMPLING PROCEDURES

A. Wet Rod

1. Cut cross sectional disc from (preferably from near one one end) a freshly prepared wet polymer rod. Cut the disc into small cubes and weigh sample (0.5 to 1.0 g) on an analytical balance.

2. Place weighed samples in iodine titration flasks, cover with water (50-75 ml) and analyze immediately.

B. Dried Material

1. Slowly, without overheating the polymer, machine fine turnings of the polymer rod with a lathe.

UNCLASSIFIED



(U) 2. Weigh out samples (1.0 gram) into iodine titration flasks; cover with water (50-100 ml) and analyze immediately.

ANALYTICAL PROCEDURE

(U) Step 1. To samples prepared by either method above, pipette 10.00 mls of standard 0.25N $KBrO_3$ solution. At the same time prepare reagent blanks containing no polyacrylamide and treat similarly throughout remainder of procedure.

Step 2. Without shaking, add to each sample 6-10 mls of 6N sulfuric acid. Stopper the flask securely and then shake vigorously for 10-20 seconds.

Step 3. Allow samples to stand for 45 min. to 1 hour with occasional shaking.

Step 4. Fill funnel mouth around the stopper of the iodine titration flask with 20% KI solution and gently allow KI to drain into flask by partially removing the stopper. Avoid loss of $Br_2$ vapor. Repeat addition of KI once or twice and shake stoppered flask vigorously.

Step 5. Titrate liberated $I_2$ in the flask to a faint yellow with 0.1N $Na_2S_2O_3$ and add 2-5 drops soluble starch solution.

Step 6. Continue titration until colorless endpoint of solution persists for 2-3 min.

UNCLASSIFIED

(U) NOTE: Some coloration in the polymer bits may persist. This is not however enough to significantly effect results.

Step 7. Calculate % monomer in polymer as follows:

For Wet rod

$$\% \text{ monomer} - \frac{\left[\text{mls } BrO_3^- - (\text{mls } S_2O_3 \times CT)\right] (N)(M)(100)}{(W)(C)}$$

For Dry Material

$$\% \text{ monomer} \quad \frac{\left[\text{mls } BrO_3^- - (\text{mls } S_2O_3 - \times CT)\right] (N)(M)(100)}{W}$$

Where $CT = \frac{\text{mls Bromate in blank}}{\text{mls thiosulfate in blank}}$

N = normality of $BrO_3^-$ solution

M = milliequivalant weight of acrylamide (.0355)

W = weight of sample

$C = \frac{\text{weight monomer in original solution}}{\text{weight original solution}}$

INTERIM REPORT

(U) RADIATION POLYMERIZED ACRYLAMIDE

14 August 1968 - 13 September 1968

13 September 1968

(U) Work subsequent to that reported in the Interim Report of 14 August 1968 consisted of preparing and analyzing fifteen additional rods of acrylamide polymer. In accordance with the original plan, these rods were prepared individually from a new lot of monomer by operator previously unfamiliar with the process. The operator was given verbal instructions and copies of the recommended procedures for preparing and analyzing the polymer.

(U) The lot of raw material was that received from Distillation Products on Purchase Order #3173.

(U) In general this material differed from that previously prepared only in that it contained perhaps a few, but not significantly, more bubbles and had a slight amber color in the monomer solution. This discoloration was not apparent in the irradiated material and the dried rod.

(U) Results of the analyses of each individual rod in the wet state and of selected dried rods are shown in the table on the next page. This work concludes that currently scheduled. Future effort will be performed as requested by the program manager.

UNCLASSIFIED

ANALYTICAL RESULTS

| (U)Rod # | % Monomer in Wet Rod (Avg.) | % Monomer In Dried Rod |
|---|---|---|
| 1 | 5.7 | 0.8 |
| 2 | 2.8 | < 0.1 |
| 3 | 3.9 | -- |
| 4 | 2.7 | < 0.1 |
| 5 | 3.5 | -- |
| 6 | 2.8 | < 0.1 |
| 7 | 4.1 | -- |
| 8 | 2.7 | -- |
| 9 | 3.8 | -- |
| 10 | 4.2 | < 0.1 |
| 11 | 3.5 | -- |
| 12 | 3.2 | < 0.1 |
| 13 | 3.3 | -- |
| 14 | 11.5* | < 0.1 |
| 15 | 4.1 | -- |

* Analysis believed to be high and unrepresentative because of sampling at extreme upper end of rod. However, material was not submitted for LAD use.

UNCLASSIFIED