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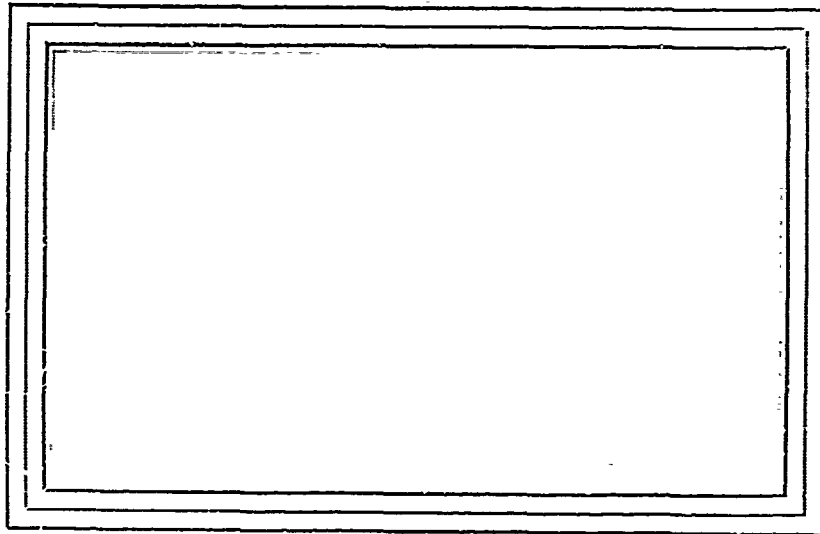


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Crane, Indiana

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BINDING PROPERTIES AND OTHER CHARACTERISTICS
OF SEVERAL POLYESTER RESIN BINDERS
USED IN PYROTECHNIC FORMULATIONS

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ABSTRACT

The binding strengths of pellets subjected to tensile and shear stresses, and the burning time and candlepower of flares pressed from compound aged 0-6 hours in increments of one hour prior to pressing were determined after curing periods of 5 and 30 days. Three formulations were used, each containing the same ratio of magnesium/sodium nitrate/binder; but, with three different polyester resin binders--Laminac 4110, Laminac 4116, and Aropcl 7720M.

The variation in physical strength, candlepower, and burning time with respect to delay time between mixing and pressing was found to be greatest for pellets and candles containing Laminac 4110. Likewise, the physical strength of pellets containing Laminac 4110 was significantly higher than the other pellets after curing 5 days; however, tests after 30 days indicated that by this time the Laminac 4116 had essentially fully cured, and now had binding properties similar to Laminac 4110. The physical strength of pellets cured at 75 - 85°F with Aropcl 7720M was considerably less than pellets containing the other binders.

All units cured for 16 hours at 150°F, and then tested after 5 days exhibited considerably higher strengths than

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pellets not subjected to an elevated temperature. However, tests after 30 days showed that for the Laminac binders, greater binding strength is obtained by curing at room temperature for the duration of the curing cycle.

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I. OBJECTIVE

The purpose of this study was to determine the binding properties of several polyester resins used in pyrotechnic compositions as a function of the polymerization time before and after pressing. Additionally, the variation in candle-power and burning time of the formulation under consideration was to be investigated.

II. EXPERIMENTAL METHOD

Composition Formulation

A pyrotechnic formulation containing 61.4% magnesium (granulation 18), 33.8% sodium nitrate (Class 2), and 4.8% binder was selected as the composition to be investigated. Three different polyester binders were utilized--Laminac 4110 and 4116 (American Cyanamid Co., Wallingford, Conn.), and Aropol 7720M (Archer-Daniels Midland Co., Minneapolis, Minn.). After mixing, the compound was stored in a closed container until pressed.

Pressing and Curing Procedure

Pellets one inch in diameter and two inches long were pressed immediately after mixing, and in increments of one hour after mixing up to a maximum of six hours. Each pellet contained two increments of 22 grams each, pressed at a dead

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load of 8,000 pounds. Twenty-four pellets were pressed immediately after mixing, and 12 during each time interval thereafter. The diameter, length, and weight of each pellet was recorded immediately after pressing, and in some cases after five and 30 days curing. All pellets were cured in a sealed can at 75 - 85°F, except for 12 pellets pressed immediately after mixing which were cured for approximately 16 hours at 150°F. The pellets were tested five and 30 days after pressing.

Three flares were pressed immediately after mixing, and during each hour thereafter, up to six hours after mixing. Each flare contained three 200 gram increments, pressed at a deadload of 22,000 pounds into a fish paper tube which was 1.96" OD and 1.75" ID. The flares were cured at 75 - 85°F, and burned after 30 days.

Testing Apparatus and Equipment

The testing apparatus and equipment used in the determination of the shear and tensile binding properties of the various resins are illustrated on the following page. The testing machine used to perform both the tensile and shear tests is shown in Illustration A. The movable jaws were clamped together to hold the cables which were connected to

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the test fixtures, and then the jaws were moved vertically apart to supply the required force. The shear and tensile test fixtures are shown in Illustrations B and C respectively. For the shear test, the pellet was placed in the test fixture and then the assembly was mounted in the test machine. In the tensile test, a pellet with an end plate attached to each end was held in the fixture by the removable pin, and then the assembly was placed in the testing machine.

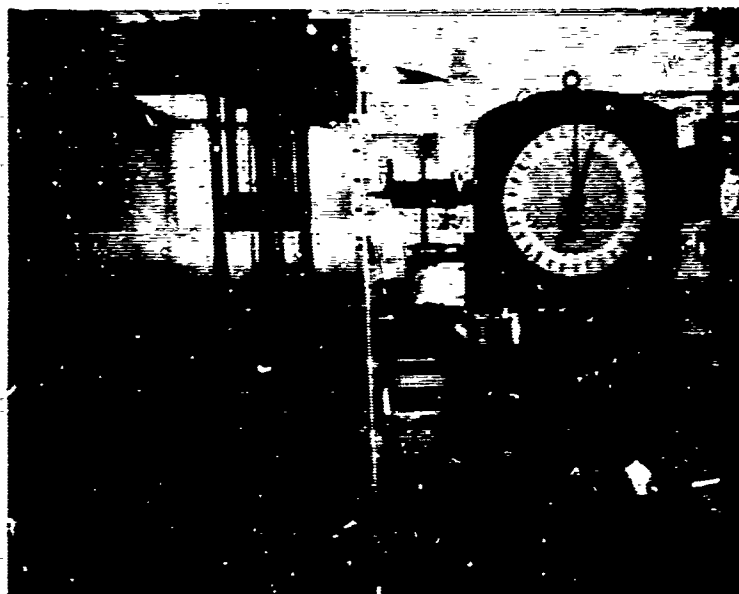
Orbond 121 was used to bond the end plates required for the tensile test to the pellets. The tensile test data for the Laminac 4116 units is rather incomplete because an inadequate amount of adhesive was used to bond the end plate to the pellet; thus, failure occurred at the plate-pellet interface rather than at the increment.

IV. DISCUSSION

Binders

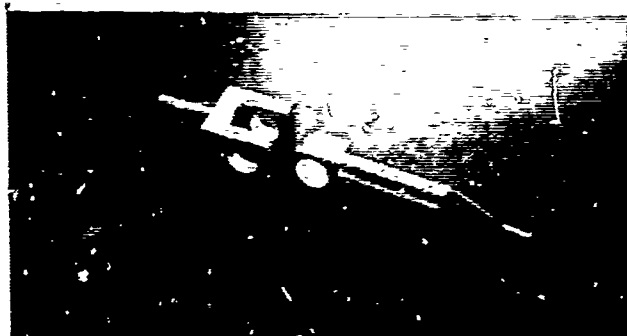
The binders selected for this investigation were ones believed to be suitable for use in pyrotechnic flare compositions, and which would essentially fully cure at temperatures between 75 - 85°F when adequately catalyzed. Cobalt Naphthenate, which promotes room temperature cure when catalyzed by methyl ethyl ketone (Lupersol DDM), is present in a

ILLUSTRATION A



Riehle Testing Machine
Mfg. by American Machine & Metals Co.
East Moline, Illinois

ILLUSTRATION B



Shear Testing Fixture

ILLUSTRATION C



Tensile Testing Fixture
with end plate

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sufficient amount in Laminac 4110 to effect a complete cure at room temperature. On the other hand, Laminac 4116 contains a lesser amount of Cobalt Naphthenate; therefore, this resin will not cure as quickly as Laminac 4110, and in some cases an elevated temperature may be required. The Aropol resin does not contain any of the Cobalt promoter, hence, the promoter must be added to obtain a room temperature cure.

Company literature indicates that the gel time for Laminac 4110 and 4116 with 1.5% Lupersol DM catalyst, is approximately 15 minutes and 35 minutes respectively, and that Aropol 7720M should gel in about 25 minutes when catalyzed by 0.5% Cobalt Naphthenate and 1.0% Lupersol DM. Although the composition was stored in closed containers between mixing and pressing, it would be suspected that the influence of the delay time between mixing and pressing upon the physical properties and burning characteristics of the pressed composition would be most noticeable in the Laminac 4110 composition. The literature also states that the tensile strength of the fully cured Laminac resins is approximately 9,000 PSI, and about 2,500 PSI for the Aropol binder.

Tensile Strength

Graph I shows a binder--binder comparison between the

tensile strength of the three formulations after five and 30 days curing, while Graph II illustrates the difference in tensile strength after five and 30 days for a given binder. As stated previously, the experimental data for the Laminac 4116 units is incomplete because the end plate-pellet bond proved to be weaker, in many cases, than the increment-to-increment bond. All of the data considered was for failures which occurred at the increment-to-increment junction. Table I gives the experimental tensile failure loads after five and 30 days for the various mix-press delay times, and Table II contains the average of the above figures for a given resin and delay time.

Considering the accuracy of the testing equipment used, and the variation in the data for a given binder and delay time, the delay time between mixing and pressing does not appear to have a significant effect upon tensile strength. The tensile strength of the Aropol pellets did seem to exhibit somewhat of a trend toward increasing as the delay time increased; however, the magnitude of the increase can hardly be judged significant. A binder-binder comparison shows that the Laminac 4110 pellets sustained a 40% greater tensile force before failure than the Laminac 4116 units, and 80% greater

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than the Aropol pellets.

On the other hand, the difference in tensile strength after five and 30 days ambient cure for a given binder was found to be very significant. The Laminac 4110 units failed at a 65% greater tensile load after 30 days than five days, the Laminac 4116 pellets increased 100% in tensile strength, and the Aropol pellets exhibited an increase of 50%. Once again, the Laminac 4110 pellets were superior to the other units, since their tensile strength was 10% and 80% greater, respectively, than the Laminac 4116 and Aropol 7720M pellets. It is interesting to note that the tensile strength of the pellets increased substantially between the five and 30 day period, and that after 30 days cure, the Laminac 4110 and 4116 pellets exhibited somewhat comparable tensile binding properties. This data agrees with the company literature for the various binders, which indicated that Laminac 4116 does not cure as quickly as 4110, and that when fully cured, the tensile strength of the two Laminac resins are basically the same, and considerably greater than for Aropol 7720M.

Shear Strength

The shear strength data is presented in Tables III and IV. Graphs III and IV give a binder-binder comparison of

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shear strengths after five and 30 day curing cycles, respectively and Graph V shows the variation in the shear strength for a given binder, resulting from a five and 30 day cure.

Referring to Graph V, it is seen that as the time from mixing to pressing increases from 0 to 6 hours, the shear strengths of the Laminac 4110 units increased rather consistently, amounting to an overall increase of approximately 200%. Such a trend was not evident after the 30 day cure in the Laminac 4110 units, nor did it exist after either five or 30 days for the other binders.

After five days cure, the pellets containing Laminac 4110 failed at about a 50% greater shear load than the other two pellet formulations. However, tests after 30 days revealed that although the Laminac 4110 had a shear strength approximately 200% greater than the Aropol units, they now failed at a 25% lower shear load than the Laminac 4116 pellets. This data substantiates that found during the tensile tests, in that although the physical properties of Laminac 4116 are inferior to Laminac 4110 after a short ambient curing cycle, as the curing period is increased, the physical properties of the Laminac 4116 are enhanced until the two binders have basically the same properties. The per cent increase in the

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shear strengths after five and 30 days was 125%, 350%, and 15% for the Laminac 4110, Laminac 4116, and Aropol resins, respectively.

Effect of 150°F Curing Cycle

Table V contains the experimental data for pellets cured approximately 16 hours at 150°F, immediately after pressing, and then stored in closed containers at 75 - 85°F until tested after five and 30 days. The average values for the above data are given in Table VI, along with the corresponding values of pellets cured at room temperature.

If the tensile and shear strengths of pellets subjected to the elevated temperature are compared to those cured at room temperature, it is seen that the elevated temperature cure has a marked effect upon the physical properties of the resins. After the 150°F cure, the Aropol units tested after five and 30 days failed at tensile and shear loads which were at least 75% and 20% greater, respectively, than the Laminac resin pellets. Referring to Table VI it is seen that the Laminac pellets cured at an elevated temperature and then tested after five days, possessed significantly greater tensile and shear strengths than pellets not subjected to the 150°F cure; however, the results of similar units tested after 30

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days indicated that binding strengths of units cured solely at 75 - 85°F, were approximately 25% greater than those cured for 16 hours at 150°F and the remaining time at 75 - 85°F.

Burning Time and Candlepower of Flares

There was found to be a significant variation in candlepower, burning time, and the emitted candlepower-seconds with respect to the delay time between mixing and pressing. A similar difference also existed between the three binders. For the flares containing Laminac 4110, 4116, and Arcopol 7720M, an overall decrease of 6%, 5%, and 3% in burning time, an increase of 14%, 8%, and 8% in candlepower, and an increase of 7%, 4%, and 4% respectively in candlepower-seconds were obtained for compound pressed between 0 - 6 hours after mixing. This experimental data may be found in Table VII. Average values are given in Table VIII and plotted on Graph VI. Generally consistent data was obtained for a given delay time and formulation.

If an average value of the emitted candlepower-seconds is calculated, it is found that the Arcopol and Laminac 4110 units are approximately equal, while the Laminac 4116 flares emitted about 13% fewer candlepower-seconds. The pressed length of flares of a given formulation were essentially the

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same; therefore, the variation in burning time is indicative of the variation in burning rate existing for the various units.

Pellet Density

No significant difference existed between the density or weight of pellets containing a given binder for the various delay times, nor between the pellets containing the three binders. Likewise, no significant change in density occurred after curing for five and 30 days. The density of the pellets was approximately 1.700 gm/cc, with less than a 3% variation between the units containing the three resins.

V. SUMMARY

Of the three resins investigated, the optimum binder for use at room temperature curing conditions appears to be Laminac 4110, when it is considered that the function of a binder is to supply physical strength to the pressed composition while at the same time impart a minimum amount of degradation to the performance of the flare. The burning performance of the Aropol flares compared favorably with the Laminac 4110 units; however, the relatively poor physical strength properties of the Aropol pellets after both five and 30 days curing, make this resin inferior to Laminac 4110.

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A comparison between the two Laminac resins indicates that after an extended curing period, the two compare favorably in physical strength; however, after only five days ambient cure, the Laminac 4110 units possessed superior strength properties. Also, the number of candlepower-seconds emitted by the Laminac 4110 flares was about 12% greater than for the 4116 units.

The results of this study also indicate that a short elevated temperature cure will substantially increase the tensile and shear strength of the compositions investigated, compared to a short ambient cure. However, for the Laminac resins, it appears that units cured 30 days ambiently have significantly higher physical strengths than units subjected to a 16 hour 150°F cure and then cured ambiently for the remaining 29 days. Theoretically, the Aropol resin should have cured at 75 - 85°F; however, considering the physical strength data after the 150°F cure, it is evident that this resin did not cure substantially at room temperature.

VI. CONCLUSIONS

1. Laminac 4110 was found to be a superior binder compared to Laminac 4116 and Aropol 7720M, considering both physical strength and burning performance of pellets and flares, respectively.

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2. Aropol 7720M exhibited poor binding properties when cured at room temperature but compared favorably with the Laminac resins when cured at 150°F for 16 hours.

3. Of the three resins investigated, Laminac 4110 will impart superior tensile and shear strength to the pressed composition after several days ambient cure; however, after approximately 30 days the two Laminac resins possess essentially the same physical strengths.

4. Flares containing the Aropol 7720M and Laminac 4110 resins emitted a significantly greater number of candlepower-seconds than the units containing Laminac 4116.

5. The burning time and candlepower of flares decreased and increased respectively, a significant amount, as the delay time between mixing and pressing increased.

TABLE I

TENSILE TEST EXPERIMENTAL DATA

Mix-Press Delay Time	Laminate 4110		Laminate 4116		Aropol 7720M	
	5 Day	30 Day	5 Day	30 Day	5 Day	30 Day
0	-	315 lbs.	-	260 lbs.	92 lbs.	160 lbs.
	-	-	-	265 "	95 "	165 "
1	173 lbs.	365 lbs.	128 lbs.	300 lbs.	102 "	150 "
	158 "	-	-	-	105 lbs.	160 lbs.
2	-	-	-	-	100 "	155 "
	185 lbs.	290 lbs.	102 lbs.	260 lbs.	102 "	160 "
3	-	-	-	215 "	85 lbs.	140 lbs.
	168 lbs.	335 lbs.	-	-	102 "	155 "
4	-	275 "	150 lbs.	-	97 "	150 "
	180 lbs.	285 lbs.	-	-	117 lbs.	170 lbs.
5	198	285 "	-	-	125 "	155 "
	195 lbs.	295 "	-	-	120 "	160 "
6	163 "	280 lbs.	125 lbs.	-	112 lbs.	170 lbs.
	180	305 "	-	-	105 "	190 "
7	193 lbs.	225 lbs.	-	-	132 "	175 "
	208 "	255 "	136 lbs.	-	137 lbs.	150 lbs.
8	173 "	210 "	142 "	-	120 "	150 "
	-	-	-	-	122 "	175 "
9	-	-	-	-	78 lbs.	170 lbs.
	-	-	-	-	140 "	185 "
10	-	-	-	-	130 "	175 "
	-	-	-	-	-	-

TABLE II

AVERAGE FAILURE LOADS (LB.) IN TENSILE

Binder Curing Period Time between mixing and pross- ing, Hrs.	Laminao 4110		Laminao 4116		Aropol 7720M	
	5 Days	30 Days	5 Days	30 Days	5 Days	30 Days
0	-	315	-	260	95	160
1	165	365	130	300	105	160
2	185	280	105	240	95	150
3	155	305	150	-	120	160
4	190	270	-	-	120	180
5	180	320	125	-	130	160
6	190	230	140	-	120	175
Average	180	300	130	265	110	165

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TABLE III

SHEAR TEST EXPERIMENTAL DATA

Mix-Process Delay Time	Laminac 4110		Laminac 4116		Aropol 7720M	
	5 Day	30 Day	5 Day	30 Day	5 Day	30 Day
0	398 lbs. 450 " 440 "	1500 lbs. 1520 " 1765 "	306 lbs. 318 " 281 "	1885 lbs. 1890 " 1665 "	332 lbs. 412 " 405 "	475 lbs. 485 " 480 "
1	465 lbs. 600 " 618 "	1655 lbs. 1540 " 1500 "	362 lbs. 382 " 380 "	2035 lbs. 1995 " 1625 "	335 lbs. 370 " 365 "	390 lbs. 425 " 445 "
2	555 lbs. 553 " 522 "	1525 lbs. 1495 " 1300 "	545 lbs. 518 " 540 "	1755 lbs. 1485 " 1625 "	455 lbs. 415 " 460 "	525 lbs. 500 " 515 "
3	665 lbs. 685 " 608 "	1425 lbs. 1425 " 1490 "	444 lbs. 484 " 482 "	2130 lbs. 1905 " 2120 "	475 lbs. 477 " 527 "	570 lbs. 550 " 610 "
4	825 lbs. 845 " 773 "	1615 lbs. 1605 " 1425 "	488 lbs. 490 " 490 "	1990 lbs. 1545 " 1740 "	480 lbs. 465 " 427 "	560 lbs. 545 " 540 "
5	613 lbs. 778 " 718 "	1435 lbs. 1550 " 1570 "	350 lbs. 347 " 377 "	2025 lbs. 2070 " 2015 "	429 lbs. 417 " 462 "	450 lbs. 510 " 510 "
6	558 lbs. 1020 " 1000 "	1500 lbs. 1510 " 1295 "	427 lbs. 418 " 426 "	2035 lbs. 2035 " 2100 "	475 lbs. 525 " 520 "	580 lbs. 555 " 545 "

TABLE IV

AVERAGE FAILURE LOADS (LB.) IN SHEAR

Binder	Luminae 4110		Luminae 4116		Aropol 7720M	
	5 Day	30 Day	5 Day	30 Day	5 Day	30 Day
Curing Period Time between mixing and pressing						
0	430	1595	305	1815	380	480
1	560	1565	375	1885	355	420
2	545	1440	530	1690	440	515
3	655	1450	470	2040	495	575
4	815	1560	490	1760	455	550
5	705	1520	360	2040	435	490
6	990	1435	425	2060	505	560
Average	670	1510	420	1900	450	515

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TABLE V

EXPERIMENTAL DATA - PELLETS CURED AT 150°F

Binder	5 Day		30 Day	
	Tensile	Shear	Tensile	Shear
Laminac 4110	215 lbs.	1308 lbs.	210 lbs.	1230 lbs.
	250 "	1425 "	180 "	1275 "
	-	1500 "	-	1180 "
Laminac 4116	234 lbs.	1375 lbs.	260 lbs.	1455 lbs.
	184 "	1215 "	-	1395 "
	-	1460 "	-	1475 "
Aropol 7720M	360 lbs.	1690 lbs.	570 lbs.	1740 lbs.
	427 "	1870 "	510 "	1705 "
	412 "	1880 "	510 "	1665 "

TABLE VI

AVERAGE TENSILE AND SHEAR FAILURE LOADS (LB.)
FOR PELLETS CURED @ 150°F AND AMBIENTLY

Binder	5 Days				30 Days			
	Tensile		Shear		Tensile		Shear	
	150°	Ambient	150°	Ambient	150°	Ambient	150°	Ambient
Laminac 4110	230	180	1410	670	195	300	1230	1510
Laminac 4116	210	130	1350	420	260	265	1440	1900
Aropol 7720M	400	110	1815	450	530	165	1705	515

TABLE VII

EXPERIMENTAL DATA - BURNING TIME
& AVERAGE CANDLEPOWER OF CANDLES

Mix-Press Delay Time	Laminao 4110		Laminao 4116		Aropol 7720M	
	B.T.	C.P.	B.T.	C.F.	B.T.	C.P.
0 Hrs.	131 Sec.	212,000	134 Sec.	193,000	140 Sec.	209,000
	130 "	223,000	130 "	212,000	141 "	216,000
	129 "	226,000	134 "	187,000	140 "	217,000
1	129 "	219,000	133 "	212,000	136 "	222,000
	129 "	245,000	130 "	198,000	139 "	226,000
	129 "	232,000	132 "	197,000	139 "	217,000
2	124 "	232,000	127 "	201,000	138 "	221,000
	128 "	242,000	130 "	200,000	135 "	224,000
	128 "	237,000	131 "	181,000	140 "	213,000
3	128 "	225,000	128 "	201,000	137 "	215,000
	126 "	235,000	131 "	206,000	138 "	230,000
	127 "	240,000	129 "	221,000	137 "	217,000
4	126 "	233,000	131 "	205,000	141 "	224,000
	126 "	231,000	132 "	209,000	134 "	227,000
	125 "	245,000	131 "	210,000	135 "	231,000
5	124 "	245,000	129 "	198,000	132 "	223,000
	124 "	247,000	130 "	201,000	137 "	231,000
	123 "	242,000	123 "	246,000	136 "	227,000
6	120 "	254,000	129 "	220,000	134 "	226,000
	126 "	247,000	122 "	218,000	136 "	228,000
	120 "	252,000	127 "	200,000	137 "	237,000

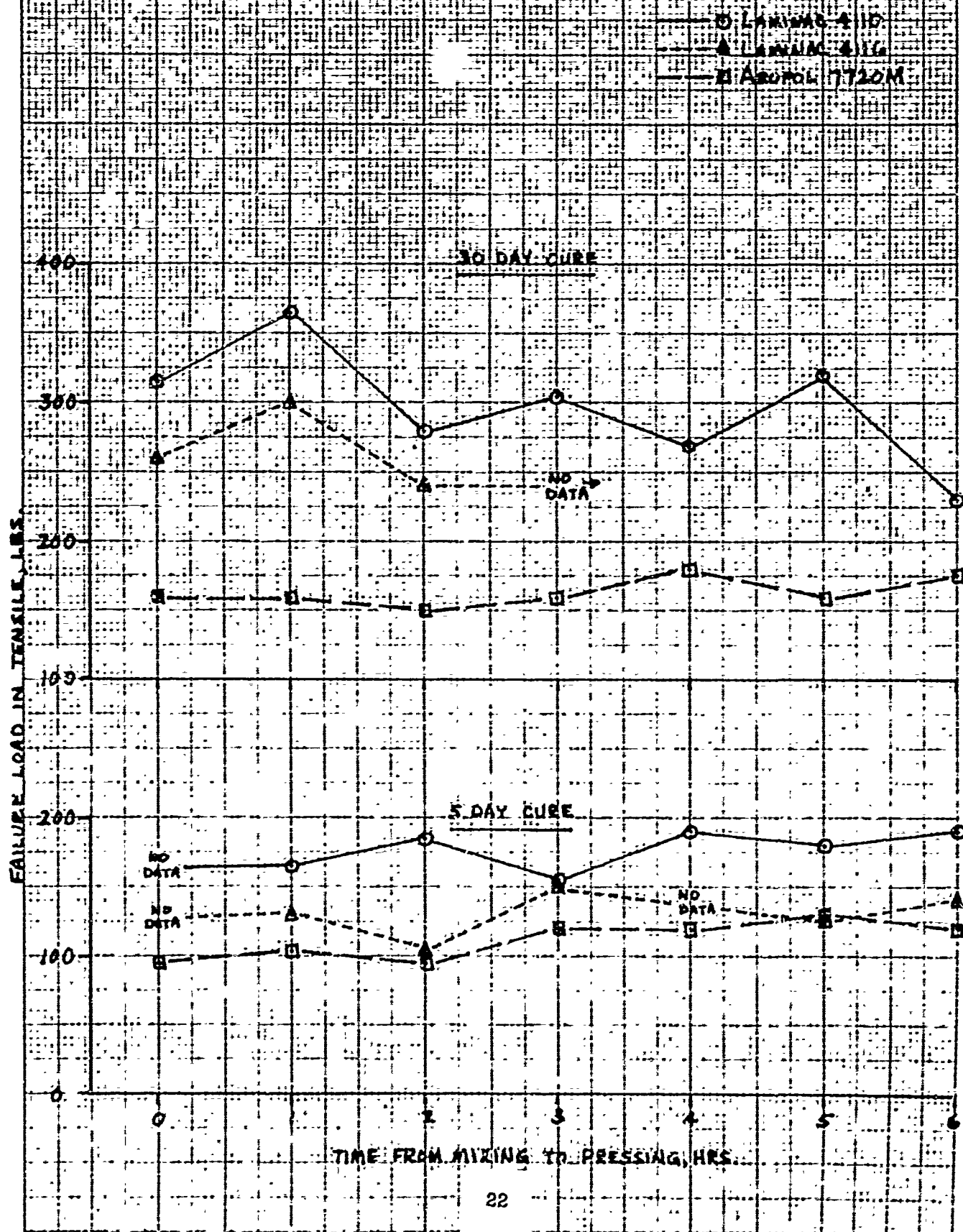
B.T. - Burning Time
C.P. - Average Candlepower

TABLE VIII
BURNING TIME, CANDLEPOWER AND
CANDLEPOWER-SECONDS OF FLARES

Time between mixing and pressing	Laminac 4110			Laminac 4116			Aropol 7720M		
	B.T.	C.P.	G.P.-Sec	B.T.	C.P.	G.P.-Sec	B.T.	C.P.	C.P.-Sec
0	130	220,000	28.6 x 10 ⁶	133	197,000	26.2 x 10 ⁶	140	214,000	30.0 x 10 ⁶
1	129	232,000	29.9	132	202,000	26.7	138	222,000	30.6
2	127	237,000	30.1	129	194,000	25.1	136	219,000	30.2
3	126	233,000	29.4	129	203,000	27.0	137	221,000	30.3
4	126	236,000	29.8	131	203,000	27.2	137	227,000	31.1
5	124	243,000	30.1	127	215,000	27.3	135	227,000	30.6
6	122	251,000	30.6	126	213,000	26.8	136	230,000	31.3
Average	126	236,000	29.8 x 10 ⁶	130	205,000	26.6 x 10 ⁶	137	223,000	30.5 x 10 ⁶

GRAPH I

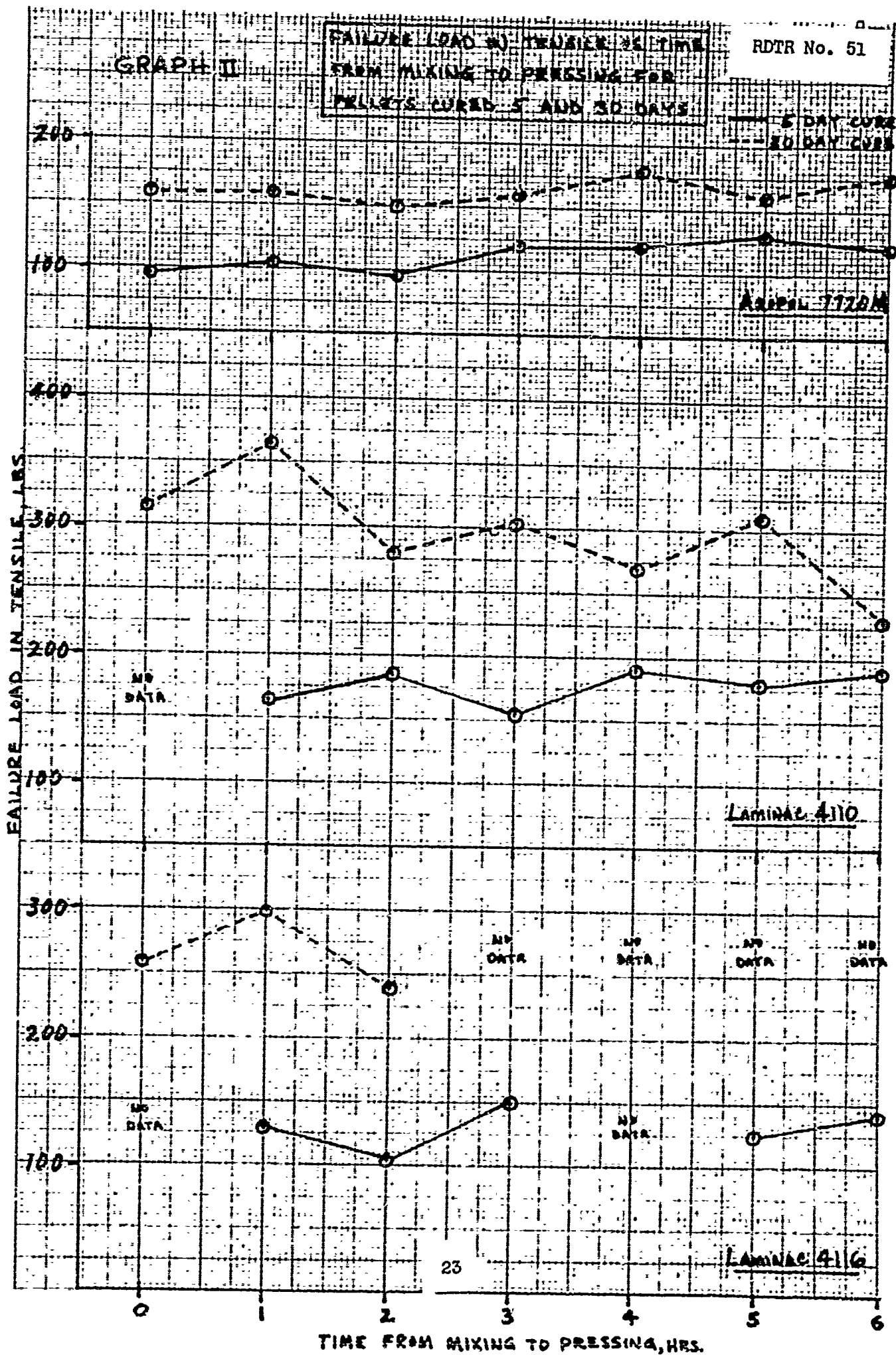
FAILURE LOSS IN TENSILE VS TIME
FROM MIXING TO PRESSING FOR
CONCRETE CURED 5 AND 30 DAYS



GRAPH II

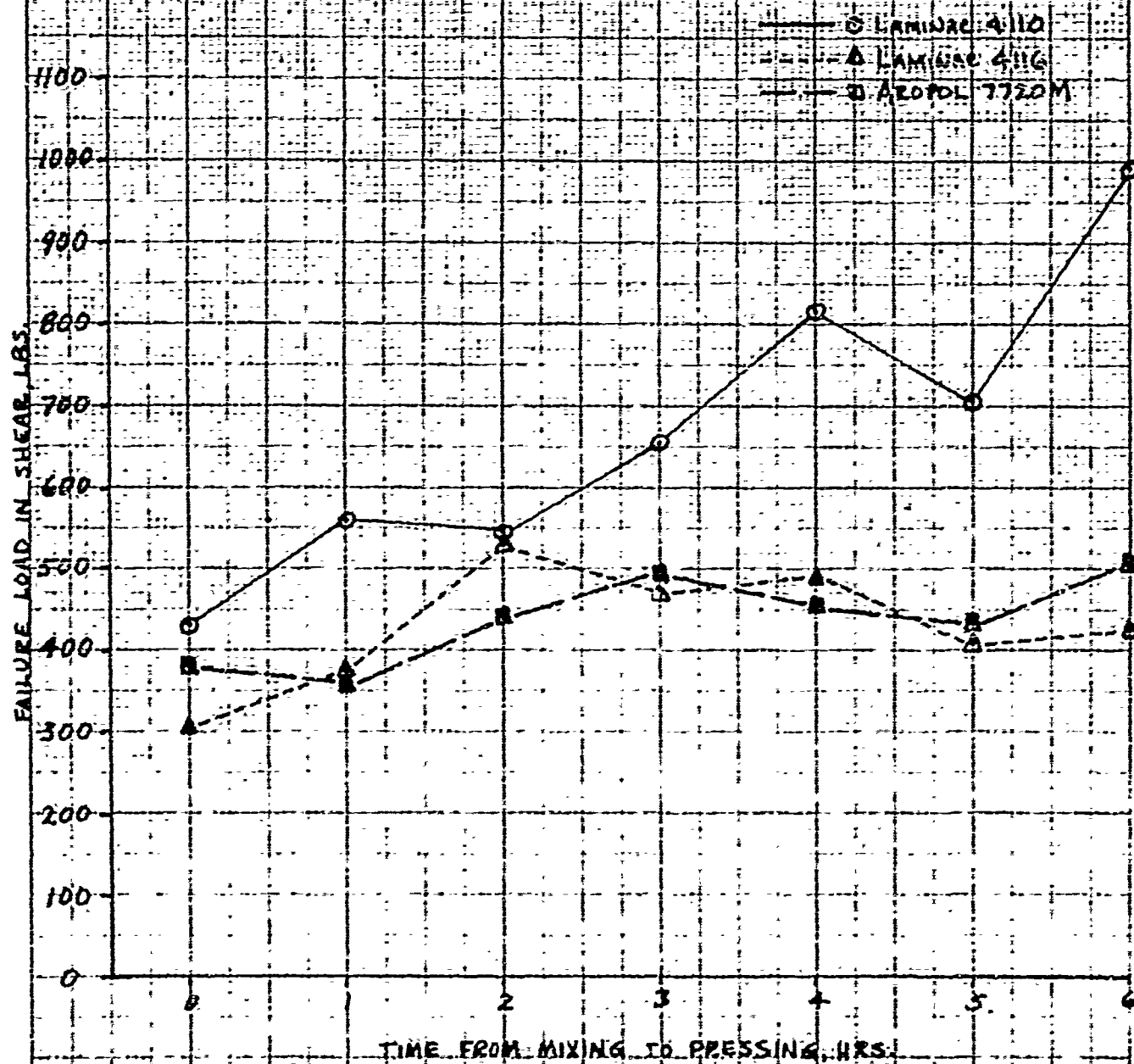
FAILURE LOAD IN TENSILE % TIME
FROM MIXING TO PRESSING FOR
PELLETS CURED 5 AND 30 DAYS

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GRAPH III

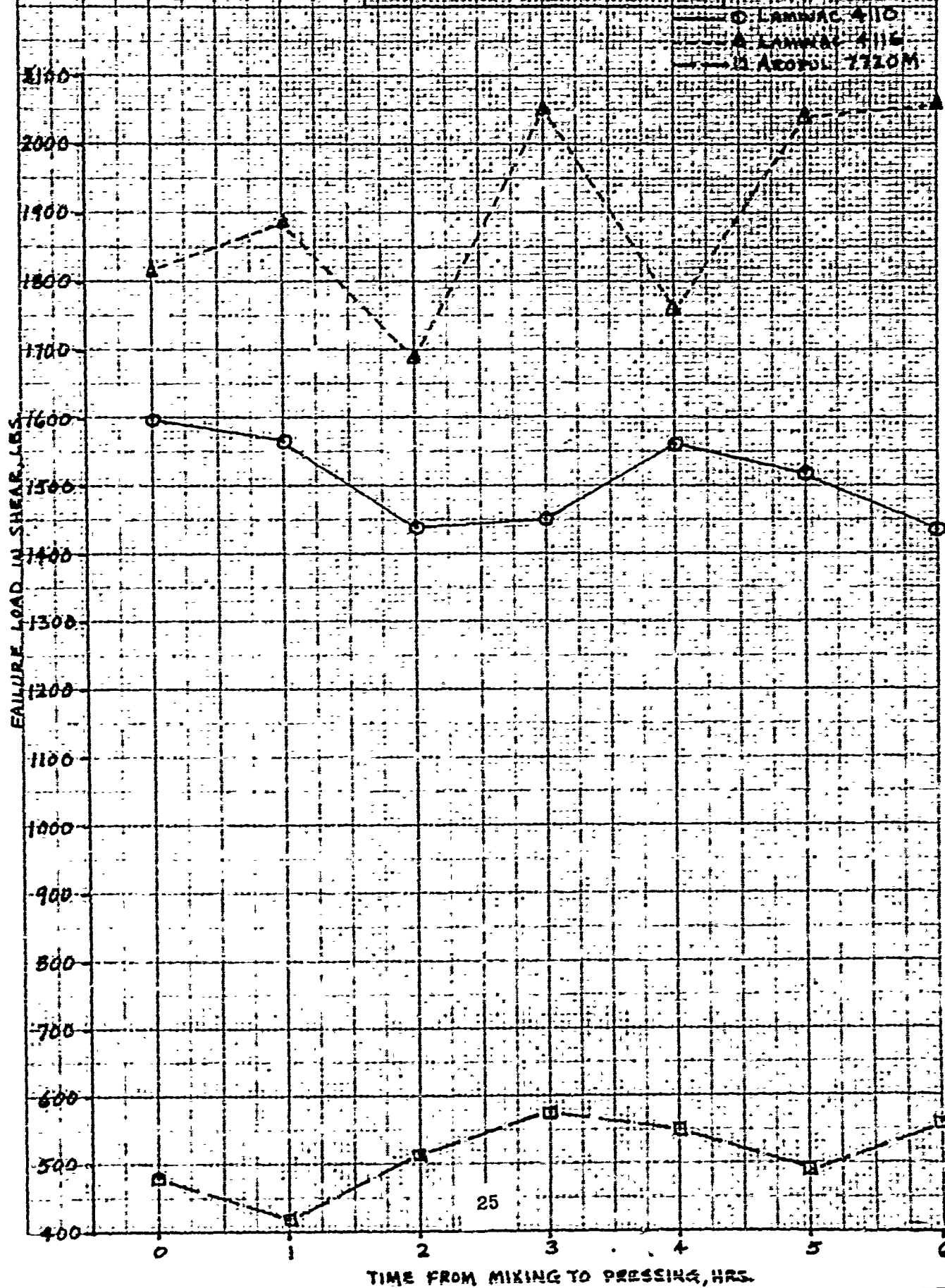
FAILURE LOAD IN SHEAR VS
TIME FROM MIXING TO PRESSING
FOR PELLETS CURED 5 DAYS

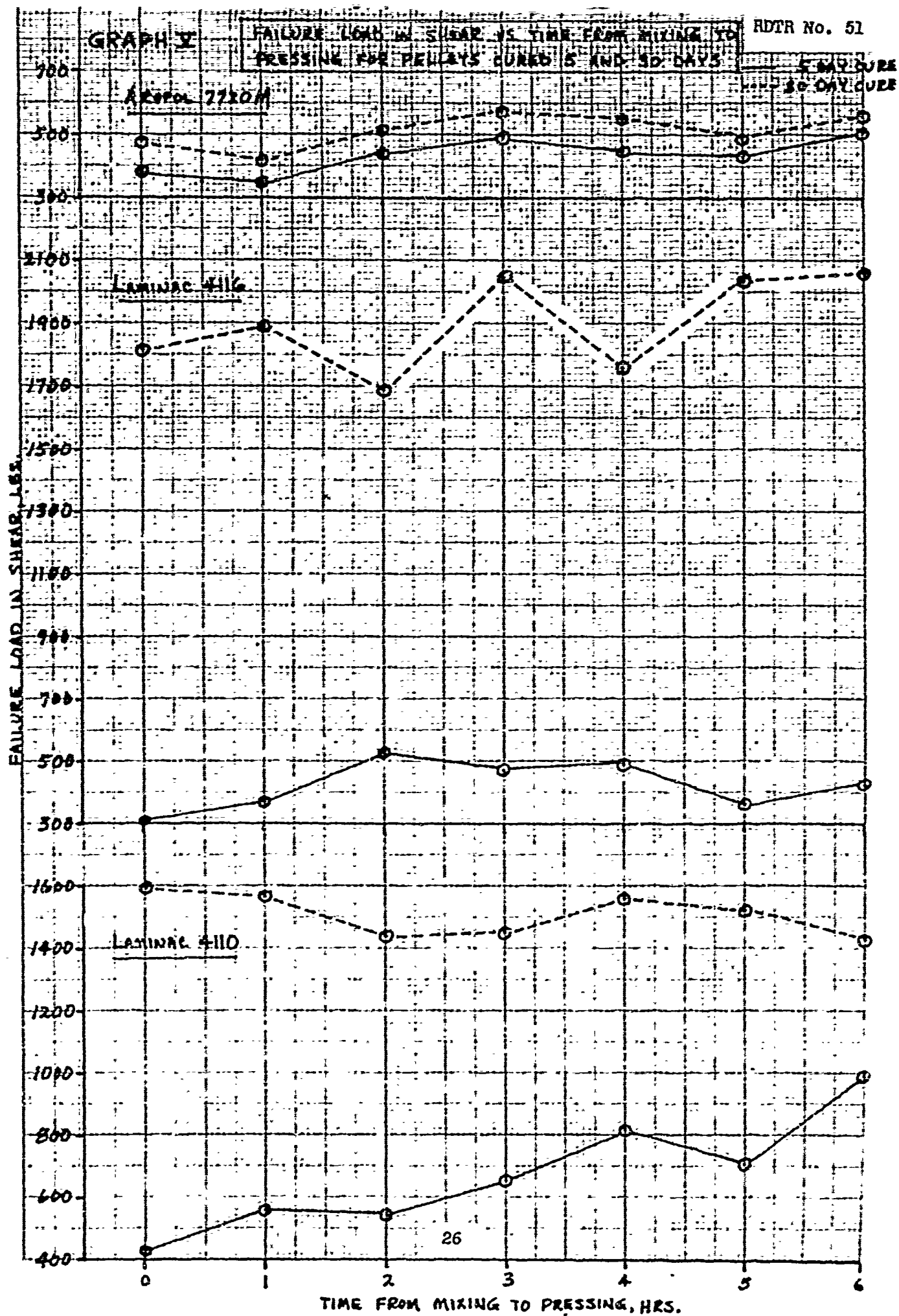


GRAPH IV

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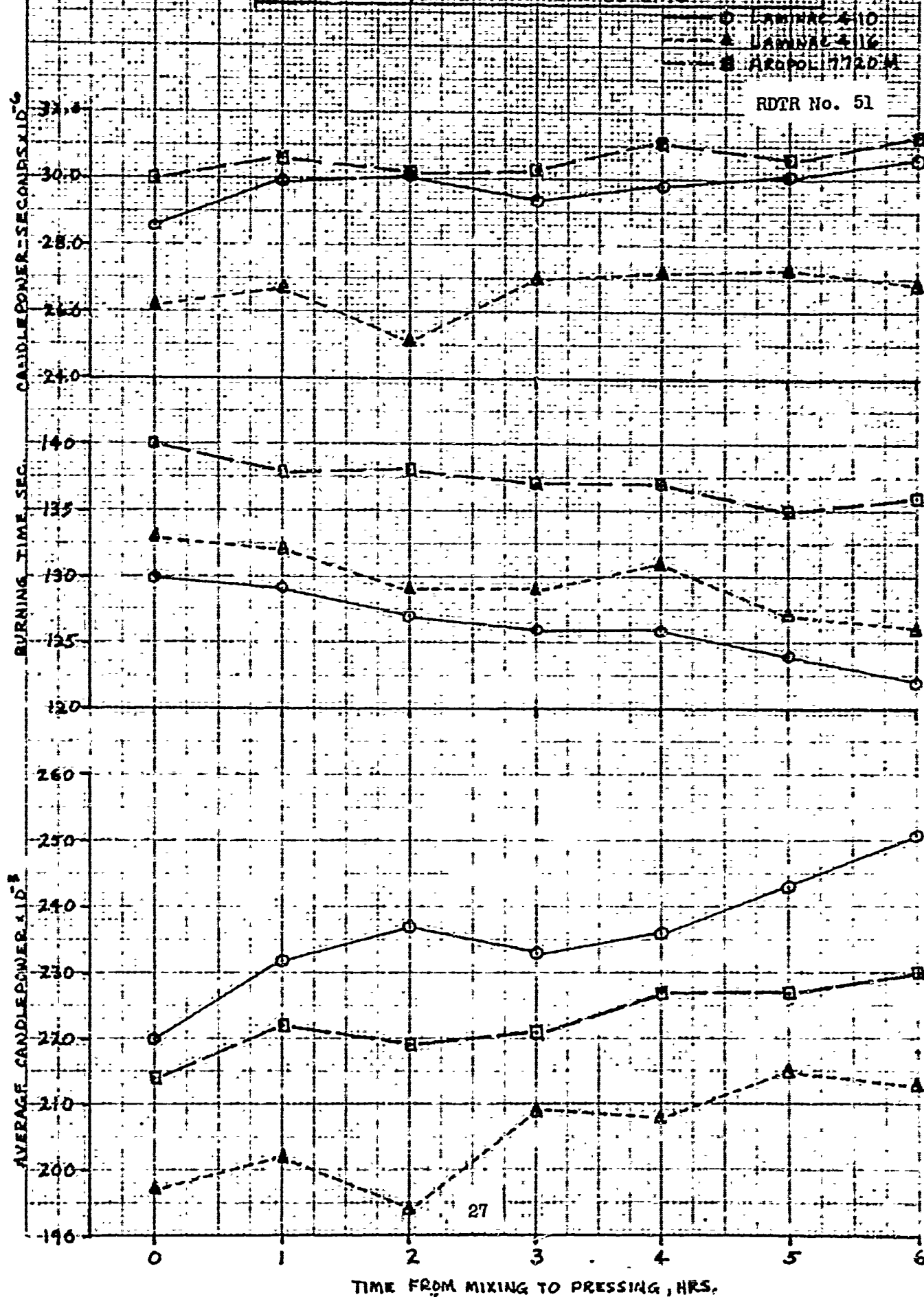
FAILURE LOAD IN SHEAR VS
TIME FROM MIXING TO PRESSING
FOR PELLETS CURED 30 DAYS





GRAPH VI

CANDLE POWER, BURNING TIME AND CANDLE POWER STRESS
VS. TIME FROM MIXING TO PRESSING FOR FLAKES CURED
AT AMBIENT TEMPERATURE FOR 30 DAYS



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11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY U. S. Naval Ammunition Depot Crane, Indiana
13. ABSTRACT The binding strengths of pellets subjected to tensile and shear stresses, and the burning time and candlepower of flares pressed from compound aged 0-6 hours in increments of one hour prior to pressing were determined after curing periods of 5 and 30 days. Three formulations were used, each containing the same ratio of magnesium/sodium nitrate/binder; but, with three different polyester resin binders--Laminac 4110, Laminac 4116, and Aropol 7720M. The variations in physical strength, candlepower, and burning time with respect to delay time between mixing and pressing were found to be greatest for pellets and candles containing Laminac 4110. Likewise, the physical strength of pellets containing Laminac 4110 was significantly higher than the other pellets after curing 5 days; however, tests after 30 days indicated that by this time the Laminac 4116 had essentially fully cured, and now had binding properties similar to Laminac 4110. The physical strength of pellets cured at 75 - 85°F with Aropol 7720M was considerably less than pellets containing the other binders. All units cured for 16 hours at 150°F, and then tested after 5 days exhibited considerably higher strengths than pellets not subjected to an elevated temperature. However, tests after 30 days showed that for the Laminac binders, greater binding strength is obtained by curing at room temperature for the duration of the curing cycle.		

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
1. Flares, Physical Strength						
2. Flares, Burning Characteristics						
3. Flares, Binders						
4. Flares, Aging						
5. Flare Composition, Aging						
6. Binders, Physical Properties						

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