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HW-24165

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April 18, 1952

TO: File

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234-5 Plant Assistance, Process Assistance
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REPORT

234-5 BUILDING RM LINE EQUIPMENT TESTS

TASK III FURNACE CALIBRATION

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Introduction

Early development work done at Los Alamos[1] on the bomb-reduction method of preparing plutonium indicated that precise control in heating the bomb at a prescribed rate is necessary in order to obtain consistently good yields. Since it is not possible to record the desired inner temperature on production runs, the heating rate is controlled by correlating this liner temperature to an externally located thermocouple attached to the outer surface of the reduction furnace. It was the purpose of the tests discussed in this report to correlate this recorder temperature with the inner liner temperature on the reduction furnaces of the RM Line.

The furnace was heated at such a rate that the average liner temperature, which was measured at three points, corresponded to the prescribed schedule. In addition to the heating rate prescribed above, it was recommended that the temperatures as recorded at the three locations should be within 75°C of each other in order to obtain best results. The test runs were evaluated with regard to this condition also.

Several dry runs using UF_4 as a standin were also made to check the newly designed furnaces. These were made following recommended procedures outlined for reduction with calcium and sulfur or iodine.

Conclusions

1. Satisfactory operation of Furnaces 1 and 3 was obtained.

2. The temperature recorded at the bottom of the crucible in the Furnace 2 calibration was low and not within the 75°C specified. Spread at firing temperature was 125°C. GEL tests showed satisfactory heat distribution for this furnace.

3. The heating rate prescribed by LA 473 is best followed by heating the furnace as rapidly as possible to 650°C and holding at this temperature until firing occurs. (11 to 13 minutes)

4. Pressure indicators do not always operate to indicate firing.

5. Using the present equipment, the liner must have backing of sand to prevent cracking of the crucible and loss of charge.

Recommendations

1. It is recommended that the furnaces and coils be used as furnished by the GE Laboratory.

2. It is recommended that Furnace 2 be operated and closely scrutinized during the startup period to compare its operating characteristics to those of the other furnaces.

3. It is recommended that only V-36 or S-816 alloy be used as a furnace material without making additional calibration runs.

4. It is recommended that the furnace assembly be recalibrated if any change in equipment or process is made in the future. (Other than Recommendation No. 3)

5. The following cycle is recommended for use with iodine, calcium and PuF_4. The power settings are only approximate for guidance. In general these should be the maximum possible until 650°C is reached and then reduced to maintain this temperature.

Time	Recorder Temperature °C	KW Input
0	25	0
1	250	22 (Maximum)
2	400	22
3	525	22
4	625	22
5	650	11
6	650	11
7	650	11
8	650	11
9	650	11
10	650	11
11	650	11
12	650	11
13	650	11
14	650	11
15	650	11

If an alternate process is used, it is recommended that the best cycle be determined experimentally.

5. It is recommended that additional work be done to provide more positive means of determining the firing point of the charge. Radiation meters as used on the RG Line might be used but must be sensitive to a firing even though the other furnaces contain unfired charges.

6. It is recommended that additional development work be done on the sanding problem to eliminate the sand or replace it with a material that does not have the objection of being strewn over the equipment.

Procedure

Three chromel-alumel thermocouples were attached to the inside wall of an S-1 type crucible with porcelain cement. The thermocouple which recorded the top temperature was located one inch (1") below the top of the crucible, the thermocouple which recorded the center temperature was located half way down the crucible wall, and the bottom temperature thermocouple was at the intersection of the inner wall and bottom. These three thermocouples were placed 120° apart circumferentially.

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The crucible was placed in a low carbon steel "tin" can and the annular space between the crucible and can filled with Blend 80 magnesium oxide sand. This "can pack" was placed in a special mild steel base that had been slotted to permit the passage of the thermocouple leads. It had the same design as the regular base in all other details. The thermocouple leads extended through a hole in the crucible lid, down the side of the can pack and out through the slot in the special base. This assembly was then raised into the furnace to be tested. The leads were protected from shorting against the can and furnace by ceramic beads.

In their design work, GEL had done much to determine the proper heating program.(2) Starting with this as a basis, heating cycles were varied until the heat input provided the same heating rate as the Time vs. Liner Temperature schedule given in Table I.

TABLE I

Time (Min)	Inner Liner Temp.°C
0	25
1	25
2	47
3	54
4	102
5	135
6	164
7	211
8	252
9	279
10	320
11	347
12	381
13	416
14	442
15	470
16	500

KW input and recorder and crucible liner temperatures were recorded at one minute intervals until an inner temperature of approximately 500°C was recorded at which time the power was shut off. Readings were continued until the liner temperatures started dropping. Further readings were felt to be unnecessary as conditions at the time of firing are not reproducible. A sketch of the test assembly is given in Figure 1.

Discussion

Information given to the GE Laboratory as to the heating program must have emphasized a desire to reach 325°C in ten (10) minutes, the firing time and temperature in the RG Line equipment. This was shown in Runs 2 and 4, which were made following GEL recommendations. In these runs the heat distribution was satisfactory and the average liner temperature did reach 325°C in ten minutes. However the RM equipment has a 2 to 3 minute lag over the RG equipment before the liner temperatures start rising which means that in order to obtain the above time-temperature condition a considerably more rapid heating rate is required.

Obviously the most important factor is not the temperature at a given time but the rate of heating. In Run 5, therefore, the heating rate was reduced by heating as rapidly as possible to 650°C instead of 750°C and holding at this point. A satisfactory liner temperature program and heat distribution pattern was obtained from this run. Because the other furnaces had such similar heating characteristics (except the bottom temperature of Furnace #2) this test was not repeated on each furnace.

There are also other advantages to be gained by using the lower temperature for the reduction. The reduction atmosphere is corrosive and by reducing the maximum temperature the corrosion rate is reduced, and as a result the life expectancy of the furnace, which is replaced only with difficulty, is increased.

Also at 750°C the nickel gasket has a tendency to weld to the furnace which presents problems in operation. By operating at the lower temperature the nickel is not softened to the degree it is at 750°C and consequently the removal of the charge from the furnace by routine remote operation is more certain.

The effect of having the charge off center in the furnace, a known factor of poor heat distribution, was not studied since the can pack cannot be more than 1/8" off center and was in this position to provide clearance for the thermocouple leads from the crucible.

The can used in Calibration Run #1 was the can furnished by GEL and had a 3.500" I.D. Following this test the can design was reviewed by Technical and Operations personnel to reconcile the problems of equipment operation, process requirements, and recovery processes. As a result of these discussions, it was decided that a can 3.312" I.D. would better fit the overall picture and all succeeding calibration runs were made in this size can.

Test data for the various runs are given in the appended tables and figures. The corresponding table number and figure number refer to the same heat run. Each figure has the program recommended by LA-473, the controller temperature, and the three recorded temperatures. The dotted line represents the average liner temperature and is added for convenience only when there is a large variation in the temperatures. A brief review of each heat run is presented in a later section of this report.

In addition to the heat calibration runs, twelve stand-in runs were made with uranium tetrafluoride, calcium, and sulfur or iodine. These runs were made to test the operability of the equipment which had some radical design changes, especially the free volume in the furnace outside the crucible. From these runs additional information regarding the operation was gained which, although not directly concerned with calibration, do affect the reduction furnace cycle.

With the exception of two runs in which the charge was partially lost, the yields from the stand-in runs were greater than 99 per cent and the appearance of the fired charge was normal. This would indicate that all the furnaces are being heated in a satisfactory manner.

The firing of the charge was not always detected by the pressure recorder provided for this purpose. Because of this inconsistency it would be desirable to study this problem and provide a better method of determining the point at which the heating can be discontinued.

The need for sand to serve as a backup material for the crucible was investigated because of the advantages to be gained by eliminating it. During subsequent operations of cutting and dumping it spills over the precision handling equipment and it adds to the recovery problem.

Several of the stand-in runs were made in crucibles having only a tight-fitting can for support. Fifty per cent of the runs of this type made in S-1 crucibles cracked and allowed the slag and metal to pour out through the supporting can into the furnace. It was decided that cans of close enough tolerance could not be provided to assure adequate support and all further runs were made in sand packed cans.

<u>Test Runs</u>

<u>Calibration Run #1</u> - This run was made in Furnace #2 using an S-1 crucible packed in a 3.5 inch can. The furnace was heated as rapidly as possible until the recorder temperature reached 750°C and was held at this point. The results show that there was a rapid heat input to the upper half of the crucible and the bottom was much cooler, lagging 175°C at the firing temperature. Even so, the average heating rate was higher than the recommended rate, being 42°C/min. compared to 31°C/min.

<u>Calibration Run #2</u> - This run was made in Furnace #3 using the same S-1 crucible from Run #1 which was removed from the can and packed in a 3.312 inch can which was felt to be better. The same heating schedule was followed as for Run #1 and gave a satisfactory heat distribution but the average heating rate was again high, 42°C/min. Two of the thermocouples gave erratic readings at the end of the heating cycle when they opened apparently from being stressed when making the change in cans.

<u>Calibration Run #3</u> - This was a repeat of Run #1 using the 3.312 inch can. It still gave an unsatisfactory heat distribution, although the lag of the bottom temperature was less, being 125°C at the firing temperature. The average heating rate was again excessive, 51°C/min.

<u>Calibration Run #4</u> - This run was made to test Furnace #1 using the same heating schedule and equipment as Runs #2 and #3. Results were the same as Run #2.

<u>Calibration Run #5</u>.- This run was made in Furnace #3, heating as rapidly as possible to 650°C and holding. A satisfactory heat distribution was

obtained and the average heating rate was identical to the recommended rate. This heating program provides the best conditions for routine plant operation of the reduction equipment.

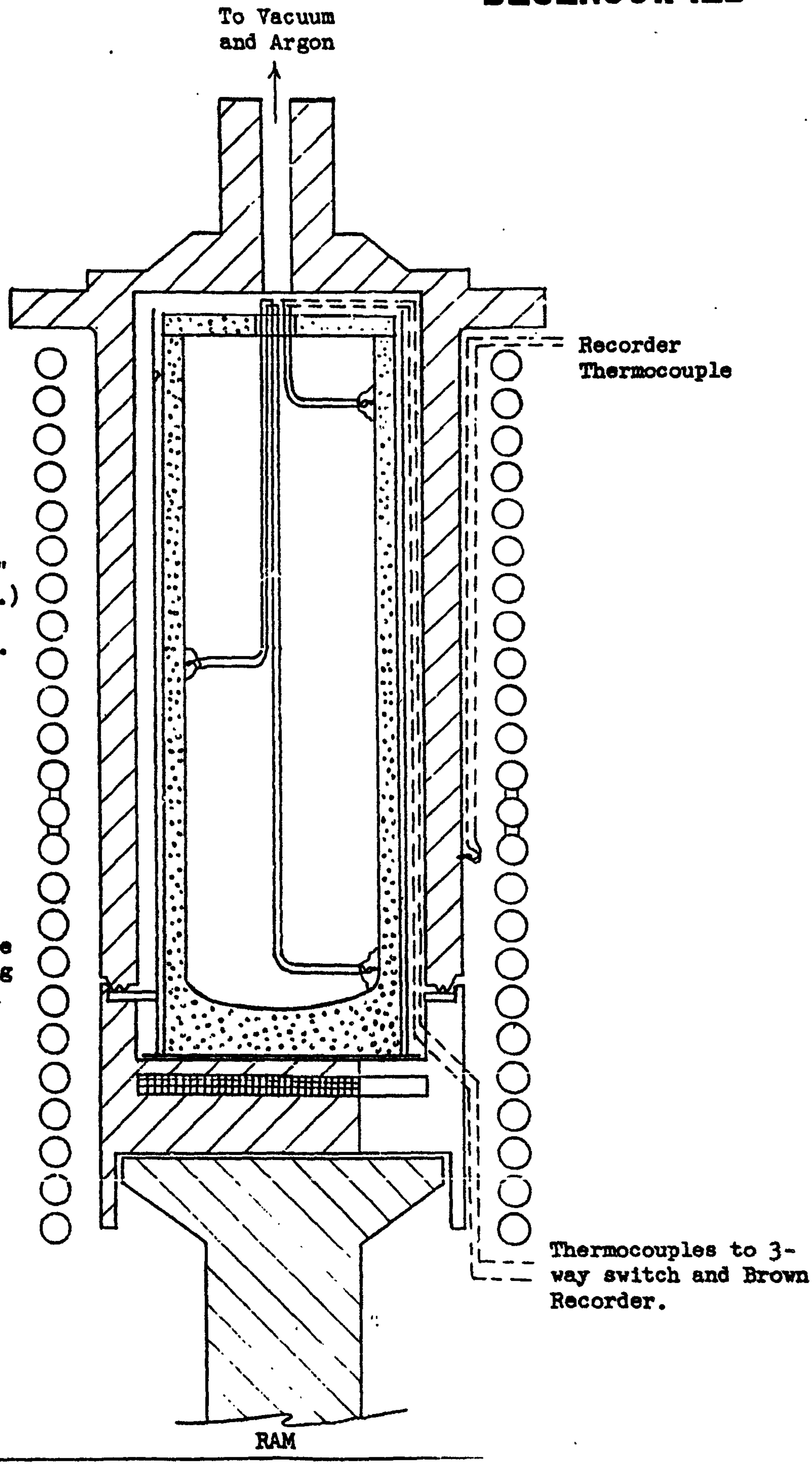
References

1. LA 473, "Preparation of Plutonium by the Bomb Method", R. D. Baker, Secret.

2. JP 369, "Schenectady Test Report, Task III Components", No Author Given, Secret.

FIGURE 1

TEST ASSEMBLY

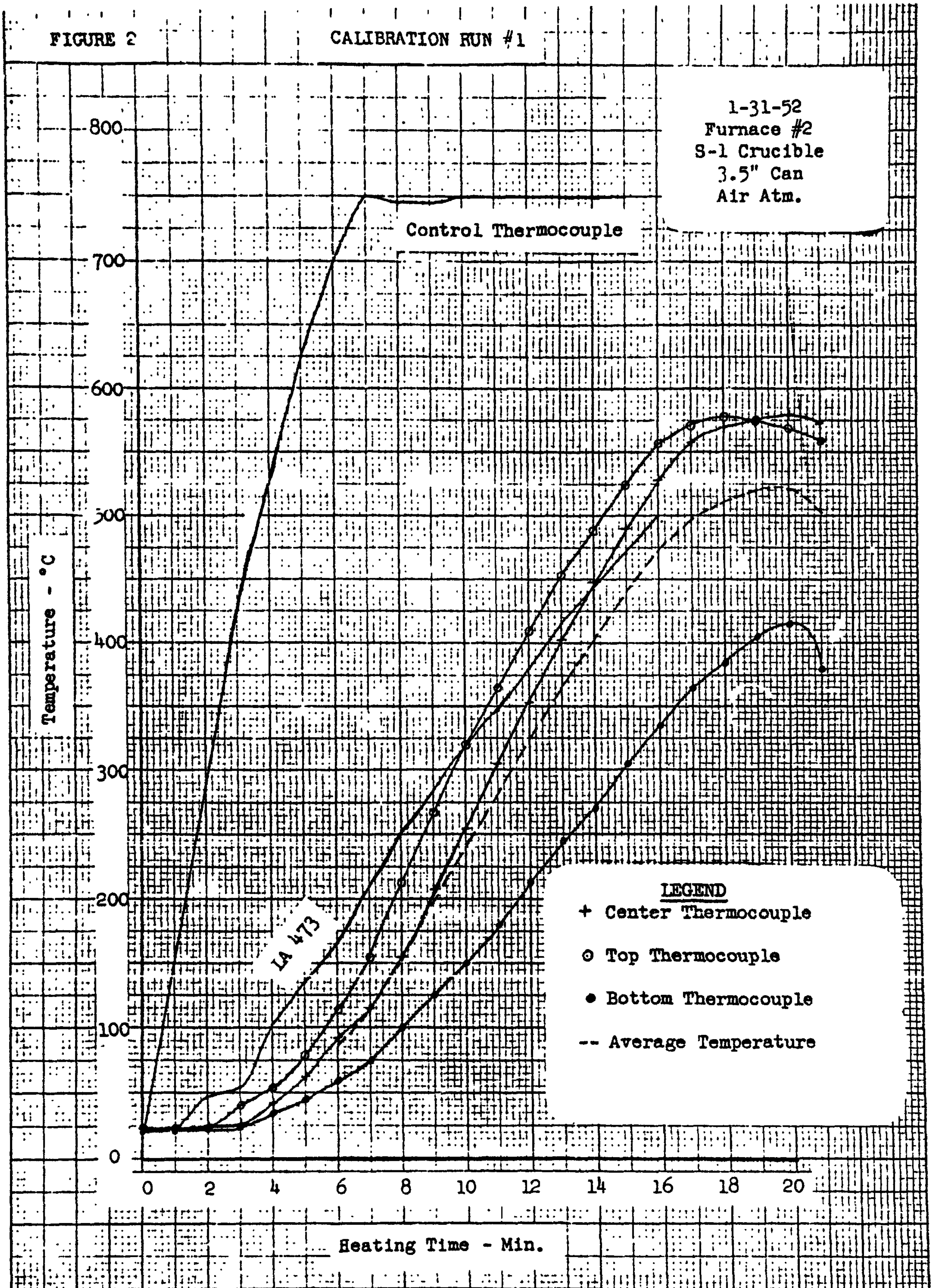


FURNACE
1. V-36 Alloy
2. 3 3/4" I.D.
3. 4 3/4" O.D.
4. 10 1/2" Inside Height.

CAN PACK
1. S-1 Crucible
2. 3.500" or 3.312" I.D. Can (28 ga.)
3. Blend 80 MgO sand in annulus.

BASE
1. Mild Steel
2. Slotted for Thermocouple entry.
3. Glass wool insulator pad.
4. Nickel gasket.

COIL
1. 3/8" Copper Tube
2. 6" Dia. 12" Long
3. 24 Turns - Even
4. 3 Center Turns Shorted.



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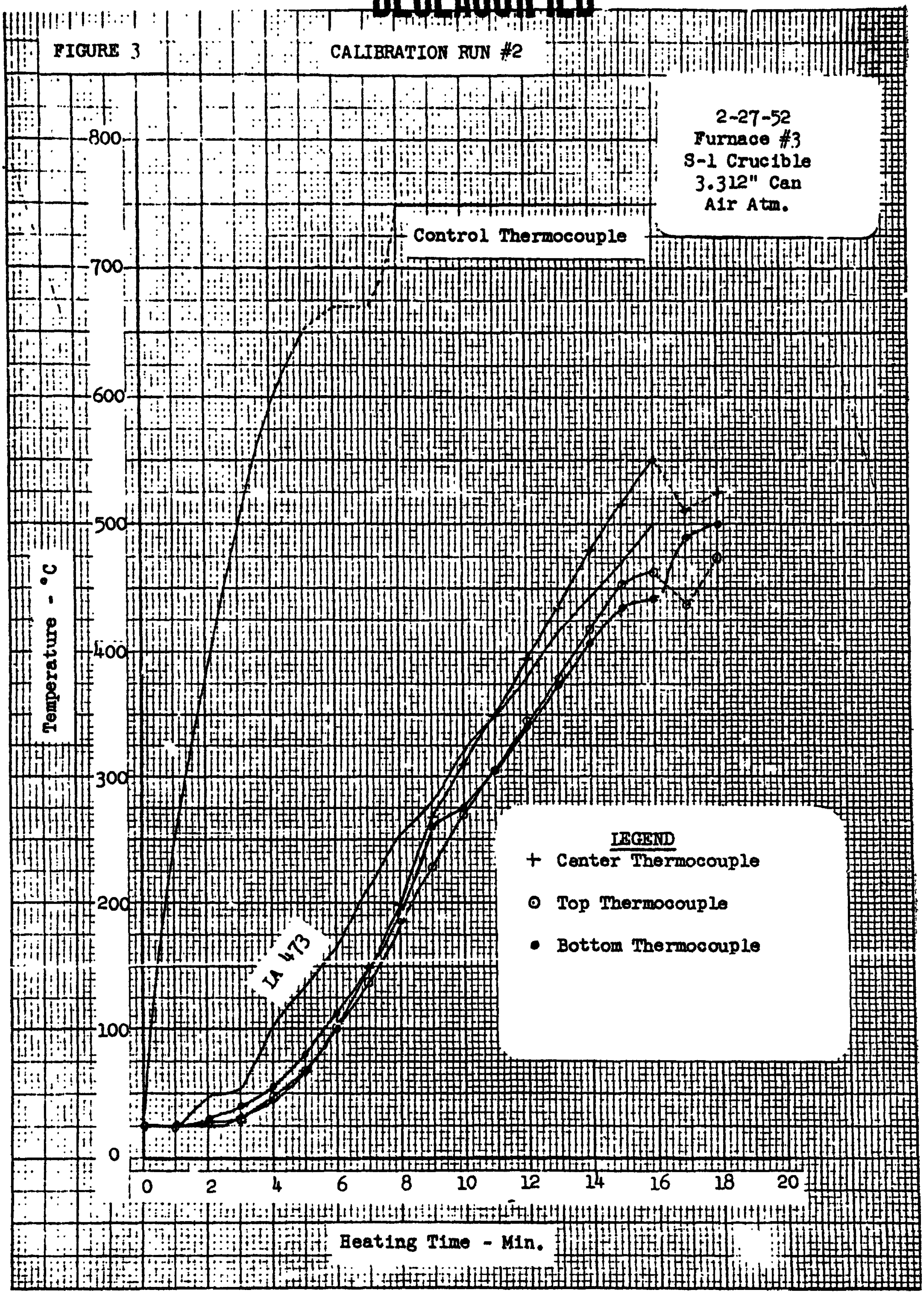

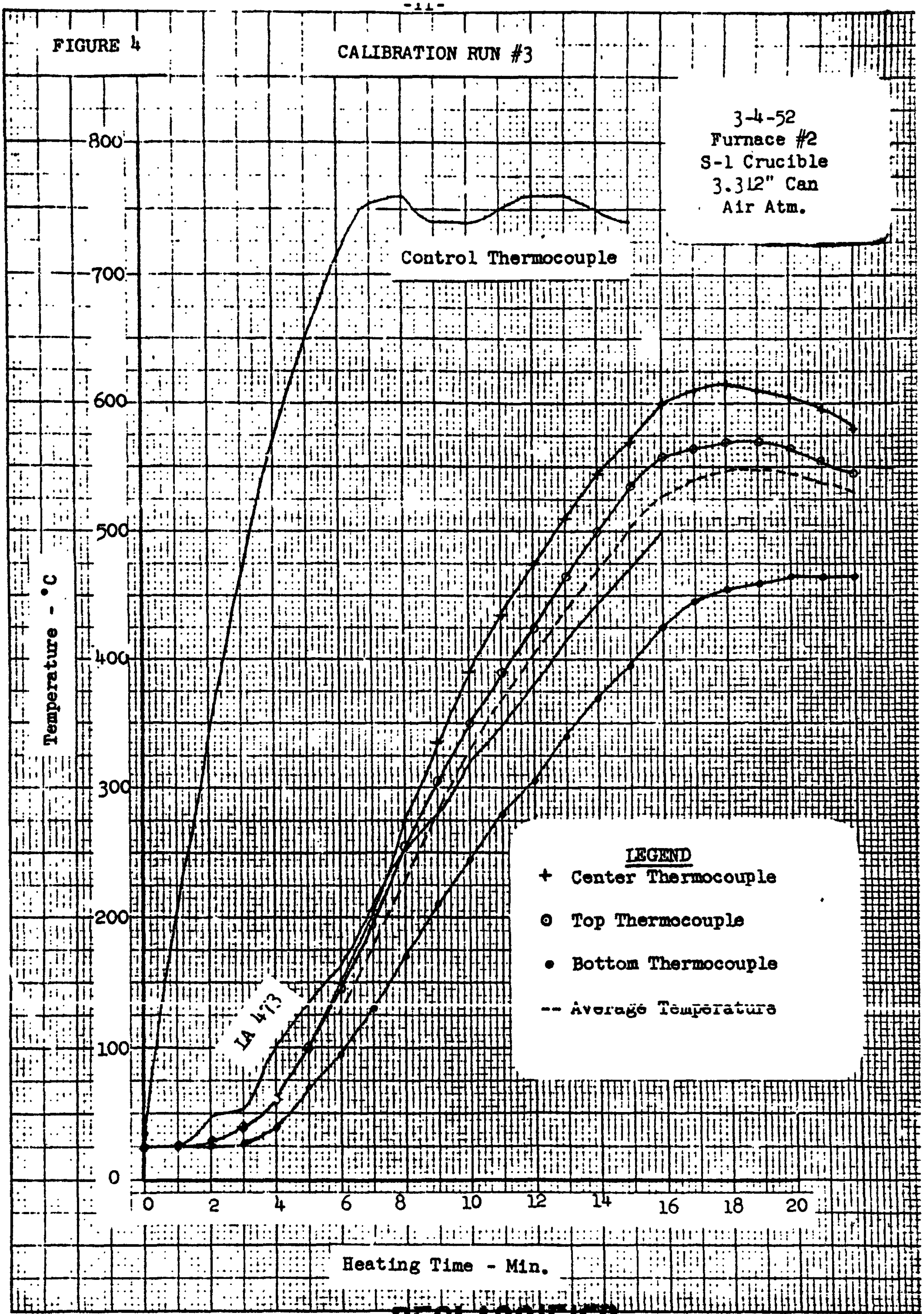
FIGURE 4
CALIBRATION RUN #3
3-4-52
Furnace #2
S-1 Crucible
3.312" Can
Air Atm.
Control Thermocouple
Temperature - °C
800
700
600
500
400
300
200
100
0
0
2
4
6
8
10
12
14
16
18
20
Heating Time - Min.
LA 473
LEGEND
+ Center Thermocouple
⊙ Top Thermocouple
• Bottom Thermocouple
-- Average Temperature

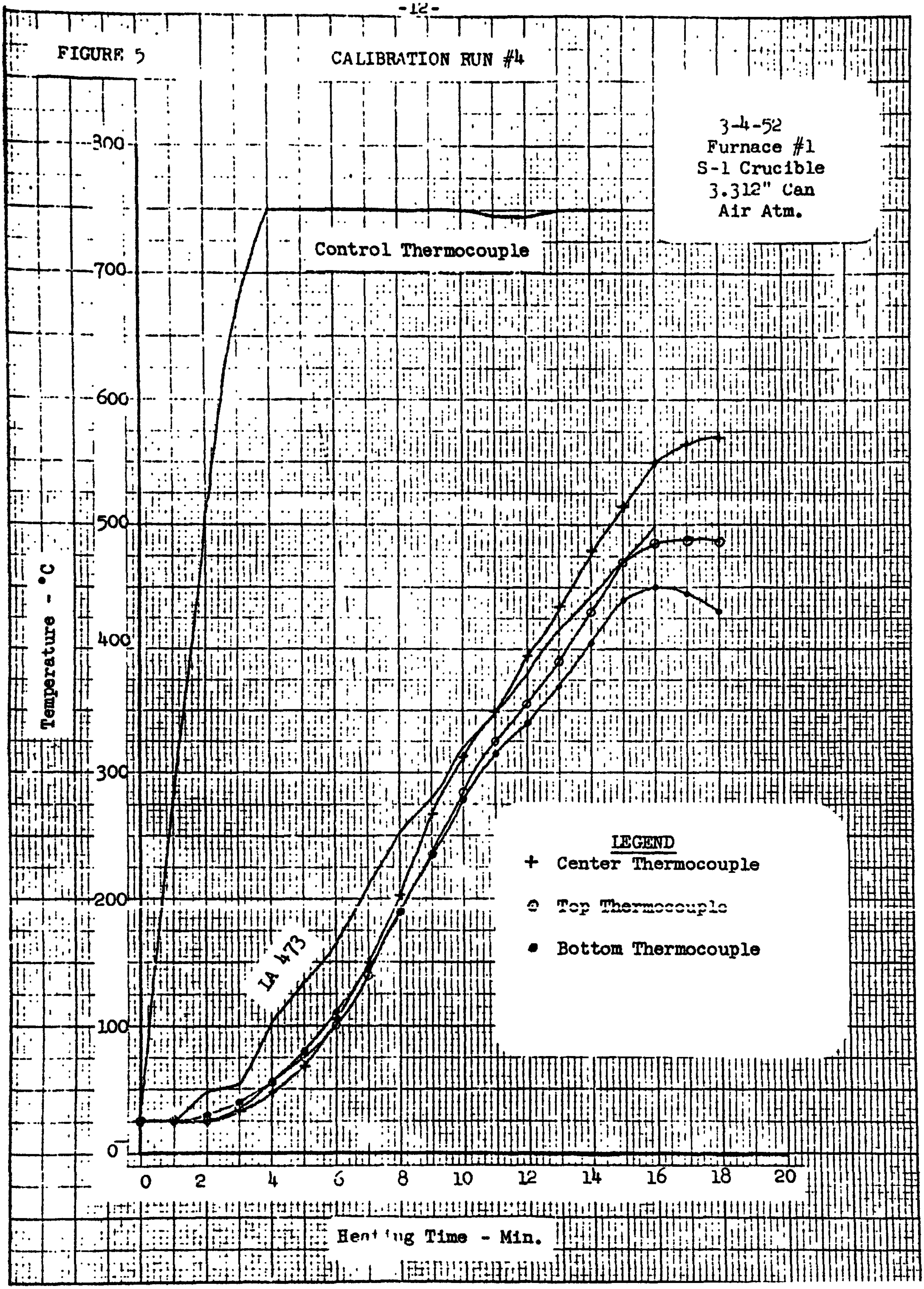
FIGURE 5
CALIBRATION RUN #4
3-4-52
Furnace #1
S-1 Crucible
3.312" Can
Air Atm.
Control Thermocouple
LA 473
LEGEND
+ Center Thermocouple
⊖ Top Thermocouple
• Bottom Thermocouple
Temperature - °C
0
100
200
300
400
500
600
700
800
0
2
4
6
8
10
12
14
16
18
20
Heating Time - Min.

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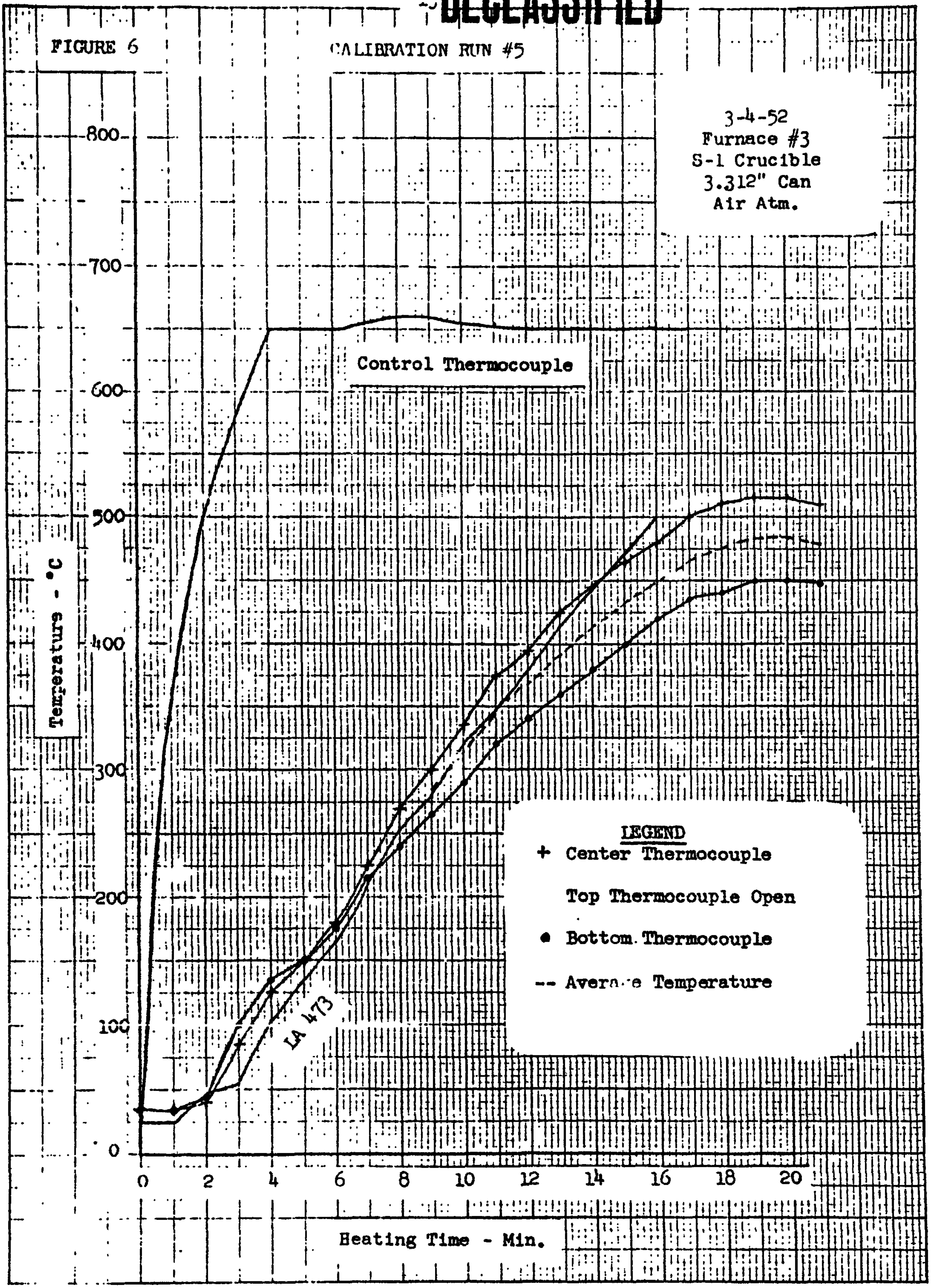

TABLE 2

TASK III FURNACE CALIBRATION

1-31-52
Run #1
Furnace #2,
S-1 Crucible
3.5" Can, Sanded
With MgO
Air Atmosphere

Time	KW Input	Recorder Temp.	Top Temp.	Center Temp.	Bottom Temp.
0	0	20	24	24	22
1	22.5	150	24	24	22
2	22.5	285	24	24	22
3	22.5	425	36	26	24
4	22.5	525	54	42	34
5	23.0	625	75	62	45
6	22.5	700	114	92	59
7	8	750	155	115	75
8	10	745	213	155	100
9	15	745	268	207	126
10	12	750	320	254	150
11	12.5	750	366	304	180
12	13	750	410	353	212
13	13	750	453	402	245
14	13	750	488	448	271
15	13	750	525	490	304
16	OFF		557	528	335
17			571	558	365
18			578	570	384
19			575	576	405
20			568	579	416
21			558	572	380

All temperatures recorded are °C.

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TABLE 3

TASK III FURNACE CALIBRATION

2-27-52
Run #2
Furnace #2
S-1 Crucible
3.312" Can, Sanded
With MgO
Air Atmosphere

Time	KW Input	Recorder Temp.	Top Temp.	Center Temp.	Bottom Temp.
0	0	35	25	25	25
1	20	250	25	25	25
2	20	375	27	25	30
3	20	500	30	33	40
4	19	590	48	45	55
5	20	650	68	68	80
6	20	675	100	103	112
7	20	675	137	149	149
8	10	750	187	197	190
9	19	745	227	267	260
10	12	750	270	310	275
11	13	745	305	350	305
12	16	745	345	395	340
13	15	750	380	436	375
14	13	755	418	480	407
15	12.5	755	453	516	440
16	OFF		463	550	442
17			437	510	490
18			475	525	500

All temperatures recorded are °C.

[redacted]

TABLE 4

TASK III FURNACE CALIBRATION

3-4-52
Run #3
Furnace #2,
S-1 Crucible
3.312" Can, Sanded
With MgO
Air Atmosphere

Time	KW Input	Recorder Temp.	Top Temp.	Center Temp.	Bottom Temp.
0	0	25	25	25	25
1	22	200	25	25	25
2	22	340	27	30	25
3	22	460	40	40	27
4	21.5	575	60	60	40
5	21.5	650	100	100	70
6	22	715	145	150	95
7	8	755	195	205	130
8	7	760	255	275	170
9	10	740	305	335	210
10	15	740	350	390	245
11	15	750	390	435	280
12	14	760	425	475	305
13	12	760	465	510	340
14	12	750	500	545	370
15	12	740	535	570	395
16	OFF		557	600	425
17			565	610	445
18			570	615	455
19			570	610	460
20			565	605	465
21			555	595	465
22			545	580	465

All temperatures recorded are °C.

TABLE 5

TASK III FURNACE CALIBRATION

3-4-52
Run #4
Furnace #1
S-1 Crucible
3.312" Can, Sanded
With MgO,
Air Atmosphere

Time	KW Input	Recorder Temp.	Top Temp.	Center Temp.	Bottom Temp.
0	0	40	25	25	25
1	22	280	25	25	25
2	22	510	25	25	30
3	22	670	35	33	40
4	12	750	55	47	55
5	12	750	75	67	80
6	12	750	100	105	110
7	12	750	140	150	145
8	12	750	190	202	190
9	12	750	235	267	235
10	12	750	285	312	280
11	12	745	325	350	315
12	12.5	745	355	395	340
13	12.5	750	390	435	370
14	12	750	430	480	405
15	12	750	470	515	440
	OFF		480	550	450
			485	565	445
			485	570	435

All temperatures recorded are °C.

[redacted]

TABLE 6

TASK III FURNACE CALIBRATION

3-4-52
Run #5
Furnace #3,
S-1 Crucible
3.312"Can, Sanded
With MgO
Air Atmosphere

Time	KW Input	Recorder Temp.	Top Temp.*	Center Temp.	Bottom Temp.
0	0	[illegible]		35	35
1	24	350		35	35
2	22	500		[illegible]	45
3	20	575		85	100
4	12	650		125	135
5	12	650		150	150
6	12	650		180	175
7	11	655		225	215
8	11	660		270	240
9	11	660		300	265
10	[illegible].5	655		335	290
11	[illegible]	650		375	320
12	[illegible].5	650		395	340
13	11.5	650		425	360
[illegible]	11.5	650		445	380
15	11.5	650		465	400
16	[illegible].5	650		480	420
[illegible]	[illegible]1.5	650		500	435
[illegible]	OFF			510	440
19	[illegible]			515	450
[illegible]	[illegible]			515	450
[illegible]	[illegible]			510	448

[illegible]res recorded are °C.

[illegible]ocouple Open.