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HIGH TEMPERATURE THERMOCOUPLE

RESEARCH AND DEVELOPMENT PROGRAM, 2 H

APPENDIX 2

CALIBRATIONS

To

SUMMARY REPORT NO. T-1097

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Prepared For

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APPENDIX 2

CALIBRATION DATA

1.0 GENERAL

The calibration data presented in this appendix was obtained from three principal sources: Auto-Control Laboratories, Southern Research Institute, and Hoskins Manufacturing Company. Others are identified in either tables or graphic presentations, where applicable.

2.0 CALIBRATIONS

2.1 Calibrations, 32°F to -320°F

In examining published calibration data for the Tungsten vs. Rhenium system, it was noted that there were no values of EMF vs. temperature for the 32°F to -320°F region. Although the primary objective of this program was operation at elevated temperatures, it was felt that the low end should be investigated in order to provide the most comprehensive data possible.

ACL, therefore, performed calibrations on a test probe at three temperatures: the boiling point of liquid Nigrogen (-320°F), the equilibrium temperature of a mixture of solid Carbon Dioxide and Acetone, and the melting point of ice.

The test gauge was fabricated from Tungsten vs. Tungsten 26% Rhenium

2.1 Calibrations, 32°F to -320°F (Cont'd.)

wire, .020 inch diameter, Hoskins Tungsten Lot No. 34, and Tungsten 26% Rhenium Lot No. 2610. The lowest output in the calibration curve provided for this matched set of wires was .292 mv at 200°F.

Copper leads attached to each of the thermocouple wires were brought out to a Type 8662 L & N Precision Portable Potentiometer, where the output was read, after the bridge was compensated.

The hot junction of the test gauge was first immersed in the ice bath, with the positive lead of the gauge attached to the positive terminal of the bridge. There was no discernible output. The leads were reversed, with the same result.

The hot junction was then immersed in the Acetone and CO_2 bath, and the same results were obtained. In the LN_2, the same result was again observed.

The junction was then placed in the ambient air, and the transition between the copper and the thermocouple materials was immersed in the LN_2. A positive output of .03 millivolts was measured on the bridge. The same test performed in the acetone and CO_2 yielded a negative output of .005 mv. In the ice bath, there was no discernible output, either negative or positive.

2.1 Calibrations, 32°F to -320°F (Cont'd.)

The effects as measured above are of particular interest in applications where the transition section of the gauge might be maintained at some low temperature, while the hot junction was operating at an elevated temperature. The tests indicate that Tungsten vs. Tungsten 26 Rhenium has a negligible output at low temperatures, and that errors in the output are very low if copper lead extensions are used when the transition is maintained at temperatures from -320°F to near ambient. This opens up the possibility of dispensing with a reference junction while still maintaining accuracy within acceptable limits, in applications where the transition area is held at low temperatures. At ACL's request, Hoskins Manufacturing Company repeated these tests in their facility, at a larger number of discrete points within the range.

The graph of Figure 1 is a plot, showing both ACL and Hoskins data. Considering experimental error, it is felt there is a good agreement.

2.2 Test Oven, Low Temperature

Because of the well known difficulties in obtaining accurate temperature readings at low temperature with an optical pyrometer, and under conditions other than "blackbody", a small oven was constructed for use in calibrating. The oven was made per recommendations of the National Bureau of Standards*. It consisted of an alumina tube fitted with a side port for viewing, surrounded by an electrical heater. The heater

*NPS Monograph 41,
Theory and Methods of
Optical Pyrometry

[redacted]

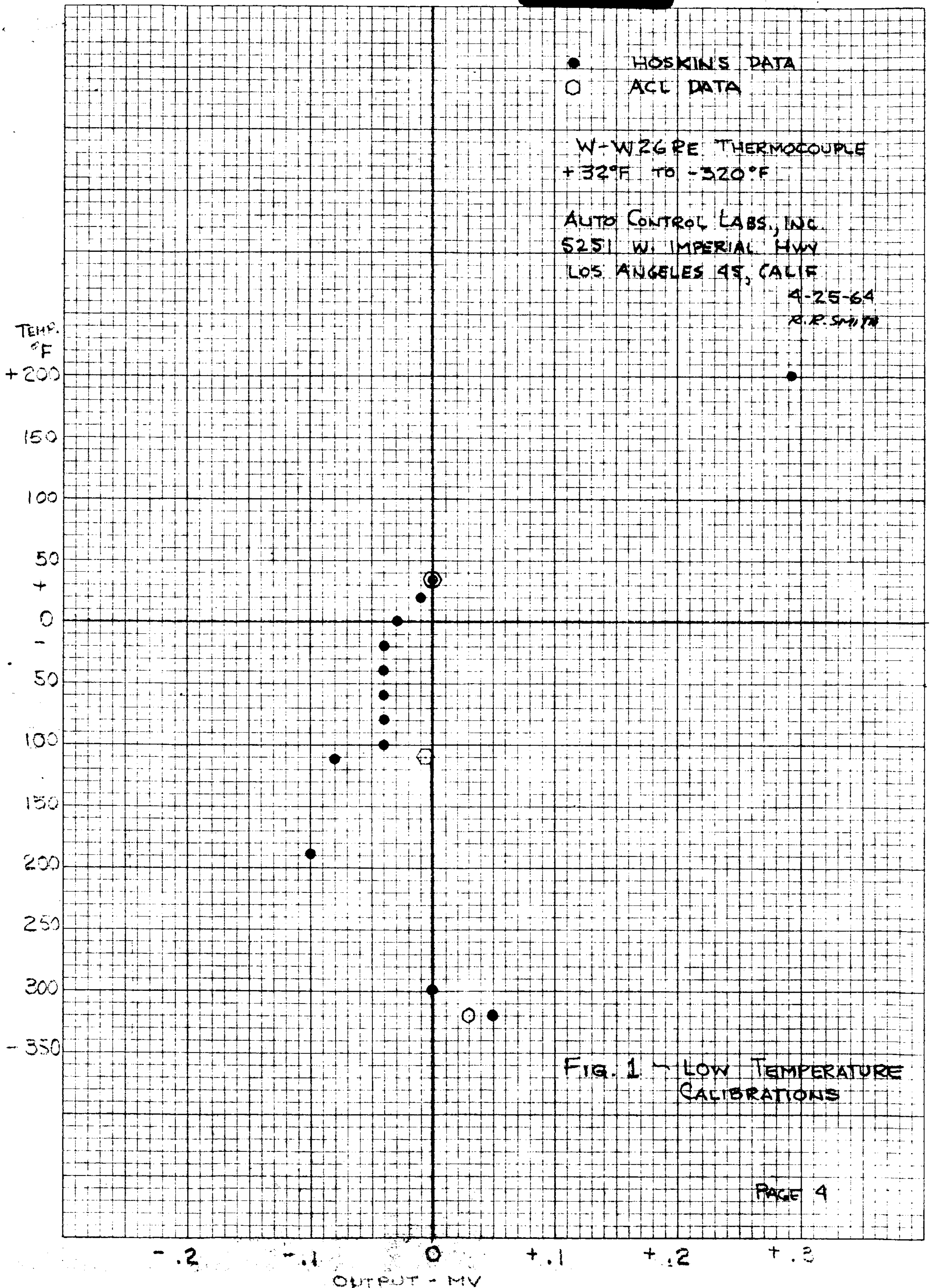

FIG. 1 – LOW TEMPERATURE CALIBRATIONS

2.2 Test Oven, Low Temperature (Cont'd.)

was helically wound over the alumina tube except in the region of the viewing port, where parallel windings were used. High temperature cement (Sauereisen No. 1) was used to coat the heater windings. The tubes were insulated with Fiberfrax, and the whole assembly was enclosed in a metal can.

The heater wire was Tophet A 28 gauge, with a total resistance of 22 ohms. 115 VAC, 1 phase, 60 cycle power was used, controlled with a 7.5 ampere Variac. The voltage settings for approximate temperatures were determined with a calibrated Chromel-Alumel thermocouple, and a 9.34 gram Tungsten load in the oven. When temperature stability was reached, the voltage was recorded, and a number of readings were taken on the thermocouple junction with the optical pyrometer. Typical comparative readings are given in Table I below.

Table I

OVEN CALIBRATION

Calibration Temperature °F	Optical Pyrometer Reading	Calibration Temperature °F	Optical Pyrometer Reading
1513	1517	1965	1960
1513	1513	1980	1976
1729	1733	1980	1978
1745	1742	2146	2140
1960	1944	2148	2130

2.2 Test Oven, Low Temperature (Cont'd.)

The readings listed in Table I represent the best and worst readings taken by both experienced and inexperienced personnel. Experienced individuals are able to repeat readings to a high degree. Individuals among them are capable of tracking the thermocouple readings within a few degrees F.

2.3 Calibrations, 1300°F to 2200°F

A series of calibration runs were made in the low temperature oven to establish calibration of the Type 4735 gauges. These are presented in Figure 3. A schematic diagram of the test setup is shown below in Figure 2. During the runs, an attempt was also made to determine an emissivity correction for use of the optical pyrometer.

Run No. 1

Basic setup conditions for Run No. 1 were as follows:

Gauge Serial No.:	001
Immersion Depth:	2.3 inches
Atmosphere:	Argon
Temperature Range:	1300°F to 2300°F

This run was made without a reference thermocouple in the oven. All observations were made directly on the gauge tip, for a large number of points within the range. These data are shown in Figure 3 as circles (ascending) and filled circles (descending). The run showed excellent

2.3 Calibrations, 1300°F to 2200°F (Cont'd.)

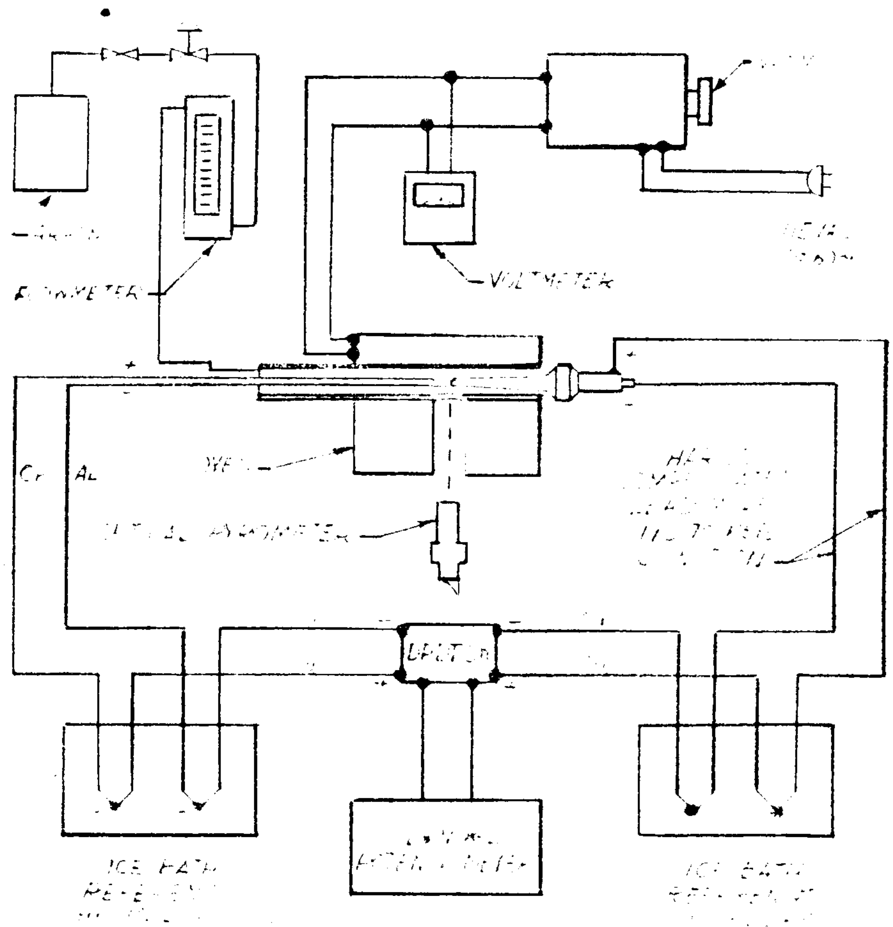

Figure 2

CALIBRATION TEST SETUP

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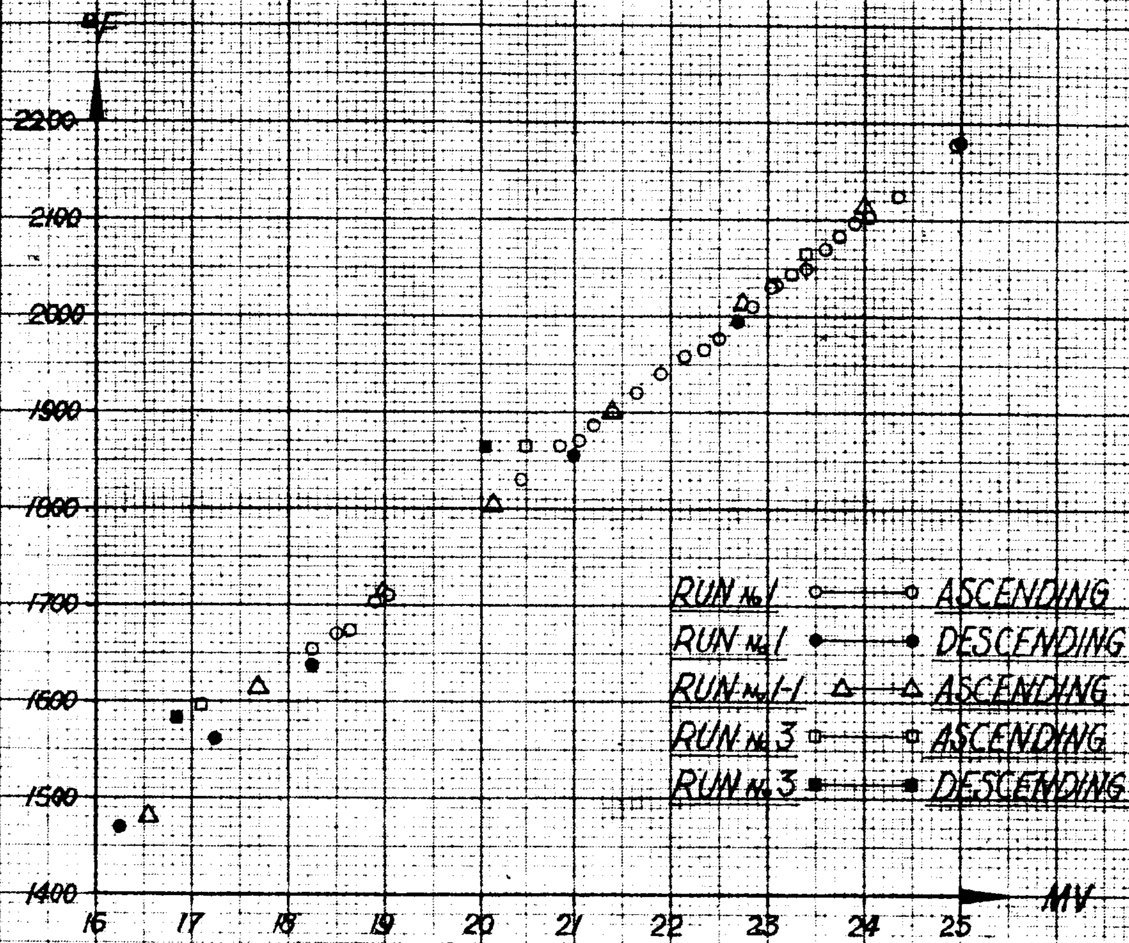

FIG. 3

CALIBRATION, ACL THERMOCOUPLE, TYPE 4735

1400°F TO 2200°F

[redacted]

2.3 Calibrations, 1300°F to 2200°F (Cont'd.)

repeatibility and very low hysteresis. Running time was 1 hour 10 minutes.

Run No. 1-1

Using the same setup as in Run No. 1, optical pyrometer readings were taken (ascending only) over the same temperature range. The data points are plotted as triangles in Figure 3. The curve was shifted slightly, but still showed good repeatibility within the range. Running time was 30 minutes.

Run No. 2

Run No. 2 was aborted, after one data point was taken, when it was discovered that copper wire, rather than compensated lead wire, had been run to the reference junction. The single point was 6.2 mv at 700°C (1292°F) with a reference thermocouple in the oven.
Total running time was 30 minutes.

Run No. 3

Run No. 3 was made with a calibrated reference thermocouple in the oven, located in proximity to, but not touching, the 4735 gauge. The installation was made such that the pyrometer filament could be matched alternately with both thermocouple junctions.

2.3 Calibrations, 1300°F to 2200°F (Cont'd.)

Run No. 3 (Cont'd.)

Basic setup conditions for Run No. 3 were as follows:

Gauge Serial No.:	001
Immersion Depth:	2.0 inches
Atmosphere:	50% Argon - 50% Air
Temperature Range:	1300°F to 2100°F

At each stabilized temperature, at least two optical pyrometer readings were taken on both the reference thermocouple and the 4735 gauge. At the same time, the emf outputs of both thermocouples were recorded.

Ascending readings for the 4735 gauge are shown as open squares in Figure 3. Descending readings are shown as filled squares. As plotted the curve shows good repeatibility compared with Runs No. 1, and 1-1. Total running time was 2 hours 27 minutes.

Discussion

When the curves developed during Runs No. 1, 1-1, and 3 are plotted as a composite curve to the same scale as the Southern Research Institute final calibration curve, (see Figure 4) it is seen that the curves taken by ACL are very closely parallel, although shifted to a higher emf value for the same temperature.

It should be noted that the SRI curves of immersion depth vs. output

[redacted]

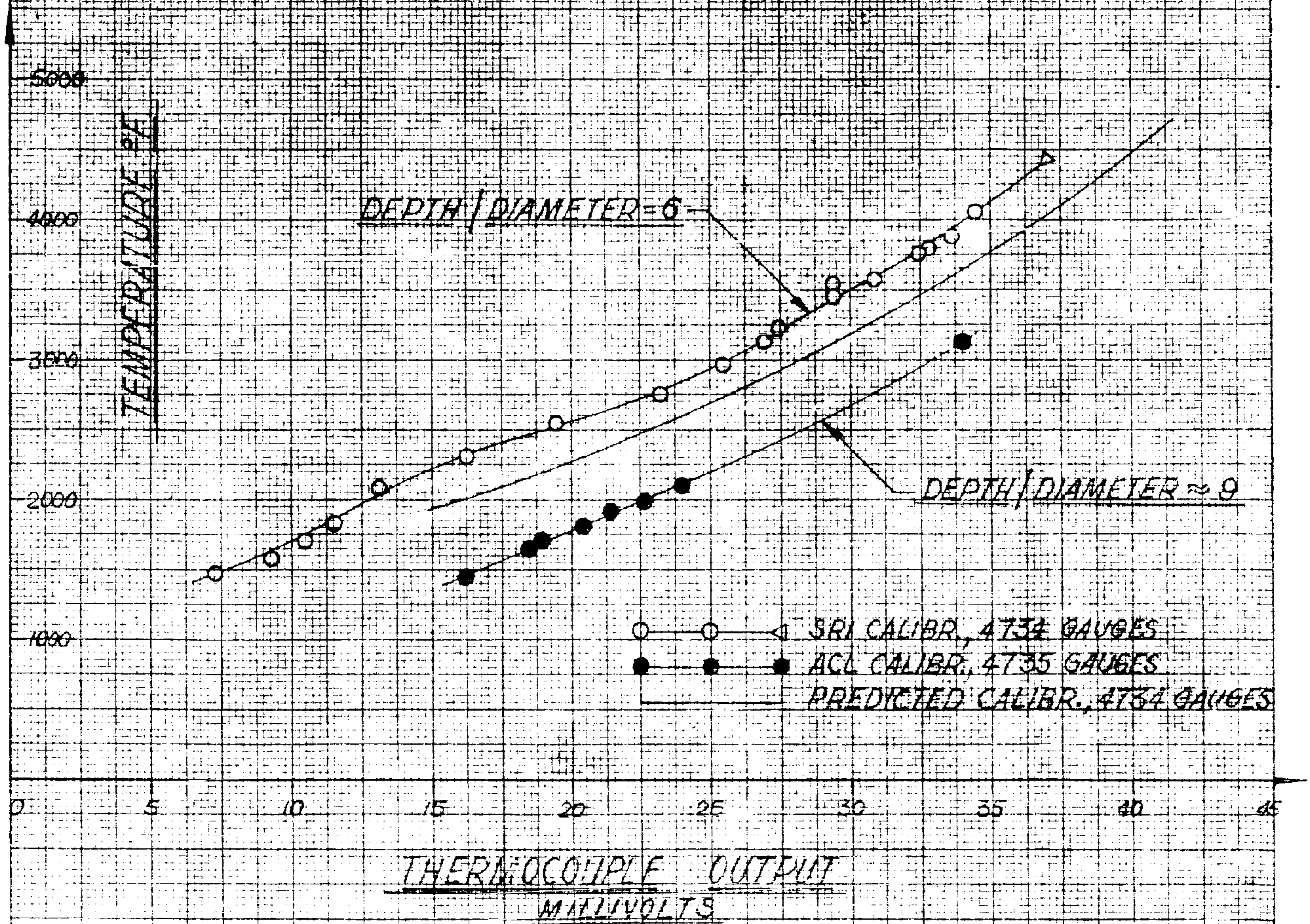

FIGURE 4

[redacted]

2.3 Calibrations, 1300°F to 2200°F (Cont'd.)

Discussion (Cont'd.)

were taken for the Type 4734 gauges at immersion depths of from 1/4" to 1-5/16". Their final calibration curve was apparently taken at 1-7/16" immersion depth, which was the maximum penetration possible with the SRI blackbody cavity. All of the SRI curves showed an increase in emf for the same temperature with increase in immersion depth, i.e. - a shift to the right. The ACL calibrations show a similar shift. If the shift per increment of immersion depth on the Type 4734 gauges is considered when comparing the two sets of curves, the emf shift in the Type 4735 gauges can be partly accounted for.

The effect of different lead wires is of interest because the Type 4734 gauges incorporated Minneapolis-Honeywell compensated lead wire (copper - copper nickel) whereas the ACL tests of the Type 4735 gauges incorporated Harco compensated lead wire.

Thus far in the calibrations, it was concluded that the Type 4735 gauges are capable of extended operation within the temperature range investigated, and the output of the gauge vs. temperature is highly repeatible for a given thermal equilibrium condition. (Immersion depth and ambient temperature constant).

2.3 Calibrations, 1300°F to 2200°F (Cont'd.)

Calibration, 3000°F

In order to establish a calibration point higher in temperature than those taken in the calibrations between 1300°F and 2300°F, the same gauge, Serial No. 001, was mounted in a steel deflection shield, and run in an oxy-acetylene burner. The point shown near 3000°F in Figure 4 is the average of twenty-eight readings. The large number of readings were taken because of fluctuations in temperature seen during the run. The highest temperature was 3182°F, the lowest 2948°F.

2.4 General

Because of serious questions, regarding calibrations of the ACL gauges, raised during a conference at M-ASTR-I on 26 February, 1964, a major effort was directed toward accumulating a large body of calibration and stability data. It was ACL policy in this program to proceed methodically toward the higher temperature ranges in order to obtain a maximum of information from each gauge fabricated for test. From early results, which were encouraging, there was a temptation to proceed immediately to the upper limits of temperature. However, this was resisted because of the complexity of inter-related factors affecting the performance of the gauges, and the desirability of failure analysis without the difficulty of multi-parameter obscurement of results.

Steady progress was made in overlapping temperature ranges from

2.4 General (Cont'd.)

temperatures of 1000°F to 5000°F. The curves and analyses presented in this section cover in detail the range from about 2000°F to 4400°F.

It was the firm belief of ACL that, in particular, the detailed examination of the range from 3800°F to 4300°F was critical to continued progress because of shift characteristics in this range revealed by others working on similar materials in the same temperature range, and to verify readings taken previously.

Calibration Set up, High Temperature Oven

In high temperature tests performed with the ACL gas burner, considerable difficulty was experienced in obtaining stabilized temperatures. Much time was expended also, in reducing test data to usable form because of uncertainties in making optical measurements due to flame swirls, and in correcting readings for emissivity. ACL set up, therefore, an existing high temperature furnace for high temperature calibrations. The burner was subsequently used only for oxidation tests.

Modification of the ACL high temperature furnace was made to accommodate the Type 4735 gauges. The furnace is fabricated as follows: An outer shell, made of Zirconium Oxide coated graphite, surrounds a calibration cavity, which is made of Tungsten. A sighting port for optical observation is located opposite the tip of the gauge. A small hole in the

2.4 General (Cont'd.)

Calibration Set up, High Temperature Oven (Cont'd.)

bottom of the cavity permits its interior to be flooded with Argon gas. The space between the cavity and the shell is also flooded with Argon. For viewing the tip, an optically flat quartz viewing port is located in the shell.

The furnace is heated by a heliarc drawn between an electrode and the Tungsten cavity. Temperatures of at least 5000°F were reached. Optical temperature measurements of the gauge were made with a micro-optical pyrometer.

The oven set up is illustrated in Figures 5 and 6 of this report. The oven was quite easily controllable up to about 4500°F. As power was increased beyond that point, great care had to be exercised in increasing temperature. A relatively large power increase can cause the Tungsten cavity to melt.

Calibrations

Two test gauges, whose essential characteristics were identical to those of the Type 4735 gauges, were used in the series of tests. The only significant difference was in the depth-to-diameter ratios resulting from a smaller sheath diameter employed to minimize the effect of immersion on the millivolt output for discrete cavity temperatures.

BY K. DATE 1-30-64 SUBJECT CALIBRATION OVEN SKETCH SCALE 1:1 SHEET NO. 1 OF 1

CHKD. BY ______ DATE ______ JOB NO. T-097

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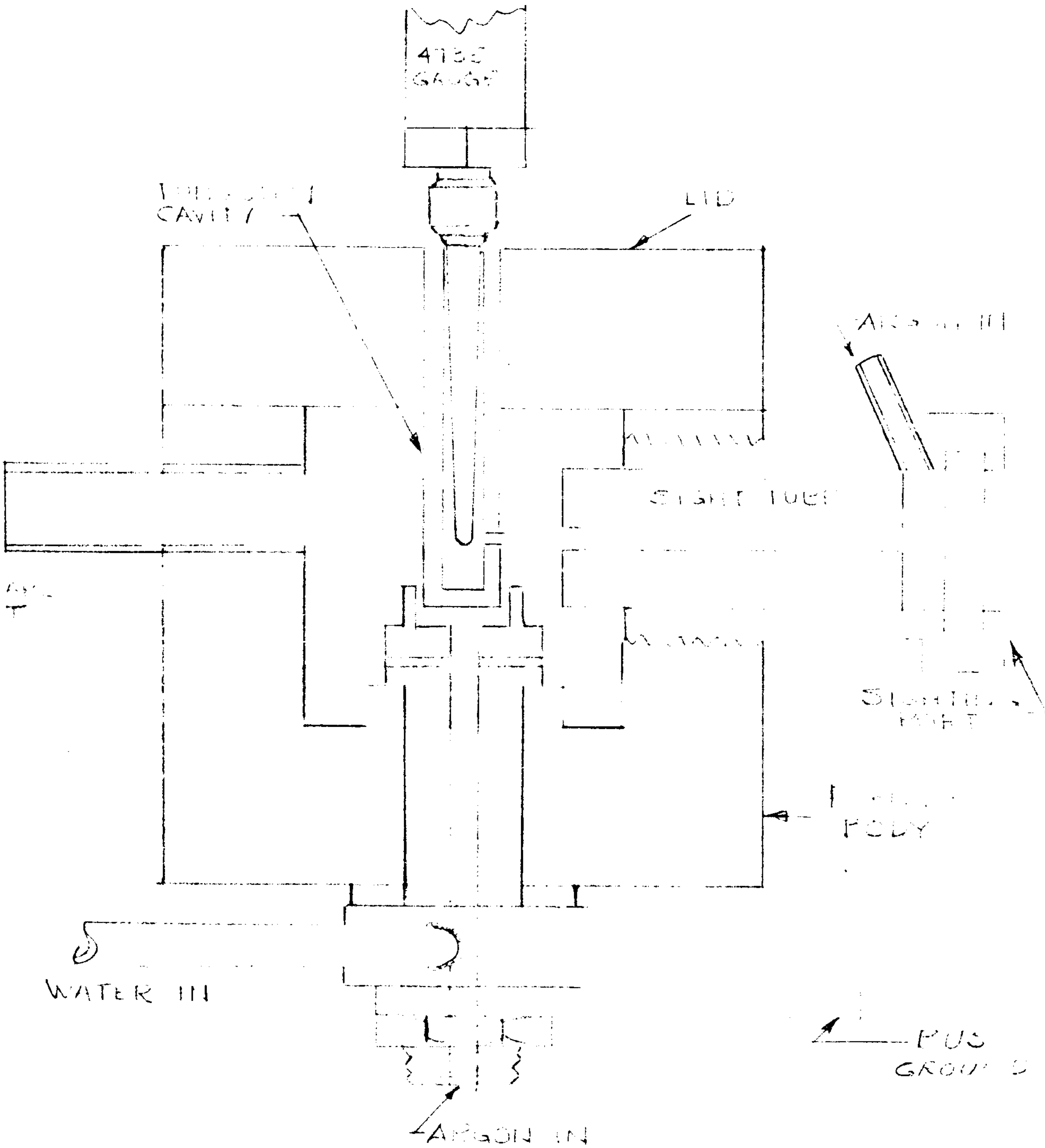

FIG. 5 – CALIBRATION OVEN

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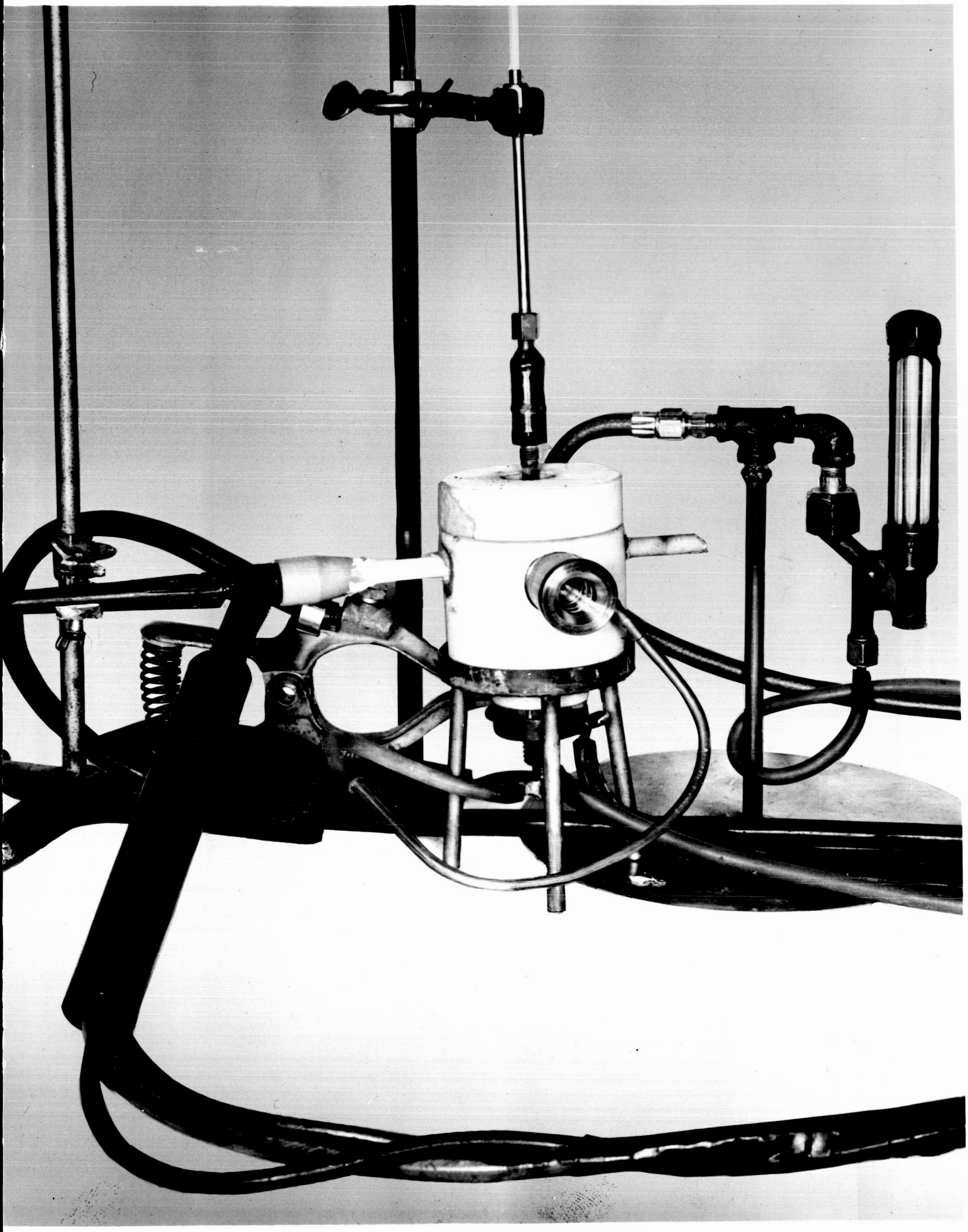

2.4 <u>General</u> (Cont'd.)

<u>Test Method</u>

The gauge was mounted in a holding fixture which permitted depth adjustment such that the tip of the gauge could be viewed directly, through a 1/16 inch sighting hole in the wall of a Tungsten cavity. The atmosphere in the oven was Argon.

Continuous leads of the thermocouple materials were brought out to an ice bath reference junction. Individual copper leads were then connected to a calibrated Leeds & Northrup Type 8662 Precision Potentiometer for reading the millivolt output. The potentiometer was carefully balanced between runs.

A Pyrometer Instrument Company Model 95-C Micro-optical Pyrometer was used to establish brightness temperatures of the tip of the gauge at each stabilized oven setting. Stability was determined by a cross-check between the optical pyrometer and the potentiometer: i.e., when there was no observable change in brightness, and the millivolt output remained steady at a given oven power setting. Sightings were made through a quartz viewing port whose interior surface was constantly irrigated with clean Argon gas. The thickness of the quartz was .306 inch. Brightness temperatures were not corrected for the quartz. The discussions in the following paragraphs cover each run made.

2.4 General (Cont'd.)

Run No. 1

Run No. 1 was made from about 1700°F to just over 4000°F and return to about 2100°F. The data taken during this run is shown in Table II and is shown plotted in Figure 7. Figure 8 is a comparison of the observed temperature versus indicated temperature for perfect agreement with the Hoskins supplied curve for Tungsten vs. Tungsten 26% Rhenium wire. The line at 45° in Figure 8 represents perfect agreement. All points above the 45° line are negative Delta T's, all those below the line are positive. In light of subsequent runs the crossovers from negative to positive at about 3300°F to 3600°F are believed due to operator error. One operator took all pyrometer readings. Total time for the run was 1 hour, 30 minutes, with about three minutes at 4000°F.

Run No. 2

After Run No. 1 the gauge was permitted to cool to room ambient temperature, and was again heated to just over 4000°F. The same operator as in Run No. 1 took all pyrometer observations. The plot of emf vs. temperature is shown in Figure 7. The comparison plot is shown in Figure 8. This run was not made descending because of a minor malfunction in the oven control. Total running time was forty minutes, with about 2 minutes at 4000°F. Tabulated data is shown in Table III.

2.4 General (Cont'd.)

Run No. 3

Run No. 3 was made from about 1700°F to about 4250°F. A different operator than in Runs 1 and 2 took the pyrometer readings. Results of Run 3 were in good agreement with Runs 1 and 2. Tabulated data is shown in Table IV. The plot of temperature vs. emf is shown in Figure 9. The comparison plot is shown in Figure 10. Total running time was 40 minutes, with about 4 minutes above 4000°F.

Run No. 4

Run No. 4 was made from about 1800°F to about 4400°F, and return to about 2600°F. The pyrometer operator was the same as in Runs No. 1 and 2. Agreement with Run No. 3 was generally good. A trend toward agreement with the 45° line in the comparison plot was seen between this run and previous runs at about 3500°F. The temperature vs. emf curve is shown in Figure 11. The comparison plot is shown in Figure 12. Tabulated data is presented in Table V. Total running time was 29 minutes, with about 5 minutes above 4000°F.

Run No. 5

After review of the first four runs, it was decided to make Run No. 5 over a longer period of time, for a check on stability, and to have four operators take observations at the same observed temperature, to determine

2.4 General (Cont'd.)

Run No. 5 (Cont'd.)

the effect of operator error.

Run No. 5 was made from about 1800°F to about 4300°F and return. To identify observations taken by the different operators, the plots for Run No. 5 are numbered 5, 5-1, 5-2 and 5-3. Run No. 5 is shown in Figure 13, and its comparison plot in Figure 14. Run No. 5-1 is shown in Figure 15, and its comparison plot in Figure 16. The temperature versus emf plots for Runs No. 5-2 and 5-3 are shown in Figures 17 and 18 respectively. The comparison plots for Runs No. 5-2 and 5-3 were plotted together in Figure 19. Except for obvious operator errors, agreement between all of the Run No. 5 plots was good.

The total running time in Run No. 5 was 2 hours, 20 minutes, with 14 minutes above 4000°F. The tabulated data for Run No. 5 is shown in Table VI.

Run No. 6

Runs No. 6 through 8 were made on a 4700-2 gauge, which was identical in construction to the 4700-1 gauge, except that the stem of the 4700-2 gauge is oval in shape, whereas the 4700-1 stem is cylindrical. Immersion depth was the same, and the same cavity was used.

2.4 General (Cont'd.)

Run No. 6 (Cont'd.)

Run No. 6 was made from about 1800°F to a high pyrometer reading of about 4700°F. The high reading is unreliable except as a minimum, because the last pyrometer reading of 4676° was taken just before an inadvertent increase in oven power caused a "punch-through" the wall of the Tungsten cavity, and the tip of the gauge was fused to the inside wall of the cavity before the power could be cut off. Since the melting temperature of Tungsten is about 6000°F, it can be assumed that the temperature of the probe tip was between 4676°F and 6000°F. The temperature may have been below the melting point of the Tungsten-Tungsten 26% Rhenium alloy leg, which is somewhat lower than Tungsten, because upon examination after cooling, the gauge was found to be undamaged except for a small mass of melted and fused Tungsten near the tip. At the time of the incident, the technician reading the L & N potentiometer was tracking the output and recorded a high, before the galvanometer was driven off-scale, of 46.78 millivolts. This is the highest output reading yet obtained on any gauge. It is emphasized here that no attempt is made to correlate this output with a temperature, since stability was not attained. The highest reliable reading of temperature vs. emf was at just under 4000°F.

The plot of temperature vs. emf for Run No. 6 is shown in Figure 20. The comparison plot is shown in Figure 21. Tabular data is given in

2.4 <u>General</u> (Cont'd.)

<u>Run No. 6</u> (Cont'd.)

Table VII. Total running time was 33 minutes, with time above 4000°F of about 2 minutes.

<u>Run No. 7</u>

After the 4700-2 gauge had cooled, the gauge with the cavity attached, was examined, and the cavity was carefully ground away to permit removal of the gauge. An electrical check revealed no apparent damage. The cavity was replaced and Run No. 7 was made from about 2100°F to about 4300°F and return to about 2500°F. From the temperature versus emf curve shown in Figure 22, it appeared that the over temperature condition of Run No. 6 had not damaged the gauge. The comparison plot of Figure 23 shows good agreement with the data taken on the 4700-1 gauge. Tabular data is shown in Table VIII. Total running time was 59 minutes, with 2 minutes above 4000°F.

<u>Run No. 8</u>

Run No. 8 was made from about 2400°F to about 4100°F and return to about 2500°F, with the same pyrometer observer as in Run No. 6. The temperature vs. emf plot is shown in Figure 24, and the comparison plot in Figure 25. Total running time was 34 minutes, with about three minutes above 4000°F. The tabulated temperature vs. emf is given in Table IX.

2.4 General (Cont'd.)

Discussion

A comparison of all runs with curves previously developed shows good agreement with the data taken at large depth to diameter ratios in previous runs. The curves tend to show an increase in stability with temperature cycling. A recapitulation of running time is given in Table X.

2.5 Calibrations, 4200°F

To conclude tests within this range, one type 4700 gauge was subjected to tests at temperatures from about 2000°F to about 4200°F. Fewer data points were taken over the range to allow more time for stability at each point observed with the optical pyrometer. Tabular data is presented in Table XI. The temperature vs. emf data were plotted and are shown in Figure 26. The comparison plot for agreement with the published data was available at the time of the tests for the temperature range from 4200°F up, the local Hoskins representative was contacted to see whether any unpublished data was available. The Hoskins representative reported that he had been advised by his engineering staff that the curve could be exprapolated from the 4200°F point to 5000°F without an appreciable loss of accuracy. Plots of millivolts per degree F in this range do not agree with such extrapolations, however, nor do they agree with either the Englehard curve or previously taken ACL data. Therefore they were not used.

2.4 General (Cont'd.)

TABLE NUMBER II

Temperature vs EMF, Run Number 1

Temp. °C	Temp. °F	Output MV	Time	Temp. °C	Temp. °F	Output MV	Time
954	1749.0	12.99	10:05	1615	2939.0	25.57	
977	1790.6	13.17		1620	2948.0	25.67	
1000	1832.0	13.90		1620	2948.0	26.05	
1006	1842.8	13.91		1641	2985.8	26.15	11:07
1061	1941.8	15.17		1650	3002.0	27.00	
1074	1965.2	15.19		1694	3081.2	27.95	
1128	2062.4	17.00		1694	3081.2	27.85	11:36
1133	2071.4	16.90		1700	3092.0	28.15	11:15
1138	2080.4	16.85		1725	3137.0	28.85	11:33
1141	2085.5	17.00		1787	3248.6	29.60	
1154	2073.0	16.95		1805	3281.0	32.41	
1330	2426.0	20.80		1875	3407.0	30.60	
1345	2453.0	20.65		1900	3452.0	33.00	
1350	2462.0	20.42		1940	3524.0	31.85	11:22
1358	2476.4	20.75		2008	3646.4	33.30	
1525	2777.0	24.55		2040	3704.0	33.30	
1568	2854.4	24.85		2082	3779.6	.05	
1568	2854.4	25.47		2170	3938.0	35.45	
1580	2876.0	25.54		2203	3997.4	36.25	11:28
1586	2886.8	25.53		2206	4001.0	36.20	11:27
1602	2915.6	26.25		2210	4010.0	36.05	

2.4 General (Cont'd.)

TABLE NUMBER III

Temperature vs EMF, Run Number 2

Temp. °C	Temp. °F	Output MV	Time	Temp. °C	Temp. °F	Output MV	Time
1161	2121.8	16.85	2:29	1590	2894.0	24.78	2:59
1222	2231.6	17.82	2:37	1690	3074.0	26.05	
1291	2355.8	18.56	2:40	1727	3140.6	27.48	
1372	2501.6	20.61	2:43	1883	3321.4	30.16	3:04
1400	2552.0	21.04		2005	3641.0	31.75	3:05
1425	2597.0	21.70	2:47	2102	3815.6	33.40	3:07
1478	2692.4	22.43		2220	4028.0	35.90	3:08
1521	2769.8	23.13	2:52	2222	4031.6	36.11	3:09
1545	2813.0	23.59	2:57				

2.4 General (Cont'd.)

TABLE NUMBER IV

Temperature vs EMF, Run Number 3

Temp. °C	Temp. °F	Output MV	Time	Temp. °C	Temp. °F	Output MV	Time
979	1784	11.10	9:26	2305	4181	36.95	
1023	1873	12.80	9:36	2320	4208	37.35	
1090	1994	14.50	9:37	2330	4226	37.50	9:56
1214	2217	17.21	9:40	2345	4253	37.52	9:57
1424	2595	20.22	9:42	2343	4249	37.60	9:58
1522	2772	22.57	9:43	2135	3875	34.65	10:00
1713	3115	26.93	9:45	1976	3589	31.66	10:01
1830	3326	29.10	9:46	1960	3560	30.92	
1800	3272	27.77	9:48	1874	3405	29.45	
1860	3380	29.00	9:50	1810	3290	28.28	
1890	3434	29.63	9:51	1704	3099	26.25	
2005	3641	32.00	9:52	1718	3124	26.87	
2060	3740	33.25	9:53	1586	2887	23.33	
2175	3943	34.87	9:54	1655	3011	24.80	
2290	4154	37.25		1465	2669	20.75	10:06

2.4 General (Cont'd.)

TABLE NUMBER V

Temperature vs EMF, Run Number 4

Temp. °C	Temp. °F	Output MV	Time	Temp. °C	Temp. °F	Output MV	Time
1010	1850	10.75	11:10	2375	4307	40.00	11:26
1050	1922	12.10	11:12	2440	4424	40.18	11:27
1093	1999	13.45	11:13	2290	4154	38.45	11:28
1163	2125	15.23	11:14	2205	4001	36.77	11:29
1372	2501	18.95	11:15	2040	3704	33.75	11:30
1545	2813	23.28	11:16	1920	3488	31.60	
1815	3299	29.58	11:17	1870	3398	30.05	
1715	3119	27.70	11:19	1722	3132	27.55	11:31
1840	3344	29.37		1770	3218	28.67	
1845	3353	30.50	11:21	1617	2943	25.23	11:33
2015	3659	33.27	11:23	1677	3051	26.47	11:32
2145	3893	36.10	11:24	1545	2813	23.52	11:34
2215	4019	37.77	11:25	1448	2638	20.60	11:35

2.4 General (Cont'd.)

TABLE NUMBER VI

Temperature vs EMF, Run Number 5

Temp. °C	Temp. °F	Output MV	Time	Temp. °C	Temp. °F	Output MV	Time
1013	1855	11.69	1:10	2340	4244	38.02	2:32
1050	1922	14.10	1:30	2370	4298	38.02	2:33
1053	1927	14.06	1:32	2230	4046	36.52	2:35
1172	2142	17.07	1:38	2237	4059	36.50	2:35
1172	2142	17.11	1:40	2240	4064	36.48	2:36
1318	2404	19.07	1:43	2100	3812	34.23	2:38
1309	2388	19.16	1:45	2117	3843	34.25	2:38
1615	2939	25.04	1:48	2100	3812	34.25	2:39
1613	2935	25.10	1:50	2101	3814	34.17	2:40
1738	3160	26.95	1:53	1937	3519	31.19	2:43
1730	3146	27.04	1:54	1957	3555	31.17	2:44
1945	3533	31.10	1:58	1935	3519	31.15	2:45
1965	3569	31.09	2:00	1848	3358	29.47	2:47
1744	3225	29.06	2:02	1840	3344	29.37	2:50
1805	3281	28.08	2:03	1838	3340	29.37	2:51
1776	3229	28.04	2:05	1845	3353	29.34	2:53
1800	3272	28.00	2:06	1753	3187	27.82	2:54
1870	3398	29.62	2:09	1770	3218	27.70	2:56
1880	3416	29.65	2:10	1753	3187	27.70	2:57
1975	3587	31.50	2:13	1700	3092	26.25	3:01
1965	3569	31.50	2:14	1655	3011	26.30	3:02
2105	3821	33.75	2:16				
2100	3812	33.81	2:17				
2105	3821	33.82	2:18				
2238	4060	36.33	2:22				
2234	4053	36.36	2:23				
2370	4298	38.10	2:26				
2330	4226	38.10	2:27				
2370	4298	38.11		1395	2543	18.72	3:24
2362	4284	38.11	2:28	1400	2552	18.59	3:26
2330	4226	38.11	2:29	1378	2512	18.57	3:27
2370	4298	38.11	2:30	1421	2590	18.50	3:30
2365	4289	38.02	2:31				

2.4 General (Cont'd.)

TABLE NUMBER VII

Temperature vs EMF, Run Number 6

Temp. °C	Temp. °F	Output MV	Time
985	1805	12.34	9:53
1002	1835	12.35	
1444	2631	23.14	
1453	2647	23.10	
1472	2682	24.68	
1526	2779	24.80	10:17
1534	2793	24.65	
1626	2959	27.10	10:19
1890	3434	31.57	10:22
2058	3736	35.00	10:26
2195	3983	37.83	
2580	4676	46.78	10:30

2.4 General (Cont'd.)

TABLE NUMBER VIII

Temperature vs EMF, Run Number 7

Temp. °C	Temp. °F	Output MV	Time	Temp. °C	Temp. °F	Output MV	Time
1148	2098	14.10	1:20	2120	3848	35.52	2:06
1167	2133	14.32	1:35	2230	4046	37.03	2:07
1158	2116	15.10	1:38	2350	4262	39.70	2:08
1260	2300	15.32	1:39	2160	3920	36.15	2:09
1438	2620	20.67		2020	3668	33.50	2:10
1453	2647	21.05	1:45	1920	3488	31.16	2:11
1820	3308	30.86	1:50	1774	3225	28.54	2:12
1810	3290	30.25	1:53	1708	3106	27.27	2:13
1816	3301	30.36	1:55	1645	2993	25.82	2:14
1820	3308	30.51	1:57	1576	2869	24.03	2:16
1910	3470	31.73	2:00	1440	2624	20.70	2:17
1980	3596	32.70	2:03	1390	2535	19.45	2:19
2035	3695	33.78	2:05				

2.4 General (Cont'd.)

TABLE NUMBER IX

Temperature vs EMF, Run Number 8

Temp. °C	Temp. °F	Output MV	Time	Temp. °C	Temp. °F	Output MV	Time
1315	2399	18.17	3:16	2160	3920	36.25	
1305	2381	18.17	3:17	2280	4136	38.20	
1380	2516	19.67	3:20	2290	4154	38.25	3:37
1438	2620	20.93	3:21	2175	3947	36.42	3:37
1475	2687	21.87	3:23	2085	3785	34.95	3:38
1508	2746	22.65	3:24	1920	3488	31.75	3:39
1536	2797	23.45	3:26	1835	3335	30.32	3:40
1570	2858	24.25	3:27	1760	3200	28.65	3:41
1600	2912	24.95	3:27	1700	3092	27.40	3:42
1640	2984	26.10	3:28	1650	3002	36.42	3:43
1680	3056	27.23	3:29	1600	3912	25.40	3:44
1760	3200	28.85	3:31	1541	2806	24.00	3:45
1803	3277	30.25	3:32	1480	2696	22.65	3:46
1970	3578	32.70	3:33	1411	2572	20.78	3:47
2080	3776	34.70	3:34	1380	2516	19.88	3:48

2.4 General (Cont'd.)

TABLE NUMBER X

Recapitulation of Running Time

Run No.	Gauge No.	Run Time Hrs	Run Time Min	Total Time Hrs	Total Time Min	Above 4000°F Min	Total Above 4000°F Min
1	4700-1	1	30	1	30	3	3
2	4700-1	0	40	2	10	2	5
3	4700-1	0	40	2	50	4	9
4	4700-1	0	29	3	19	5	14
5	4700-1	2	20	5	39	14	28
6	4700-2	0	33	0	33	2	2
7	4700-2	0	59	1	32	2	4
8	4700-2	0	34	2	06	3	7

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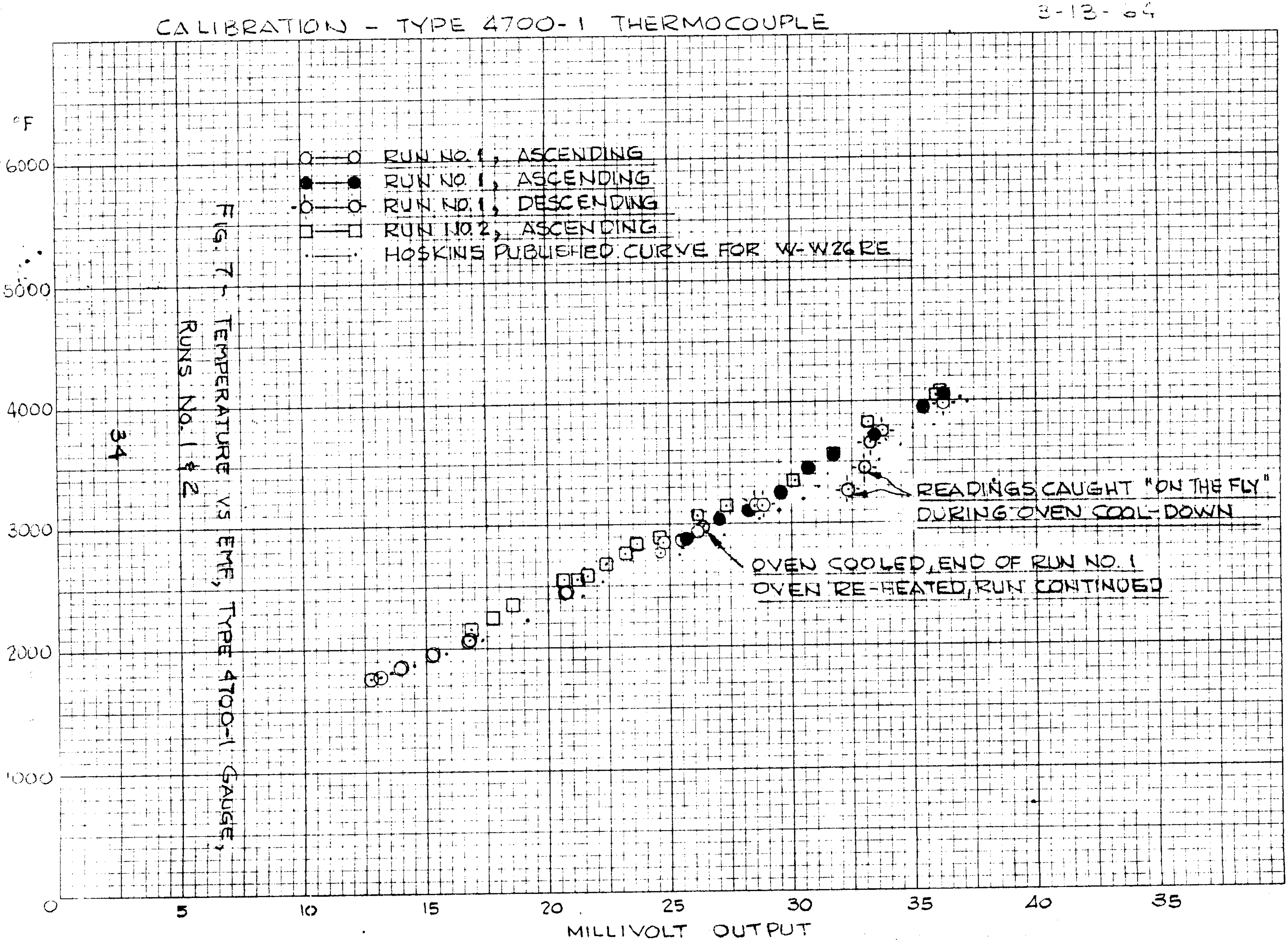

FIG. 7 - TEMPERATURE VS EMF, TYPE 4700-1 GAUGE,
RUNS NO. 1 & 2

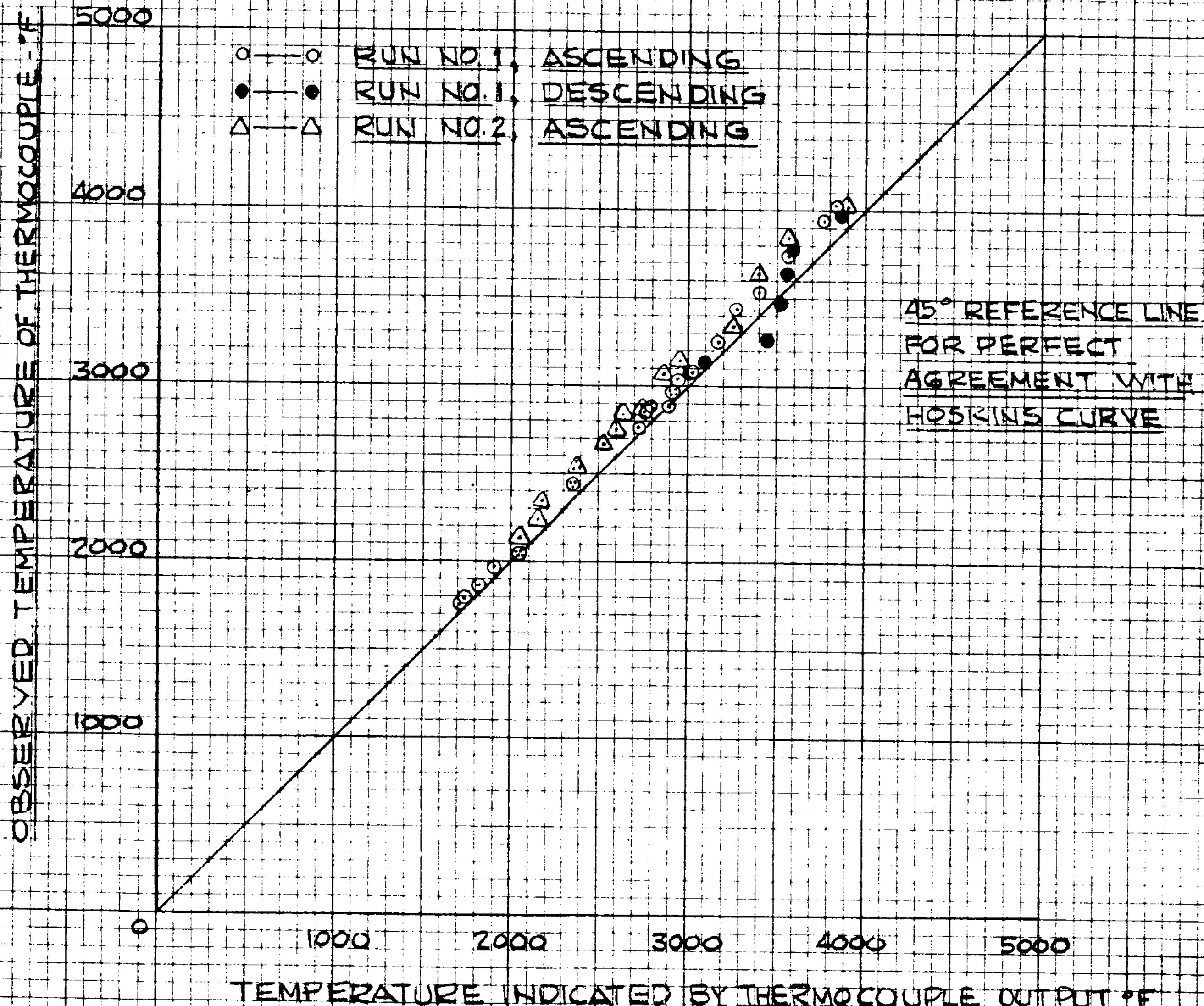

FIG. 8 - COMPARISON OF OBSERVED TEMPERATURE AND INDICATED TEMPERATURE VS HOSKINS CURVE, RUNS NO. 1 & 2

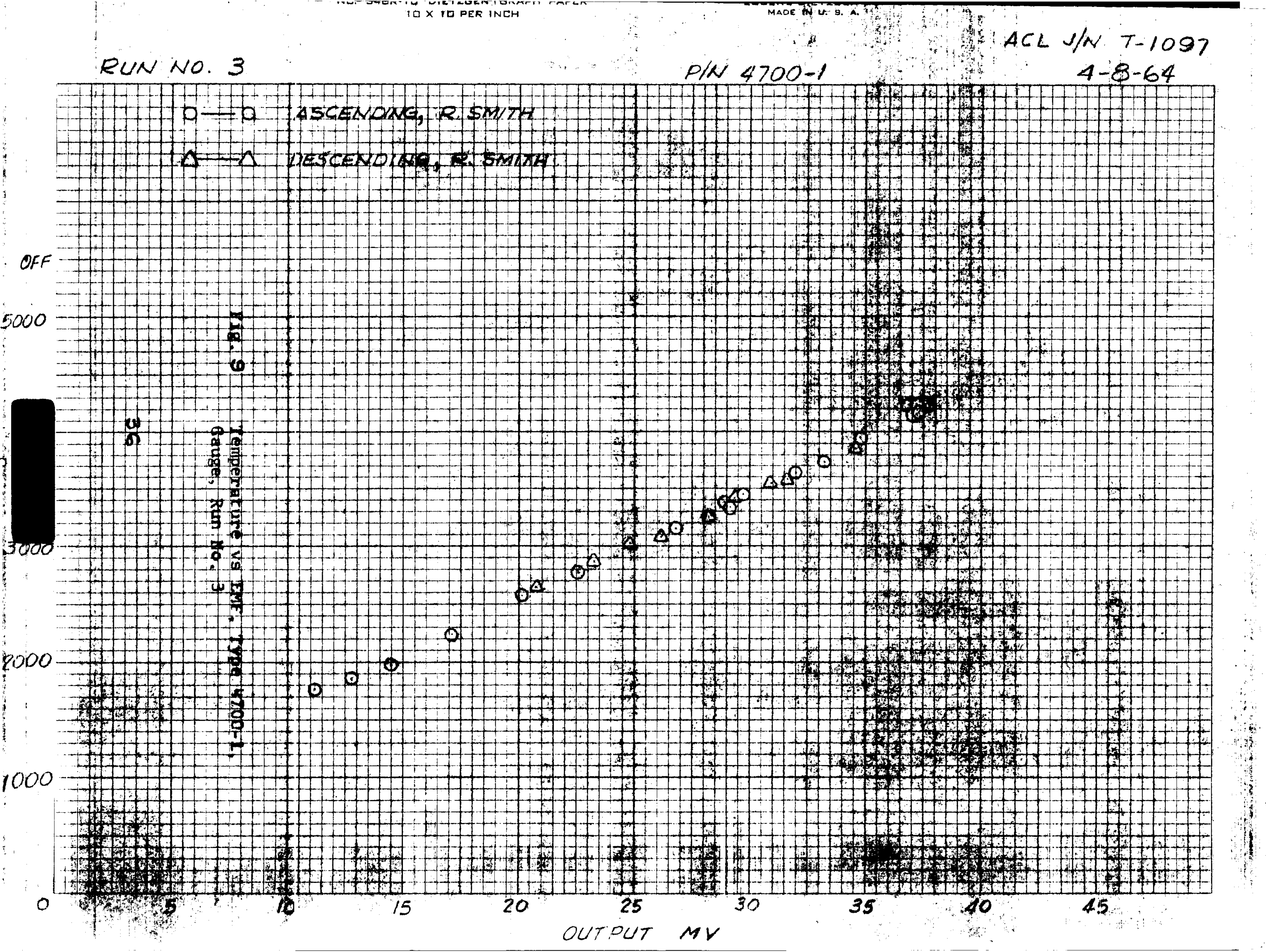

Fig. 9 Temperature vs EMF, Type 4700-1 Gauge, Run No. 3

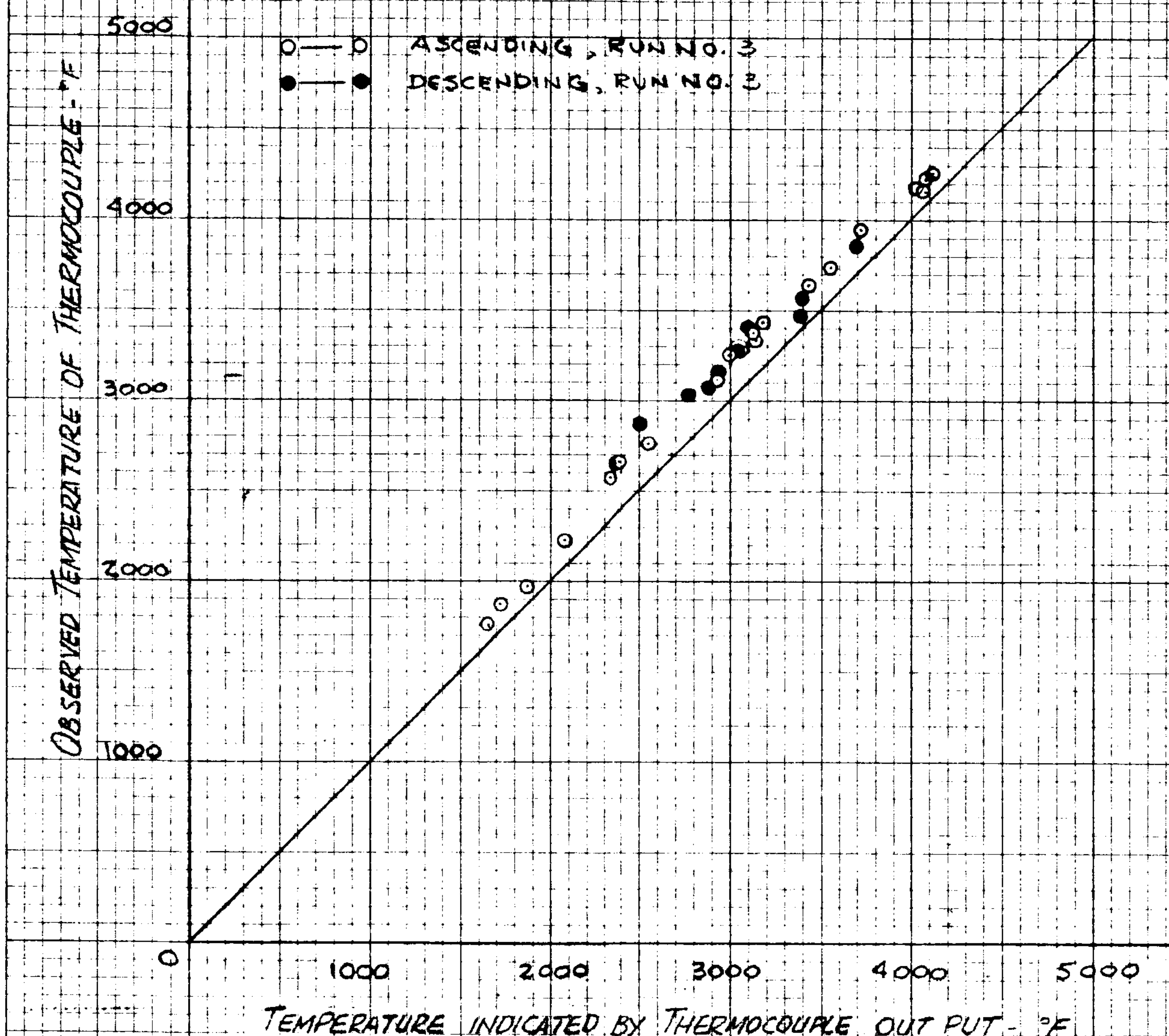

Fig. 10 Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve, Run No. 3

[redacted]

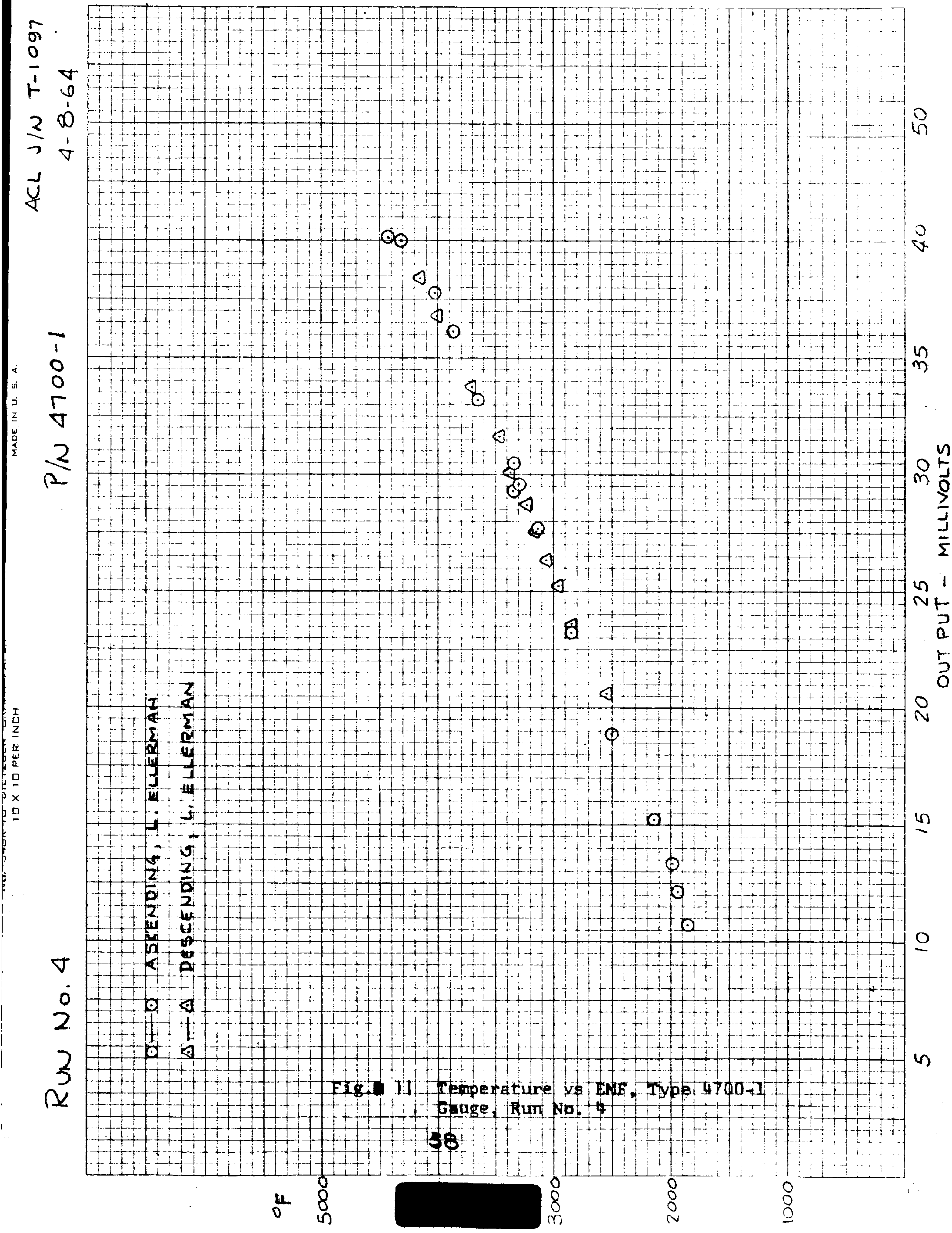

Fig. 11 Temperature vs EMF, Type 4700-1 Gauge, Run No. 4

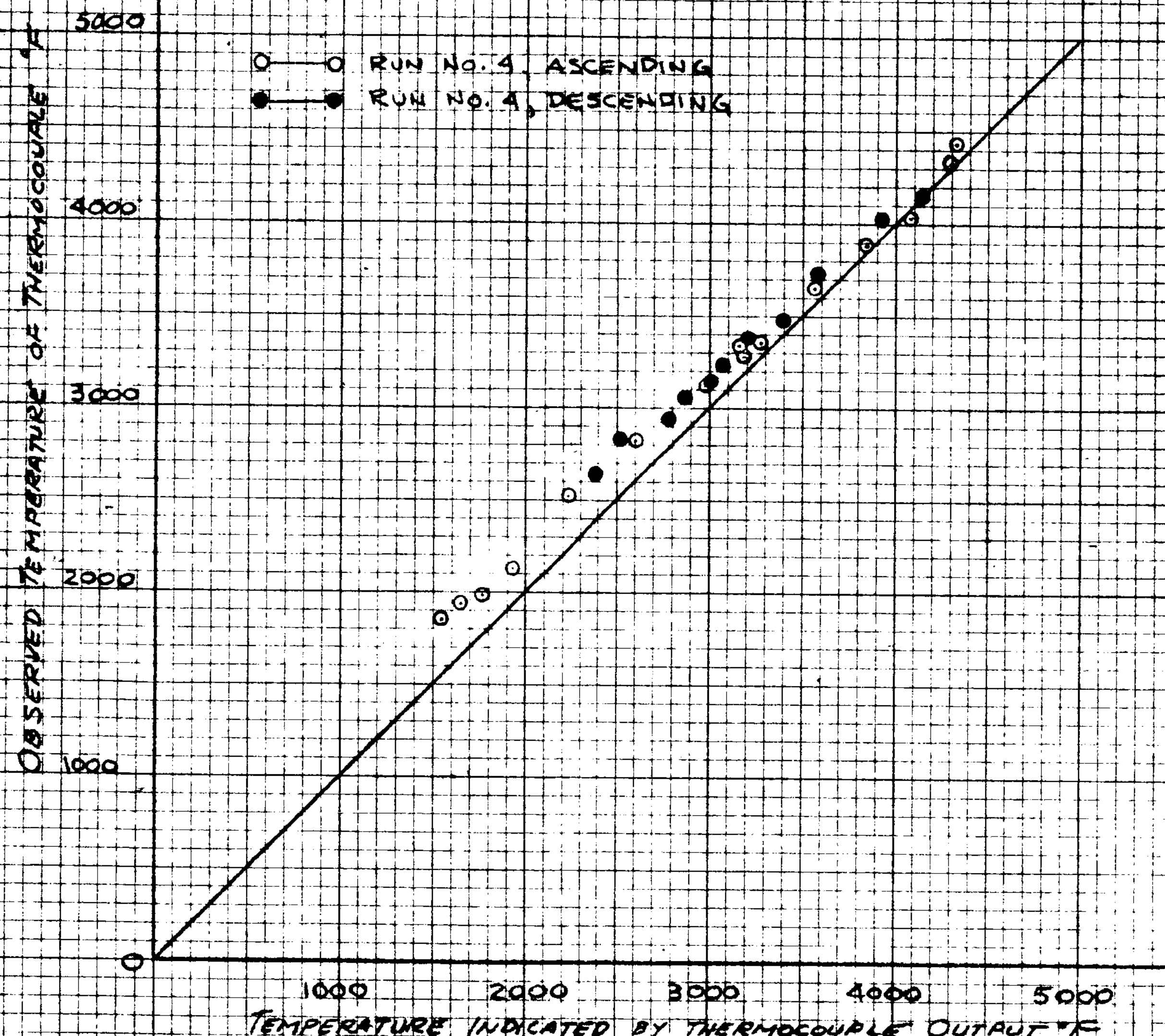

Fig. 12 Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve, Run No. 4

[redacted]

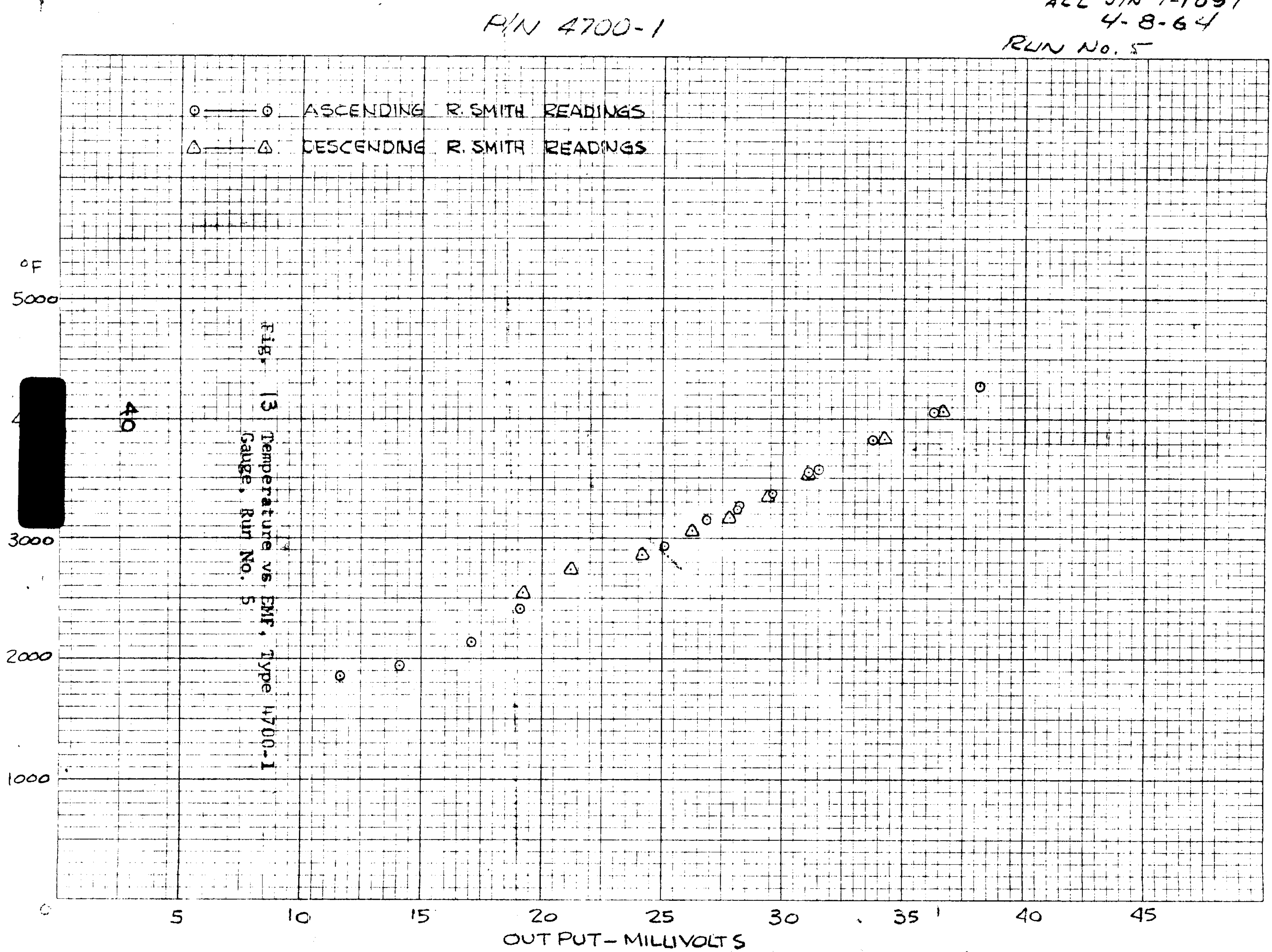

Fig. 13 Temperature vs EMF, Type 4700-1 Gauge, Run No. 5

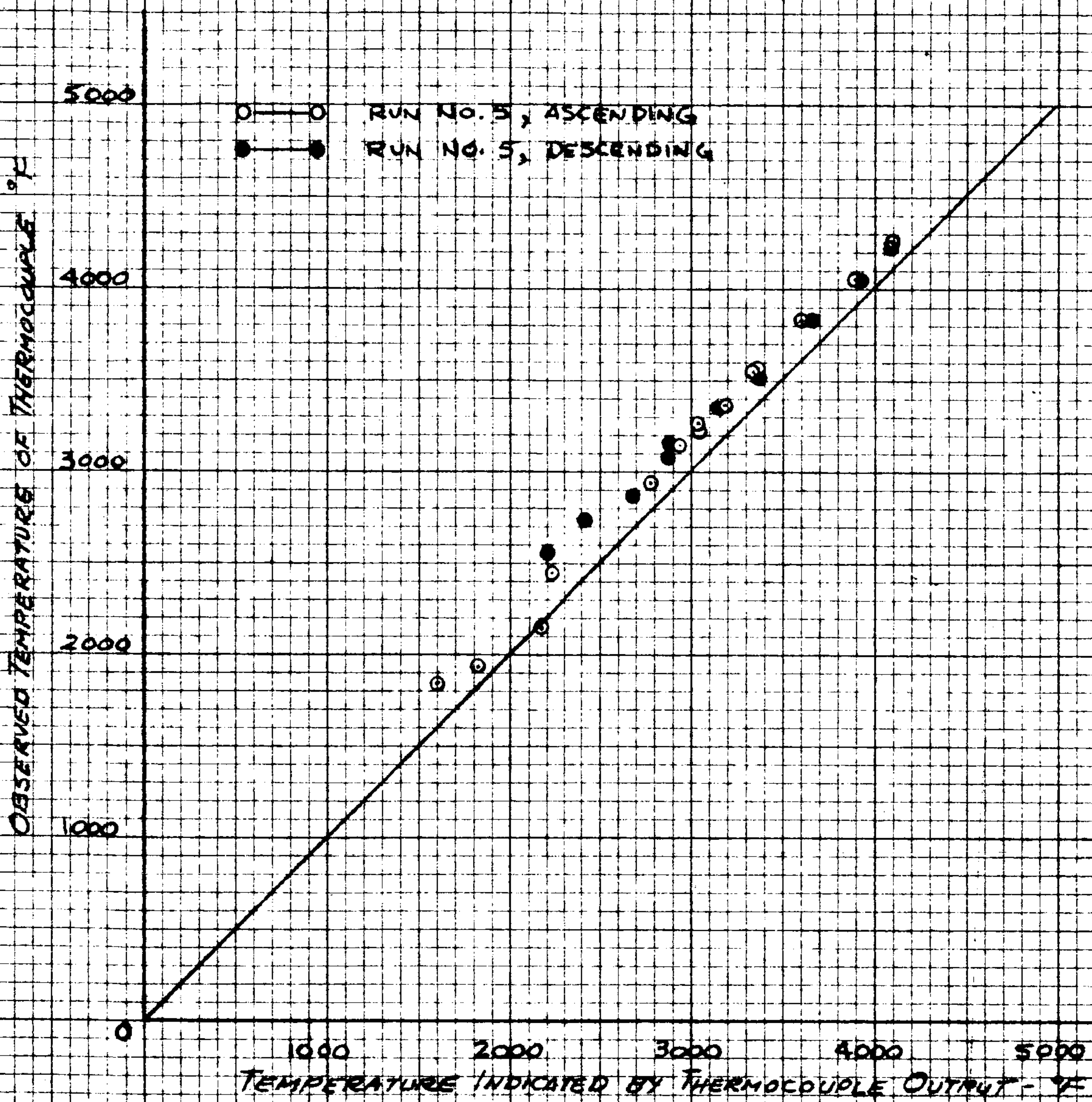

Fig. 14. Comparison of Observed Temperature
and Indicated Temperature versus
Hoskins Curve, Run No. 5

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RUN NO.5-1 P/N 4700-1

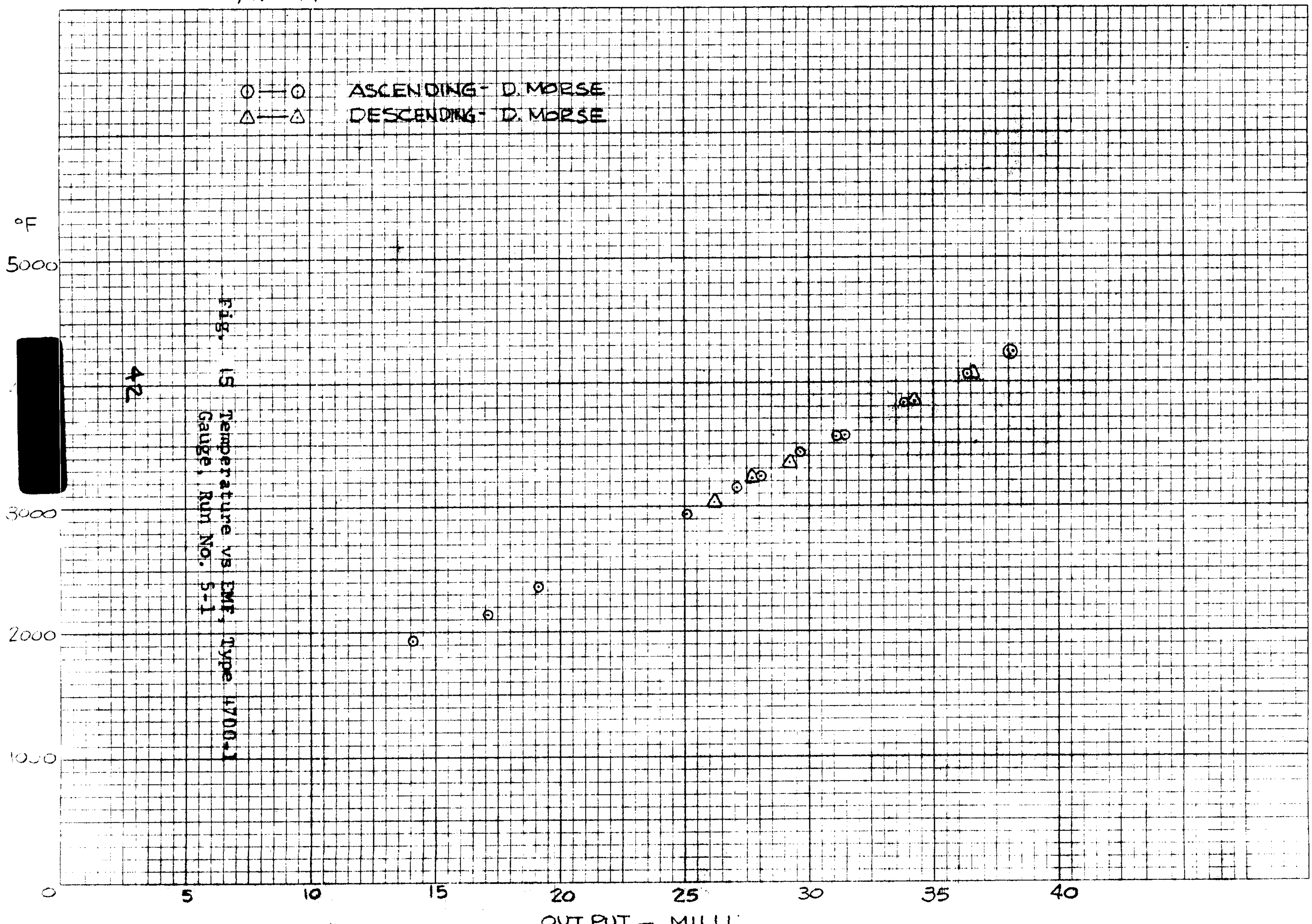


Fig. 15 Temperature vs EMF, Type 4700-1 Gauge, Run No. 5-1

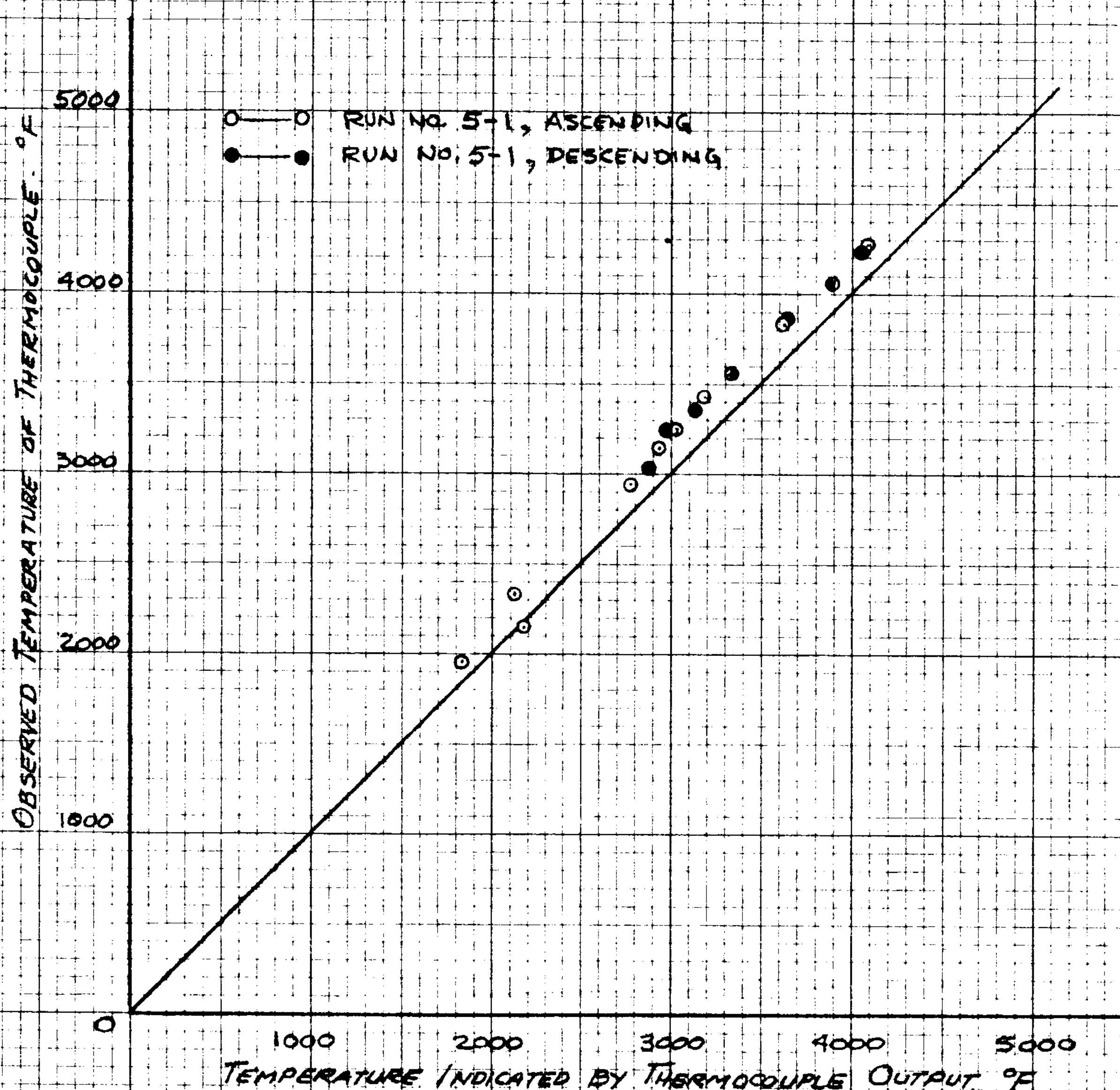


Fig. 16 Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve, Run No. 5-1

[redacted]

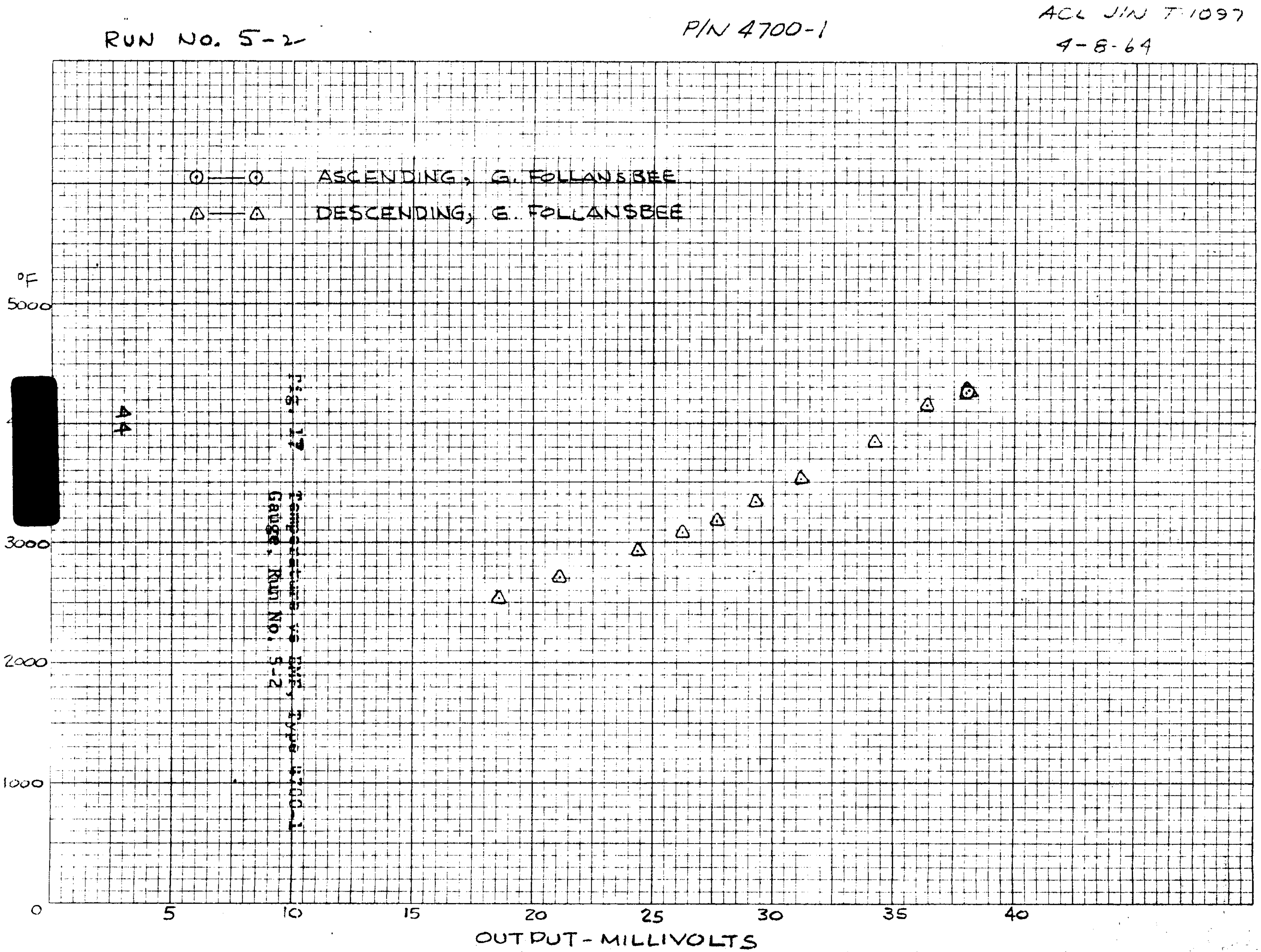


Fig. 17 Temperature vs EMF, Type 4700-1 Gauge, Run No. 5-2

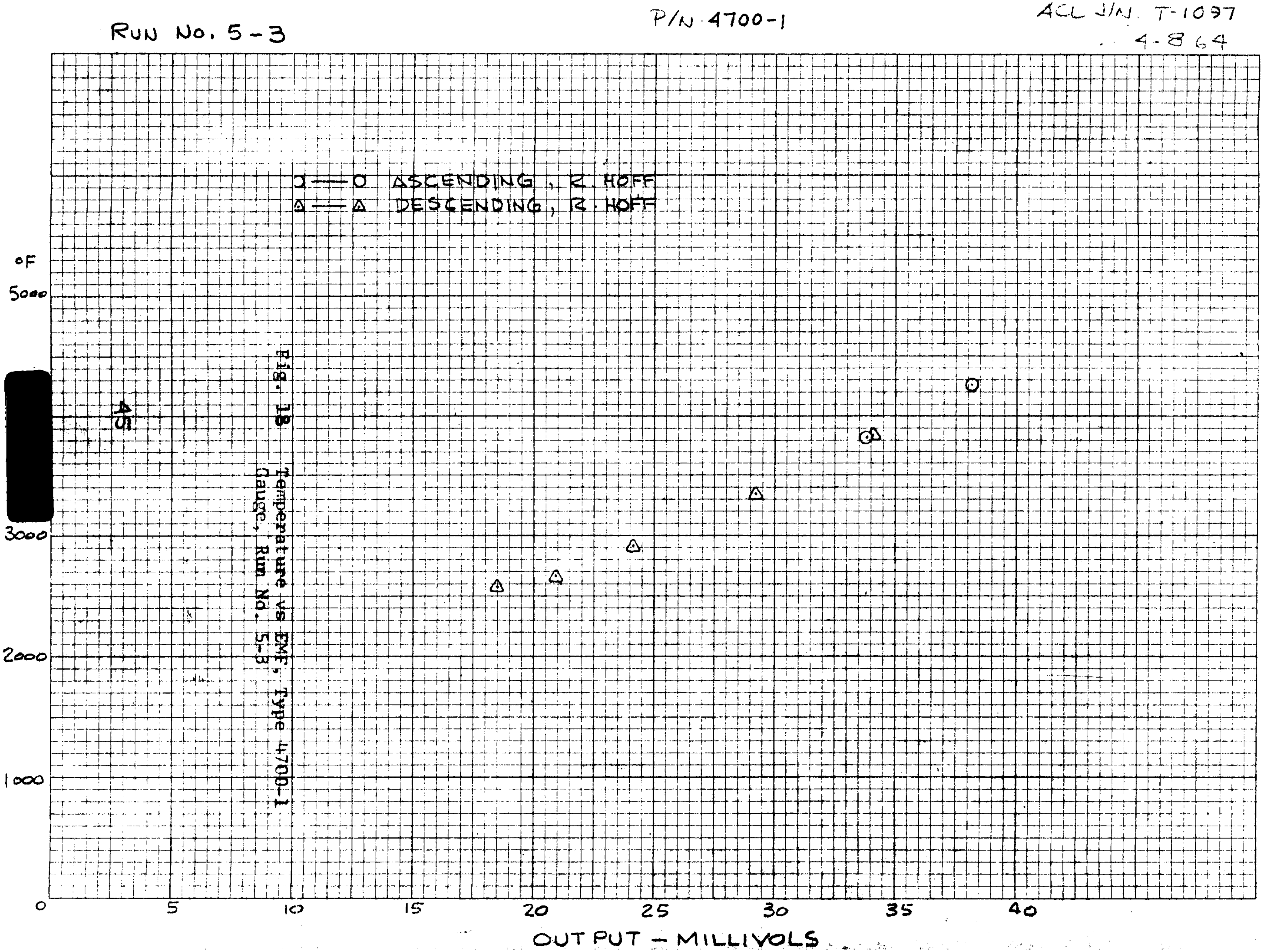


Fig. 18 Temperature vs EMF, Type 4700-1 Gauge, Run No. 5-3

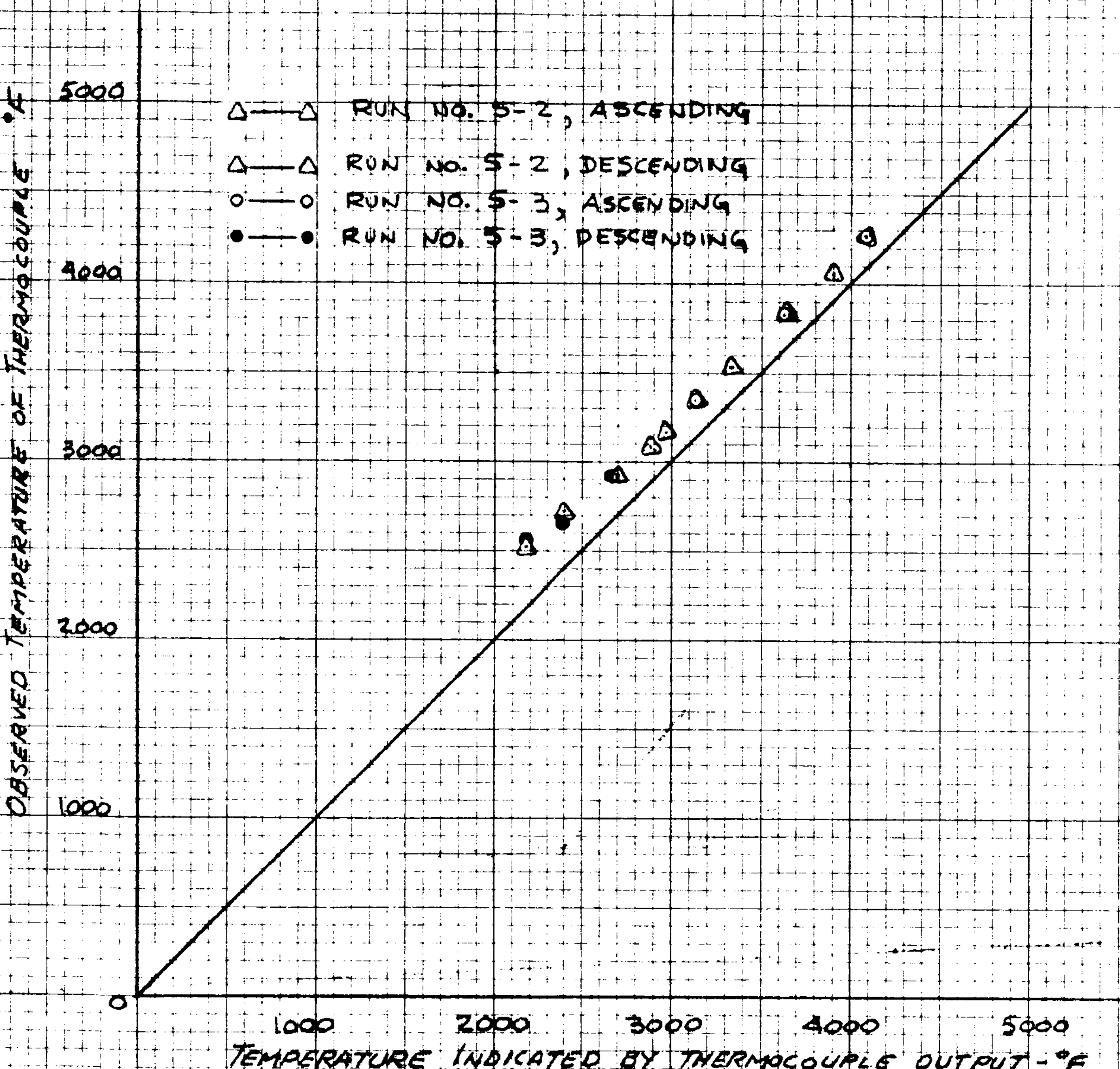


Fig. 19 Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve, Runs No. 5-2 and 5-3

[redacted]

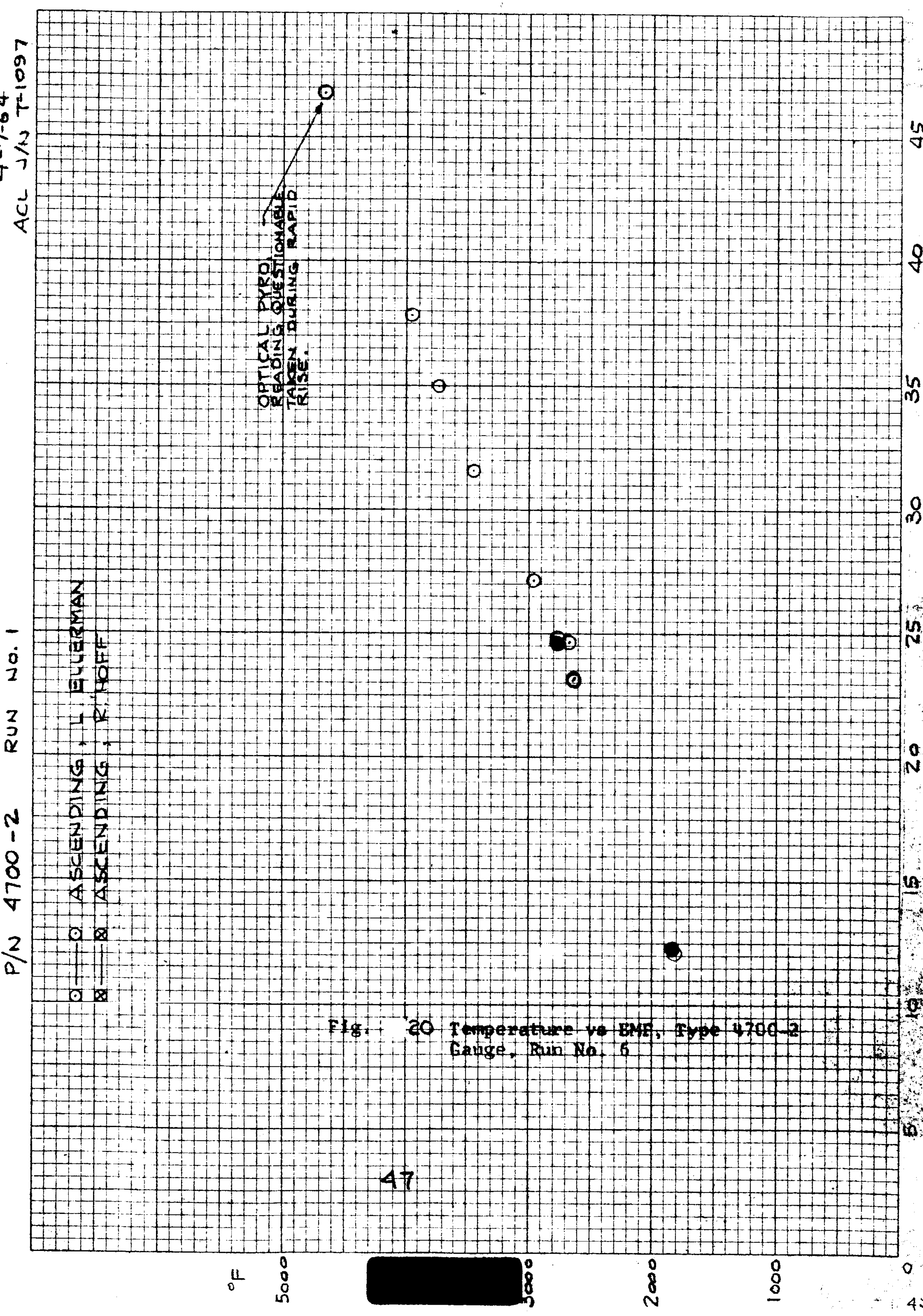


Fig. 20 Temperature vs EMF, Type 4700-2 Gauge, Run No. 5

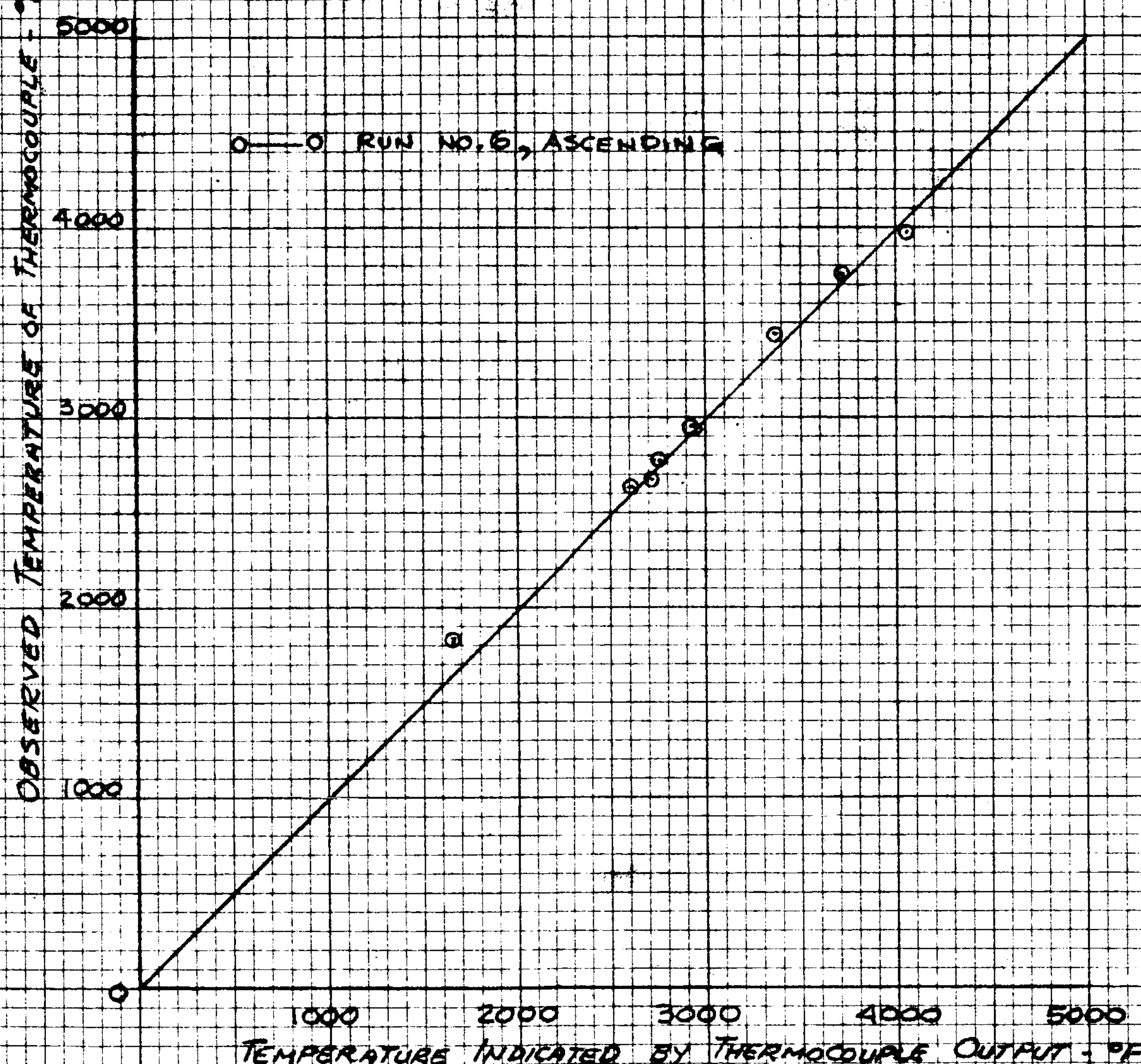


Fig. 21 Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve, Run No. 6

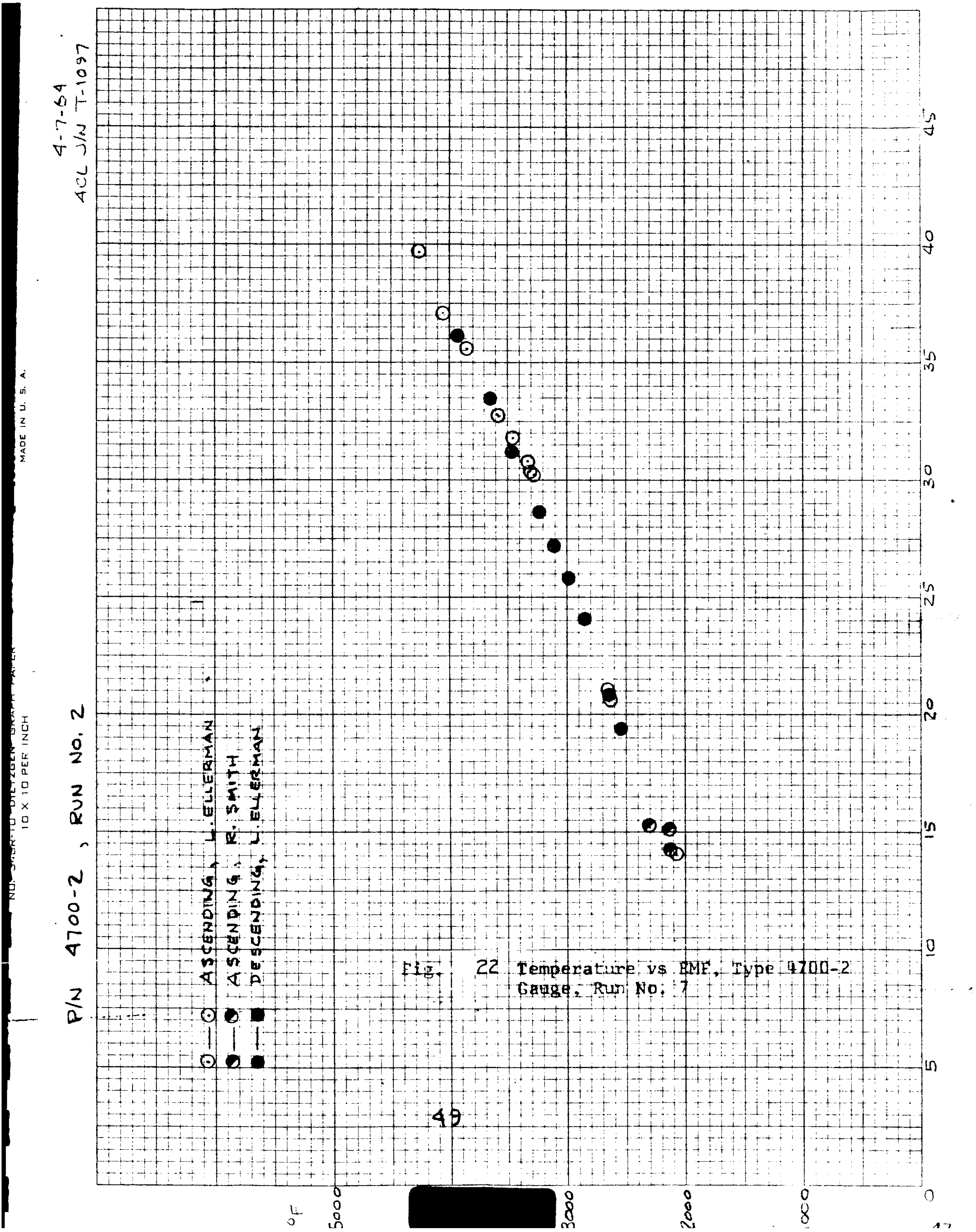


Fig. 22 Temperature vs EMF, Type 4700-2 Gauge, Run No. 7

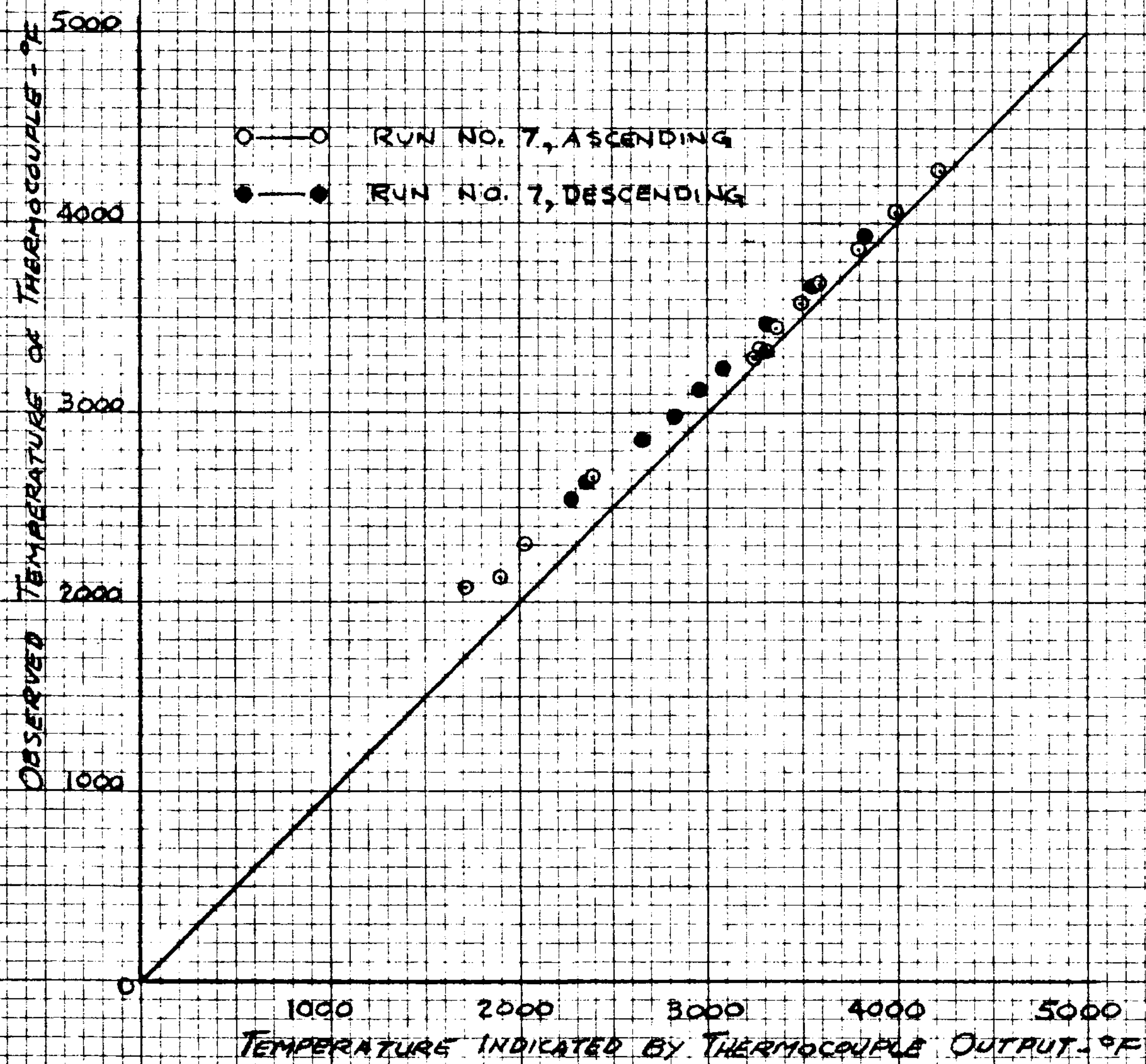


Fig. 23 Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve, Run No. 7

[redacted]

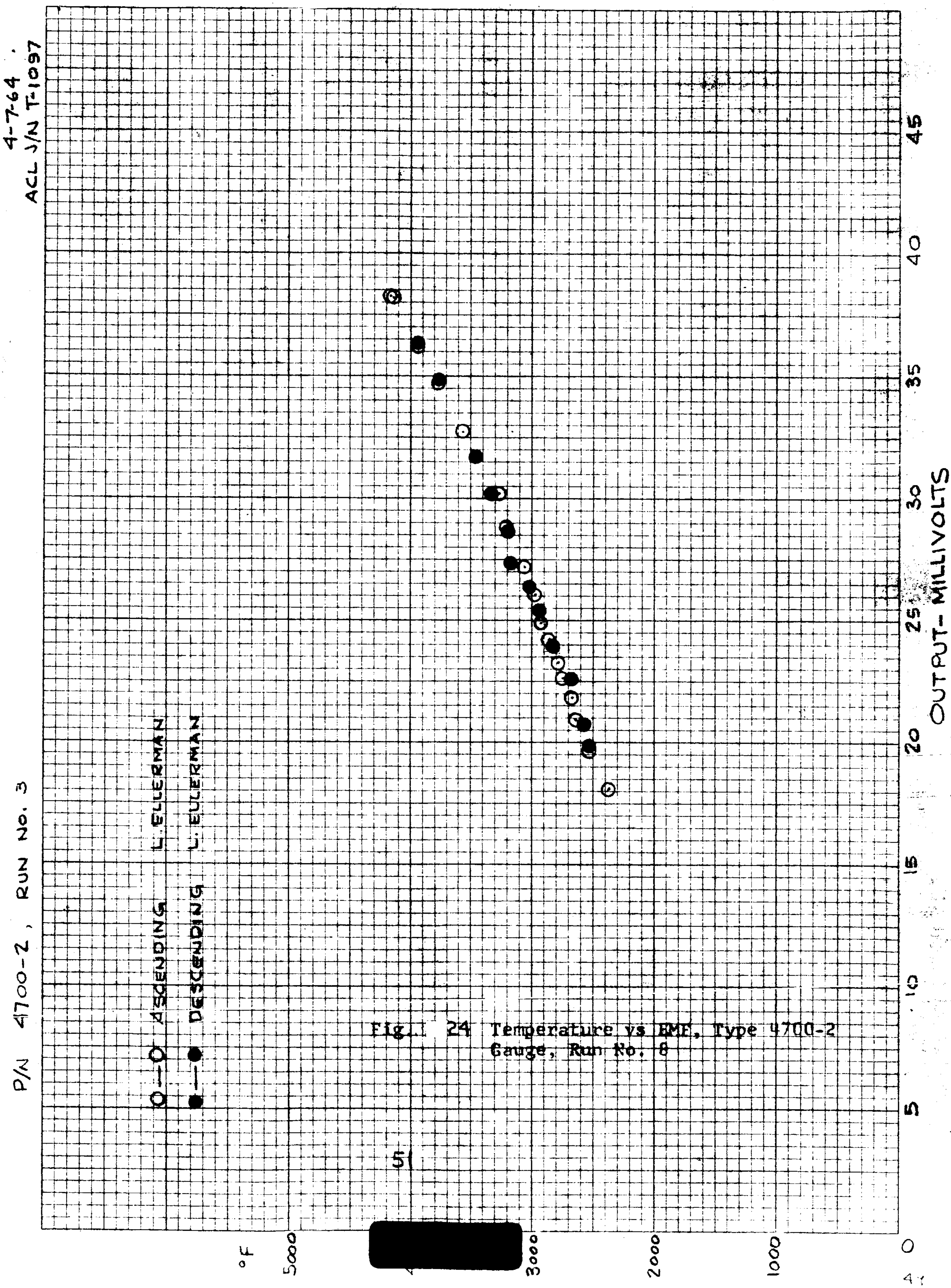


Fig. 24 Temperature vs EMF, Type 4700-2 Gauge, Run No. 8

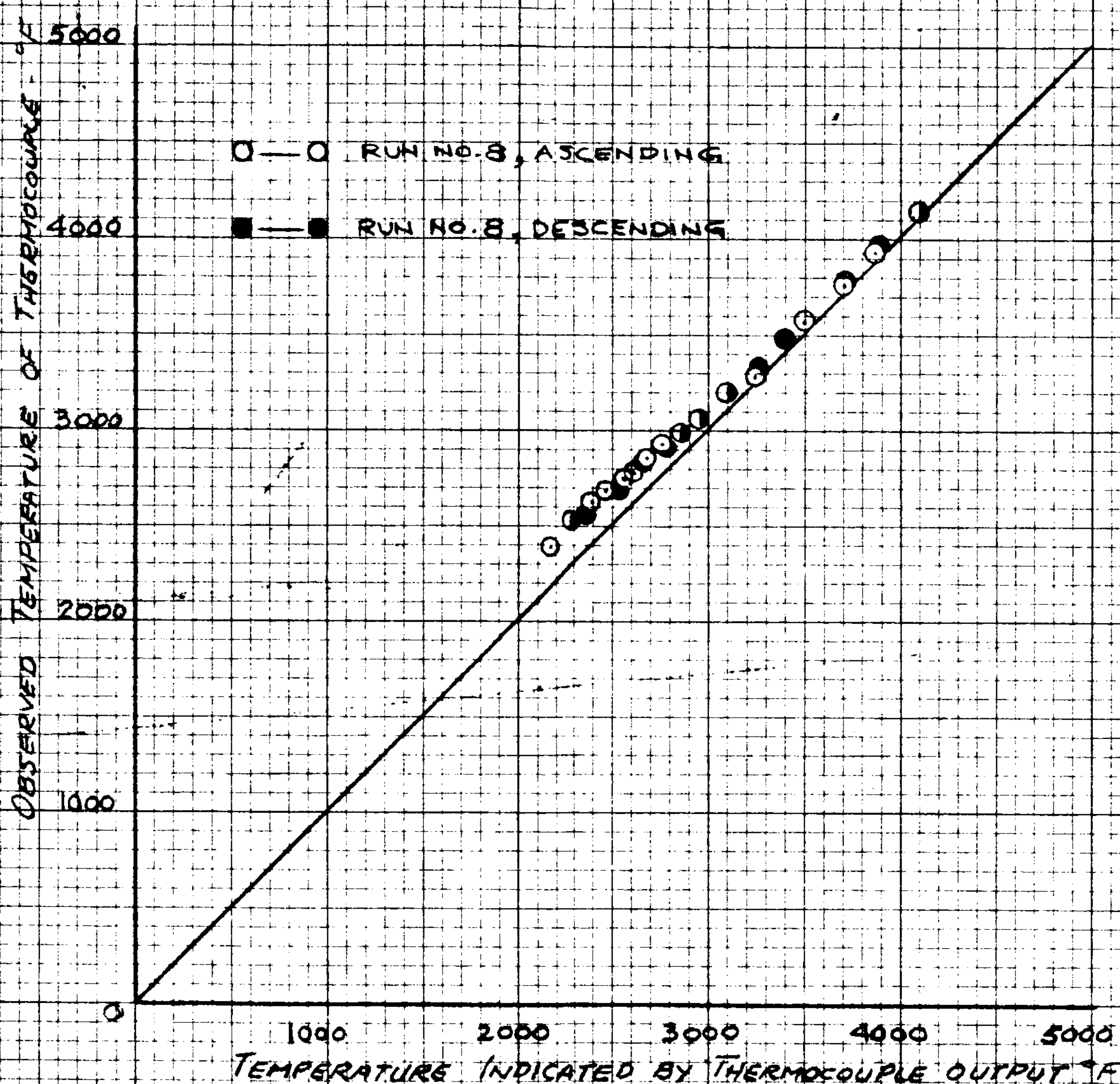


Fig. 25 Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve, Run No. 8

[redacted]

2.5 Calibrations, 4200°F (Cont'd.)

Test Method

The test methods employed in these calibrations were identical in every essential detail with those described previously.

Run No. 9-1

Run No. 9-1 was made from 2093°F to 4217°F and return to 2257°F. Total running time was 3 hours, 40 minutes with about 1 hour at 4217°F. After cool-down, the gauge was examined. No evidence of adverse effects were observed. The output curve from the ascending portion of the run matched the Hoskins curve, point for point, over nearly the entire range. Any deviations were within the experimental error limits. A positive drift was observed during the descending run.

Run No. 9-2

Run No. 9-2 was made from 2138°F to 4082°F. No descending run was made. Total running time was 2 hours, 20 minutes with about 1 hour above 4000°F. No adverse effects were observed as a result of this run. The positive drift observed during the descending portion of Run No. 9-1 had apparently stopped.

Run No. 9-3

Run No. 9-3 was made from 2241°F to 4136°F. No descending run was made.

2.4 General (Cont'd.)

TABLE NUMBER XI

Calibration Data - Type 4700 Gauge

Runs 9-1, 9-2, and 9-3

Temp. °C	Temp. °F	Output MV	Time
Run Number 9-1			
1145	2093	16.582	2:20
1430	2606	23.666	2:40
1651	3004	27.515	3:00
1990	3614	34.367	3:20
2325	4217	38.856	3:40
2325	4217	38.111	4:40
2019	3708	33.531	5:00
1731	3148	27.780	5:20
1517	2763	23.250	5:40
1236	2257	16.649	6:00
Run Number 9-2			
1170	2138	14.952	10:40
1401	2554	20.455	11:00
1626	2959	25.436	11:20
1938	3520	31.675	11:40
2236	4057	36.706	12:00
2250	4082	36.880	1:00
Run Number 9-3			
1227	2241	16.452	2:55
1515	2759	23.362	3:15
1643	2991	26.215	3:35
1951	3544	32.264	3:55
2236	4057	37.240	4:15
2280	4136	37.438	5:15

4-24-64

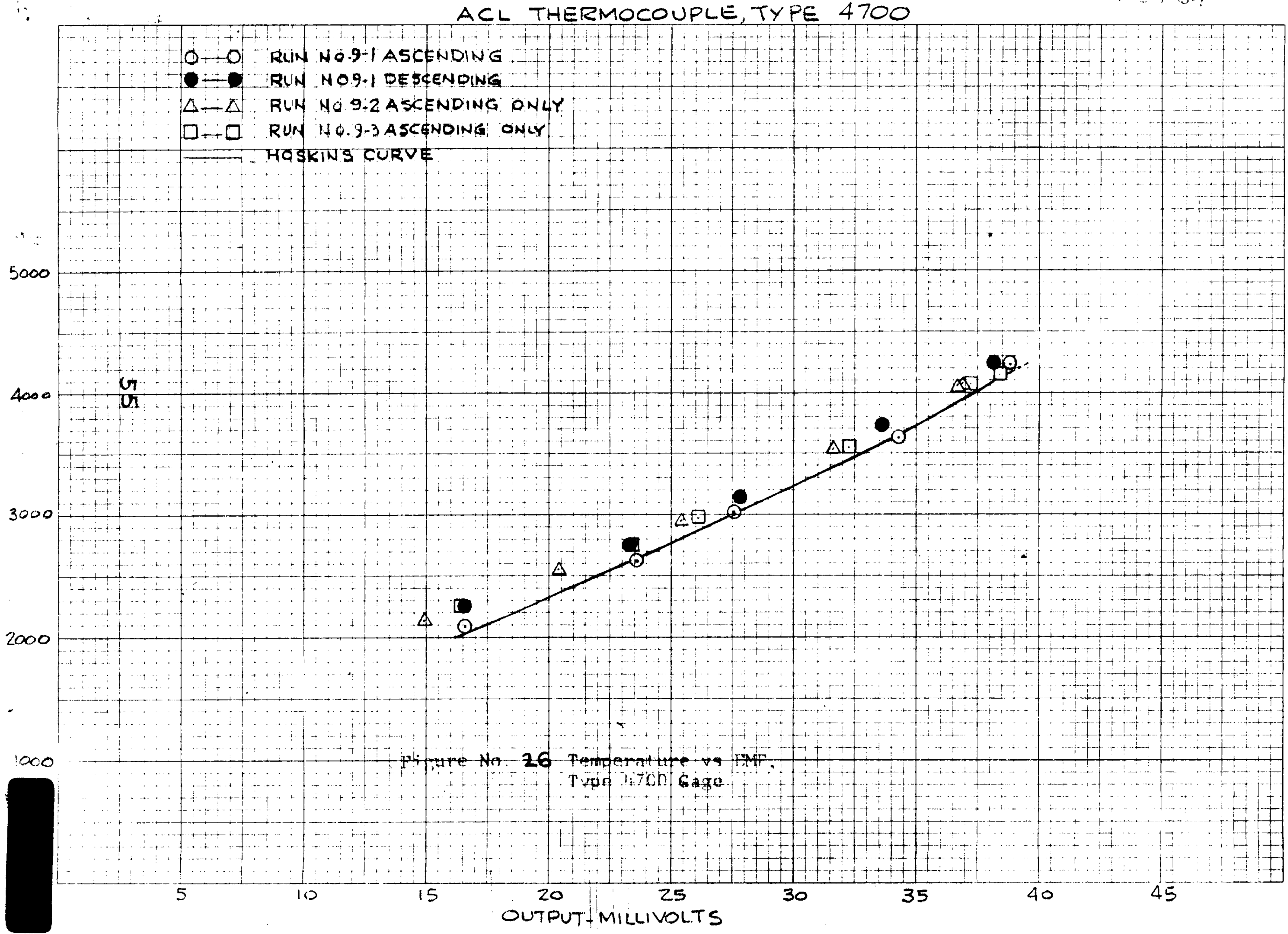


Figure No. 26 Temperature vs EMF,
Type 4700 Gage

[redacted]

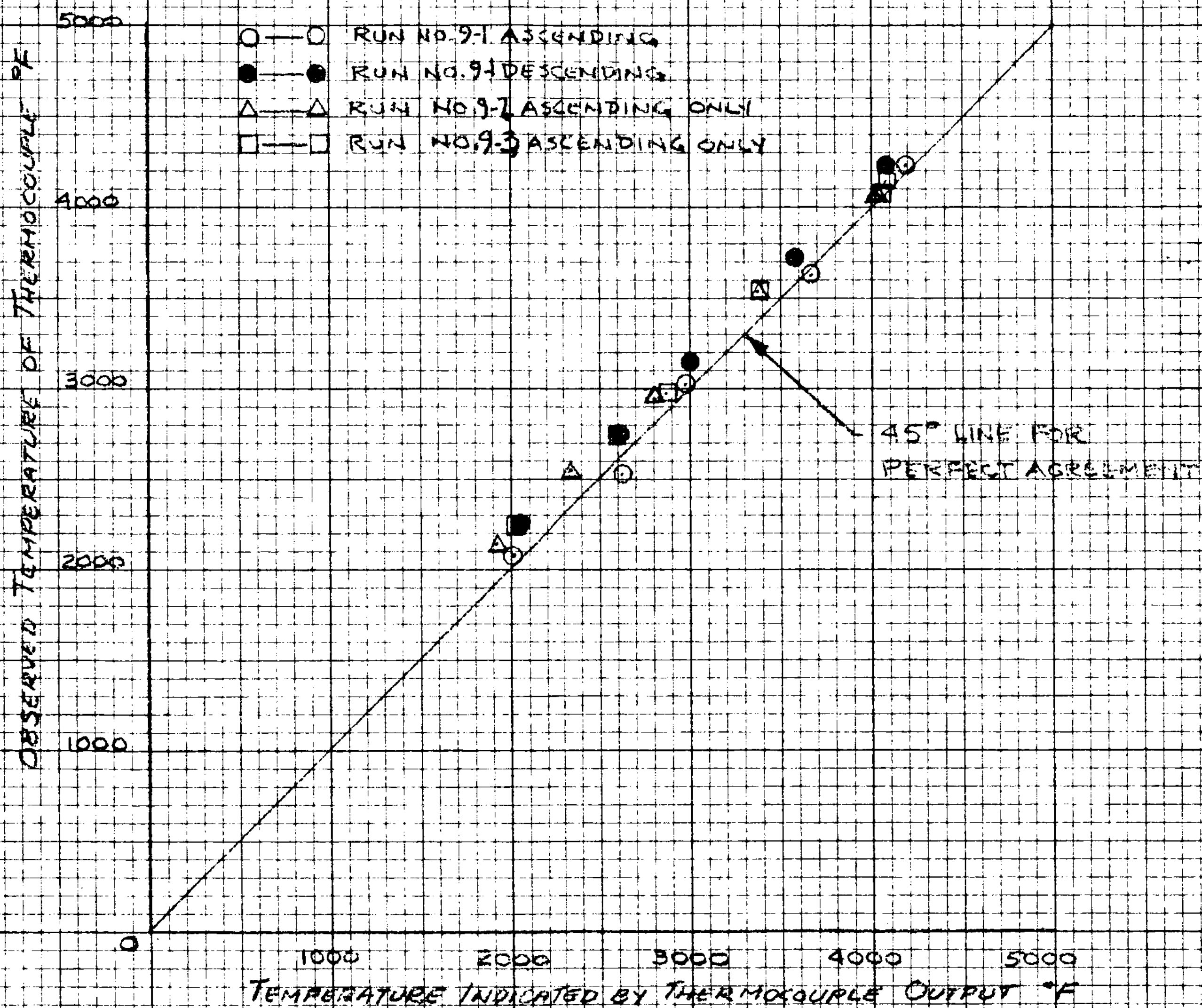


Figure No. 27 Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve

2.5 Calibrations, 4200°F (Cont'd.)

Run No. 9-3 (Cont'd.)

Total running time was 2 hours, 20 minutes with about 1 hour above 4000°F. Excellent agreement was seen between Run No. 9-2 and Run No. 9-3. The positive drift was still believed to be present, but had evidently diminished to the extent that its presence was difficult to detect, when pyrometer operator error and the limits of accuracy of the measuring apparatus are taken into account.

2.6 Calibrations, 5400°F

Two gauges were available for calibration and test. Serial No. 003 was assembled with a sheath from the first production run. The other, Serial No. 010, was assembled with a sheath from the second run. The principal difference between the two was a greater wall thickness in the second generation gauge sheaths. The principal interest in calibrating the two gauges under the same conditions was to verify whether it was possible to duplicate the output characteristics of the gauges between runs. Previous tests had demonstrated that the characteristics of gauges fabricated with sheaths from a single run were very close to identical. However, the question of repeatibility had been raised by both ACL and M-ASTR-I and a determination was deemed advisable.

Test Method

The high temperature calibration oven described in the last report was

2.6 Calibrations, 5400°F (Cont'd.)

Test Method (Cont'd.)

used in all the calibrations reported herein. The micro-optical pyrometer was set up with a focal length, such that the standard Tungsten lamp could be compared in brightness, with the thermocouple within the cavity and in proximity radially, with the thermocouple junction. Except as noted, the brightness comparisons were made only when the thermocouple tip had been scanned and there was no evidence of gradients. Since the Tungsten thermocouple sheath was entirely enclosed within the Tungsten cavity (with the exception of the .062 inch diameter sighting hole) it was assumed that no correction for emissivity need be made. The very good agreement with the Hoskins and Englehard curves for Tungsten-Tungsten 26% Rhenium Thermocouples at a large number of points seems to validify this assumption. Immersion depths were recorded for each run. Since the gauge sheaths are tapered, rather than cylindrical, the effective immersion depth is greater than would be the case with a cylindrical gauge, since the ratio of immersion depth to probe diameter would be calculated on the basis of the mean sheath diameter for the immersion used. This ratio is estimated at six, rather than four. Again, good agreement with the literature on the effect of immersion depths is evidenced in the calibration curbes. The literature recommends that immersion ratios of six to ten be used.

The thermocouple output was referenced to 32°F (ice bath) in all runs. The output was measured with a Leeds & Northrup Type 8662 Precision

2.6 Calibrations, 5400°F (Cont'd.)

Test Method (Cont'd.)

Potentiometer.

The output curves for three immersion depths, .875 inch, 1.00 inch and 1.25 inches are shown in Figure 28.

The data points taken at .875 inch immersion during Run No. 10-1, were taken with the thermocouple enclosed in a graphite cavity. These were halted, however, and tests continued with the Tungsten slug, in Runs 10-2, 10-3, and 10-4. The separation apparent between the curves for different immersion depths is in good agreement with data taken by SRI and ACL on earlier probes.

During the runs, the immersion depths were changed from 1.00 to 1.25 inches several times to verify the repeatibility of the two different gauges. Thus, the curves are representative of two different production runs.

A tabulation of the data taken during the runs is shown below in Table XII. A tabulation of the Hoskins Certification for the wire is shown in Table XIII. It should be noted that the Hoskins curve is for their lot No. 5 Tungsten wire vs. Lot No. 2603 Tungsten-26% Rhenium Alloy Wire. In the Type 4735 gauges, the Lot No. 2603 Tungsten Rhenium alloy

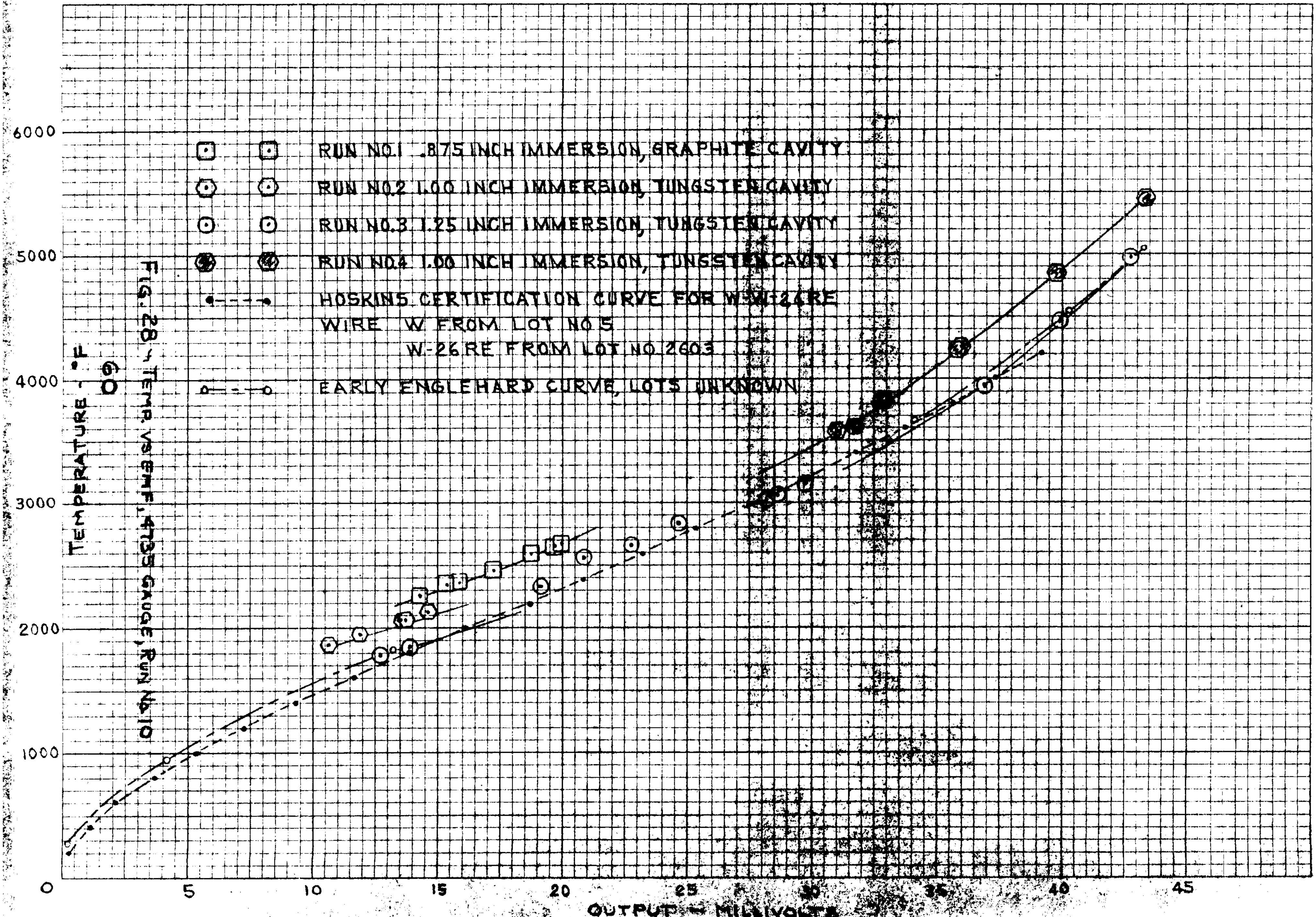


FIG. 28 - TEMP. VS EMF, 4735 GAUGE, RUN No 10

2.6 Calibrations, 5400°F (Cont'd.)

Test Method (Cont'd.)

TABLE XII

Calibration - Type 4735 Gauge

(Reference Junction - 32°F)

Run No.	Pyrometer °F	Output MV	Run No.	Pyrometer °F	Output MV
10-1	2264	14.47	10-4	3596	30.95
(.875 inch	2327	15.45	(1.00 inch	3614	31.60
Immersion)	2352	15.95	Immersion)	3821	32.80
	2458	17.25		4208	35.83
	2592	18.75		4289	35.90
	2622	19.67		4830	39.75
	2640	19.95		5430	43.43
10-2	1886	10.68			
(1.00 inch	1958	11.94			
Immersion)	2052	13.55			
	2067	13.66			
	2111	14.68			
10-3	1796	12.75			
(1.25 inches	1854	13.98			
Immersion)	2318	19.12			
	2566	20.85			
	2651	22.75			
	2813	24.67			
	3011	28.02			
	3074	28.64			
	3164	29.62			
	3937	36.90			
	4460	39.90			
	4975	42.70			

HOSKINS MANUFACTURING COMPANY

CABLE ADDRESS "THERMO"

4445 LAWTON AVENUE • DETROIT 8, MICHIGAN

January 8, 1964

Auto-Control Laboratories Inc.
5251 W. Imperial Highway
Los Angeles 45, Calif.

A F F I D A V I T

This is to certify that the 30 double feet of .020" diameter Tungsten versus Tungsten-26% Rhenium thermocouple wires shipped Jan. 8, 1964 against your Order 17768 has the following temperature/emf characteristics:

Tungsten Lot # 5
Tungsten-26% Lot # 2603

Temperature (°F)	EMF (Mv.)	Temperature (°F)	EMF (Mv.)
200	.287	2200	18.537
400	1.049	2400	20.875
600	2.159	2600	23.112
800	3.615	2800	25.441
1000	5.429	3000	27.580
1200	7.297	3200	29.713
1400	9.424	3400	31.893
1600	11.583	3600	33.805
1800	13.853	3800	35.668
2000	16.199	4000	37.485
		4200	39.185

Any liability under this affidavit is limited by the terms and conditions appearing on our General Order.

HOSKINS MANUFACTURING COMPANY

L. C. Lane

Sales Service Section

TABLE XIII

2.6 Calibrations, 5400°F (Cont'd.)

Test Method (Cont'd.)

was used, but the Tungsten leg is made of thermochemically formed Tungsten which is of much greater purity than normal Tungsten wire. Therefore, some difference in the output characteristic could be expected.

The ACL calibration at 1.25 inch immersion shows a similar "hump" in the curve between 2000°F and 3000°F as does the Hoskins curve. The reason for this "hump" is not known. Data points were not taken in this range during Runs No. 10-2 and 10-4 at 1.00 inch immersion. However, it is likely that the curve would have taken the same shape. The early Englehard Curve is plotted in Figure 10-1 and, although differences are seen, it is of interest that agreement is, in general, good.

2.7 Analysis of SRI Final Report, Type 4734 Gauges

A copy of the Southern Research Institute Final Report, covering calibrations and oxidation tests of ACL Type 4734 gauges, was obtained during the program. There is basic agreement on all phases of their observations and measurements except as follows:

a. In the SRI summary it was indicated that errors in measurement, or faulty techniques were responsible for differences in emf vs. temperature between the predicted curve and the SRI curves. The ACL curve was first calculated, then compared with the Englehard curve for W-W26Re. Good agreement was

2.7 Analysis of SRI Final Report, Type 4734 Gauges (Cont'd.)

obtained. Spot checks were then taken, in an isothermal zone, and when agreement between the calculated curve, the Englehard curve, and the spot checks was observed, the curve was drawn, based on the Englehard curve, with a reference junction temperature of 0°C. From the SRI curves, wherein differences in output, at the same temperature, for different immersion depths were seen, it can be inferred that conduction losses were largely responsible for the deviations from the ACL curve. The original ACL spot checks were run at an immersion depth of at least 25 diameters, whereas the SRI calibrations were taken at 5-1/4 to 6 diameters. Inspection of the SRI curves shows that the output for a discrete temperature moves toward the ACL curve as the immersion depth is increased. The deviation was plotted as a function of immersion depth. The resulting curve became asymptotic as the immersion depth was increased. This satisfies both the first law of thermodynamics and the literature regarding "end" or conduction losses.

b. Spurious emf's due to the lead wire are mentioned in the SRI report as possibly contributing to differences between the SRI calibrations and the ACL calibrations. This observation is valid. However, based on both the SRI calibrations, and later ACL calibrations, as well as the discussion in "a." above, the errors due to any spurious emf contributed by the compensated

2.7 Analysis of SRI Final Report, Type 4734 Gauges (Cont'd.)

lead wire must have been quite small. Of greater interest is the near certainty that a much greater spurious emf exists with different types of lead wire. This effect is believed to have been responsible for the appreciable differences between the SRI curves and the ACL curves. In these cases the essential difference between the Type 4734 gauges tested by SRI, which employed a copper (+) and copper-nickel (-) lead wire, and the Type 4735 gauges, which employed two different copper-nickel alloys, calibrated by ACL was in the compensated lead wire. Both employed the same type of junction, (W-W26Re) therefore, the emf for the same temperature, should have been the same. The ACL tests were made in two ways: with the sheath assembly alone (with lead wires attached) and with the sheath assembly mounted in the body. No observable differences were noted in these tests, thus further reinforcing the belief that the spurious emf, if present, was due to the copper-nickel alloy positive lead wire.

c. In the burner calibration tests, ACL agrees with the method used in the tests, except for contact of the sheath with the graphite cylinder. This method, although normally yielding good results thermally and with respect to emissivity, can contribute to an accelerated deterioration of the sheath due to the possible reaction between the Tungsten sheath and the

2.7 Analysis of SRI Final Report, Type 4734 Gauges (Cont'd.)

c. (Cont'd.)

graphite. This has been observed in previous ACL tests. The rise in observed temperature from the first to the last cycle can be explained if the graphite tube deteriorated enough, during a given cycle, to allow an increase in flame leakage. Great difficulty has been experienced by nearly all researchers in stabilizing temperatures during burner tests. Even small variations in burner pressure, flame path, or probe position can effect appreciable local variations of temperature. These are particularly evident at the higher temperatures, and are at times difficult to detect due to the time required to obtain a good brightness match in the optical pyrometer during a temperature change.

d. In the SRI evaluation of divergence between the burner exposure and cavity calibration, it is interesting to note the very high degree of repeatibility of the Type 4734 gauge, on successive cycles, with points taken on the first through the fourth cycles being nearly coincident, and certainly falling within the degree of accuracy possible with the optical pyrometer under the conditions of test.

e. One significant aspect of the SRI tests, which was not discussed in the report, but which is worth of mention is the absence of

2.7 Analysis of SRI Final Report, Type 4734 Gauges (Cont'd.)

e. (Cont'd.)

"flash" readings near the burner temperature (given as between 4760°F and 4870°F). If the flame ambient was in this range, it could be expected that flash readings could be seen, particularly when it is considered that the heat flux density was in the order of 600 $BTU/FT^2/SEC$. In previous tests such initial readings have been seen, quickly dropping off to some intermediate value in the gradient as the probe stabilized out at a temperature near the upper operating temperature of the Beryllia. It is possible, therefore, that the points in the burner flame, at which the readings were taken, were in a region of much lower temperature, and this fact was inadvertently omitted from the SRI report.

Despite disagreement with some details of the SRI report, and some discrepancies noted, ACL is in general agreement with the body of the report, and the conclusions.

Calibration vs. Immersion Depth, Type 4734 Gauges

In the evaluation program conducted by SRI, several of the ACL Type 4734 gauges were subjected to high temperature calibrations. It is assumed the published results are typical of these gauges. In examining the measured output curves, (See Figures 29 and 30) it is apparent they

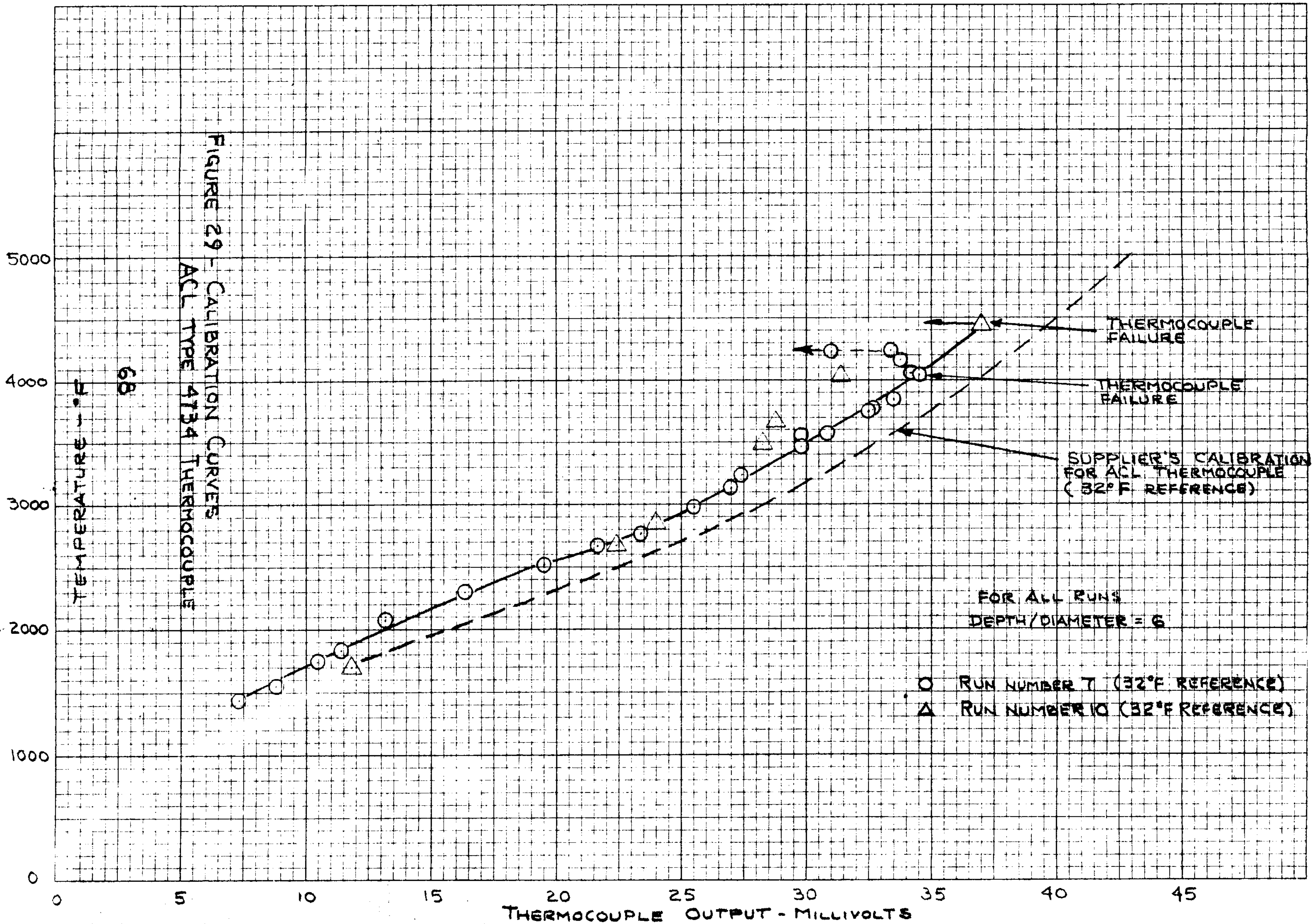


FIGURE 29 - CALIBRATION CURVES
ACL TYPE 4734 THERMOCOUPLE

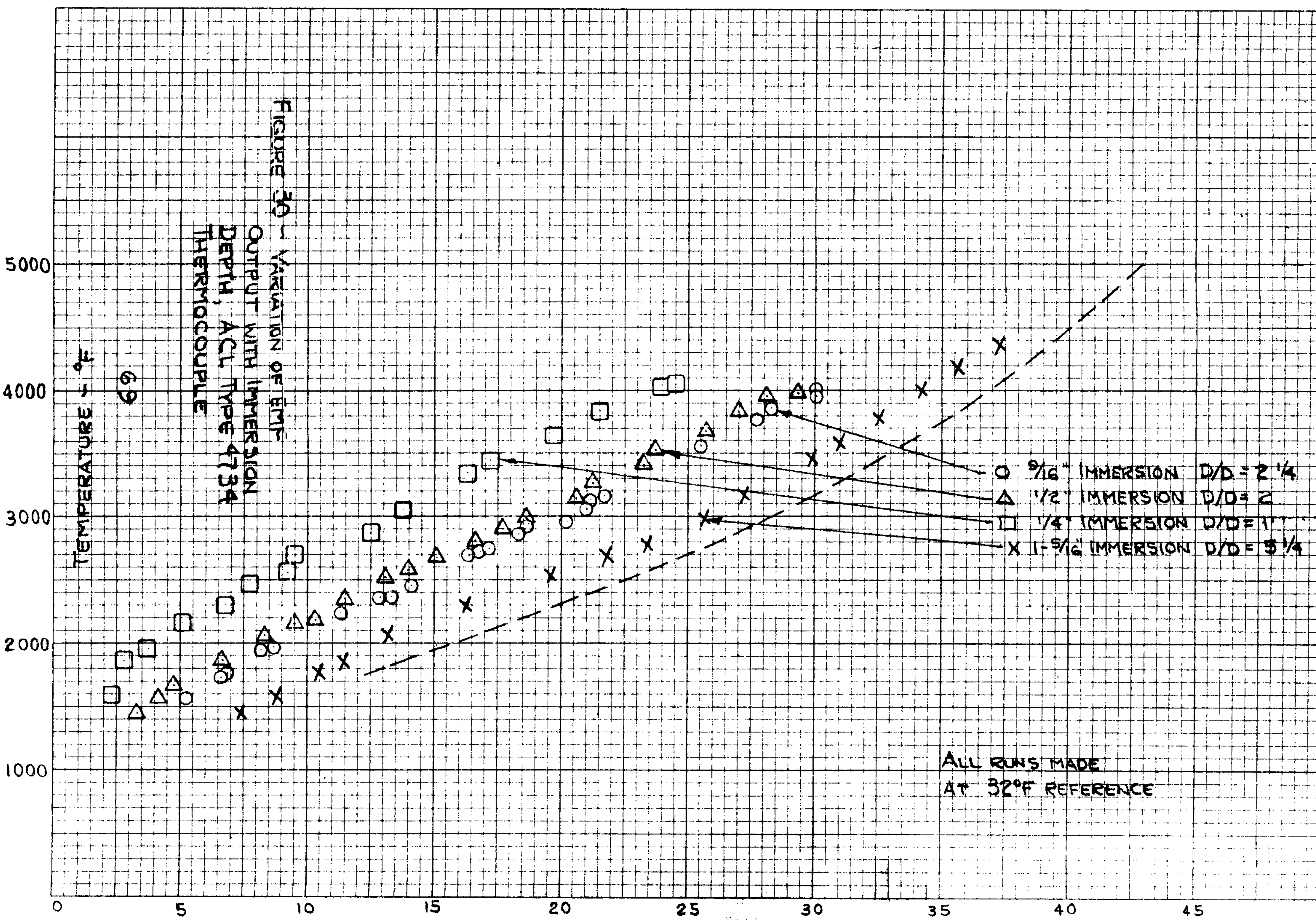


FIGURE 30 – VARIATION OF EMF OUTPUT WITH IMMERSION DEPTH, ACL TYPE 4734 THERMOCOUPLE

2.7 Analysis of SRI Final Report, Type 4734 Gauges (Cont'd.)

Calibration vs. Immersion Depth, Type 4734 Gauges (Cont'd.)

are in fair agreement with calibration curves supplied by ACL, within the temperature range covered.

The curves of output vs. immersion depth are of particular interest, in that the influence of immersion depth on deviation from previously calculated and measured value is apparent, for 2000°F, 3000°F, and 4000°F. These were plotted (See Figure 31) as mean deviation from the predicted curve vs. immersion depth. Although four immersion depths were examined, only three are of practical use because of the difficulty in reading finite emf values for the 9/16" and 1/2" immersions. A curve can be drawn with only the three points for 1/4", 1/2" and 1-5/16" immersions. However, extrapolation to an exact immersion depth where there is minimum deviation did not seem feasible, because of the uncertainity of predicting the rate at which the deviation slope changes.

It can be shown, however, that in accordance with first law principles, the curve becomes asymptotic with increase of immersion depth, probably approaching 1 mv. mean deviation at about 2" immersion. The parallelism of the curves indicates that an output curve very close to the predicted curve can be obtained with an optimum immersion depth.

[redacted]

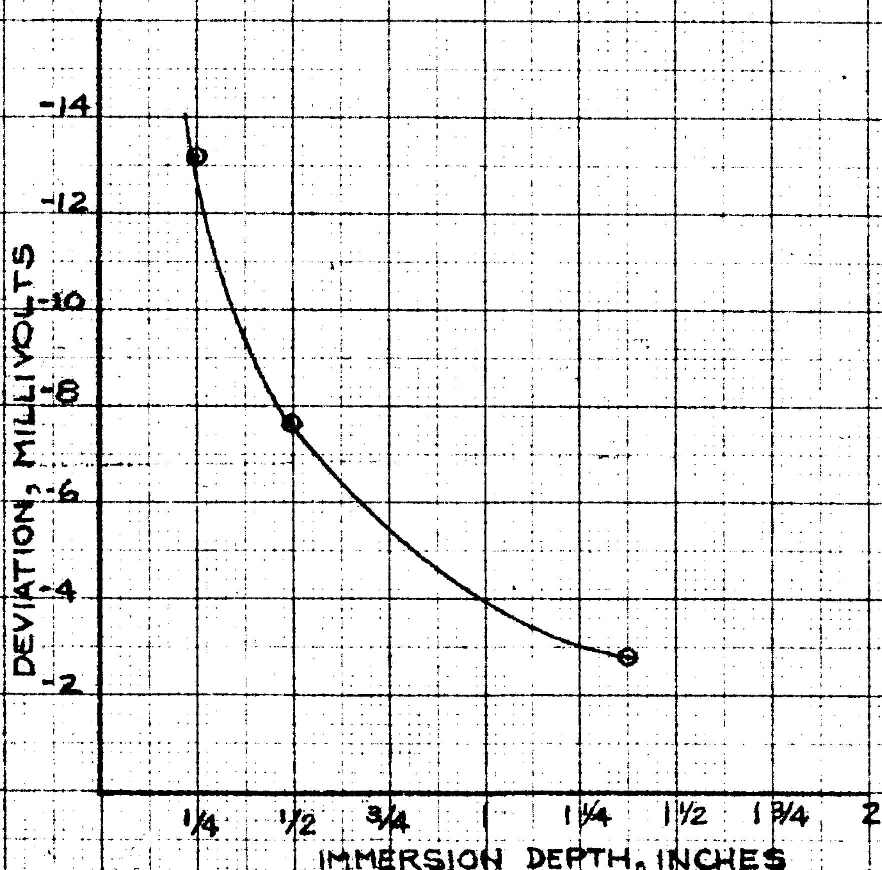


FIGURE 31

MEAN DEVIATION OF THERMOCOUPLE EMF OUTPUT WITH IMMERSION DEPTH. ACL TYPE 4734 THERMOCOUPLE.

[redacted]

2.7 Analysis of SRI Final Report, Type 4734 Gauges (Cont'd.)

Evaluation of Calibration Curves

The calibration curves for the ACL Type 4734 gauges obtained by Southern Research Institute were discussed previously, and a curve of mean deviation vs. immersion depth was presented. A more comprehensive tabulation of the data was made, including measurements taken at 1-1/2 inches immersion, and is presented in Table XIV, below.

TABLE XIV

Calibration vs. Immersion Depth

Output MV	Immersion, Inches				Full Scale Deviation**	Percent of Full Scale
	1/4	1/2	1-5/16	1-1/2		
5.0	*2175	1700	1250	----	---	---
7.5	2500	1975	1450	1450	-0-	-0-
10.0	2700	2200	1700	1700	-0-	-0-
12.5	2900	2450	1950	1950	-0-	-0-
15.0	3200	2700	2200	2150	-50	-2.3
17.5	3500	2900	2350	2375	+25	+1.0
20.0	3650	3100	2500	2550	+50	+2.0
22.5	2900	3400	2750	2725	-25	-1.0
25.0	4100	3600	2900	2925	+25	-1.0
27.5	----	2900	3200	3200	-0-	-0-
30.0	----	4000	3450	3500	+50	+1.0
32.5	----	----	3800	3750	-50	-1.0
35.0	----	----	4100	4100	-0-	-0-
36.5	----	----	4350	4300	-50	-1.0
37.0	----	----	----	4400	----	----

*All Temperatures in °F
**Deviation defined as °F difference between 1-5/16" and 1-1/2" immersion depths.

2.7 Analysis of SRI Final Report, Type 4734 Gauges (Cont'd.)

Evaluation of Calibration Curves (Cont'd.)

The behavior of the gauges versus immersion depth indicates that thermal equilibrium was being approached at the 1-5/16" to 1-1/2" immersion. The mean deviation curve mentioned above did not take into account the data taken at the 1-1/2" immersion. The tabulation above accounts for the full scale deviation from the calibration temperature, which is assumed to be well known. Percent of full scale deviation is also shown over the range. In preparing the above tabulation, unexplained anomolies in the SRI curves were not used. Since the values were picked off the curves, rather than selected from test data sheets for the emf values shown, there is room for argument as to the accuracy of the percentages shown. Despite any such inaccuracies in finite values, there seems to be ample evidence that equilibrium was achieved, and that errors in measurement approach a reasonable value.

The literature reports that 5-10 diameters immersion are required to minimize the so-called conduction losses in an isothermal cavity. Since the gauges were of 1/4" diameter, the ratio of diameter to immersion depth is 6, at 1-1/2" immersion.

Of interest also are the points at which thermocouple failure is indicated. In the failures at or near 4500°F, the cause is believed to be due to melting of the Beryllium oxide insulator. At the 4000°F failure, it is believed that a small piece of steel, inadvertently left in the tip

2.7 <u>Analysis of SRI Final Report, Type 4734 Gauges</u> (Cont'd.)

<u>Evaluation of Calibration Curves</u> (Cont'd.)

of the probe, melted, and the Tungsten in the region of the junction went into solution with the molten metal. This analysis would fit the mode of failure; i.e. deterioration of output, followed by sudden opening.

2.8 <u>SRI Calibrations, High Temperature, Type 4735 Gauges</u>

It had been considered by ACL that calibration of the Type 4735 gauges had been established in the range 2000°F to over 5000°F as a result of the accumulation of a large number of measurements within the range. These measurements had been made without a single failure, except for those deliberately induced during oxidation tests.

During a telephone conference with M-ASTR-I personnel, it was related that two of the second generation Type 4735 gauges, being tested by Southern Research Institute, failed to perform beyond 3500°F. It is understood by ACL that the following occured: 1) The output of the gauges appeared to deteriorate after 3500°F. 2) that some material, presently unknown, appeared to melt and drip from the sheath, 3) SRI, at this point, terminated the tests.

The SRI report was in wide variance with results observed during ACL tests by, not only ACL technicians and observers, but by representatives of other companies as well. In fact, other ACL gauges, fabricated in a similar manner, and tested by the user, repeated the ACL curves with

2.8 SRI Calibrations, High Temperature, Type 4735 Gauges (Cont'd.)

very close agreement, from 1500°F to about 4500°F. These independent tests were conducted by Aerojet on probes intended for use in the control loop for the NERVA propulsion system. Their calibrations were conducted under such stringent procedures, and under such extremely close observation and critical supervision by SNPO that the results were accepted without question by ACL. ACL, therefore, visited M-ASTRI to ascertain the facts in conference with M-ASTRI and SRI personnel. At that time, ACL verbally requested an extension of the contract to permit an adequate review of the SRI tests, to permit further ACL tests, to allow for any redesign indicated, and to allow time for review of further N.A.S.A. or SRI tests whose desirability was agreed upon in the conference. This verbal request for additional time was followed by a confirming letter on 15 June 1964. Permission was subsequently granted.

It was agreed, in the conference, that ACL would attempt to provide answers to three questions:

1. Why was there a variation between outputs of the 4735 gauges S/N 007 and 009, during the two calibrations performed by SRI?
2. Why did a "bulb" of material form on the tip of both gauges?
3 Why was there an irreversible change in the output of both gauges above 3500°F?

The answers to all three questions are closely related to one significant fact: The sheaths of both gauges were treated with a vapor deposited coating of elemental silicon.

2.8 SRI Calibrations, High Temperature, Type 4735 Gauges (Cont'd.)

The silicon was applied to provide an oxidation resistant coating to the gauges. When the gauges were run in a non-oxidizing atmosphere (Argon) as in the SRI tests, the Silicon could not combine with Oxygen (since there was none present) to form the two oxides: Silicon Oxide (SiO), and Silicon Dioxide (SiO_2), and would not react with Argon, which is inert. As a consequence, the Silicon melted, and ran down the sheath to the tip, where, like a drop of water on the end of a rod, it remained. As the gauges cooled after the run, the Silicon solidified, and the bulb remained on the tip. The zone of most intense heating in the SRI furnace can be seen in an examination of the 007 and 009 gauges. There is a very sharply defined transition about one inch from the tip of the gauge, above which the coating was undisturbed.

Below this line, and extending to the bulb, is a zone wherein the Tungsten sheath presents the typical high luster, shiny appearance of Tungsten run in Argon at elevated temperatures. This effect has been observed many times by ACL in other tests. In fact, this process is useful to remove surface contamination and oxides from Tungsten. Any Silicon that dropped from the gauge tip to the bottom of the cavity was due to the mass of the material exceeding the surface tension of the molten mass of Silicon.

The discussion above explains the formation of the bulb, and is fairly straightforward. The variation in the output between the two gauges and

2.8 SRI Calibrations, High Temperature, Type 4735 Gauges (Cont'd.)

the irreversible changes are twofold, and are explained as follows.

It is ACL's position that a true comparison of output vs. temperature can not be made unless all factors influencing the output are identical. This is particularly true where mounting method and immersion depth are concerned. Neglecting for the moment any differences in measuring technique between those used by ACL and SRI in calibrations, the mounting method should simulate the conditions of use as closely as possible.

Since ACL permitted the body of the gauge to heat as the furnace was raised in temperature, it is felt that errors due to stem conduction are smaller because of the smaller temperature differential between the sensing junction and the body. The exact extent of any such differences cannot be estimated because no measurements of body temperature were taken by either ACL or SRI. It was agreed by SRI in the conference that future tests would take mountings into account.

The irreversible changes in the outputs of both gauges may be explained as follows. The Silicon, upon exposure to temperatures above its melting point, and as explained previously, ran down the sheath of the gauge. Without having Oxygen with which to combine, the Silicon reacted with the Tungsten to form Tungsten Silicides. This was as planned, to take advantage of the oxidation resistance of the silicide to extend the life of the gauge. However, without Oxygen to use up excess Silicon, the

2.8 SRI Calibrations, High Temperature, Type 4735 Gauges (Cont'd.)

formation of the silicide continued until chemical equilibrium was reached. Thus, the junction between the Tungsten and the Tungsten-Rhenium alloy in the gauge was "poisoned" by the presence of either diffused Silicon, the silicide, or both.

The SRI spectrographic analysis of the tip materials of both gauges, although qualititive, seems to bear out the argument above. The sample from gauge S/N 007 shows Silicon and Tungsten present. The first sample from gauge S/N 009, which was taken from "drippings" on the cavity floor shows Silicon, but not Tungsten. The second sample from 009, taken from the ball at the gauge tip shows both Silicon and Tungsten. Rhenium did not appear in any of the samples. If the junction was poisoned, a new thermoelectric system was in effect, and a different output curve would be seen. Unfortunately, the effect cannot be reversed, thus little information of value can be obtained from more tests of these gauges.

ACL's position in the above explanations is greatly strengthened by the existence of test data covering more than two hundred hours of cycling, not only type 4735 gauges, but also other types of Tungsten-Rhenium alloy gauges over the same temperature range, and in the presence of Oxygen as well as inert gases. Output curves from these gauges are within a few percent of each other, and it can be concluded that the irreversible changes must be due to some effect common to the 007 and 009 gauges, rather than a fault common to the system. In every case,

2.8 SRI Calibrations, High Temperature, Type 4735 Gauges (Cont'd.)

ACL tests on type 4735 gauges have been run only after the sheaths were oxidized. It is recommended therefore, that gauges to be run in future be oxidized to take care of excess elemental Silicon. The SiO_2 melts at 3100°F and, being volatile, is easily driven off. The Silicon oxide (SiO) does not melt until 4406°F, and tends to form a tenacious film.

2.9 Response Tests

Response of the gauges developed under this contract was a matter of deep interest, because of the necessity of providing response compatible with the overall loop control and indication characteristics of associated systems.

Definition of Response

Response is defined as the time required to achieve 63.2% of a step increase in temperature, under stipulated conditions of the media, and the mass velocity in the medium of the step change.

Discussion

In the background of this project, as well as in many previous projects, response, and its measurement, has at times become a highly controversial subject. The controversy has usually resulted from a failure, by both parties, to adequately define response itself as meaning a change to

2.9 Response Tests (Cont'd.)

Discussion (Cont'd.)

63.2%, 95%, or 100% of the step function, and further, a failure to define, or misunderstanding of the characteristics of the medium in which the sensor is at a stable condition and the medium in which the response measurement is to be made. These may be the same, or they may be different. They may be at the same velocity, or their velocities may be different. They may have the same mass or not, as the case may be. It is of prime importance that the various parameters described above be well defined because, for a given probe configuration; i.e. open, enclosed, grounded, or insulated, response under various combinations of the above may vary widely.

In most cases, the designer is required to attempt to provide the most rapid response possible. He must, therefore, carefully consider the means available to him for designing a probe that has the ability to perform its intended function of operating within some specified temperature range, to withstand the effects of the medium in which operation is intended, to survive the dynamic loads imposed by the medium if moving, and the installation, to respond to the step temperature change in the required time, to continue to operate for a useful period of time, and finally, to be capable of the maximum number of repeated cycles of operation.

2.9 Response Tests (Cont'd.)

Discussion (Cont'd.)

The velocity of the medium and its mass may be combined in the term, mass velocity, as for example, lbs/ft^2/sec. The other characteristics affecting response; such as coefficients of thermal conductivity of the materials, their thermal resistances at interfaces, thermal capacities, film coefficients, etc., must be considered individually and collectively as regards their effect on response. Trade-offs are normally required to meet the other operating conditions. Because of the extreme complexity of the inter-relationships of all the design considerations, as well as the many assumptions that must be made, calculations of response must be regarded skeptically until proved by test. On the other hand, a large body of data exists regarding response of a large number of different types of thermocouple probes in various media. These data are very useful in estimating the response of new types of probes, as well as in verifying results obtained from tests.

In previous tests at NASA, two ACL Type 4734 gauges were tested in a scale rocket motor. Response of these probes, under the conditions of the test, was estimated by test personnel at 250 milliseconds. The mass velocity must have been quite high, although no value has been, to ACL knowledge, assigned.

M-ASTR-I personnel had previously assigned a maximum of 500 ms, with

2.9 Response Tests (Cont'd.)

Discussion (Cont'd.)

250 ms as an objective. Although the tests mentioned above seemed to indicate that the ACL probes could meet the response requirement, ACL could not verify the 250 ms estimate because a suitable means of reproducing the M-ASTR-I tests was not available. Therefore, a search was made of specification requirements used in the missile industry to establish response.

Two specifications* were selected, both of which used the same technique for simulating conditions of use, but at much lower temperature and stress levels. These means consisted of plunging the gauge from room ambient air into water with a velocity of 3 feet per second in one case, and into agitated boiling water in the other. ACL selected the method employing boiling water at 3 ft/sec. Results of the tests are described below.

*Aerojet-General Corporation, Component Specification, Thermocouples, General Specification for, AGC-42136B, Amend. 1
Rocketdyne, Specification ETI-3-004, and Amendments.
Rocketdyne Spec. Control Dwg. SK-9420

2.9 Response Tests (Cont'd.)

Response of ACL Type 4735 Gauge, Procedure

The test probe was fitted with two sets of W-W26Re compensated lead wire, as used in the lead wire tests. (Harco Laboratories and Minneapolis Honeywell). The lead wires were connected to a dpdt switch, and copper extension wires were, in turn, connected to the indicating or recording instrument. The test setup is shown schematically in Figure 32.

The flask was filled to approximately 500 ml with tap water and subjected to free boiling. The output of the thermocouple was stabilized at room temperature, 80°F ±5°F. The probe was then rapidly inserted into the boiling water to a depth of approximately 1", and permitted to remain for 15 seconds. It was then removed and permitted to stabilize at room temperature in preparation for the next run. Four successive runs were made, and these were correlated against an open junction, 24 AWG thermocouple.

Test Results

Copies of typical visicorder recordings are shown in Figure 33. Response of the Type 4735 gauge measured 45 milliseconds. The response of the bare wire thermocouple was 80 milliseconds under the same conditions. No reference junction was used because interest was confined to a step temperature rise, rather than to a discrete level. As was expected, there was no difference between results from the different lead wires.

TEST SETUP

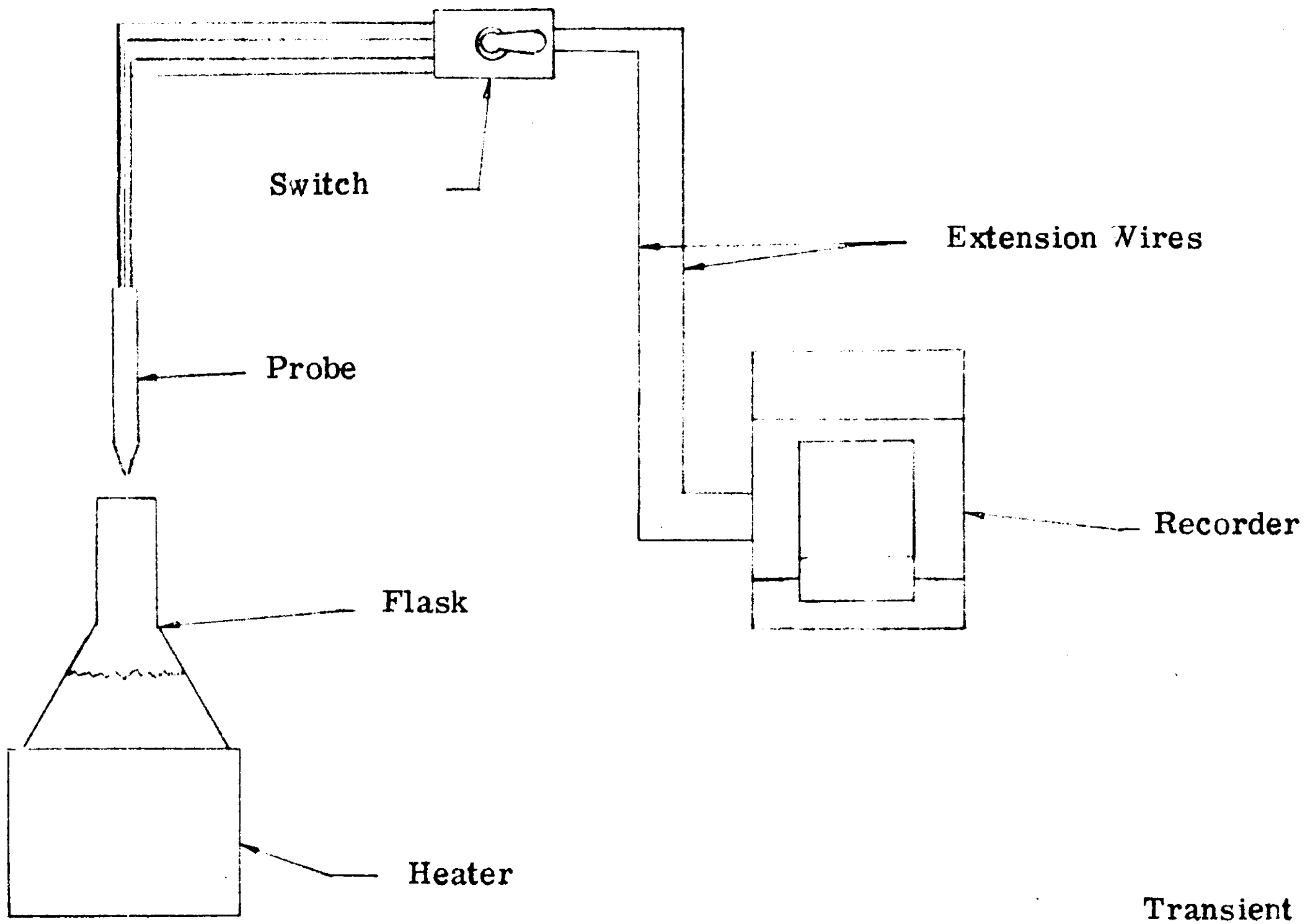
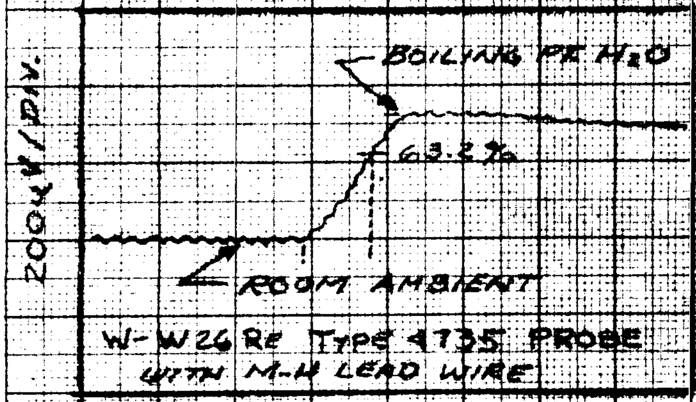
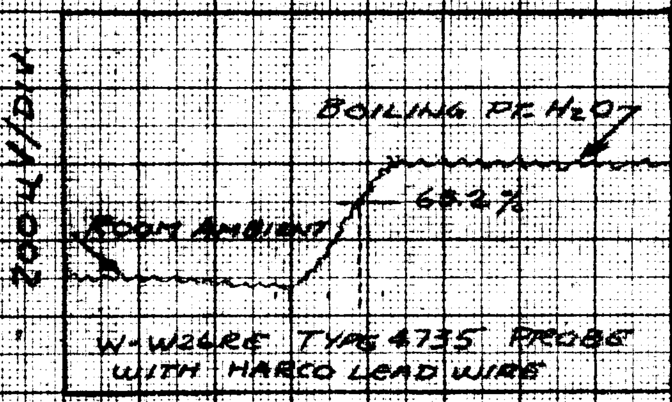


FIGURE 32 - RESPONSE TEST SETUP

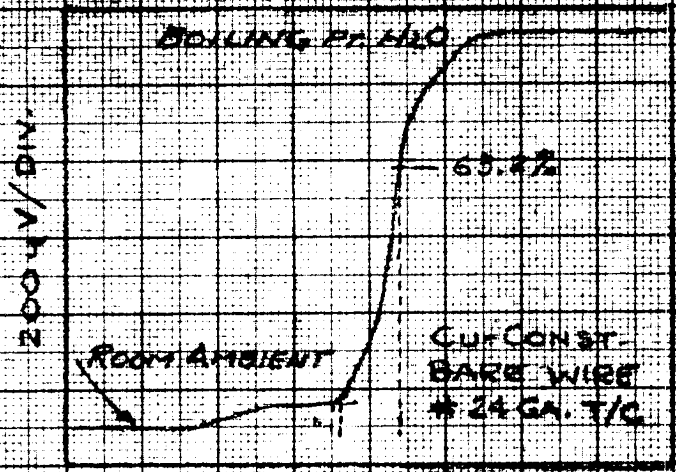
[redacted]



2 DIV = 100 MS
RESPONSE ≅ 45 MS



2 DIV = 100 MS
RESPONSE ≅ 45 MS



1 DIV. = 100 MS
RESPONSE = 80 MS

FIGURE 33

TRANSIENT RESPONSE TEST
T-1087
30 December 1963

[redacted]

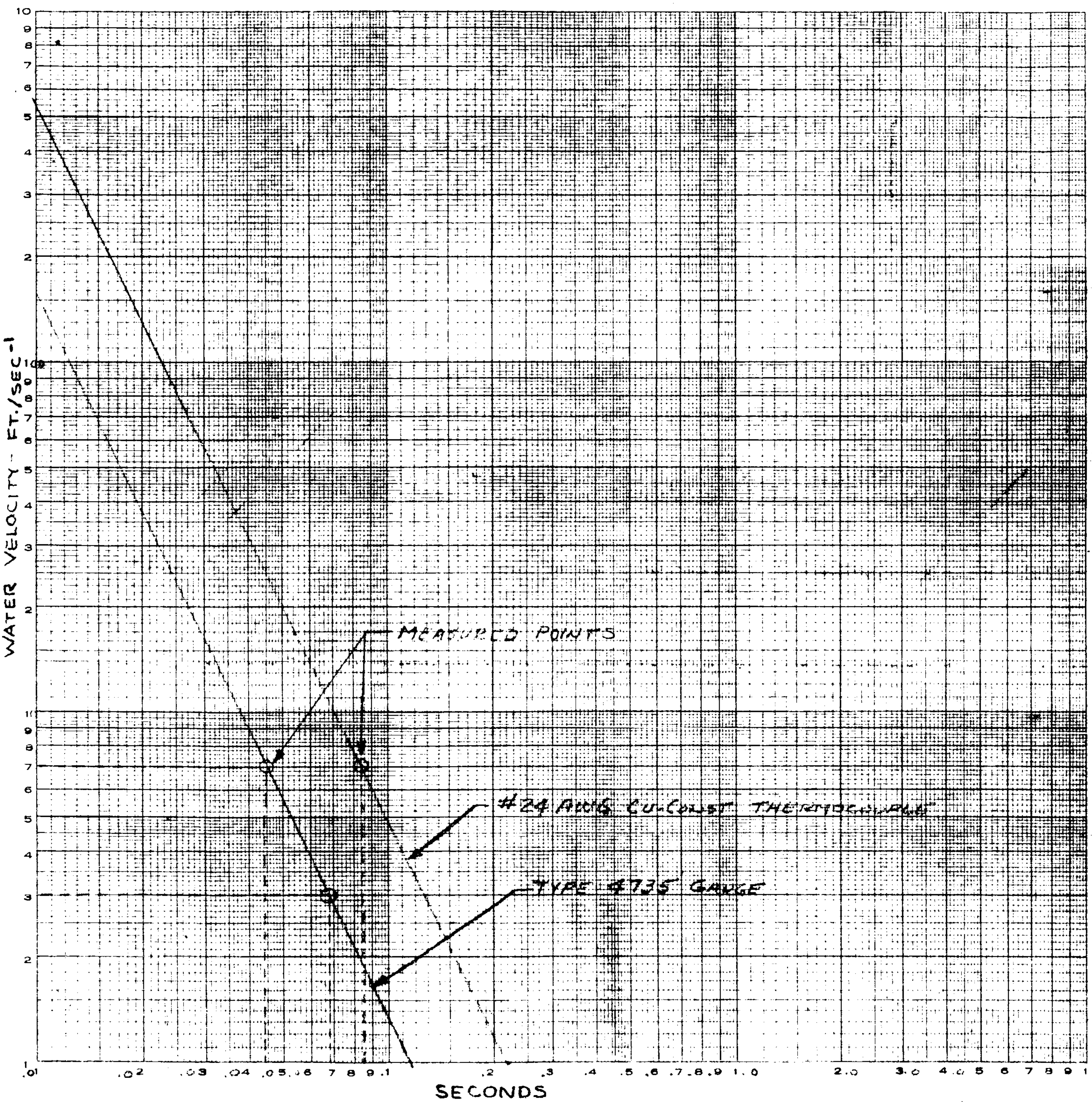


FIGURE 34 - RESPONSE COMPARISONS

[redacted]

2.9 Response Tests (Cont'd.)

Test Results (Cont'd.)

A comparison was made between response, as obtained during the ACL tests, and standard, published response curves common in the art. See Figure 34.

The bare wire thermocouple is common to both curves. The response of the bare wire thermocouple, plotted as measured by ACL, shows that the velocity of the agitated boiling water was about 7 ft/sec in the ACL test. If the line representing the slope of the response, is plotted on a two cycle logarithmic graph, the response of the Type 4735 gauge can be estimated at about 70 milliseconds for a water velocity of 3 ft/sec.

Conclusions

It is concluded, as a result of these response tests, that the ACL Type 4735 gauges, in prototype form, as delivered to M-ASTR-I, are capable of meeting the 250 millisecond response objective. An interesting possibility presents itself in that a considerable latitude in tip design is possible, should an increase in mass be required because of strength or oxidation resistance.

2.10 Leadwire Calibration Tests

Test Method

One objective was to determine the effect of different types of compensated lead wire on the output of the Type 4735 gauges. It had been noted, in previous tests, that an appreciable difference in output level, at discrete temperatures, existed between ACL Type 4734 gauges and Type 4735 gauges. This difference appeared as an increase in output for the same temperature. To determine whether this difference did exist, a Type 4735 gauge was fitted with two sets of compensated lead wires. One set was Harco #20 AWG, P/N SS-136-32. The Harco wire is comprised of two different copper-nickel alloys. The M-H wire is comprised of copper-nickel alloy on the negative leg, with copper on the positive leg. The Type 4734 gauges employed the M-H wire, and Harco lead wire was used in the Type 4735 gauges.

Each set of leads was run out to an ice-bath reference junction, to a dpdt switch, then through copper leads, to an L & N potentiometer. When a stabilized temperature was reached, the output was read for both types of leads, and recorded.

Test Results

The temperature - emf plot in Figure 35 shows the results of this test. When the results of the Southern Research Institute calibrations run on the ACL Type 4734 gauges are plotted against the Type 4735 calibrations,

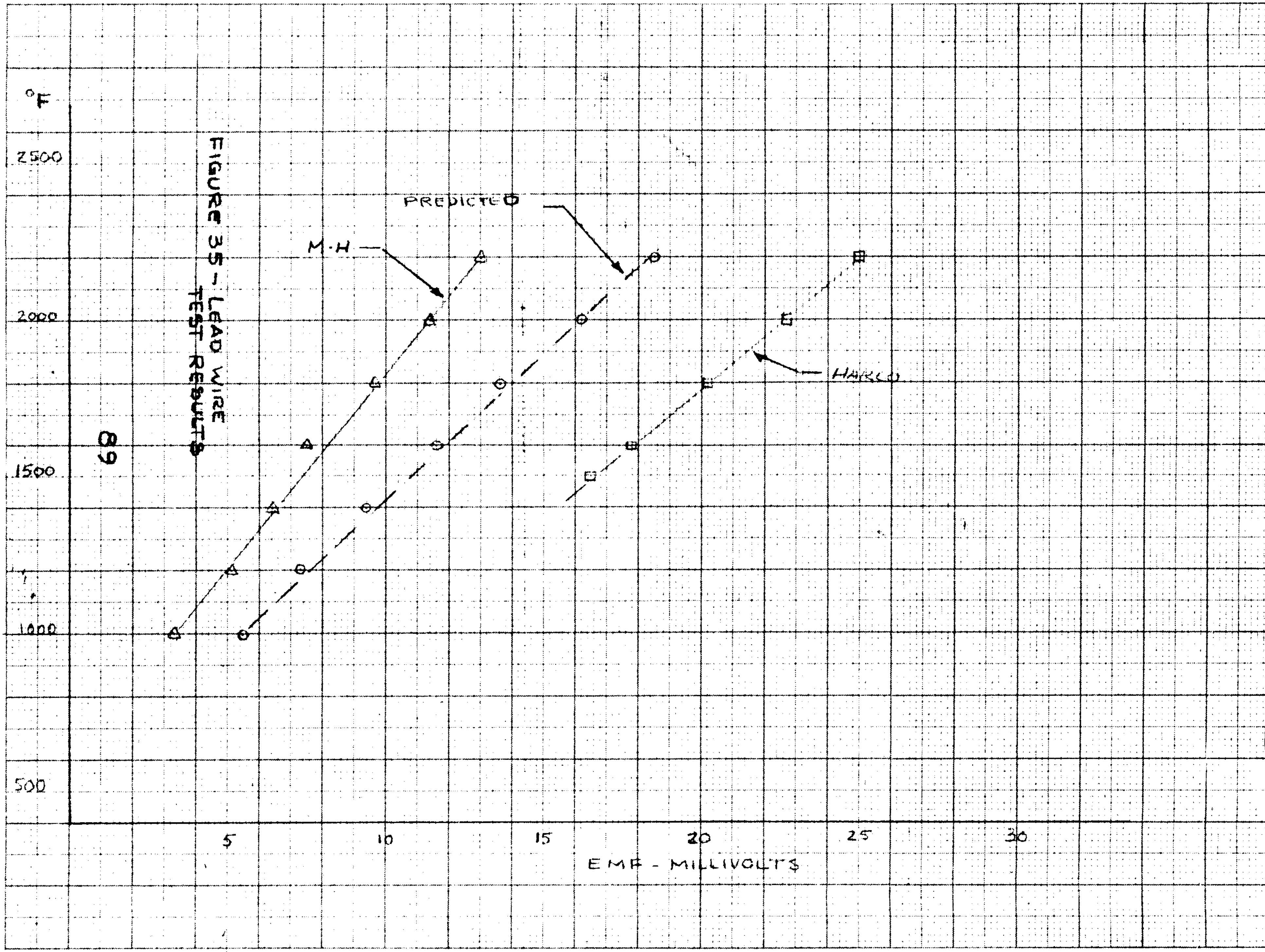


FIGURE 35 - LEAD WIRE TEST RESULTS

[redacted]

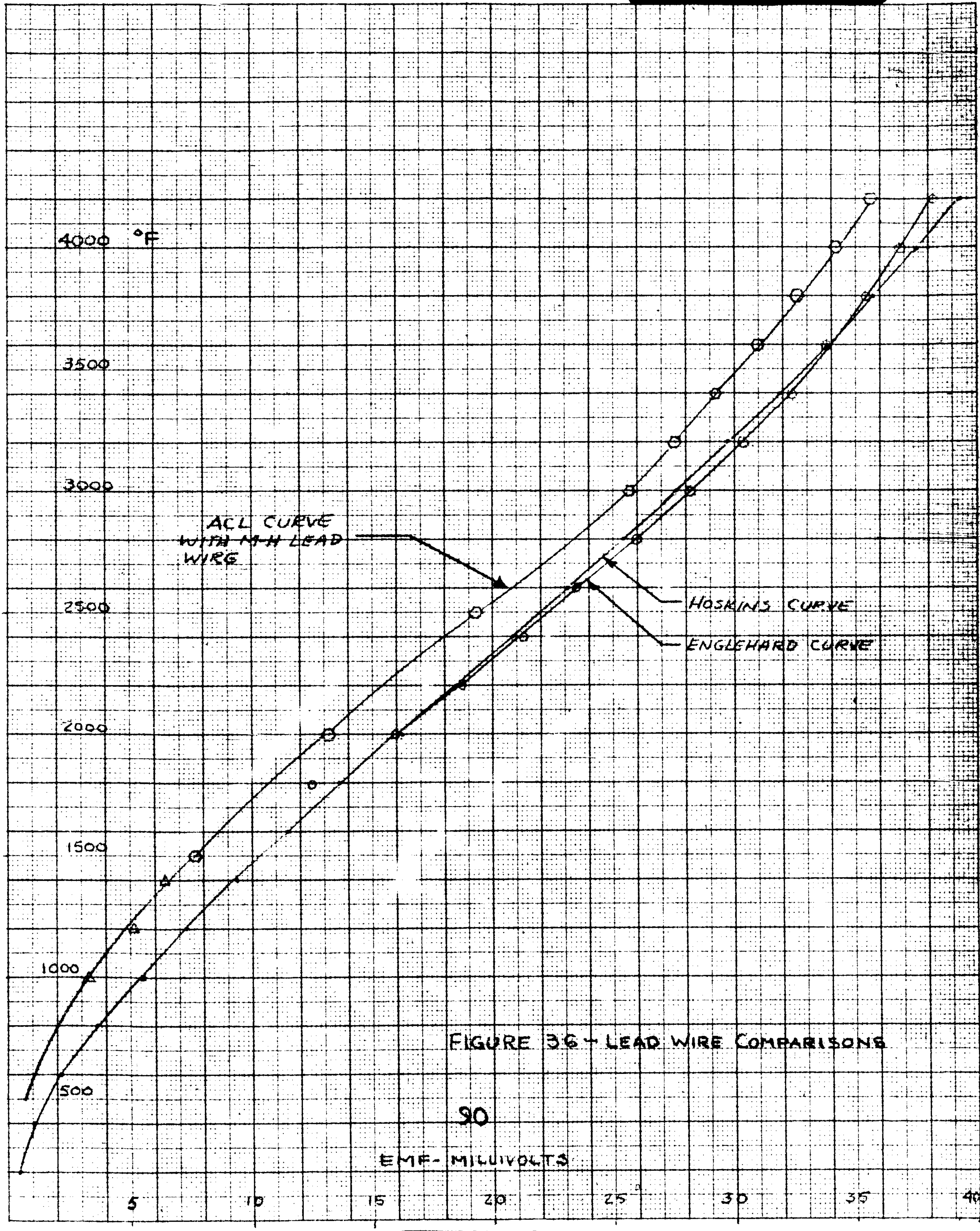


FIGURE 36 - LEAD WIRE COMPARISONS

[redacted]

2.10 <u>Leadwire Calibration Tests</u> (Cont'd.)

<u>Test Results</u> (Cont'd.)

the difference between the two types of lead wire is evident. See Figure 36.

<u>Conclusions</u>

It is concluded that in at least the two types of compensated lead wire tested, there is an appreciable difference in output of the Type 4735 gauge. The difference between the output of the M-H wire and the gauge, as compared with the Harco gauge, is less. The M-H wire more nearly approximates the predicted calibration curve. It is believed that the error is attributable to a spurious emf generated between the W and W26Re and their associated lead wires. The most desirable condition would exist where lead wires of the same materials as the thermocouple are used.

2.11 <u>Stability of W-W26Re Thermocouples</u>

Little reliable background data is available regarding stability of Tungsten-Tungsten 26 Rhenium thermocouples at and near the temperatures of interest. However, one report* was found, which discusses, generally, the Tungsten-Rhenium system.

Figure 37, reproduced from this report, shows the effect of temperature cycling a test thermocouple several times in a hydrogen atmosphere.

*Stability of Rhenium/Tungsten Thermocouples in Hydrogen, Keuther & Lachman, ISA Journal, March 1960.

2.11 Stability of W-W26Re Thermocouples (Cont'd.)

The highest temperature to which the thermocouple was cycled was 2300°C, which approaches the temperatures of interest in this program. The authors conclude that their tests were of value in that the error of less than 4% at 1000°C lessens to 1.5% at 1500°C and was not detectable at 2000°C. They believe that the instability observed during these tests is due either to changes in chemical or physical properties, or to the addition of impurities by the furnace in which the thermocouples were heated. The tests were run in the presence of Tungsten, Molybdenum and Zirconium, and impurities in the refractories of the oven. ACL agrees with the authors that the tests may be even more significant because of the presence of the contaminants.

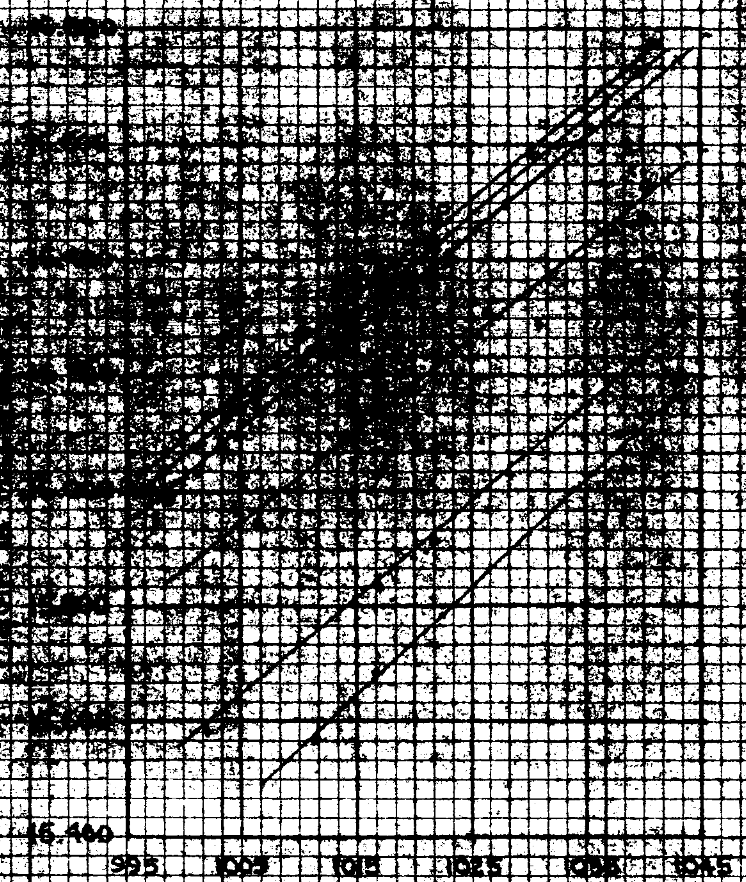


FIGURE 37 - STABILITY OF TUNGSTEN-RHENIUM