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SO_2 Reactivity Studies with BENMOL Sorbents CRADA 90-002, Final Report

December 1990

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Disclaimer

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INTRODUCTION

BENMOL sorbents have been purported to be reactive with sulfur dioxide (SO_2) in flue gas at post-air-preheater temperatures (350°F and below), thus making them possible candidates for dry sorbent injection desulfurization processes. As an initial step to determine the reactivity of these sorbents, microbalance studies were conducted at the Pittsburgh Energy Technology Center (PETC) under the Cooperative Research and Development Agreement (CRADA) program. Initial reactivity rates and total absorption capacities were determined and compared to those obtained with hydrated lime, which is the chosen sorbent for most duct injection flue gas desulfurization processes.

EXPERIMENTAL APPROACH

BENMOL Corporation provided five sorbent samples for testing. The samples were reported to have an average particle size between 50-100 microns. Three samples (B5,D,E) were homogeneous; two sorbents (D7,E7) required riffling prior to testing in order to obtain homogenized samples. The hydrated lime sorbent was an ACS certified calcium hydroxide powder obtained from J.T. Baker containing 97.4% $Ca(OH)_2$ (see Table 1).

The sorbents were tested in a microbalance unit shown schematically in Figure 1. A simulated flue gas was blended from certified gas mixtures and humidified. For humidification, the oxygen-containing gas was bubbled through a water saturator and blended with the dry gas stream containing SO_2 and CO_2. The temperature of the water saturator was controlled to obtain the desired gas humidity at a set gas flow rate; this was checked by condensing the water vapor from a stream at a set temperature and calculating the water vapor content. The water saturator and all lines to the microbalance were heat-traced to prevent condensation. Depending upon the total gas flow rate, the water content of the gas stream ranged from 5.4-

5.8% by volume. The simulated flue gas flow rates were varied from 500 to 2000 cm^3/min (1 atm, 0°C) -- or sccm on a dry basis -- with most tests being conducted at 1500 sccm. The simulated flue gas composition (on a wet basis) for tests conducted at about 1500 sccm was approximately 2866 ppm SO_2, 12.55% CO_2, 3.00% O_2, 5.8% H_2O, with a balance of N_2.

A Cahn Electrobalance (Model 1000) was used to measure the weight changes of the solid samples as reaction occurred. The microbalance is a differential measuring instrument, designed to accommodate weights up to 100 grams and is sensitive to mass changes as small as 0.5 microgram. For this work, the 10 and 100 milligram ranges were used, thus, providing an accuracy in readings of ± 0.01 and ± 0.1 mg, respectively. Prior to testing, the chart recorder and microbalance were zeroed and calibrated using standard weights for the desired experimental weight range. For the tests at 163°C, the 10 milligram weight range was used and the recorder was adjusted so that full scale deflection on the chart was 5 mg. At the higher reactor temperatures of 427°C and 650°C, larger weight gains were observed; therefore, the 100 milligram weight range was used and the full scale deflection on the chart recorder was adjusted to 50 mg.

Sorbent samples weighing approximately 50 mg were spread over a layer of quartz wool placed in a quartz sample basket (see Figure 2). The basket was positioned inside the quartz reactor tube in the furnace and suspended on a wire connected to the microbalance weighing apparatus (see Figure 3). The sample was then heated to reaction temperature under a flow of pure nitrogen gas for a typical period of 90 minutes. The gas stream entered the microbalance at the base of the reactor tube and passed through a section containing ceramic beads, which were placed in the tube to reduce the amount of dead gas volume. The temperature of the reactor was measured with a thermocouple positioned 0.5 cm below

the sample basket. Absorption experiments were conducted at reactor temperatures of 163°C (325°F), 427°C (801°F), and 650°C (1202°F).

During the heat-up period, the BENMOL sorbents were observed to lose weight. Although the reason for this loss in weight of the BENMOL sorbents is not known, some sorbents have been observed to lose weight during the heat-up period due to moisture loss.

After a steady state reactor temperature was reached and the sorbent weight stabilized, the simulated flue gas mixture, which was bypassing the microbalance, was introduced into the microbalance by switching the air-actuated four-way valves shown in Figure 1. The flow rates of the two gas streams were closely matched to minimize shock buoyancy forces that can cause inaccuracies in sample weight measurements during the initial time period. The sorbent was typically exposed to the SO_2-containing gas stream for a period of 90 minutes, during which time the system reached a steady state and no further weight change was observed. Upon completion of the absorption period, the air-actuated four-way valves were again activated, thus, allowing the sample to cool to room temperature using nitrogen gas.

After the system had cooled to room temperature, the contents of the sample basket (quartz wool and exposed sorbent) were placed in a labeled sample vial under a nitrogen atmosphere. Selected test samples were later analyzed for total sulfur content.

EXPERIMENTAL RESULTS

Prior to sorbent testing, humidity experiments were conducted to determine the bubbler temperature required to obtain approximately 5% moisture in the flue gas stream at various gas flow rates. Figure 4 shows the flow scheme used to test the flue gas humidity.

Nitrogen was bubbled through the water saturator heated at a set temperature. The humidified nitrogen was blended with a dry nitrogen stream to simulate the total flow of flue gas mixture. The gas stream then passed through a glass condenser placed in an ice bath, followed by a gas purifier containing drierite desiccant and zeolite sorbent. The amount of water condensed and absorbed was determined from weight measurements. The water vapor content of the gas stream was then calculated.

Results from the humidity tests are given in Table 2. At about 1000 sccm total dry gas flow rate, a gas bubbler temperature of 56°C gave a flue gas moisture content of 5.5%. Similarly, at approximately 500 sccm, 5.4% H_2O was obtained at a gas bubbler temperature of 70°C, and 5.8% H_2O at about 1500 sccm and a gas bubbler temperature of 56°C. These bubbler temperatures were used during the sorbent testing at the respective gas flow rates to maintain a relatively constant water vapor content (5.4-5.8%) in the total gas stream.

Fourteen microbalance tests were conducted with BENMOL sorbents and hydrated lime. The microbalance weight change versus time curves resulting from the sorbent tests can be found in Appendix A. Percent weight gains and initial absorption rates were evaluated from the weight curves using the following calculational methods. The sorbent weight gains after 45, 60, and 90 minutes of exposure to simulated flue gas were determined, from which the percent weight gains at the respective times were calculated based on the initial weight of the sorbent placed in the sample basket. The initial absorption rate was obtained by drawing the best straight line through the data at the start of absorption and determining the slope of the line which represents the weight gain occurring in the respective time period. This measurement can vary somewhat due to individual interpretation of the "best straight line" through the "initial" data. Therefore, a 5-minute average absorption rate

was also calculated based on the weight gain in the first five minutes of absorption. Sample calculations from test 11 are given in Appendix B.

Results of the microbalance sorbent experiments are shown in Table 3. The first five tests were conducted with SORCAT B5 at varying gas flow rates from approximately 500 to 2000 sccm to determine the region of negligible mass transfer resistance. If the system is gas mass-transfer-limited, then the absorption rates will increase as more SO_2 is supplied to the reaction surface per unit time. If there is negligible mass transfer resistance, then the reaction rates will remain relatively constant. From the initial five tests, an increase in absorption rates was observed from 1000 to 1500 sccm, but little change was seen at 2000 sccm gas flow rate. From these results, 1500 sccm appeared to be in the region of negligible mass transfer resistance and was chosen as the gas flow rate for the sorbent testing.

Tests 3, 4A, 6, 7, 8, and 9 were all conducted at about 163°C using various sorbents. Duplicate tests using SORCAT B5 and hydrated lime were conducted. As seen in test pairs (3 and 4A) and (6 and 9), the data replicate fairly well. The accuracy in the microbalance readings is ± 0.01 mg, which results in possible errors in the weight gain data of ± 0.04% and those in the rate data of ± 0.02 mg/min. In the tabulated calculations, the largest inherent error exists in the initial rate data since these values rely on visual interpretation of the best straight lines through the data.

At 163°C reactor temperature, hydrated lime had the highest rate of removal and largest total weight gain. Of the BENMOL sorbents studied, SORCAT B5 performed the best, achieving approximately 70% of the hydrated lime reactivity and absorption capacity. Both SORCAT D and E performed poorly in these absorption tests.

Originally, all the sorbent testing was to be conducted at 163°C (325°F), a representative flue gas temperature after the air preheater in a utility installation and a parameter often used in duct injection work. After initial testing was completed, the scope of work was modified to include two other temperatures, 427°C (801°F) and 650°C (1202°F). The former temperature simulates the flue gas temperature after the economizer but before the air preheater in a utility scheme. The latter is representative of the temperature before the economizer.

Three tests (10, 11, and 18) were conducted at a reactor temperature near 427°C. Hydrated lime performed better than the two BENMOL sorbents. Hydrated lime showed a high rate of absorption, gaining 92% of the total weight absorbed in 45 minutes. The BENMOL sorbents were significantly less reactive than hydrated lime. The 5-minute average absorption rate for hydrated lime was 7 times faster than that of SORCAT B5 and 14 times faster than that of SORCAT D7. The absorption capacity of hydrated lime was double that of SORCAT B5 and 7 times that of SORCAT D7.

Both hydrated lime and SORCAT B5 were tested at a reactor temperature of 650°C (tests 15 and 16). Increasing reaction temperature resulted in increasing absorption rates and capacities for both sorbents. However, hydrated lime continued to outperform SORCAT B5, providing three times the absorption capacity at a clearly higher absorption rate.

Microanalysis for total sulfur was performed by Huffman Laboratories in Golden, Colorado on eight test samples using ASTM test D 4239-85 Method C. The results of six of the tests are given in Table 3. The remaining two test analyses were conducted on raw sorbent samples of hydrated lime and SORCAT B5. The hydrated lime obtained from Baker contained 0.025% S and the initial SORCAT B5 sorbent had 0.073% S.

Interesting results were found from the sulfur analyses. Since both CO_2 and SO_2 are present in the simulated flue gas mixture, it is possible that the observed weight gains may be due to carbonate formation as well as sulfur removal. By comparing the results of the total sulfur analyses, more information can be gathered on the type of species being removed. Analytical results from tests 5 and 9 show that more sulfur was contained in the hydrated lime sample than the SORCAT B5 sample, corresponding to the observed difference in the sorbent absorption capacities. The total weight gain of the SORCAT B5 sorbent in 90 minutes was 67% of the weight gained by the hydrated lime sample. Similarly, the total sulfur contained in the SORCAT B5 sample was 62% of the total sulfur in the hydrated lime sample. Thus, both samples appear to be absorbing SO_2 in the same proportion. At 650°C reactor temperature, hydrated lime contained more sulfur than the SORCAT B5 sample (tests 15 and 16). The total absorption capacity of SORCAT B5 was only 32% of that observed with hydrated lime; however, the total sulfur content of the SORCAT B5 sample was 46% of the sulfur found in the hydrated lime test. This may indicate that hydrated lime sample is absorbing more CO_2 than the SORCAT B5 sorbent.

The analytical results from tests 10 and 11 conducted at 427°C were surprising, however, since the sulfur content of the SORCAT B5 sample was more than double that of the hydrated lime sample. This result was not expected because the total weight gained by the SORCAT B5 sorbent was about half the weight gained by the hydrated lime sample, and this was also opposite of the trend in the data collected at 163°C or 650°C. Huffman Laboratories was contacted concerning the sample results. It was verified that the analytical data were reported correctly. Unfortunately, due to the small sample sizes, requested duplicate analyses could not be performed. If the sulfur results are correct, then the SORCAT B5 sorbent was a better SO_2 removal agent than hydrated lime at 427°C and the larger observed absorption capacity of hydrated lime was probably

due to increased uptake of CO_2.

CONCLUSIONS

Fourteen tests were conducted in a microbalance unit studying the reactivity of various BENMOL sorbents and hydrated lime at 163°C, 427°C, and 650°C. At similar test conditions, hydrated lime was found to have faster rates of absorption and larger absorption capacities than the BENMOL sorbents. Of the BENMOL sorbents studied, SORCAT B5 gave the best removal performance. Both hydrated lime and SORCAT B5 showed increased absorption rates and removal capacities as reactor temperature was increased. Sulfur analyses of selected samples correlated with the experimental findings, except for those taken at a reactor temperature of 427°C.

DISCLAIMER

Reference in this paper to any specific commercial product, process, or service is to facilitate understanding and does not necessarily imply its endorsement or favoring by the United States Department of Energy.

Table 1

Calcium Hydroxide Powder Analysis

'Baker Analyzed' Reagent 1372-01

Actual Analysis, LOT B08339

Meets A.C.S. Specifications

Assay ($Ca(OH)_2$)	97.4%
Insoluble in HCl	<0.01%
Chloride (Cl)	<0.015%
Sulfur Compounds (as SO_4)	<0.05%
Heavy Metals (as Pb)	<0.003%
Iron (Fe)	<0.03%
Magnesium and Alkali Salts (as SO_4)	0.8%

TABLE 2
HUMIDITY TEST RESULTS

TEST NO.	BUBBLER TEMP. (°C)	N_2 FLOW THRU BUBBLER (dry sccm)	TOTAL N_2 FLOW RATE (dry sccm)	WEIGHT CHANGE of DRIERITE (g)	WEIGHT CHANGE of ZEOLITE (g)	WEIGHT of CONDENSATE (g)	TOTAL H_2O COLLECTED (g)	% H_2O (sccm H_2O per 100 sccm wet gas)
HT-1	71	314	994	1.53	0.01	6.85	8.39	14.7
HT-2	71	313	990	1.24	0.12	5.97	7.33	13.1
HT-3	71	314	994	0.76	0.01	3.01	3.78	7.2
HT-4	71	312	986	1.75	0.07	5.48	7.3	13.1
HT-5	71	314	993	2.35	0.08	4.93	7.36	13.15
HT-6	53	314	993	0.13	0.03	1.40	1.56	13.1
HT-7	60	315	996	1.1	0.01	2.9	4.01	7.6
HT-8	56	314	993	1.18	0.05	1.62	2.85	5.5
HT-9	56	312	988	1.27	0.02	1.53	2.82	5.5
HT-10	56	158	495	0.34	0.19	0.42	0.95	3.8
HT-11	62	158	496	0.26	0.10	0.8	1.16	4.5
HT-12	70	158	495	0.20	0.01	1.17	1.38	5.4
HT-13	56	473	1490	0.88	0.05	3.57	4.5	5.8

*Duration of each test was 1 hour.

TABLE 3
MICROBALANCE SORBENT TEST RESULTS

RUN NO.	SORBENT	REACTOR TEMP (°C)	INITIAL WT. (mg)	GAS FLOW RATE (dry sccm)	% WT. LOSS DURING HEAT UP	45 MIN. ABSORPTION WT. GAIN (%)	60 MIN. ABSORPTION WT. GAIN (%)	90 MIN. ABSORPTION WT. GAIN (%)	INITIAL ABSORPTION RATE (mg/min)	5 MIN. AVG ABSORPTION RATE (mg/min)	TOTAL SULFUR (%)
2	B5	162	50.44	495	3.08	1.80	1.89	---	1.14	0.115	---
1B	B5	163	50.02	982	2.84	1.90	---	---	1.14	0.111	---
3	B5	164	50.34	1483	2.82	1.77	1.88	1.98	1.39	0.121	---
4A	B5	162	50.43	1477	3.17	1.85	1.95	---	1.89	0.126	---
5	B5	163	50.04	1985	4.11	1.75	1.81	1.96	1.52	0.120	0.37
6	LIME	163	50.93	1481	1.26	2.87	3.04	3.30	2.22	0.189	---
7	D	164	50.01	1490	1.18	0.03	0.03	0.05	---	0.002	---
8	E	163	50.23	1487	0.72	0.08	0.08	0.08	---	0.006	---
9	LIME	162	50.03	1487	1.30	2.53	2.68	2.91	2.55	0.166	0.60
10	LIME	428	50.29	1487	25.85	28.53	29.43	31.02	16.98	2.53	1.48
11	B5	427	50.13	1480	12.97	11.87	13.46	15.36	1.53	0.34	3.30
15	LIME	650	50.02	1489	31.39	60.78	65.27	68.77	14.46	3.95	7.43
16	B5	650	50.59	1489	18.88	19.27	20.46	21.94	4.94	0.54	3.44
18	D7	428	50.65	1487	5.43	3.26	3.46	4.15	0.72	0.16	---

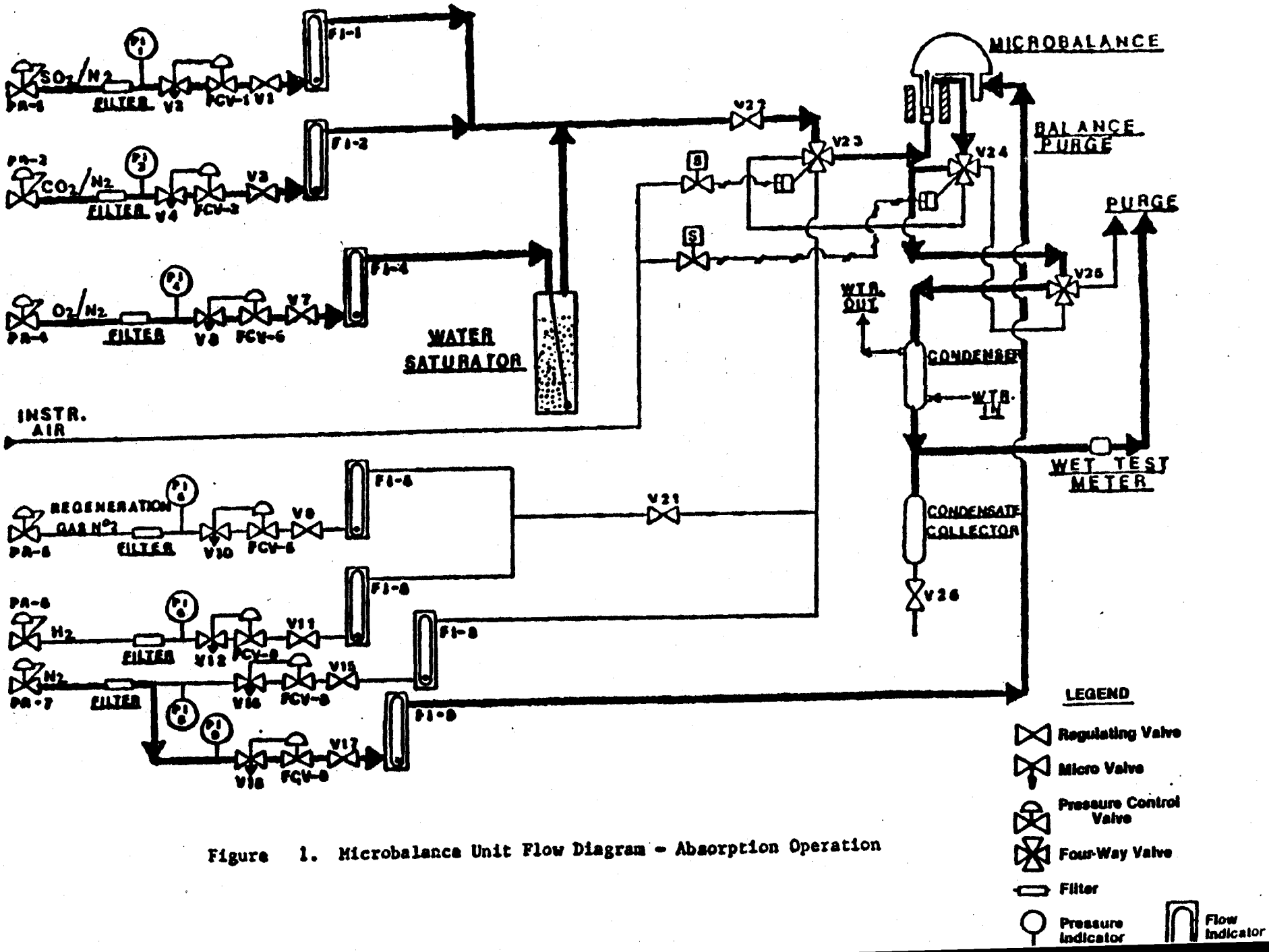

Figure 1. Microbalance Unit Flow Diagram - Absorption Operation

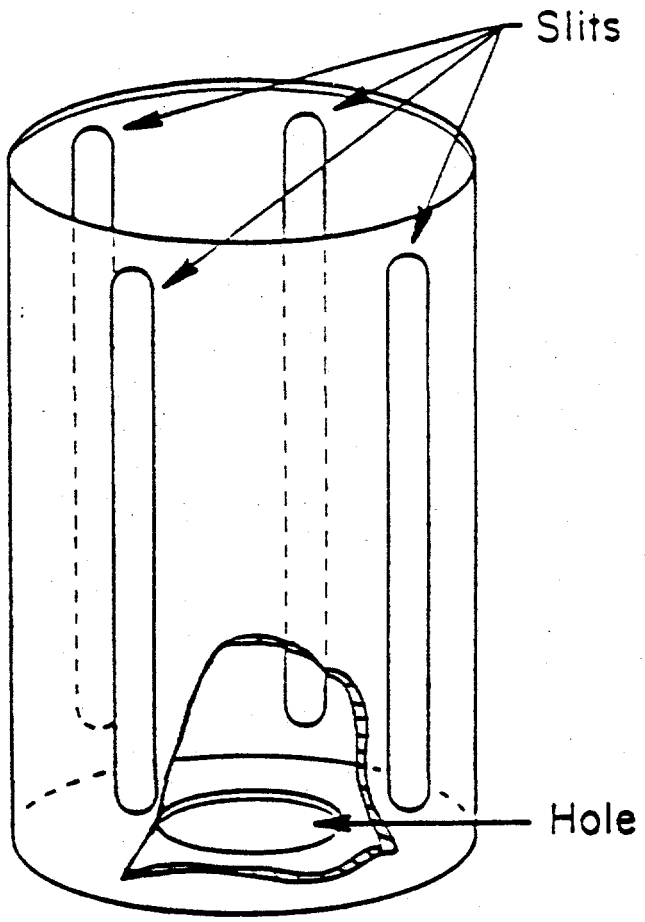

Figure 2. Microbalance Sample Basket.

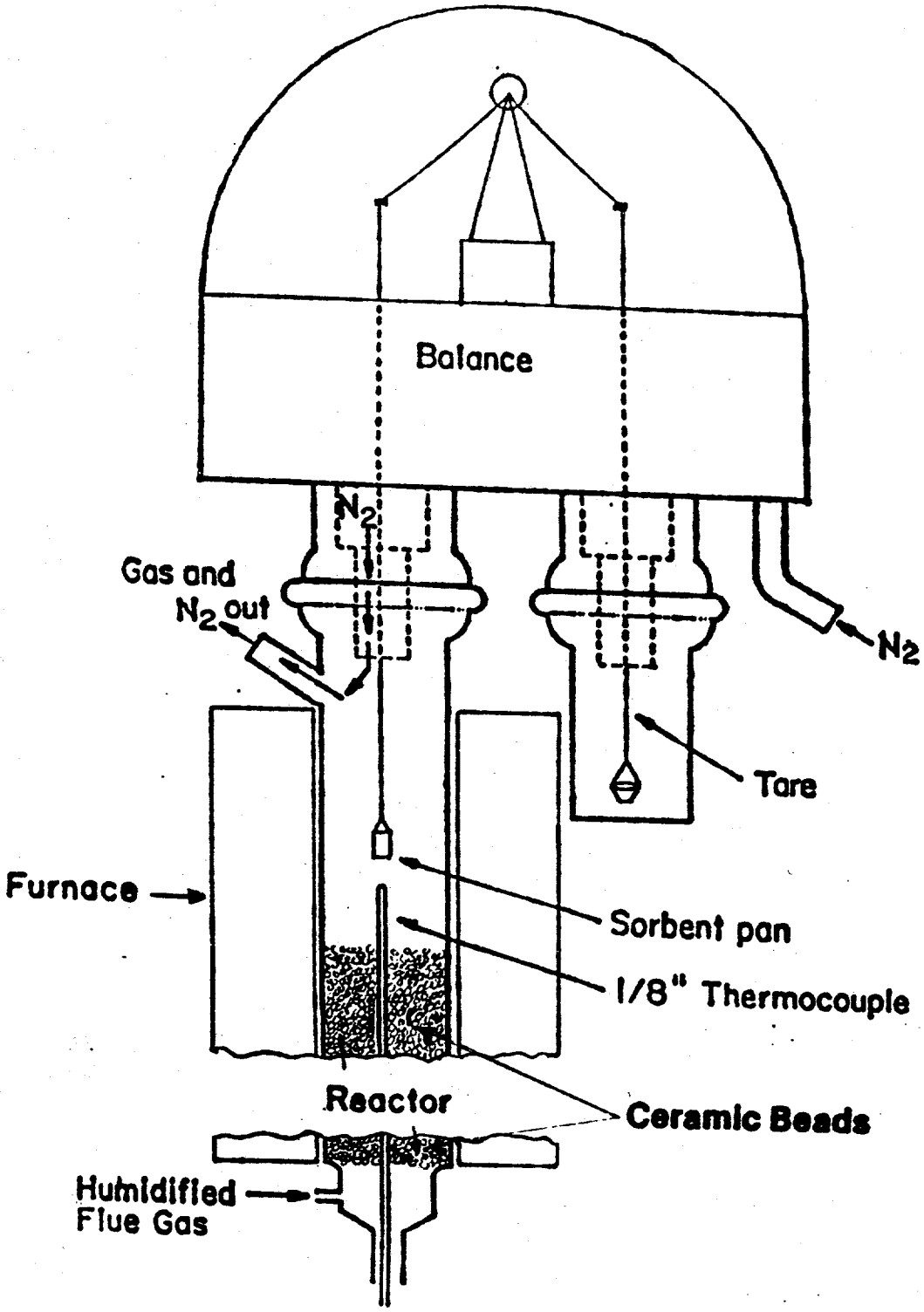

Figure 3. Microbalance Reactor Used for Kinetics Study.

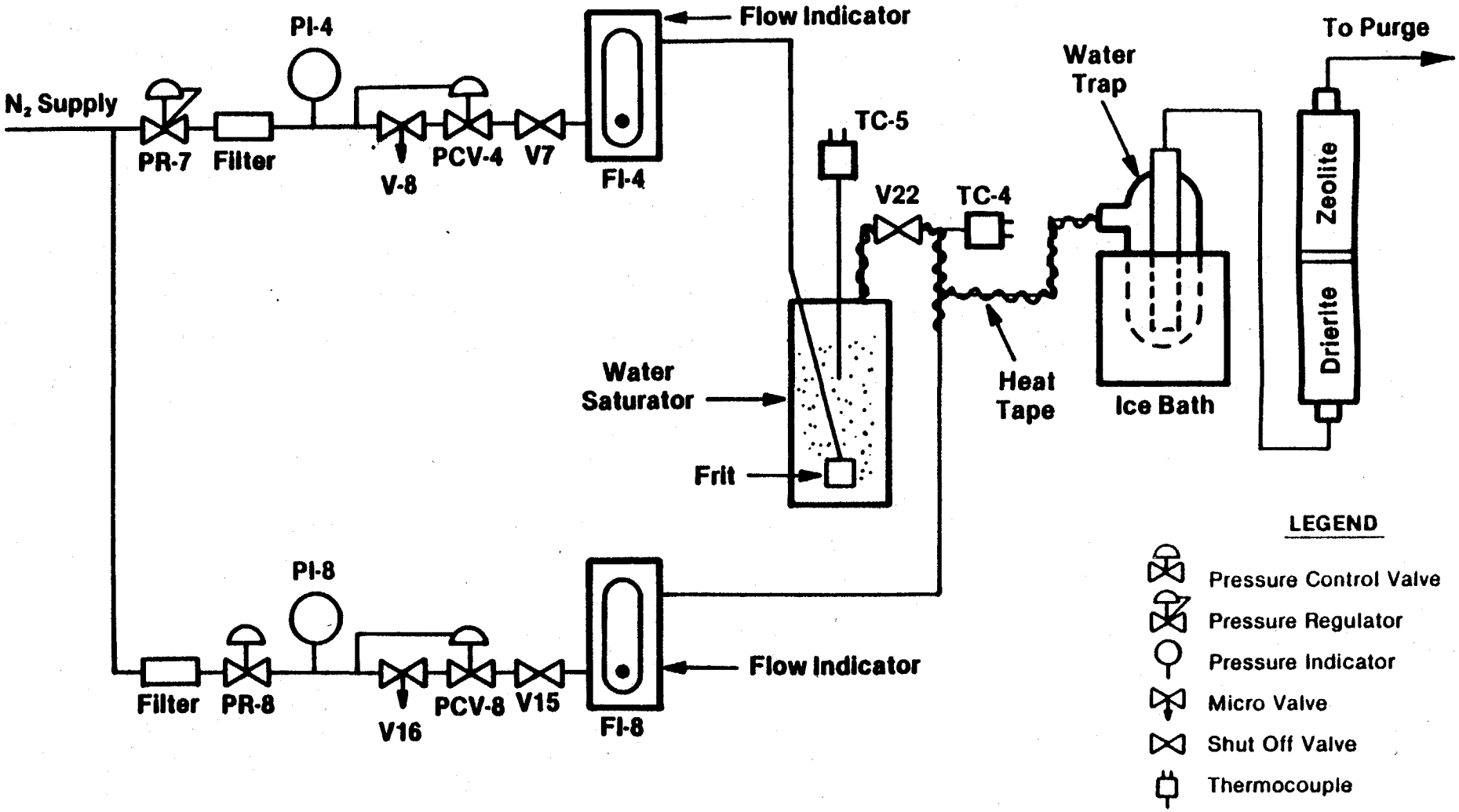

Figure 4. Flow Schematic for Humidity Testing.

Appendix A

Microbalance Experimental Test Data

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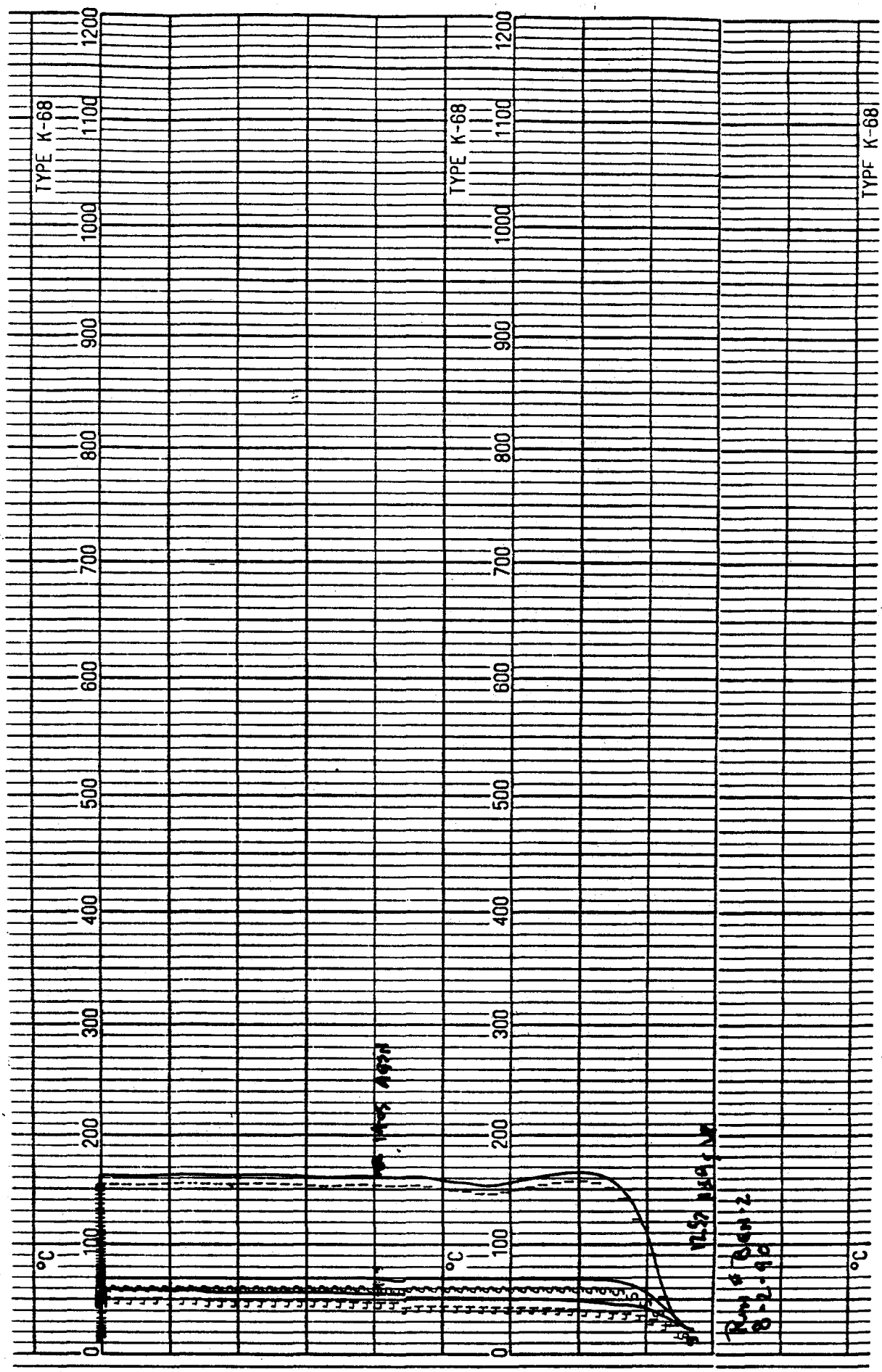
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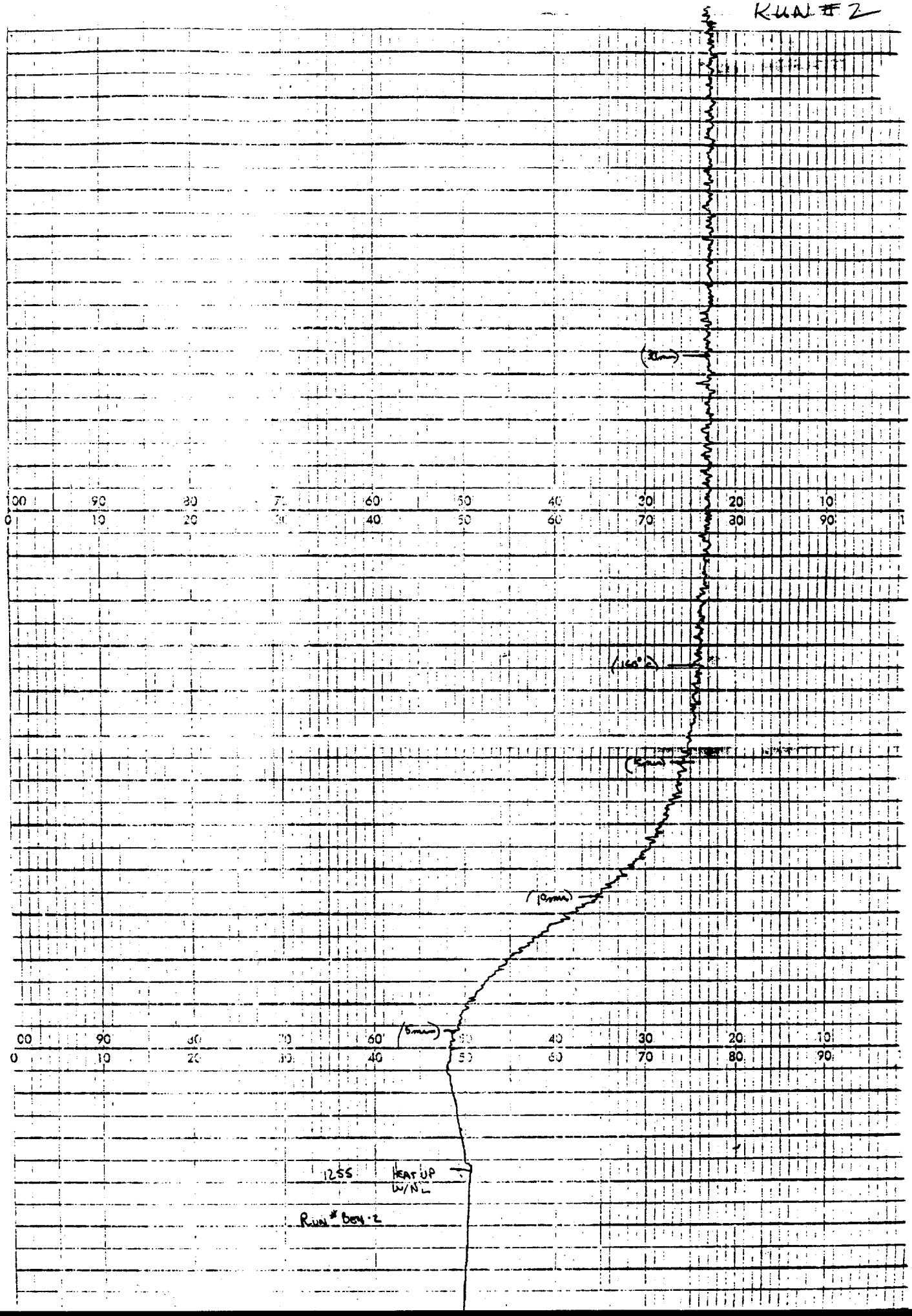
°C
TYPE K-68
TYPE K-68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(5mm)
(10mm)
(5mm)
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W/ N2
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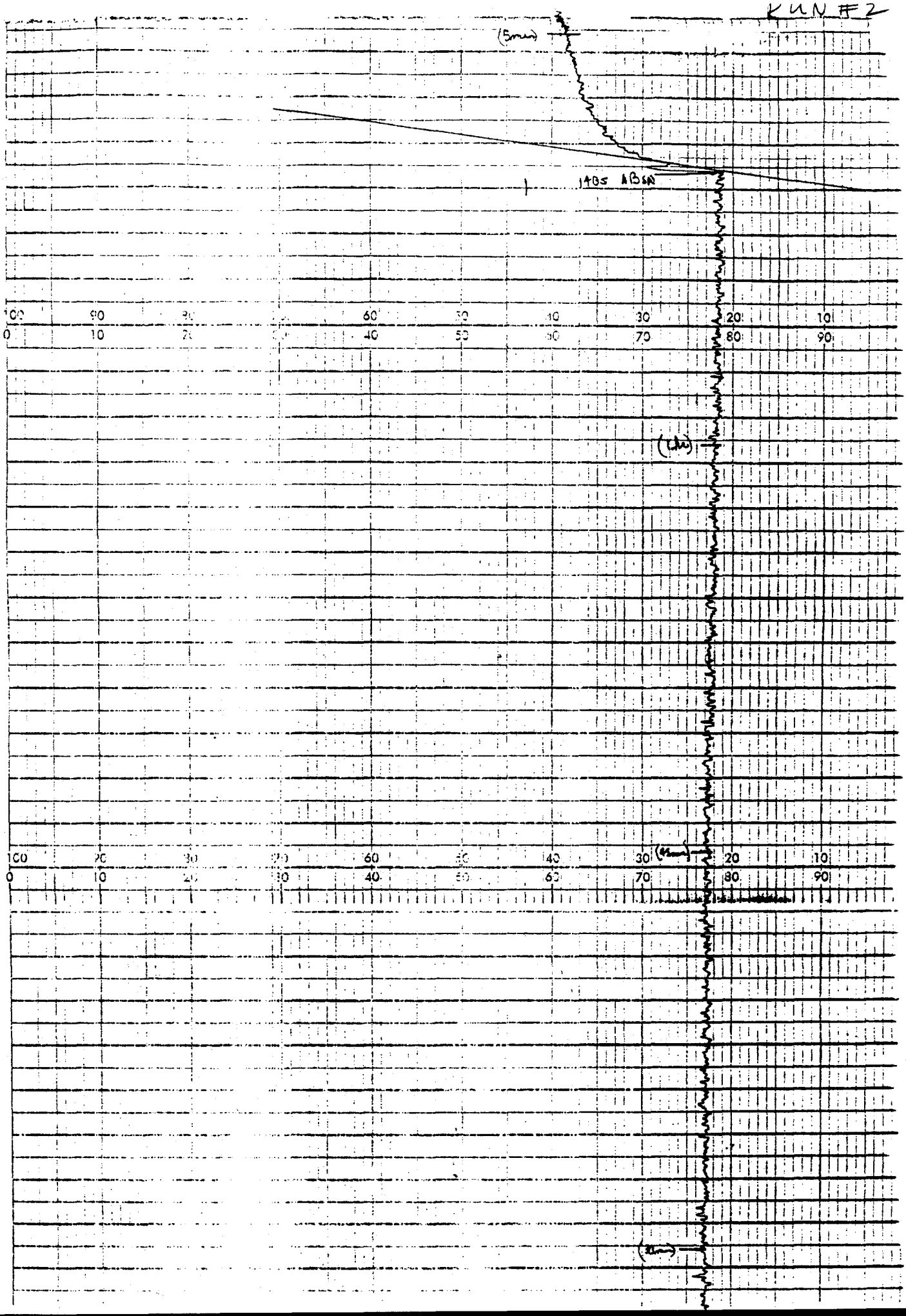
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(10mm)
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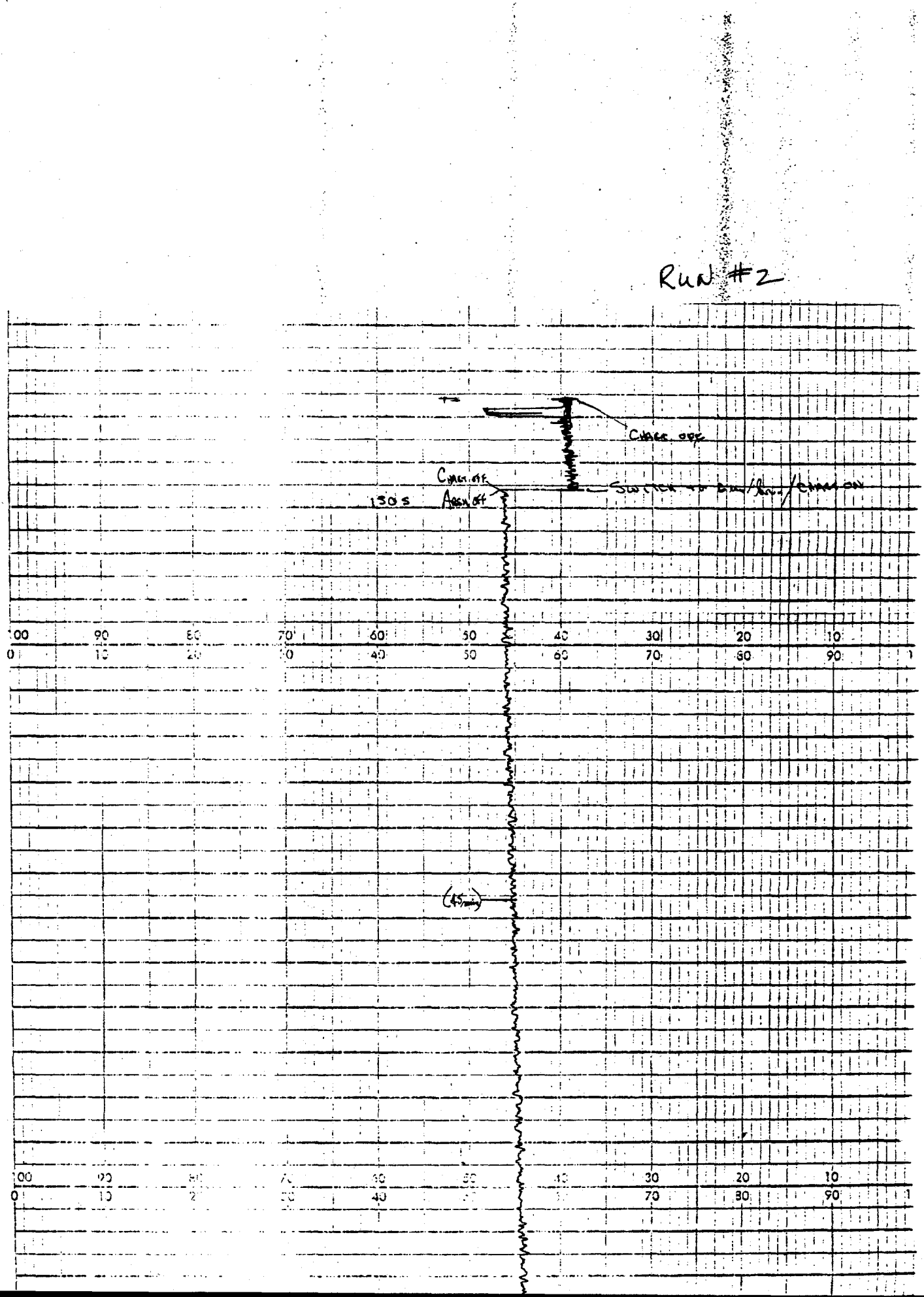
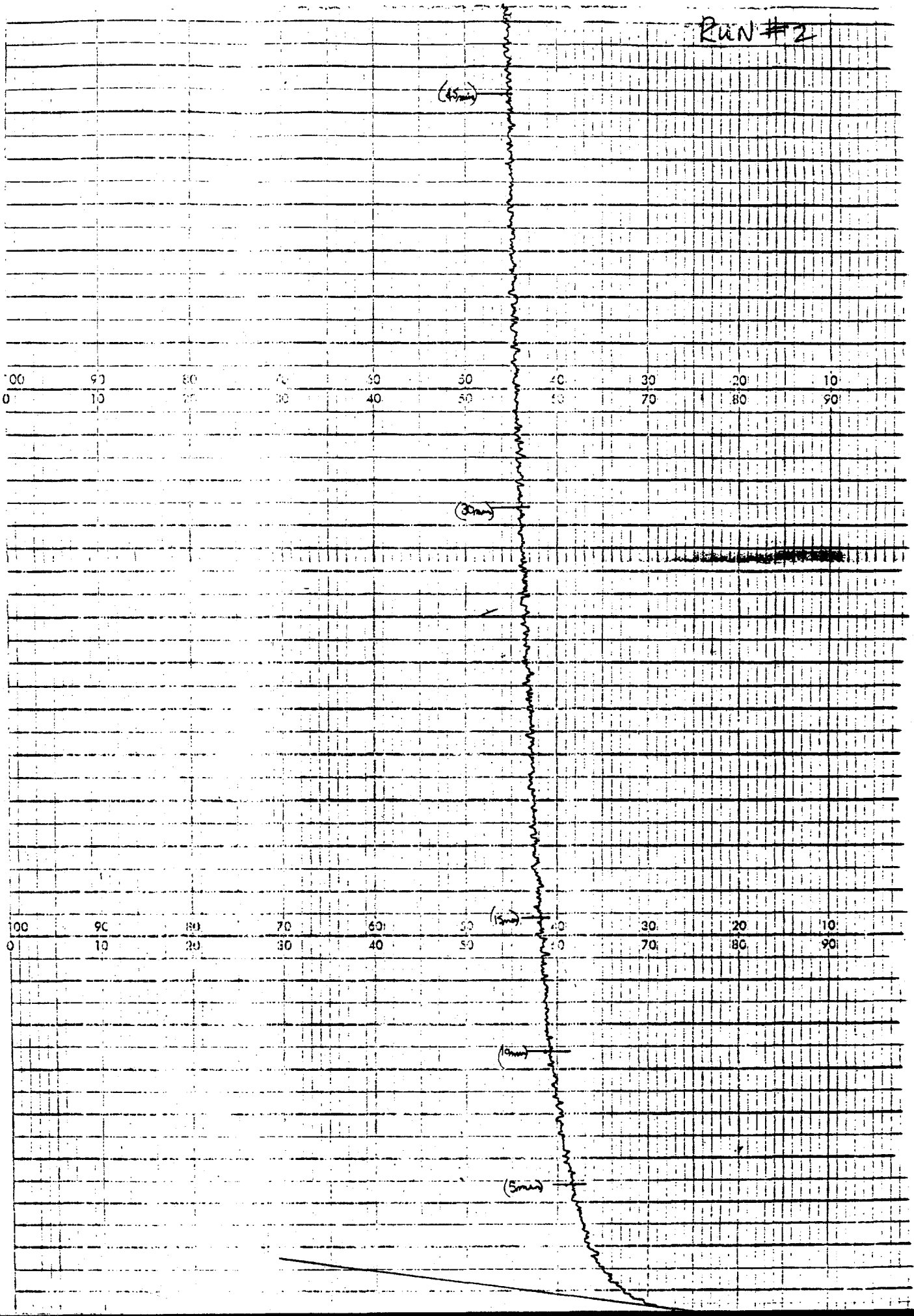
(10mm)
(10mm)

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(1min)
(5min)
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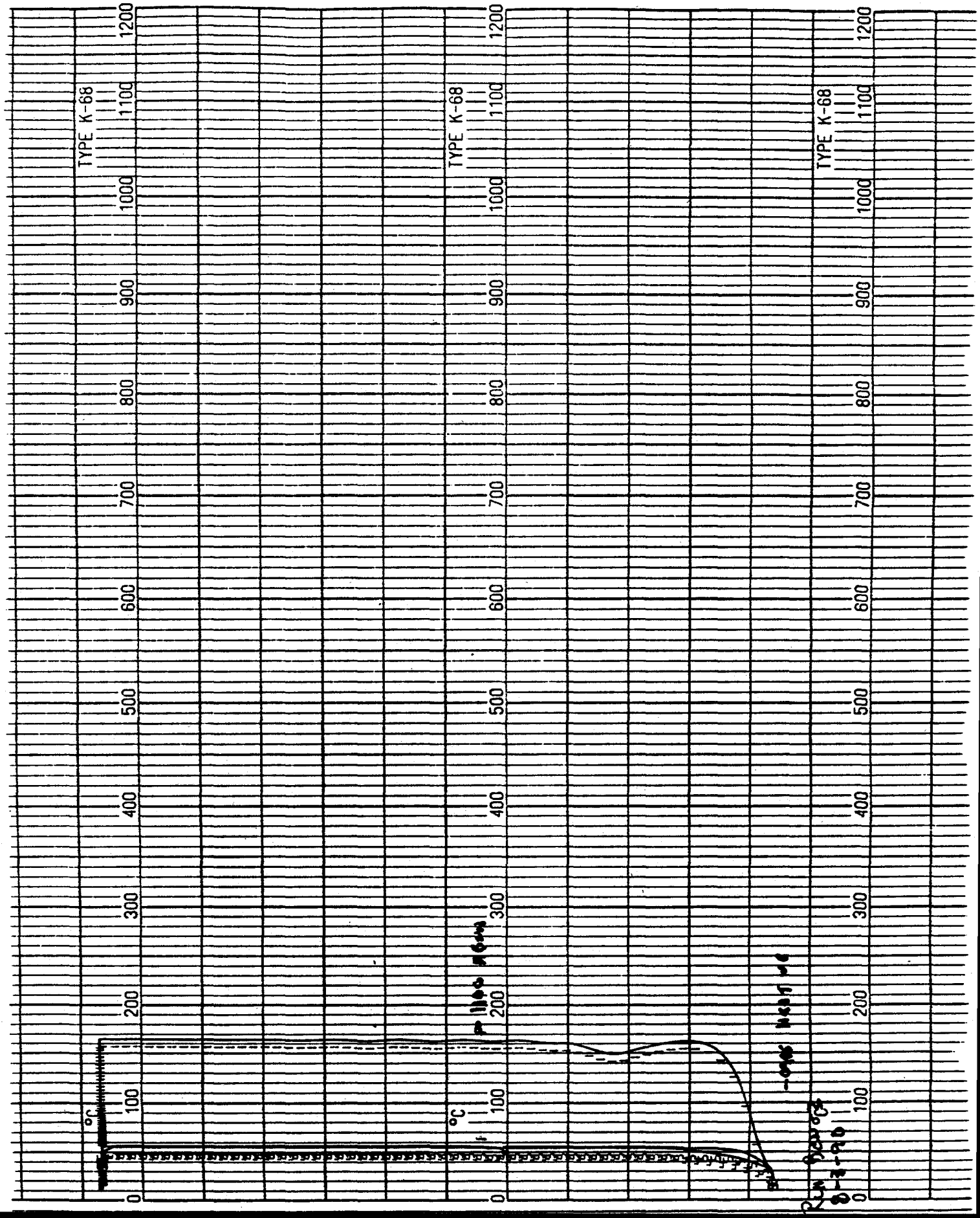
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TYPE K-68
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1100
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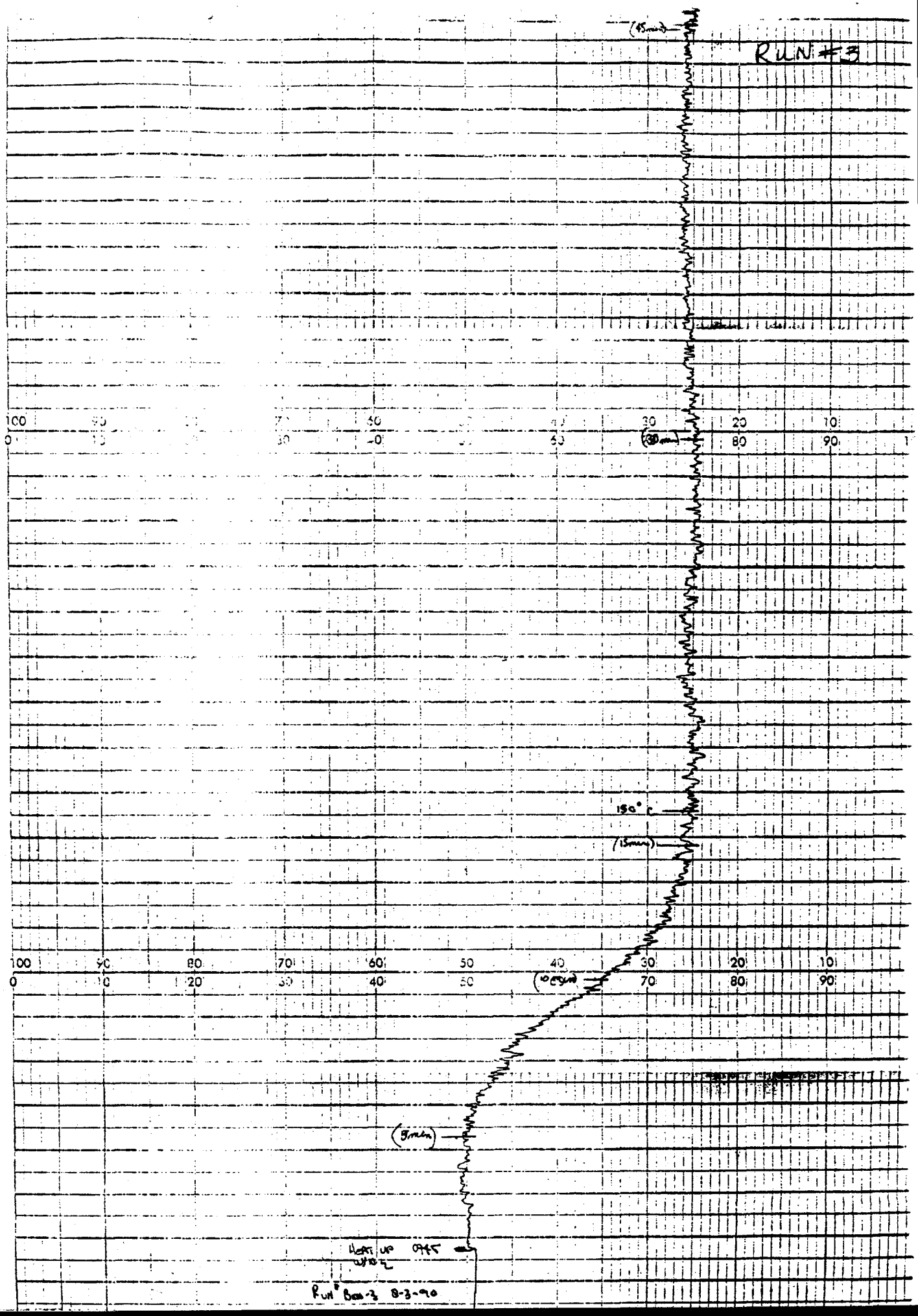

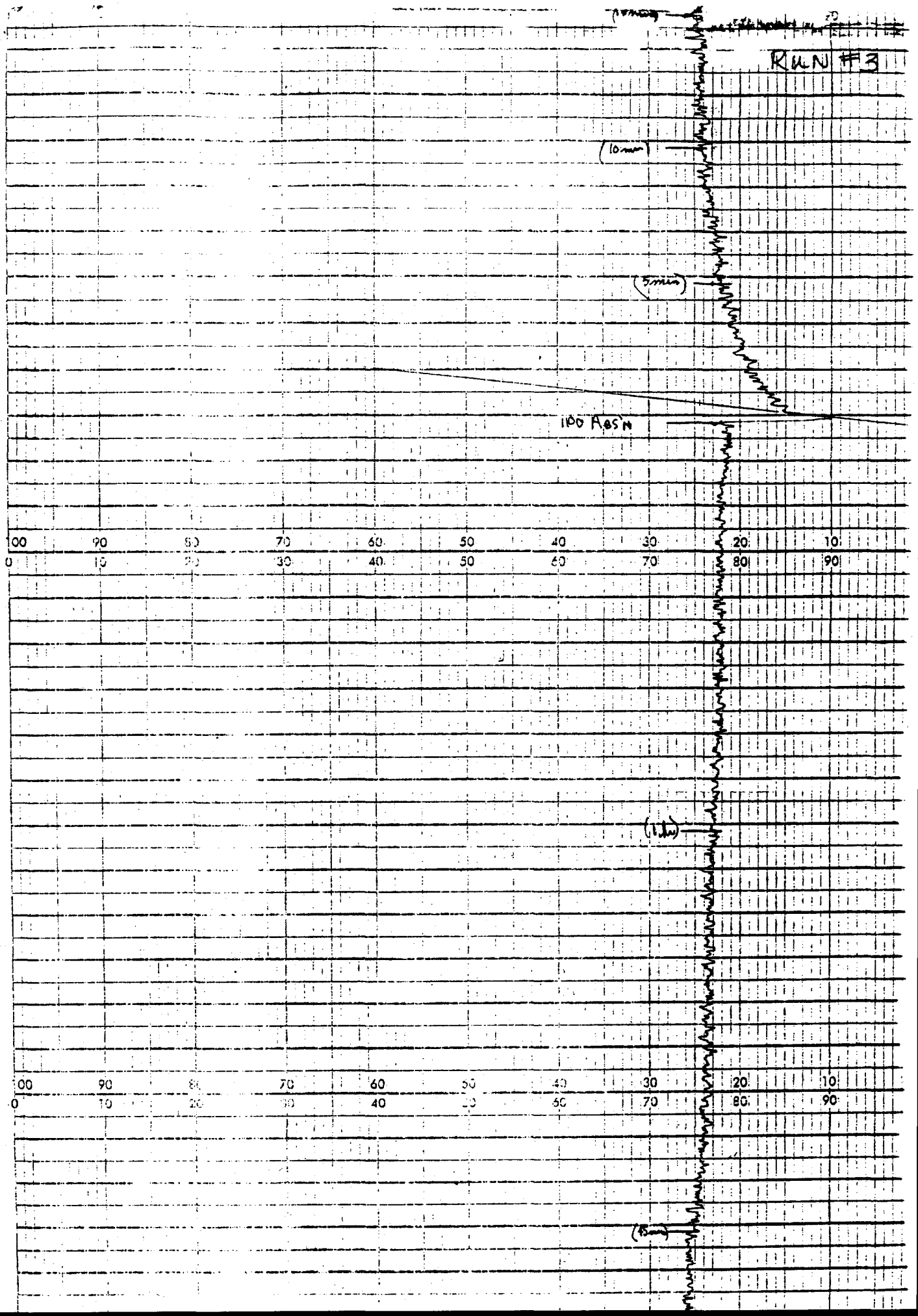

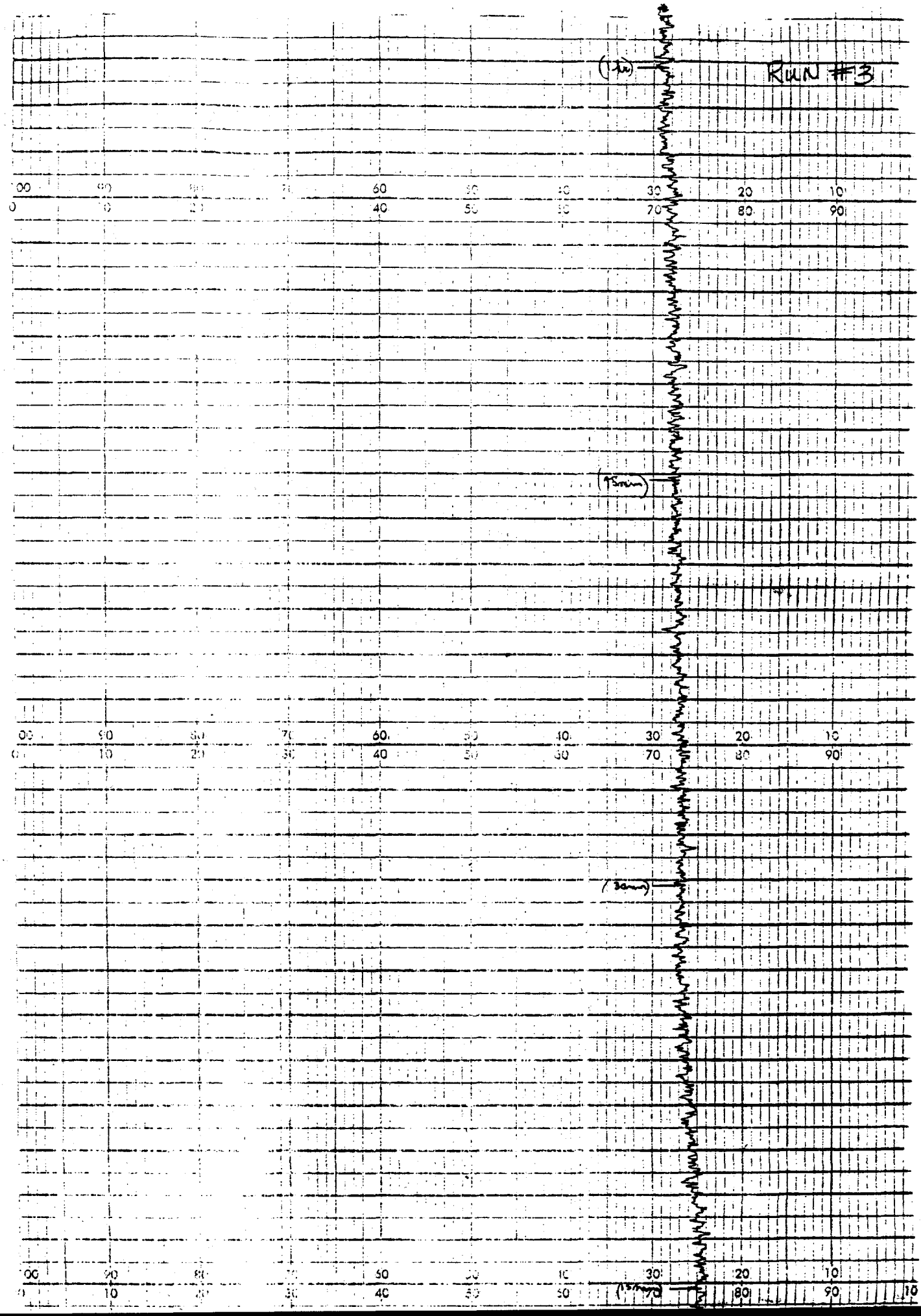
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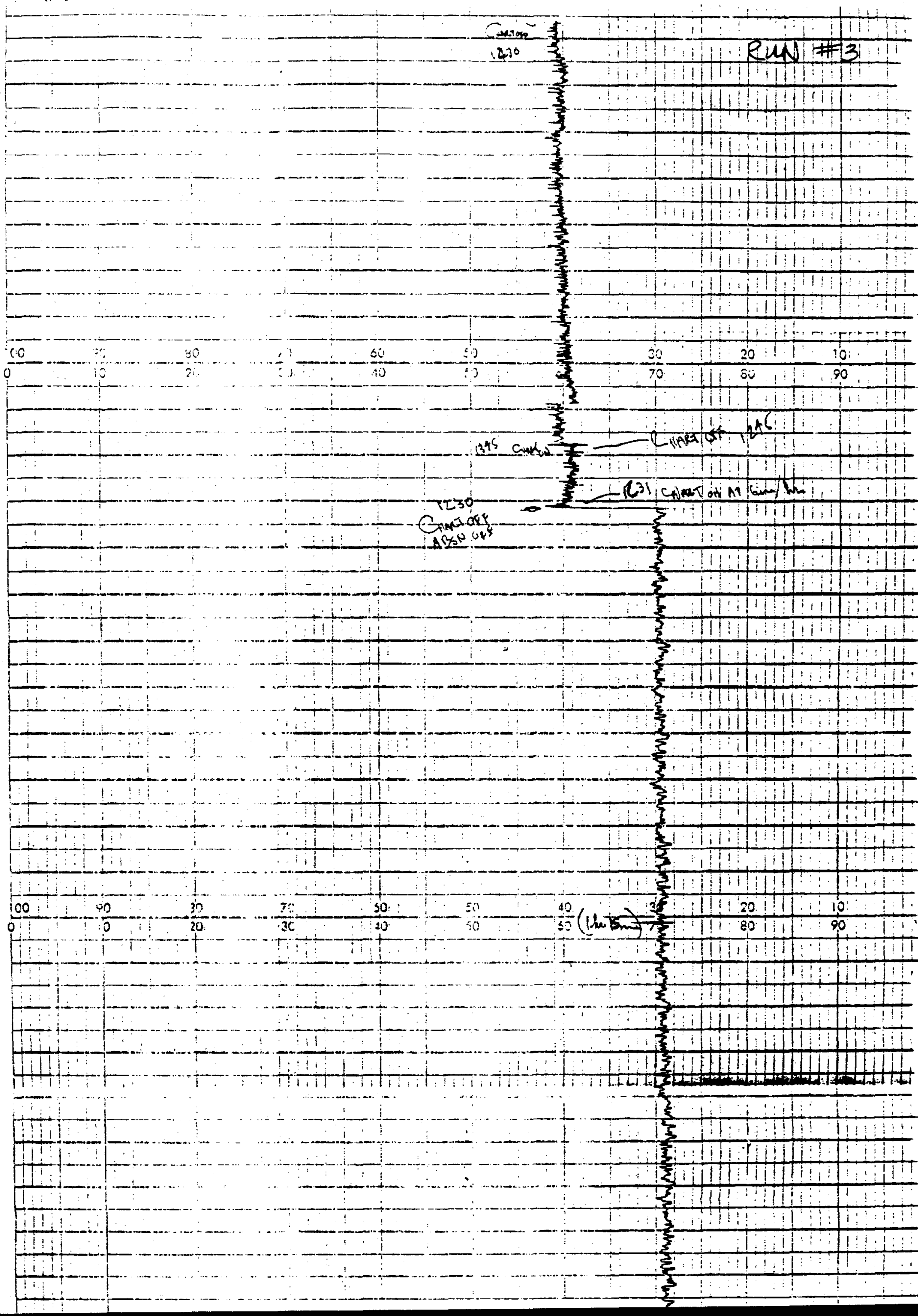

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1230
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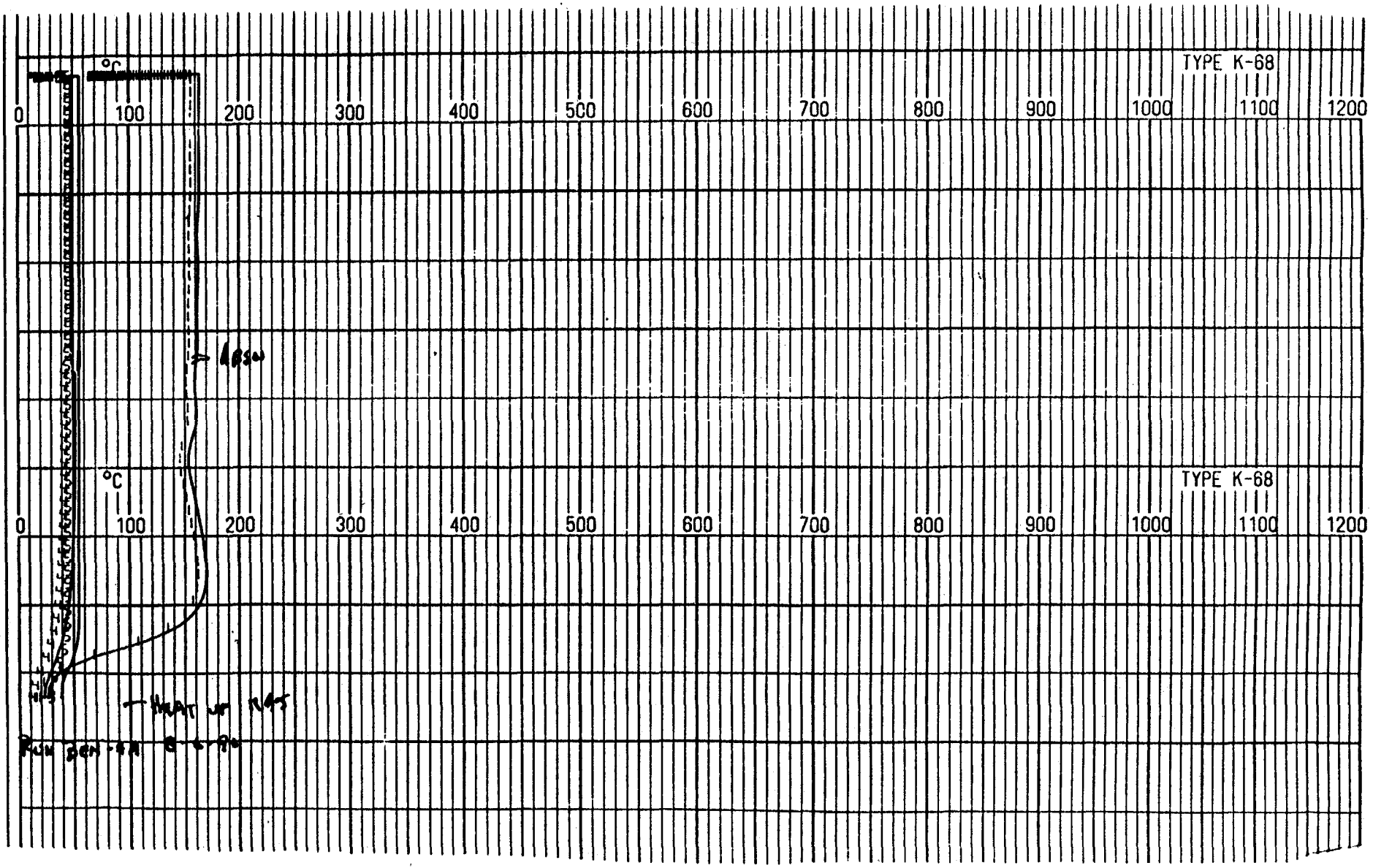
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600
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800
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1000
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1200
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400
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600
700
800
900
1000
1100
1200

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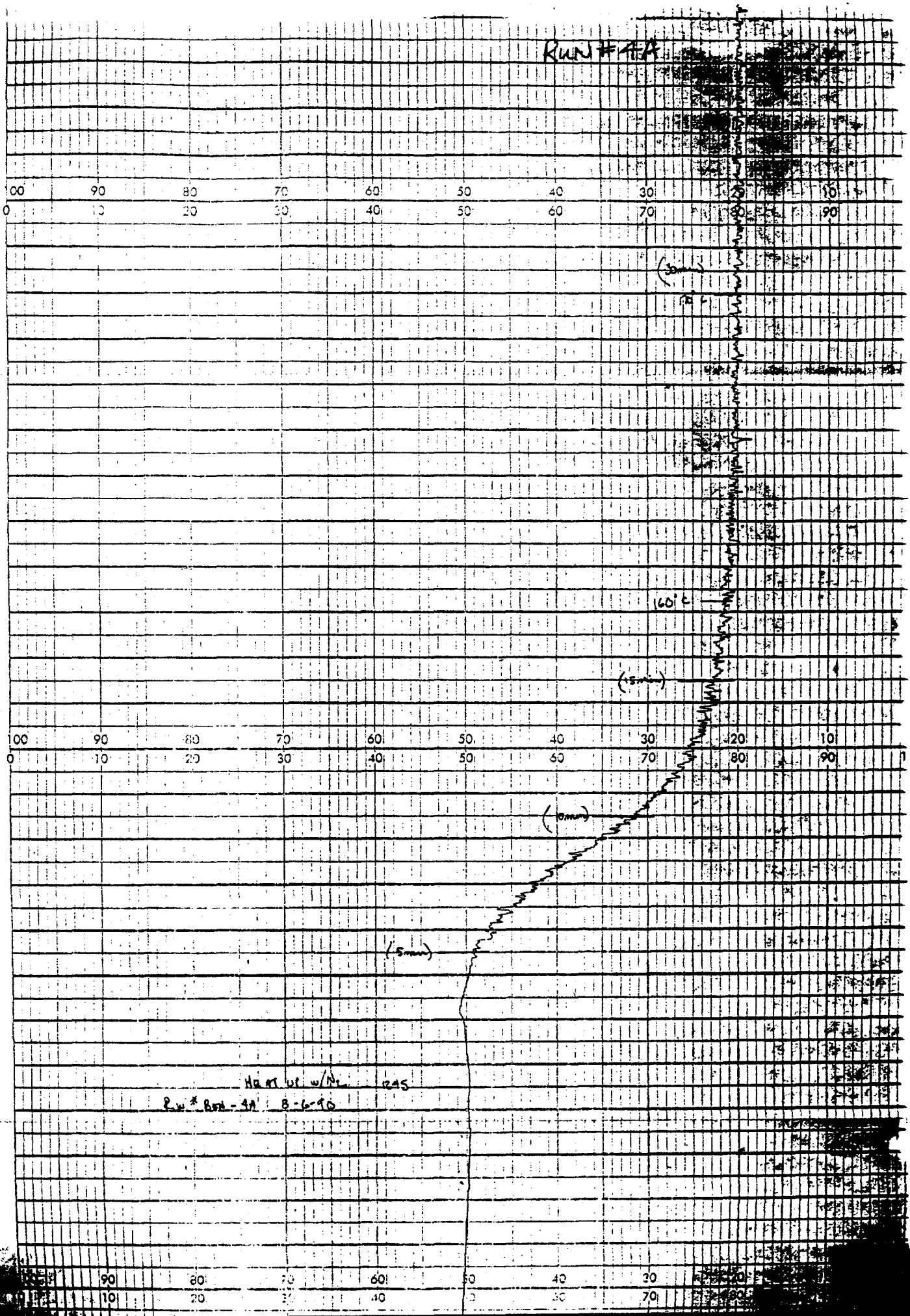

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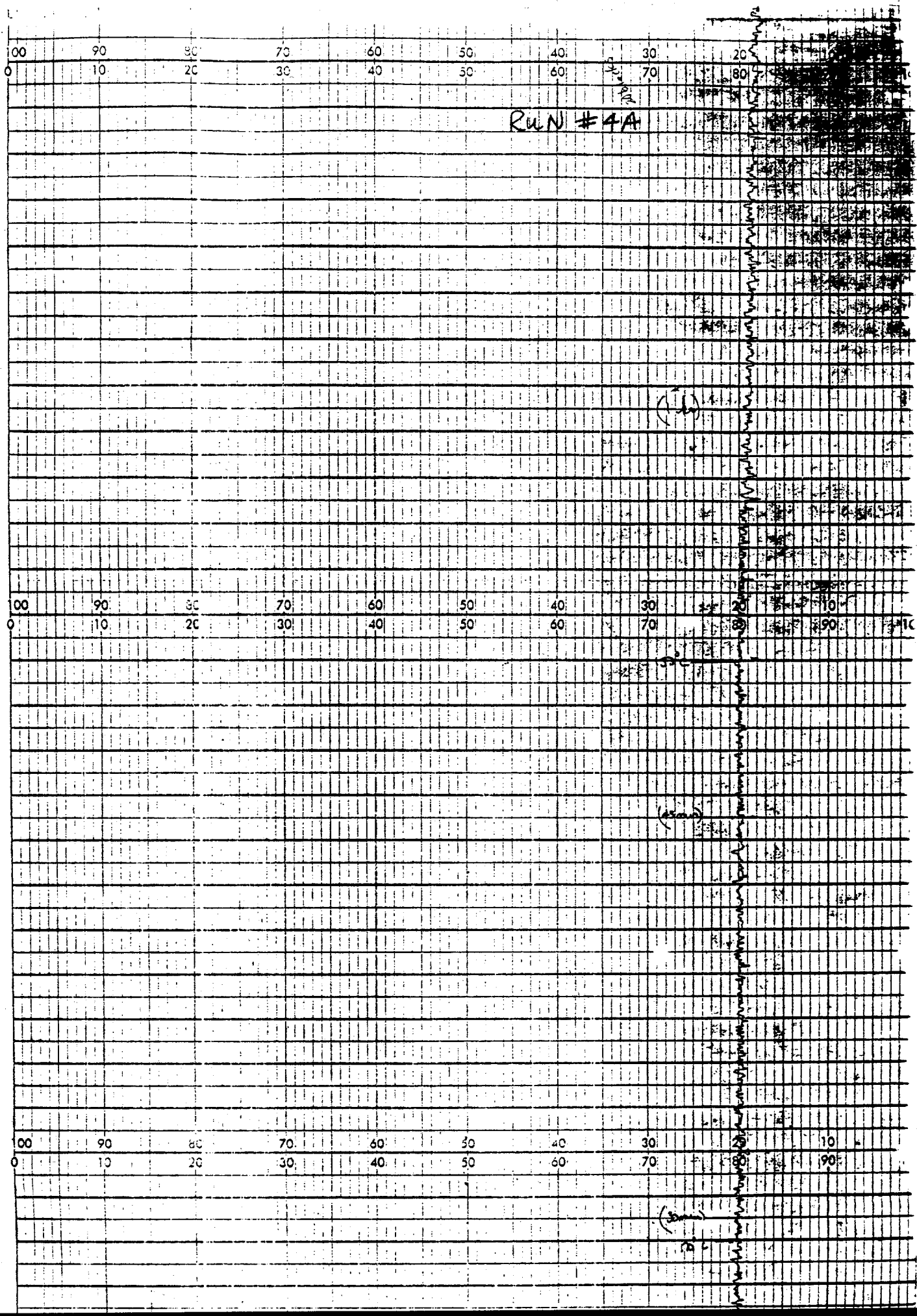
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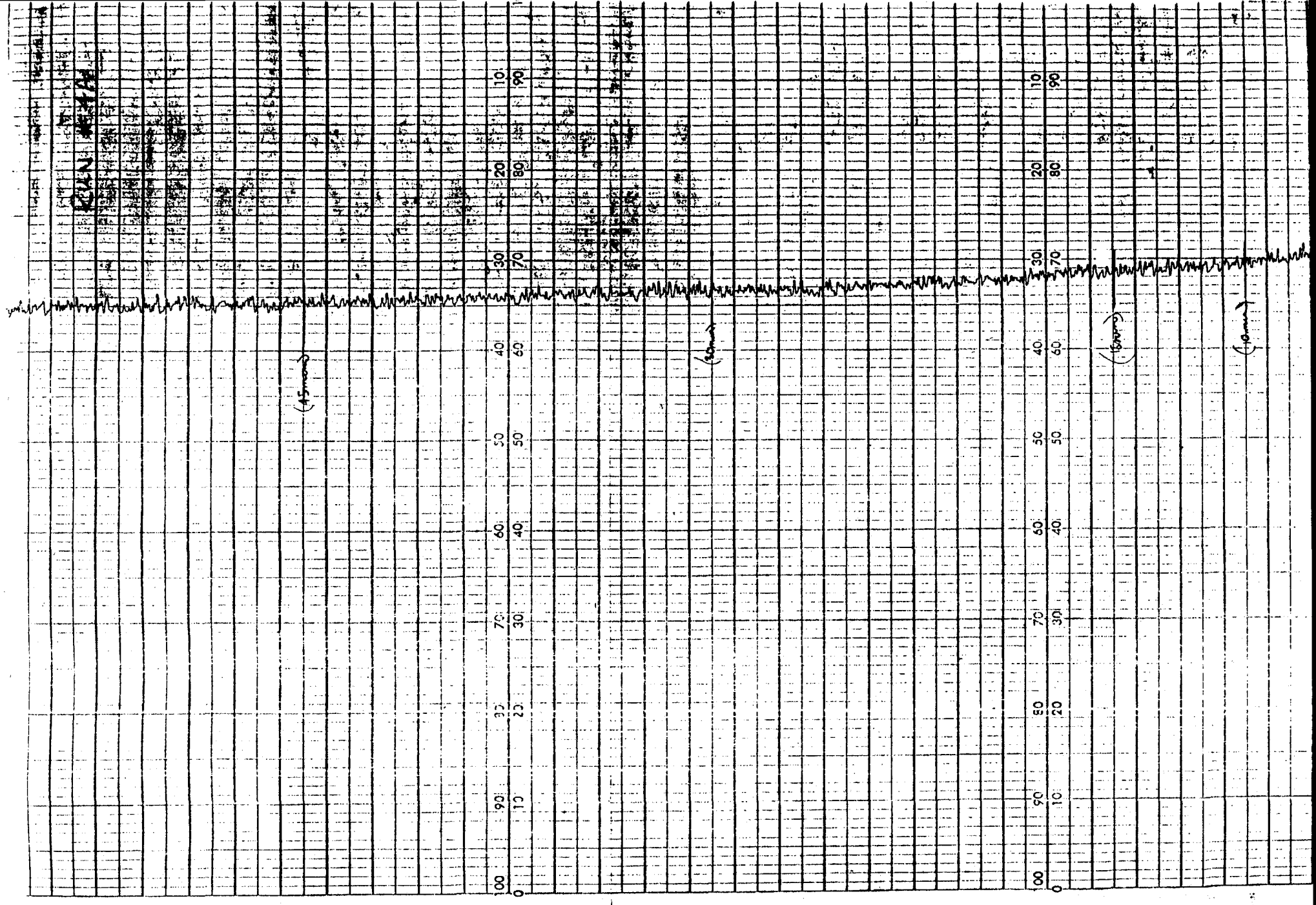
MOLYTEK INC. 3419 SMALLMAN ST. PITTSBURG, PA. 15222 (412) 761 9030

CHART NO. 414103



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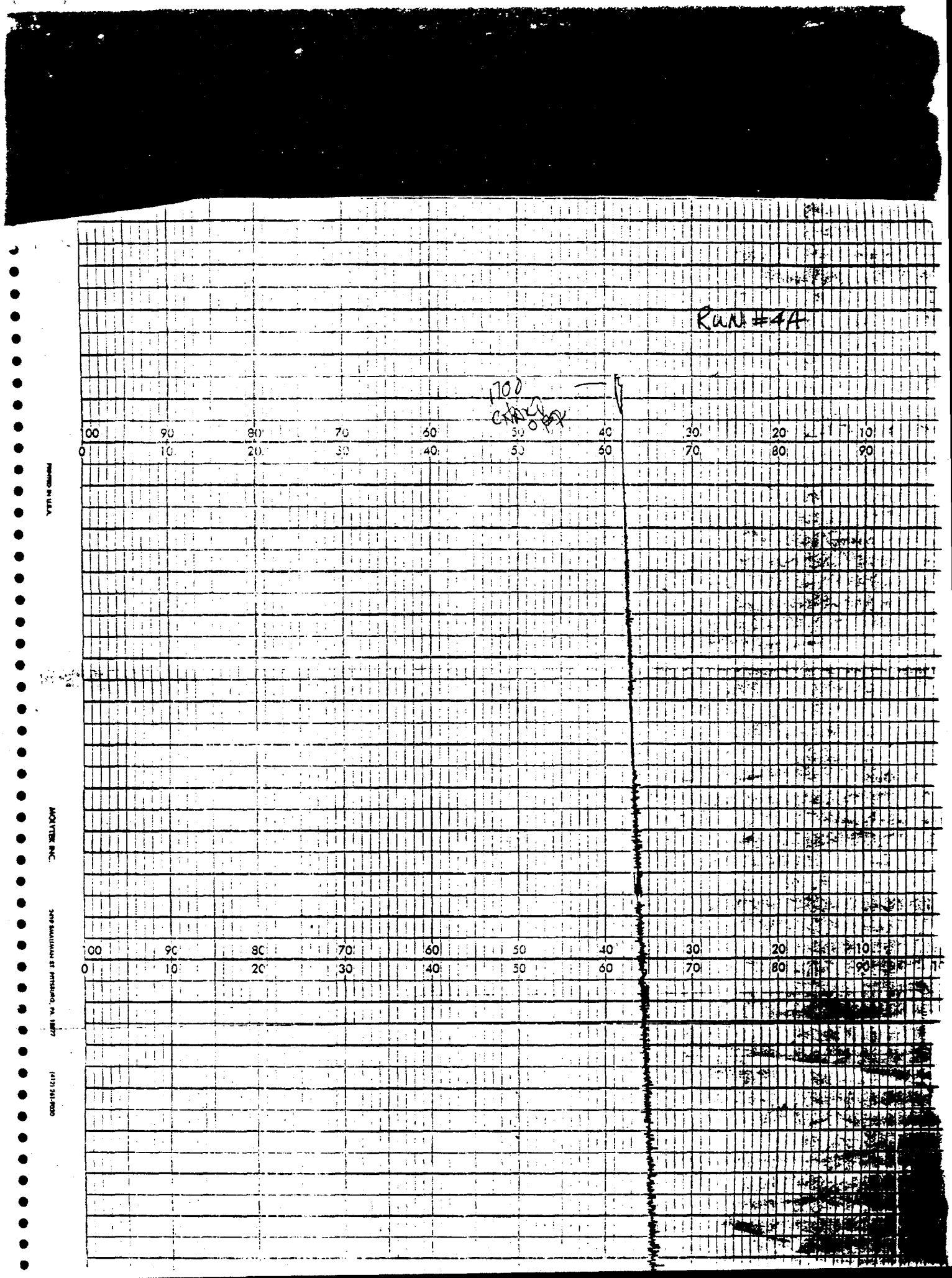
MOLYTEK INC. 3419 SMALLMAN ST. PITTSBURG, PA. 15222 (412) 261-9030

MOLYTEK INC. 2419 SMALLMAN ST. PITTSBURG, PA. 15222 (412) 761-9030 CHART NO. 414103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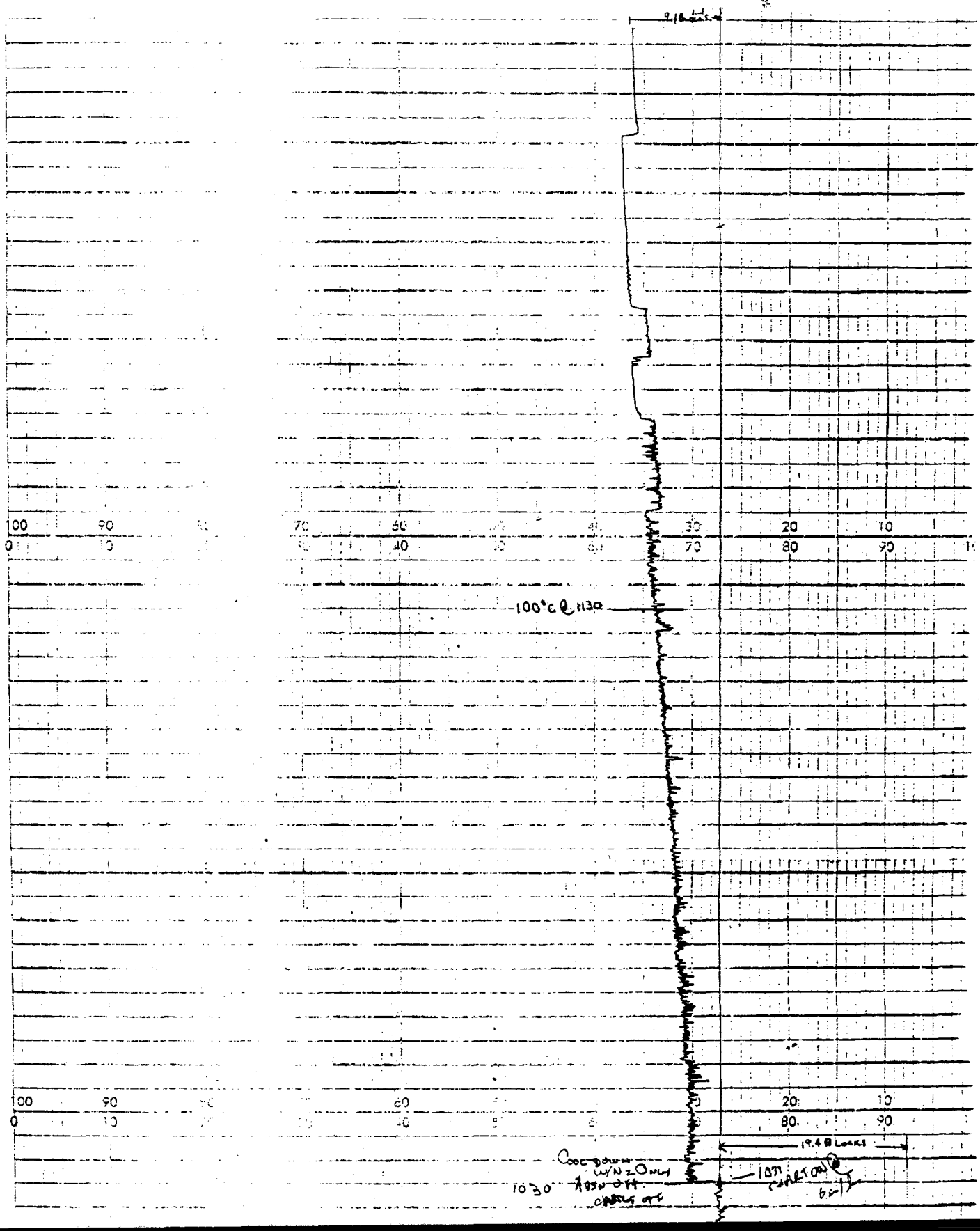
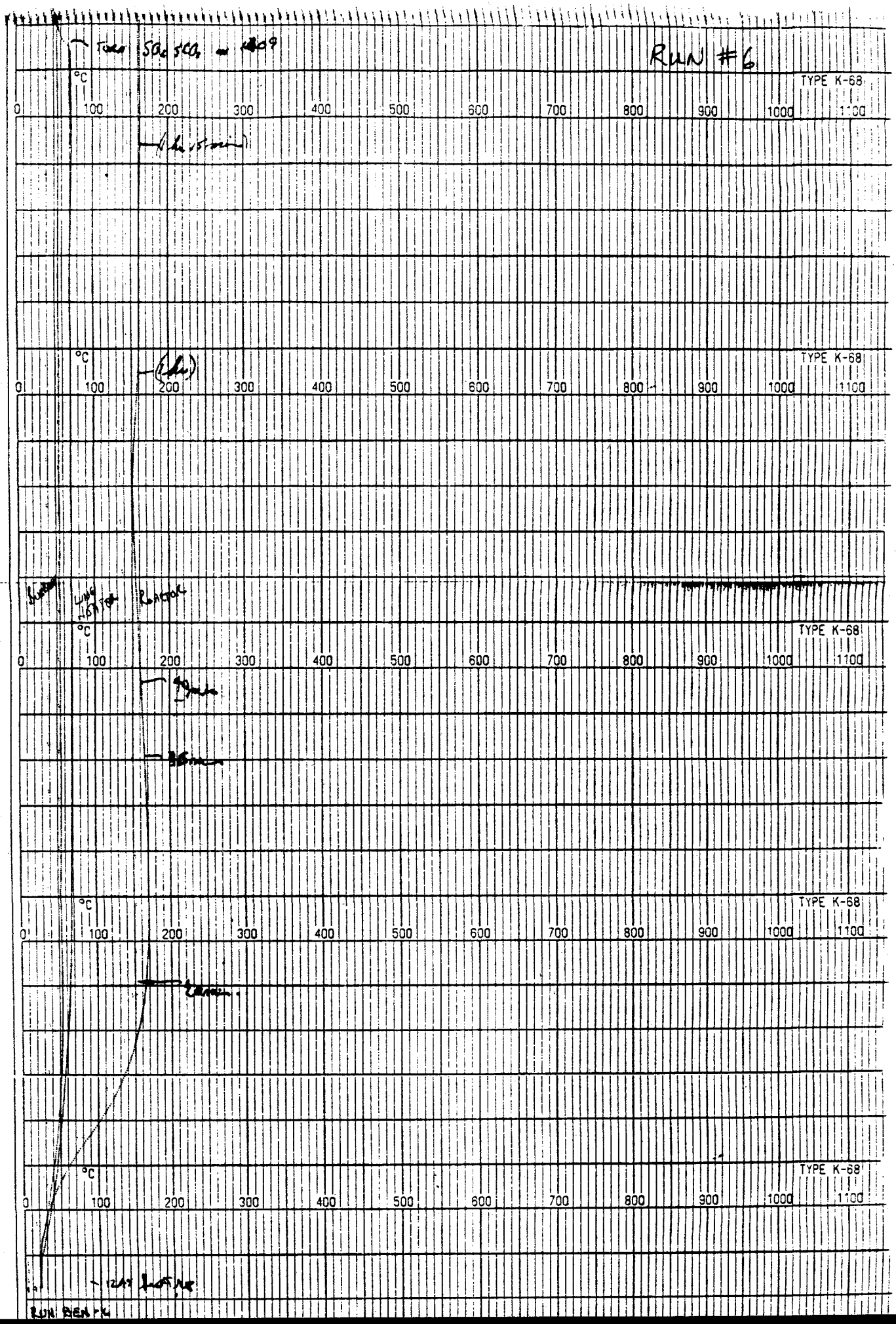
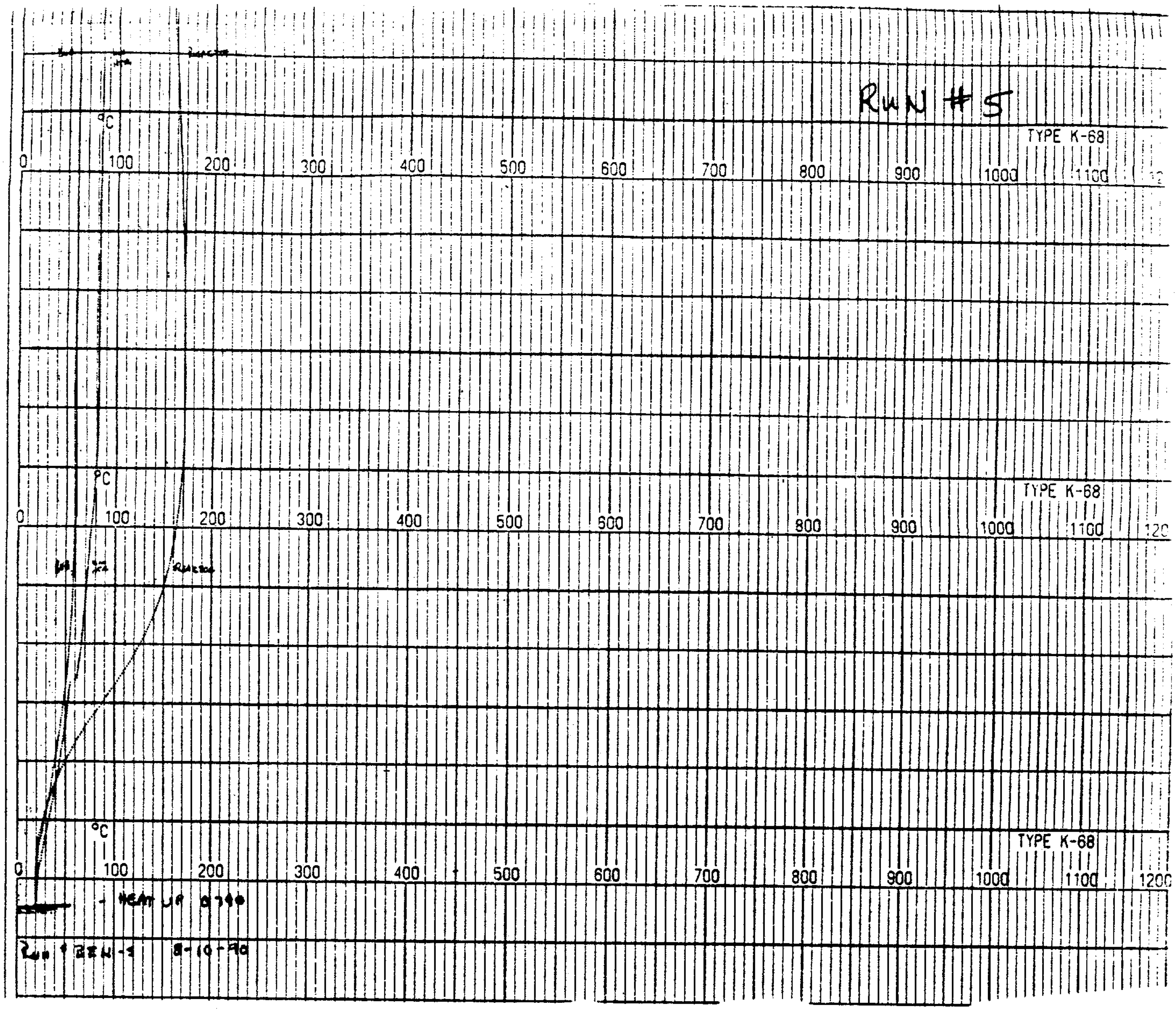
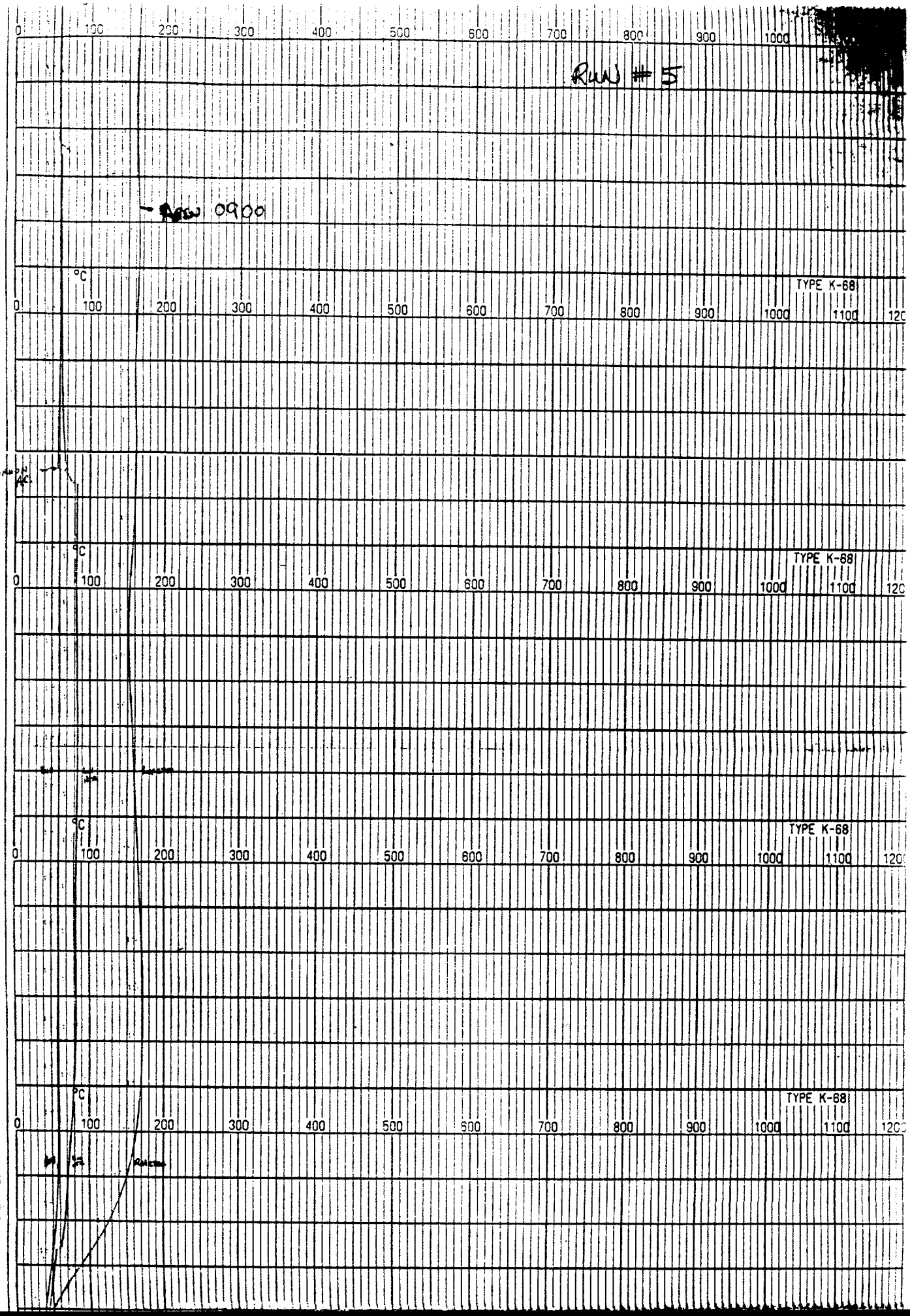
RUN #5




RUN #6
TYPE K-68
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
NO. 646042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.


RUN #5
TYPE K-68
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
HEAT UP


RUN # 51
0900
°C
TYPE K-68
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
NO. 64042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

RUN #5

°C

TYPE K-68

0 100 200 300 400 500 600 700 800 900 1000 1100 120

°C

TYPE K-68

0 100 200 300 400 500 600 700 800 900 1000 1100 120

°C

TYPE K-68

0 100 200 300 400 500 600 700 800 900 1000 1100 120

°C

TYPE K-68

0 100 200 300 400 500 600 700 800 900 1000 1100 120

°C

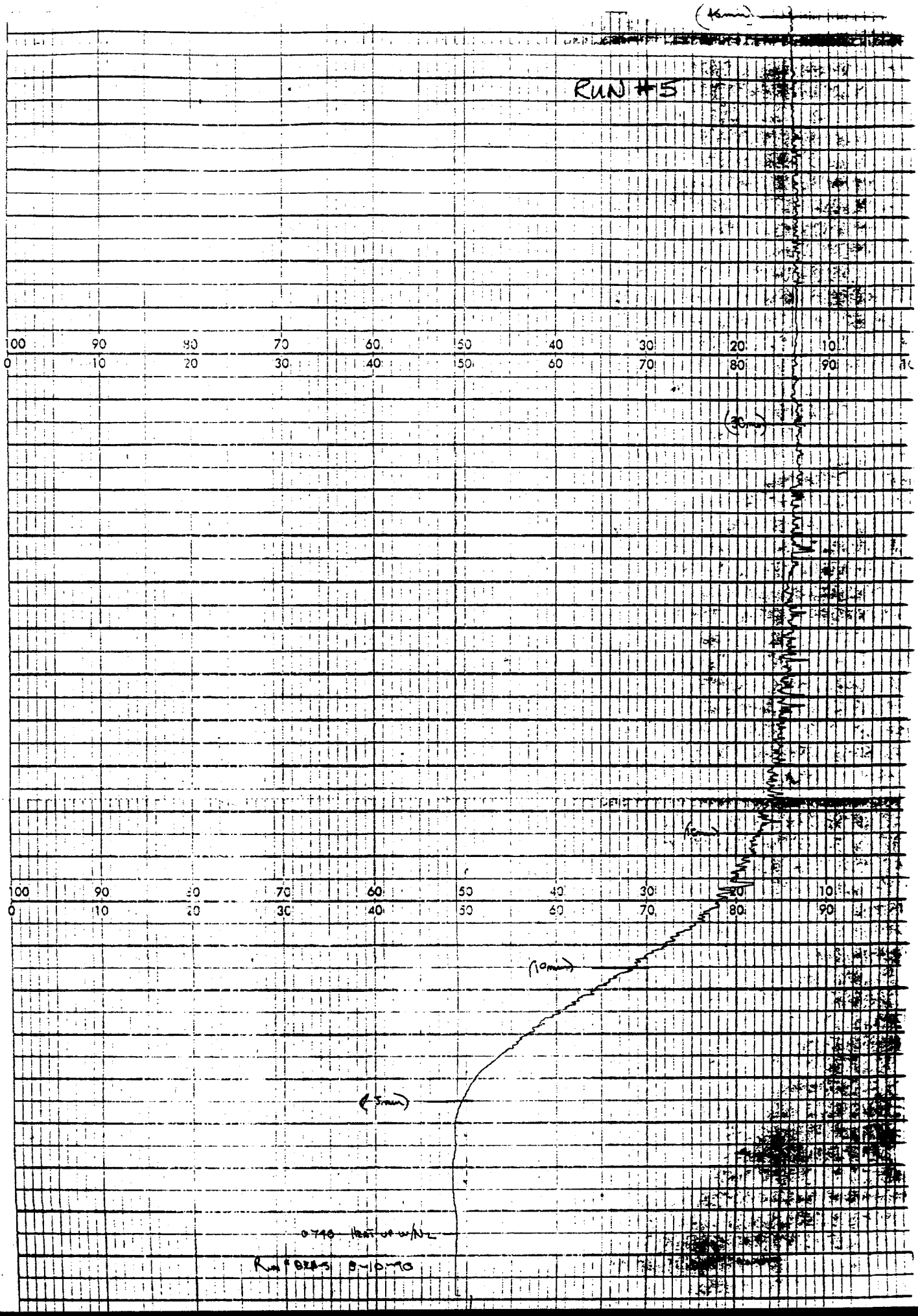
TYPE K-68

0 100 200 300 400 500 600 700 800 900 1000

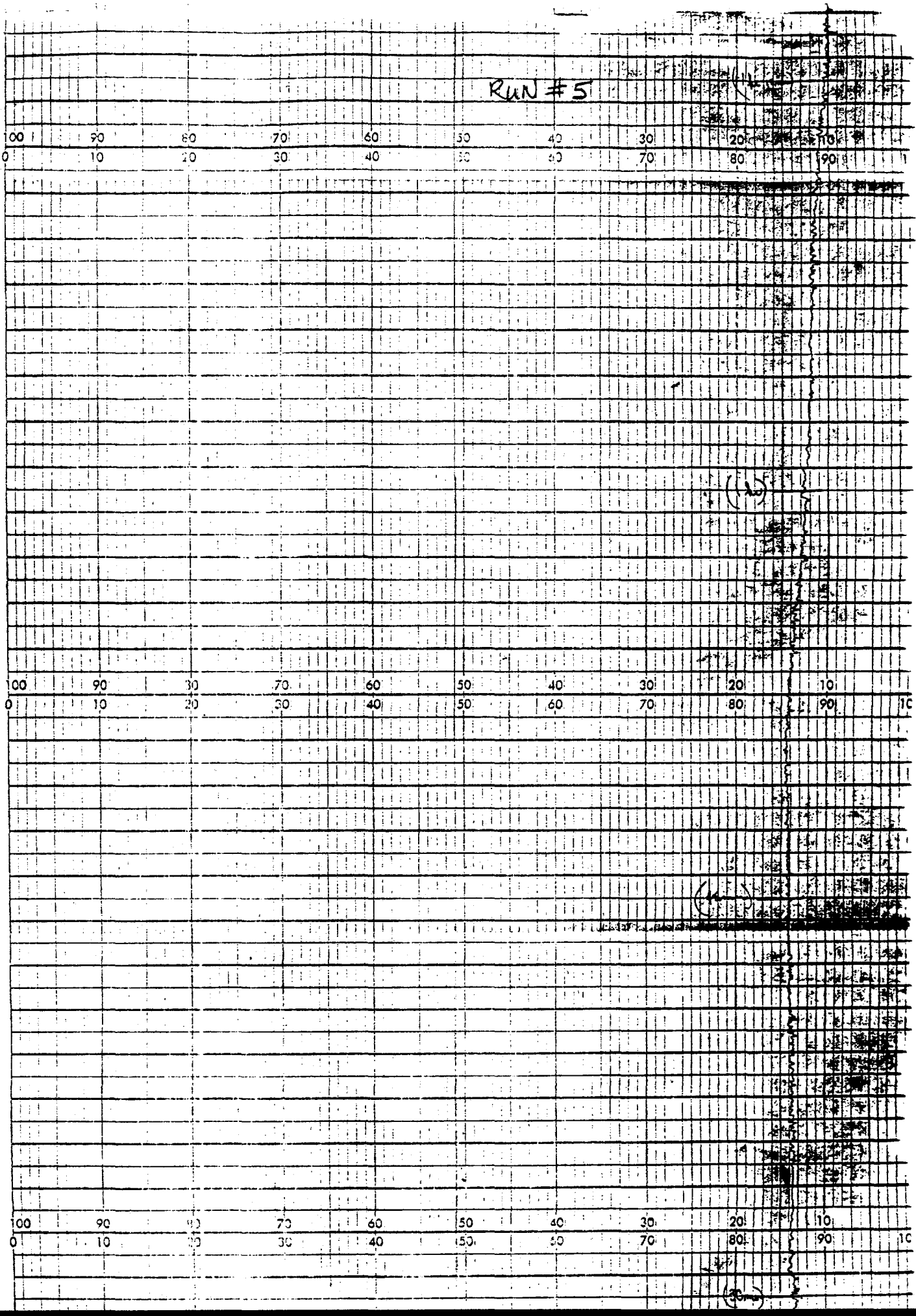
NO. 645042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

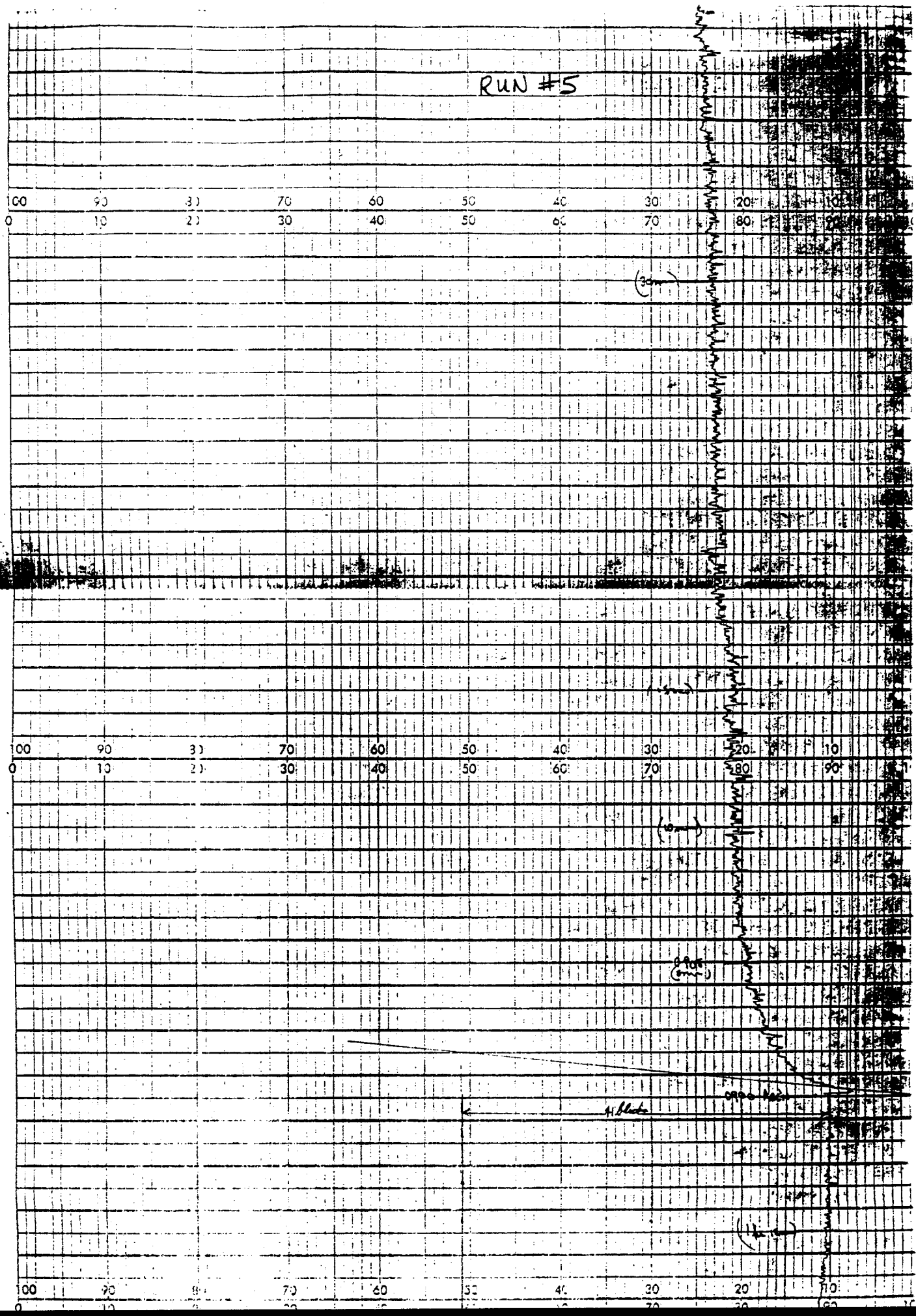
NO. 645042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

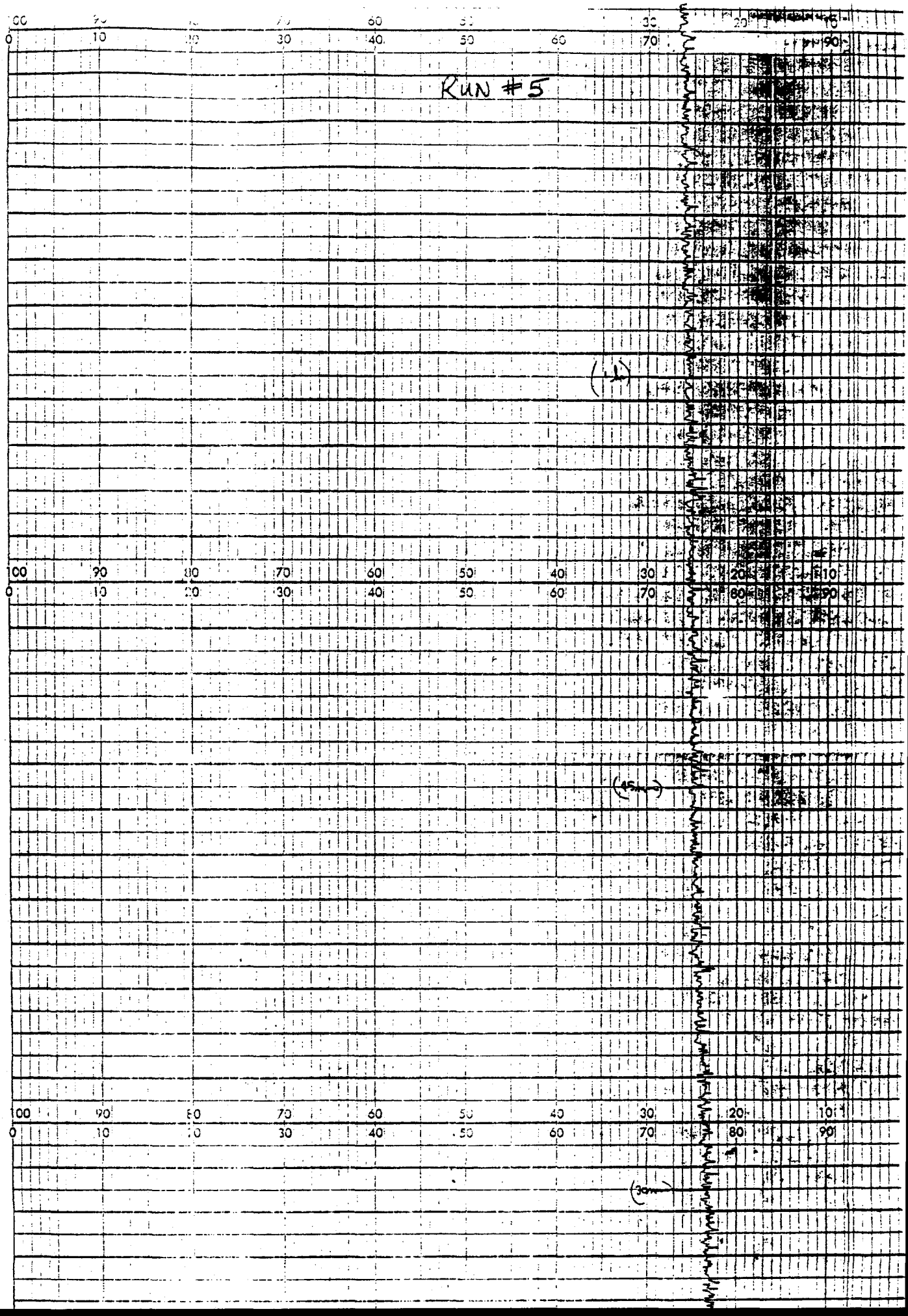
NO. 645042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

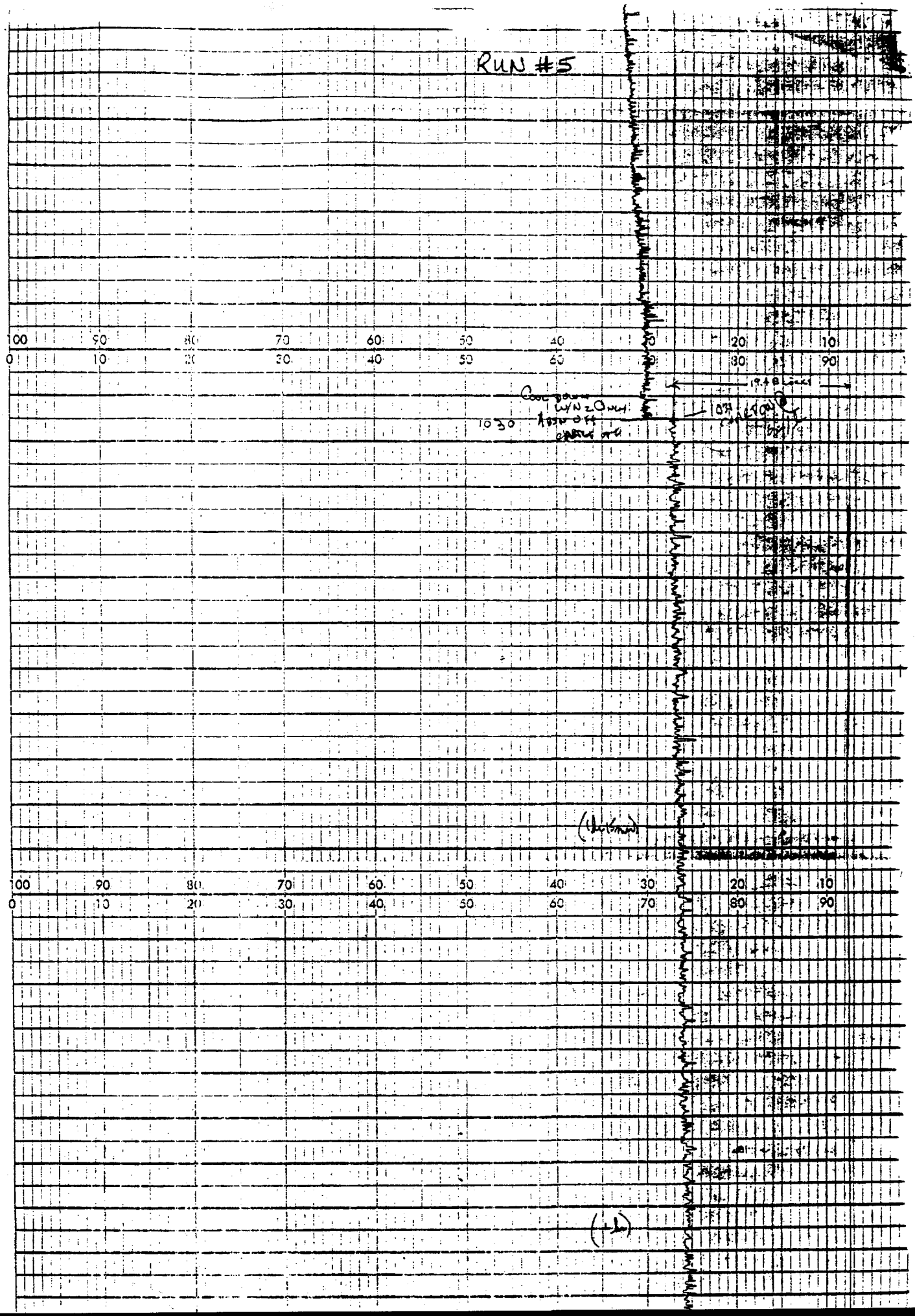

RUN #5
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90

RUN #5

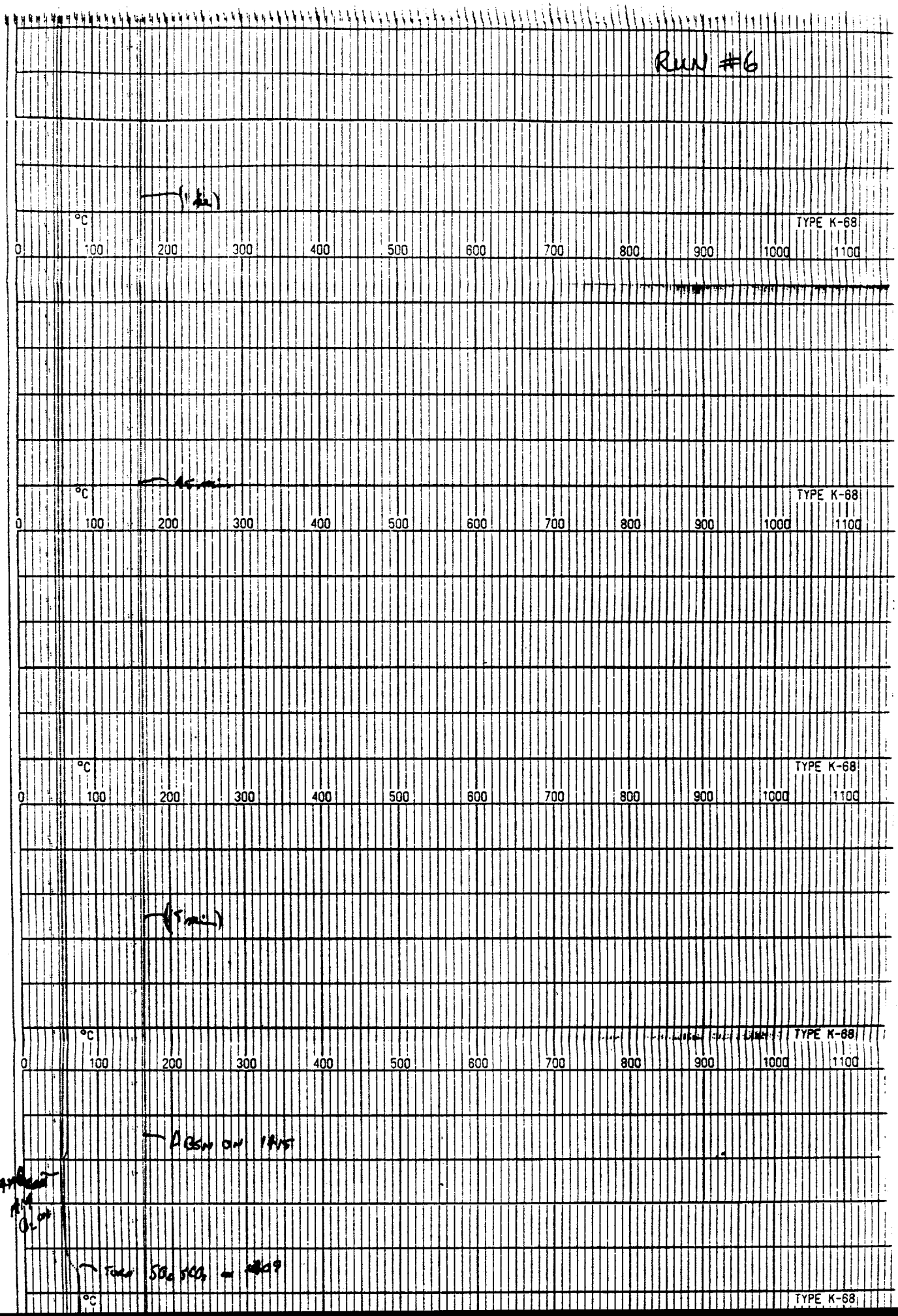





RUN #5
(1)
(45m)
(30m)



RUN #6


°C
TYPE K-68
0
100
200
300
400
500
600
700
800
900
1000
1100
NO. 64042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

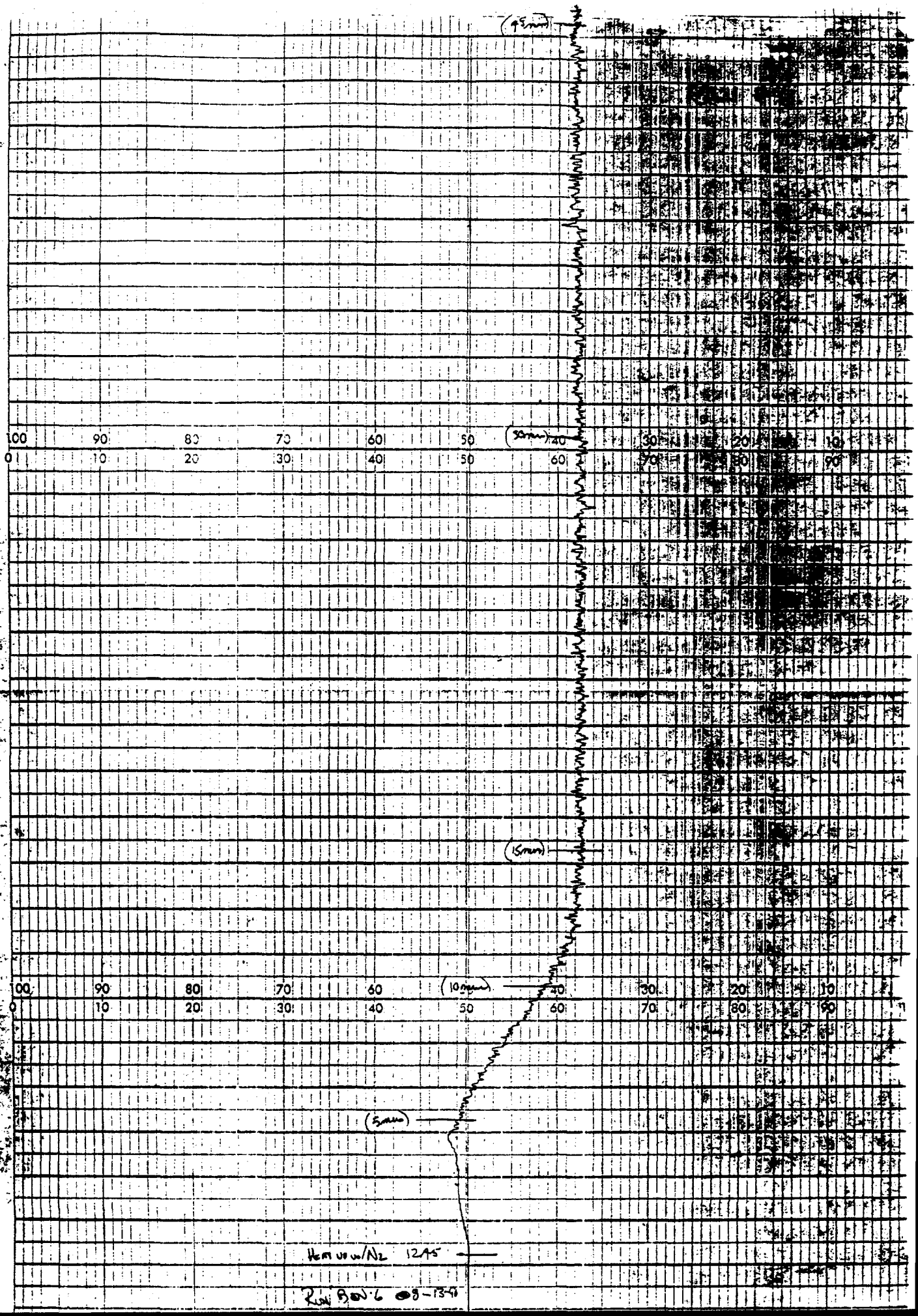
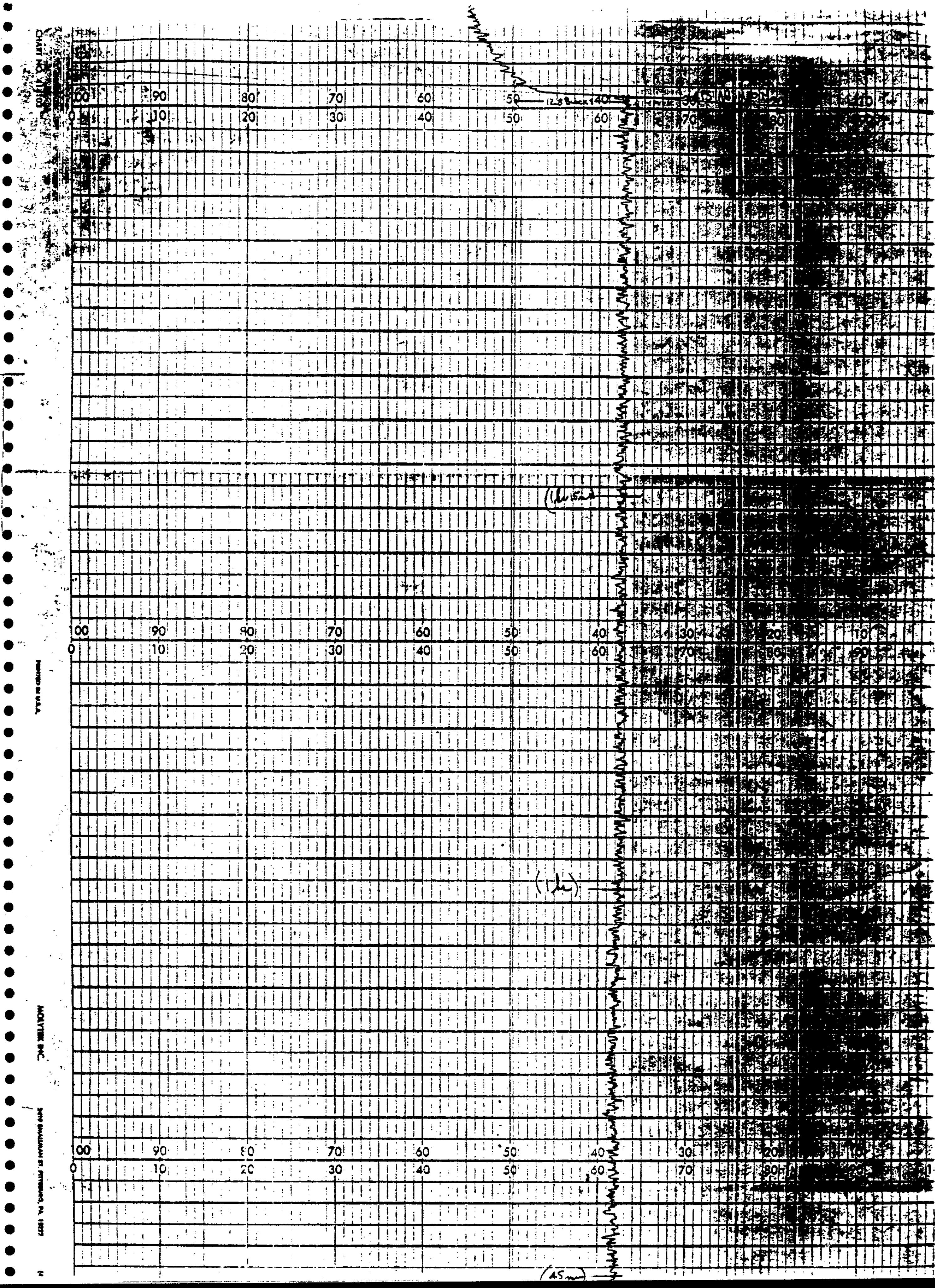
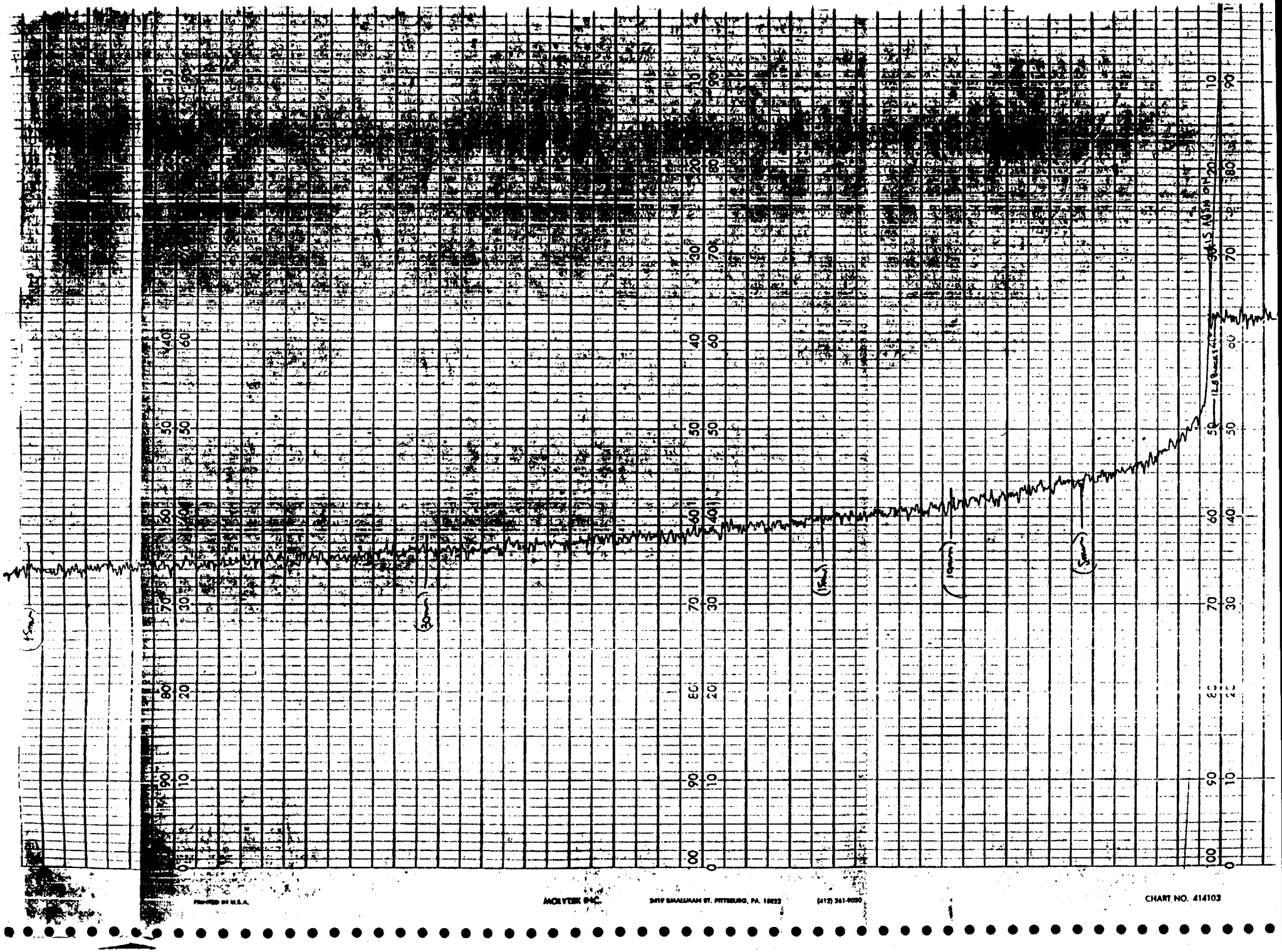


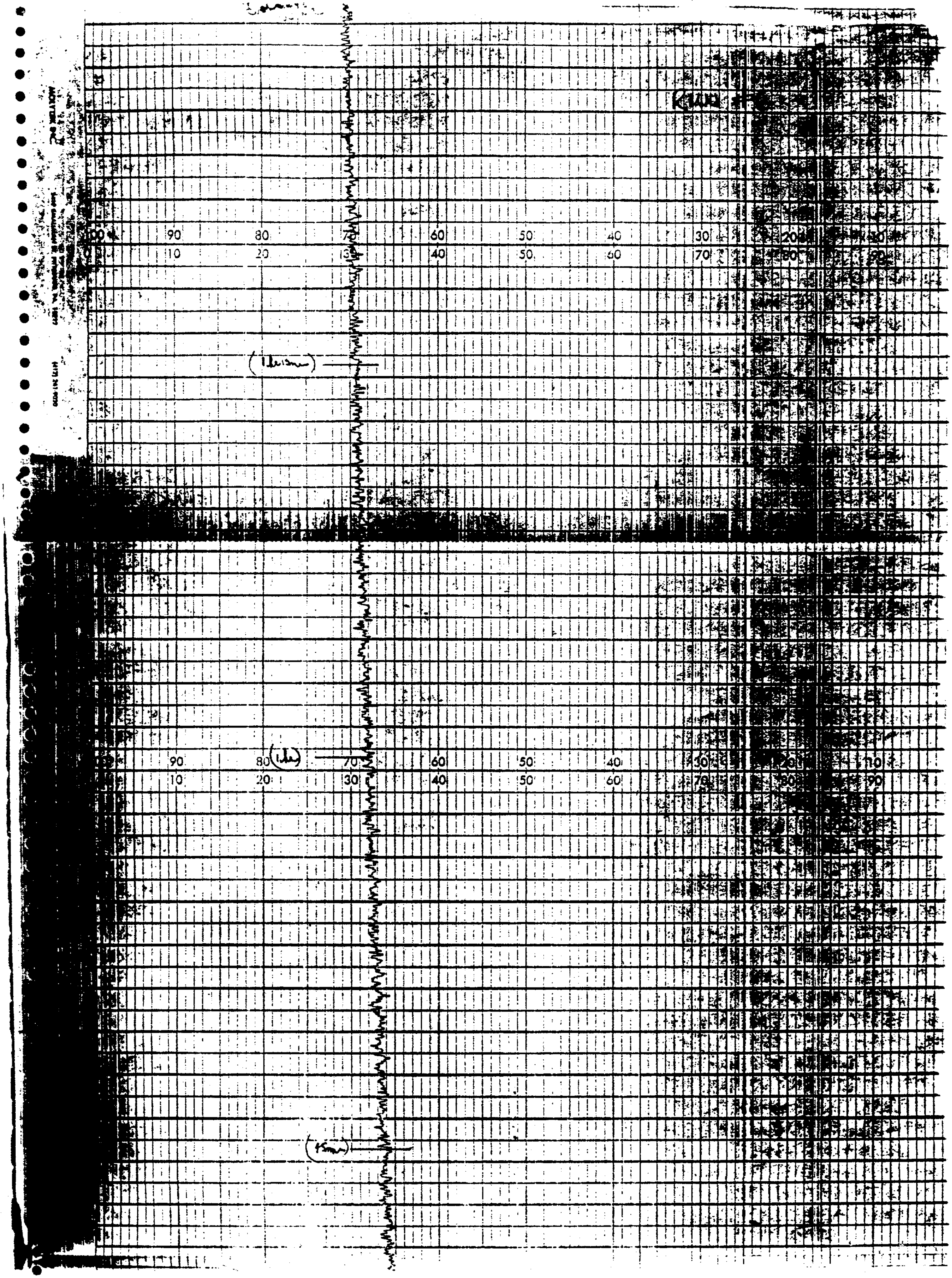
CHART NO. 414103
(45 mm)
(30 mm)
(15 mm)
(10 mm)
(5 mm)
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90
Heat up w/N2 1245
Run #6 8-13-91

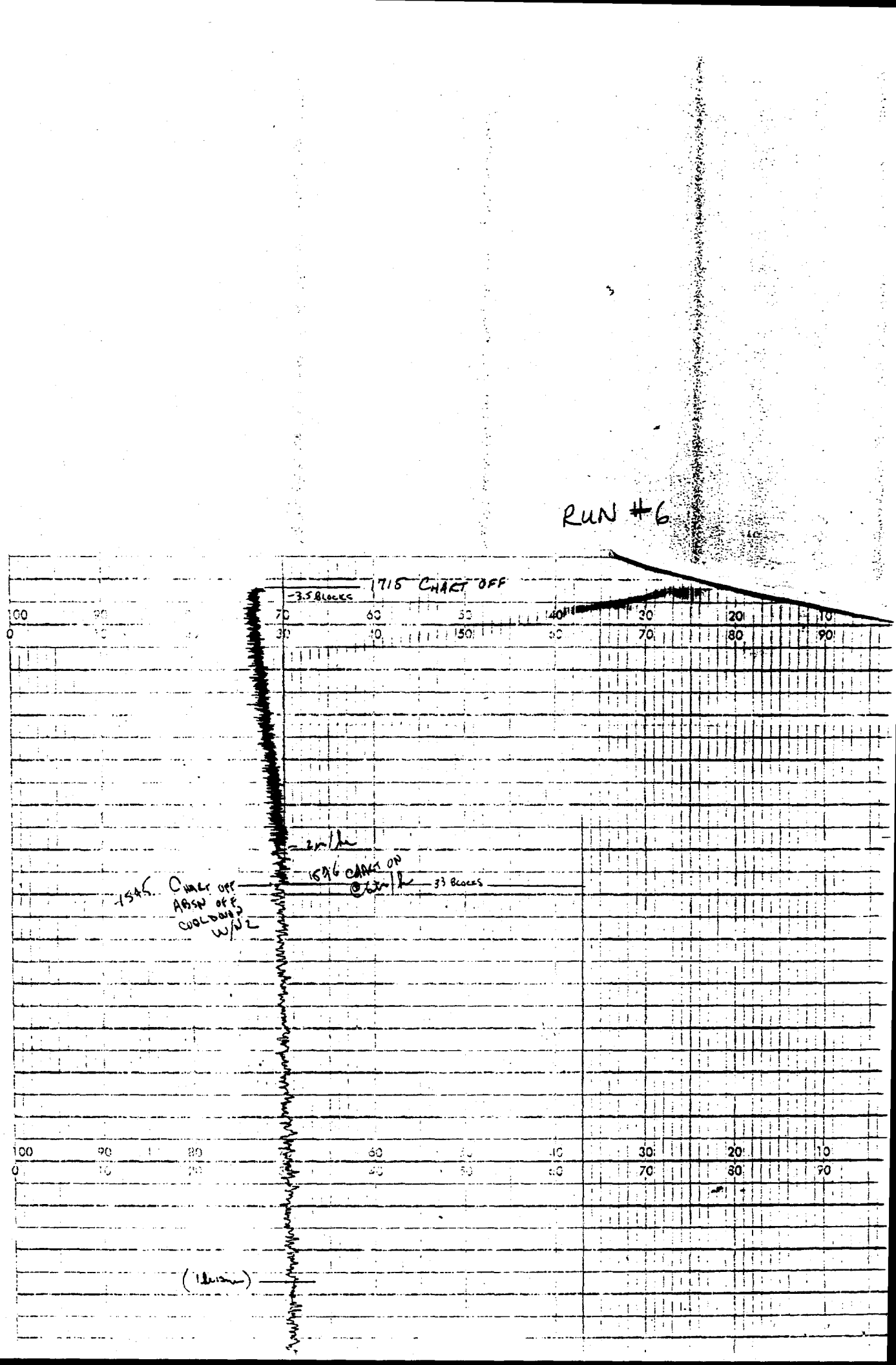
RUN #6

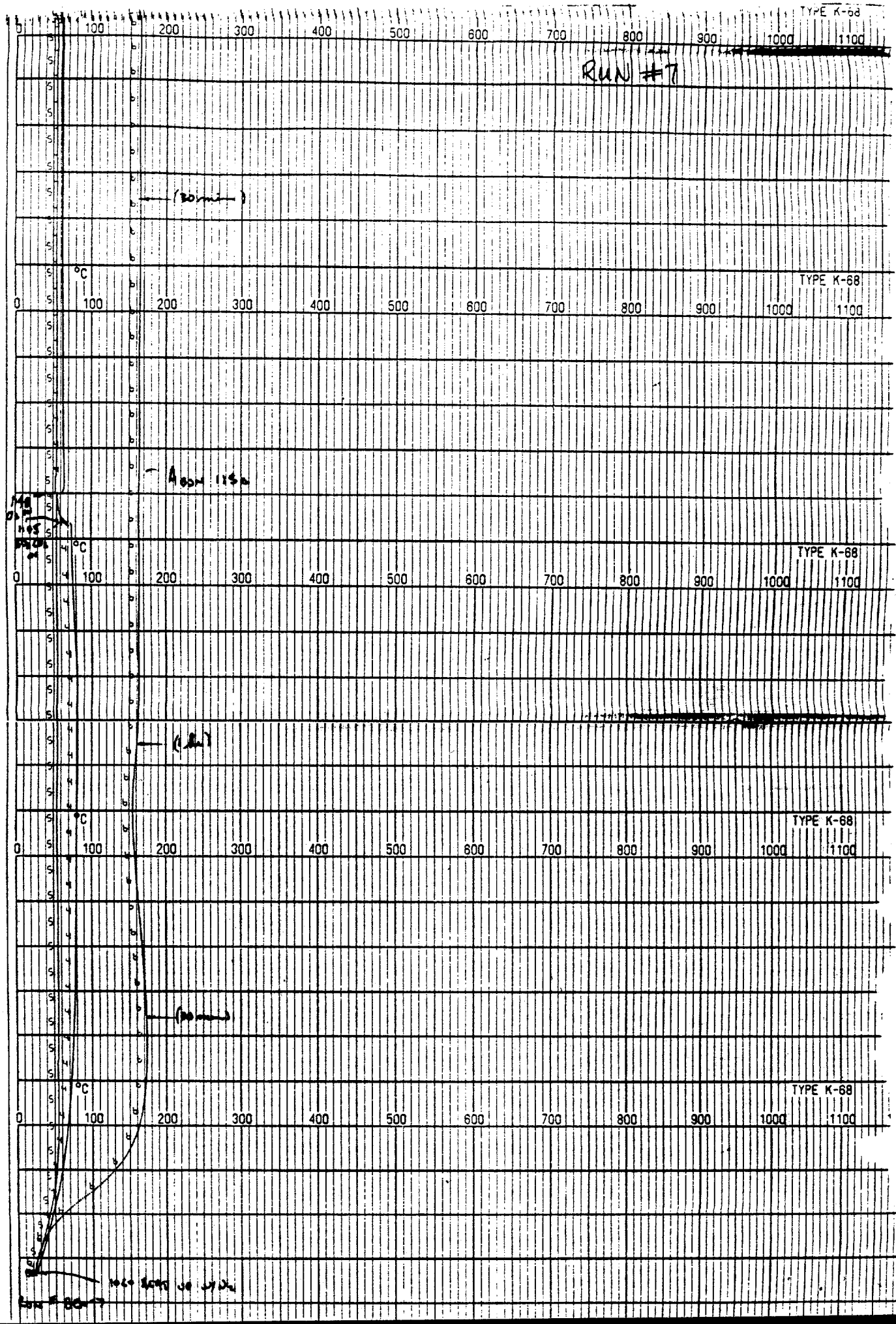
17/5 HEAT OFF
TYPE K-68
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
TYPE K-68
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
TYPE K-68
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
NO. 545042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.
NO. 545042 LEEDS & NORTHRUP CO., NORTH WALES, PA


PRINTED IN U.S.A.
MOLYTEK INC.


MOLYTEK INC.
3410 SMALLMAN ST. PITTSBURG, PA. 15222
(412) 341-9090
CHART NO. 414103


90 80 70 60 50 40 30 20 10
10 20 30 40 50 60 70 80 90
90 80 70 60 50 40 30 20 10
10 20 30 40 50 60 70 80 90


RUN #6
1715 CHART OFF
-3.5 Blocks
1545. CHART OFF
ABSN OFF
COOLDOWN
W/N2
1546 CHART ON
33 Blocks


TYPE K-68
RUN #7
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
NO. 545042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

RUN #7

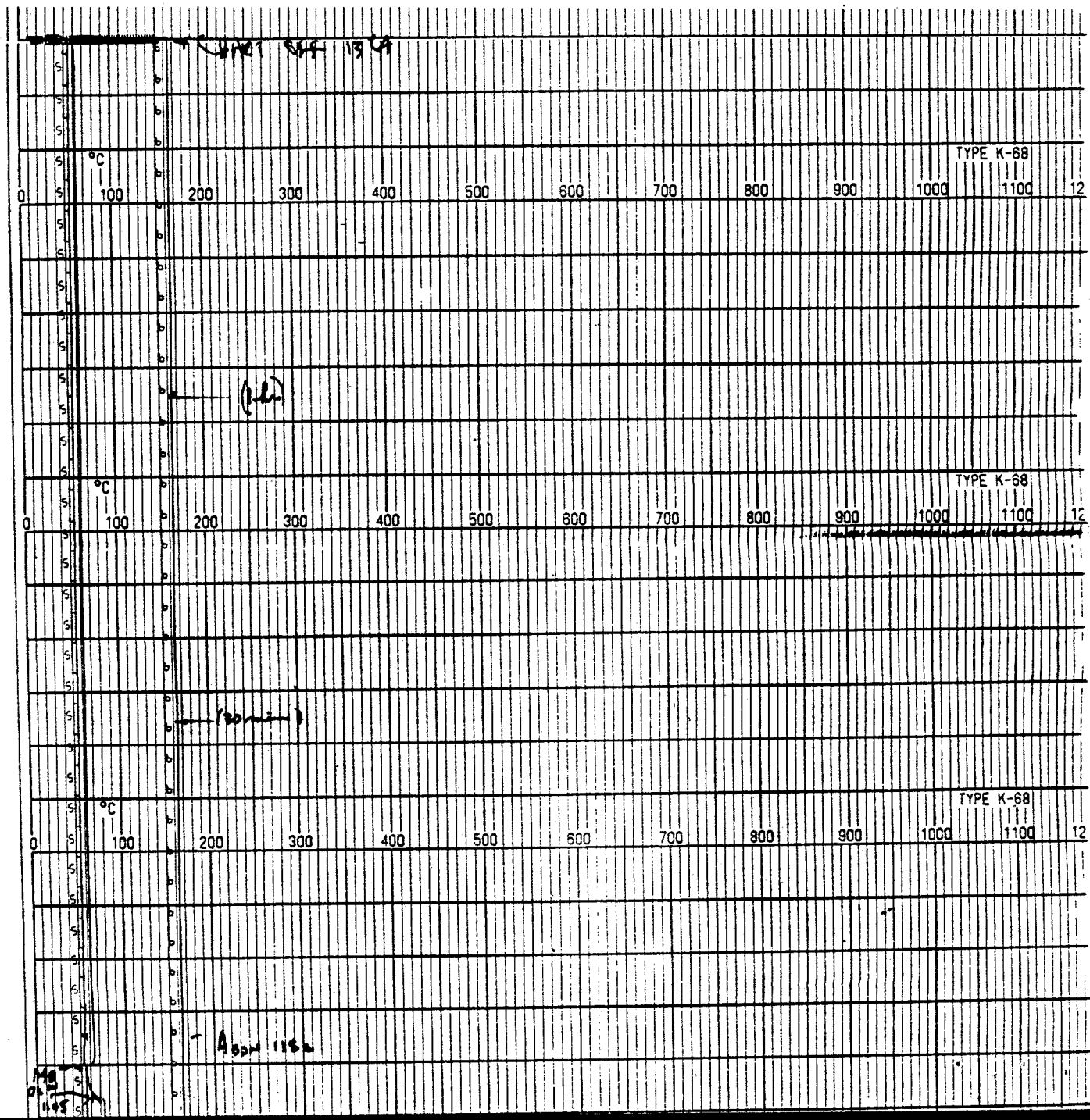
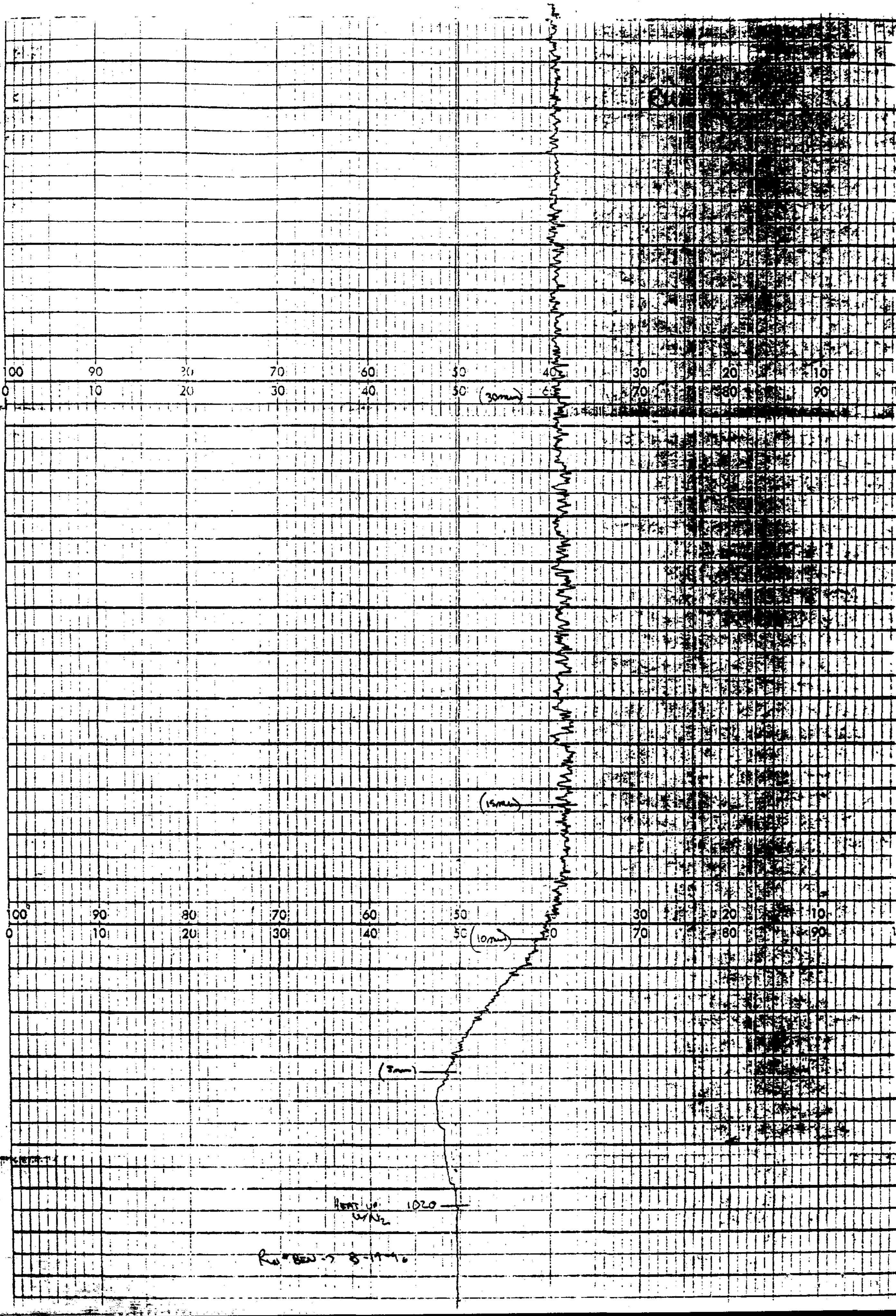
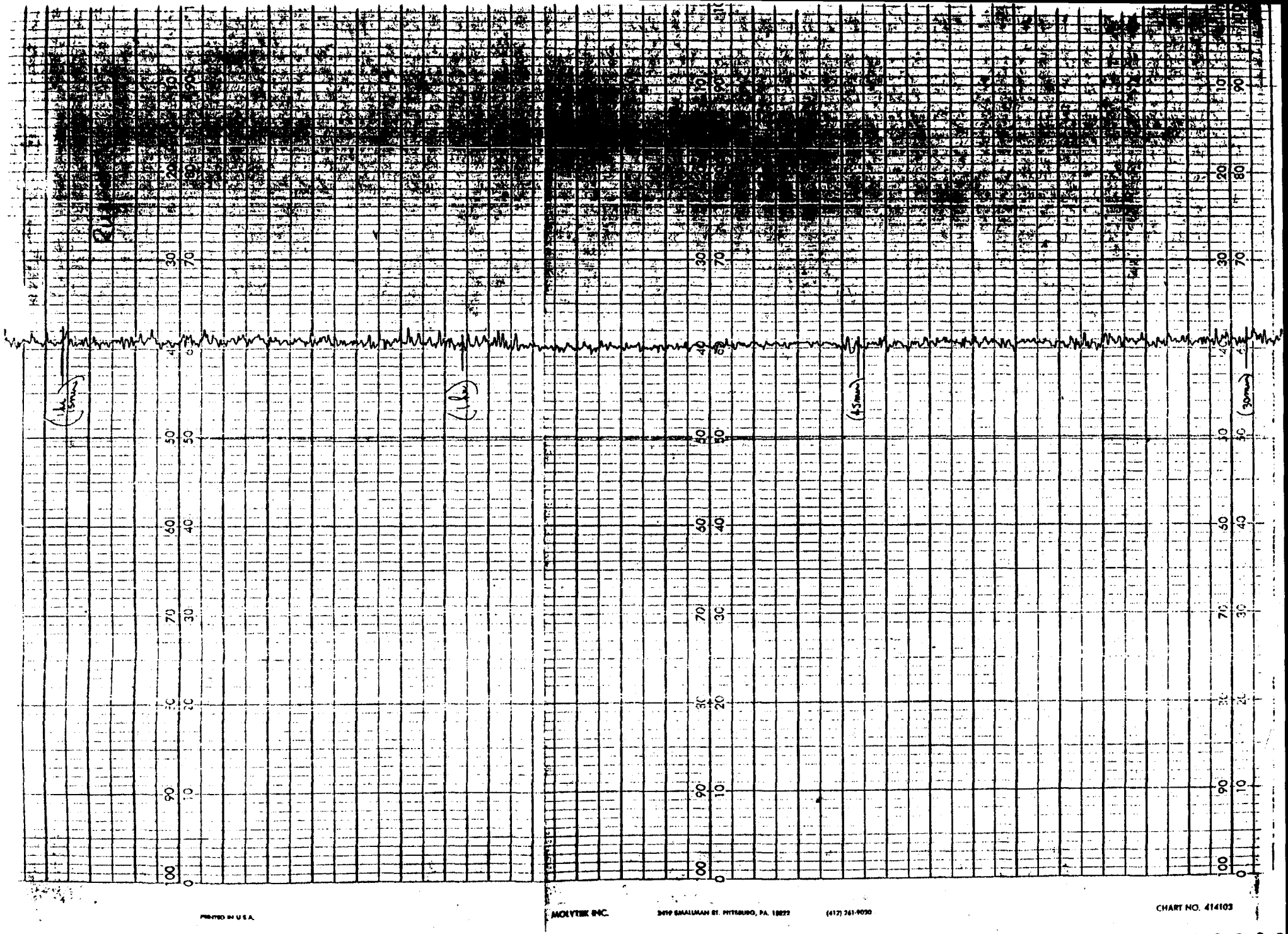


CHART NO. 414103



PRINTED IN U.S.A.



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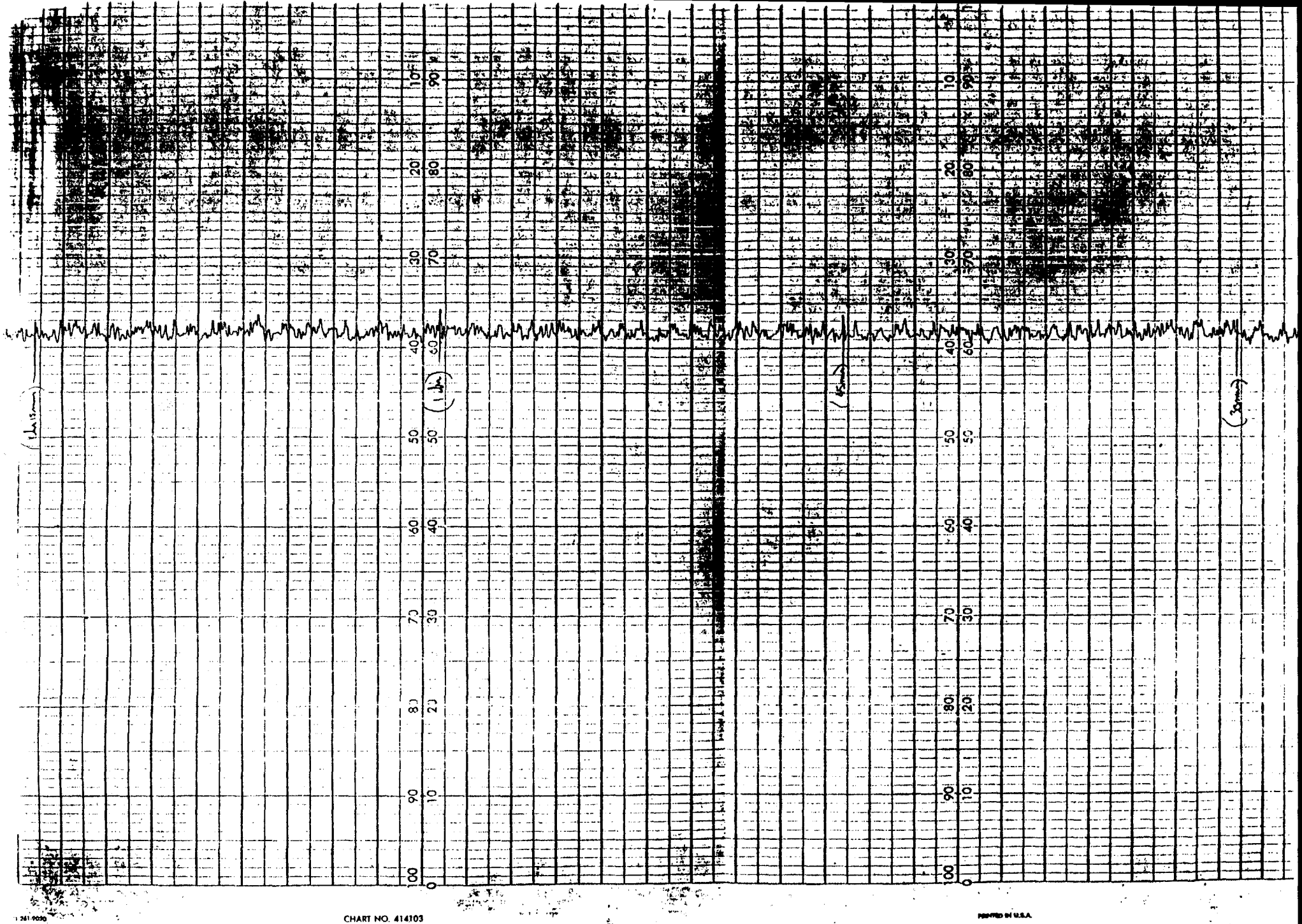
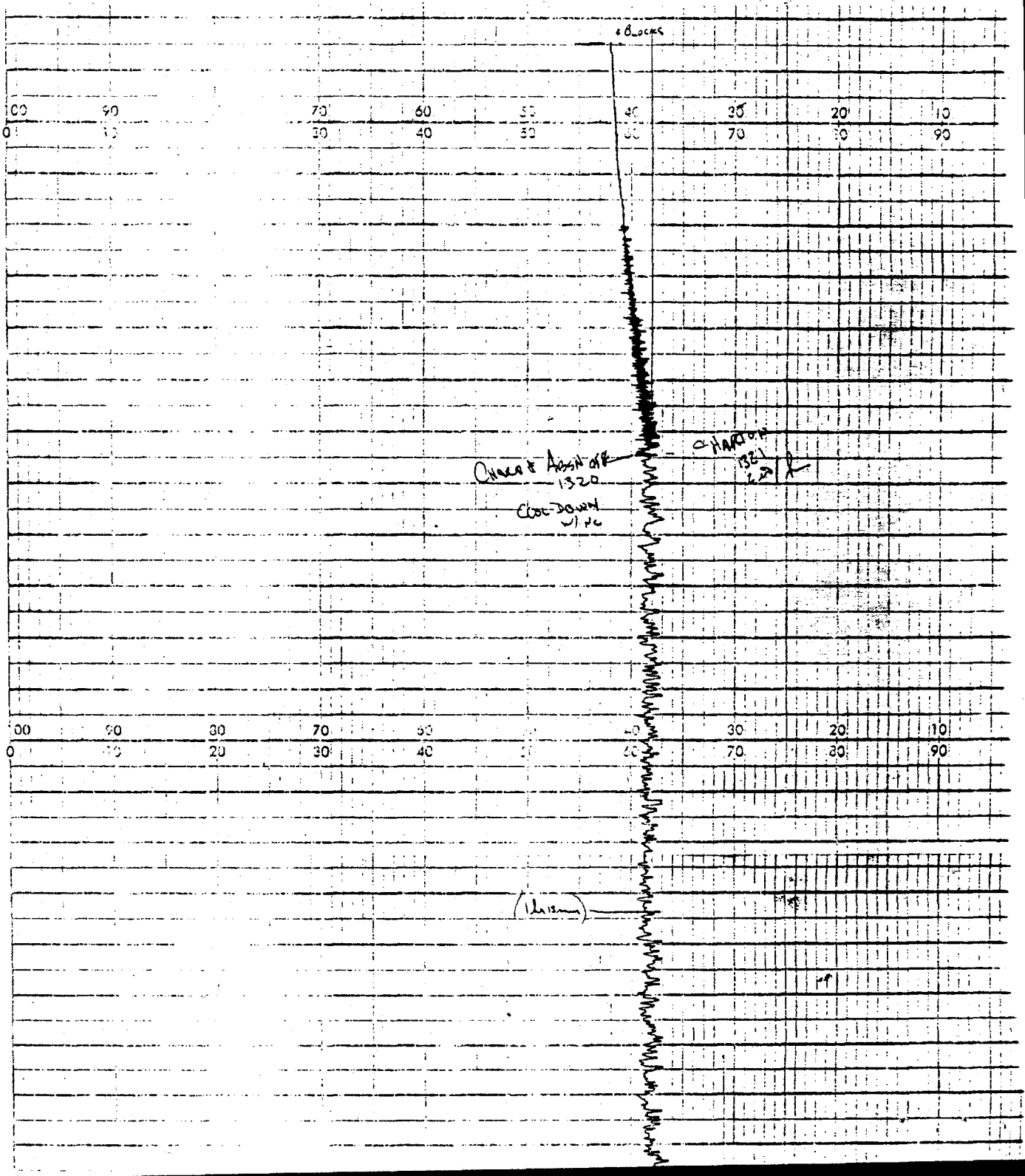
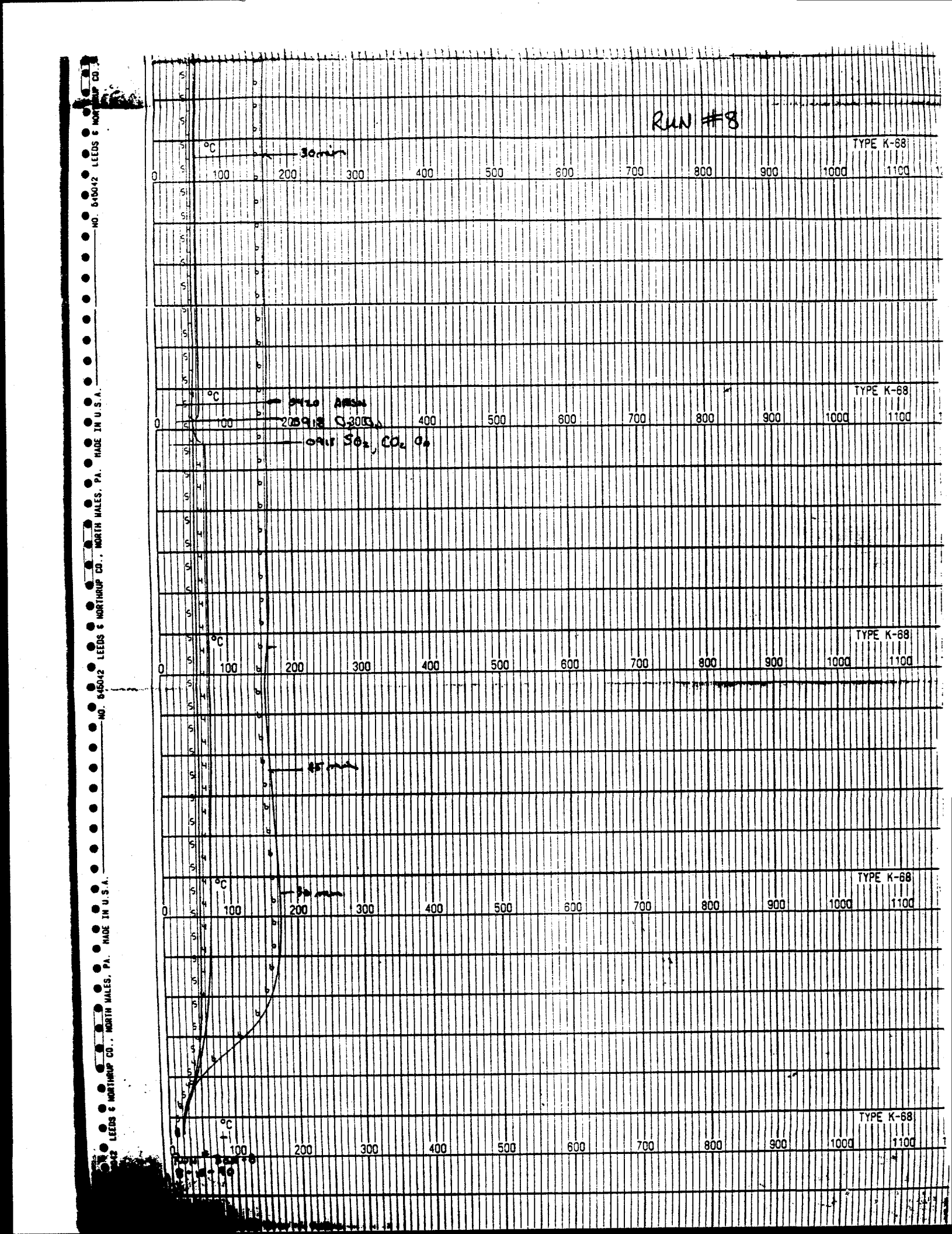
MOLYTEK INC. 3419 SMALLMAN ST. PITTSBURG, PA. 15222 (412) 761-9030
CHART NO. 414103

MICROTEK INC.
2419 BRADDOCK ST. PITTSBURG, PA. 15213
(412) 261-0100
CHART NO. 414103
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90
ABSN
1153
11:53 Blocked

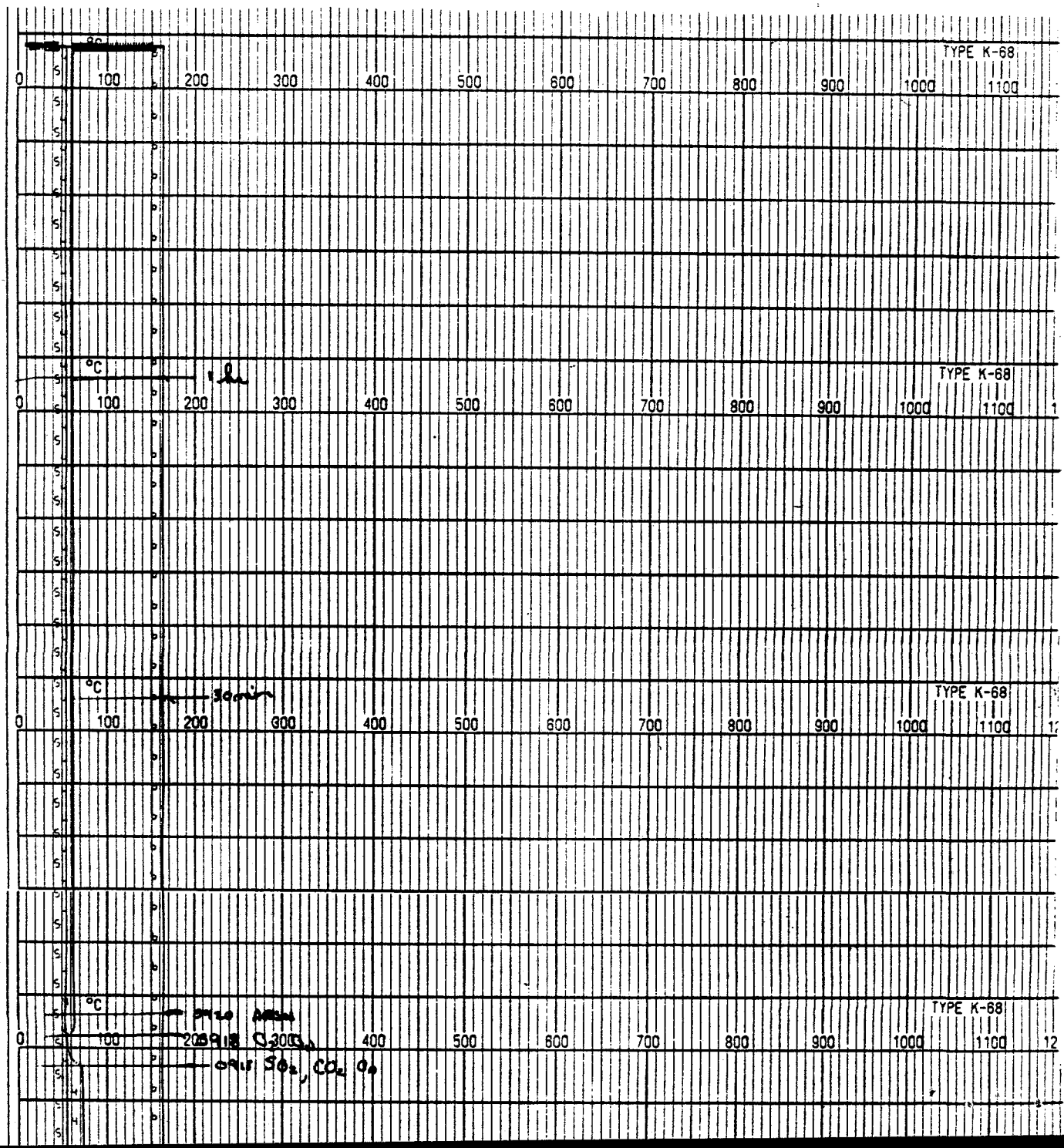

0
10
20
30
40
50
60
70
80
90
100
CHART NO. 414103

RUN #7




RUN #8
TYPE K-68
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
30 min
45 min
LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A. NO. 646042

RUN #8



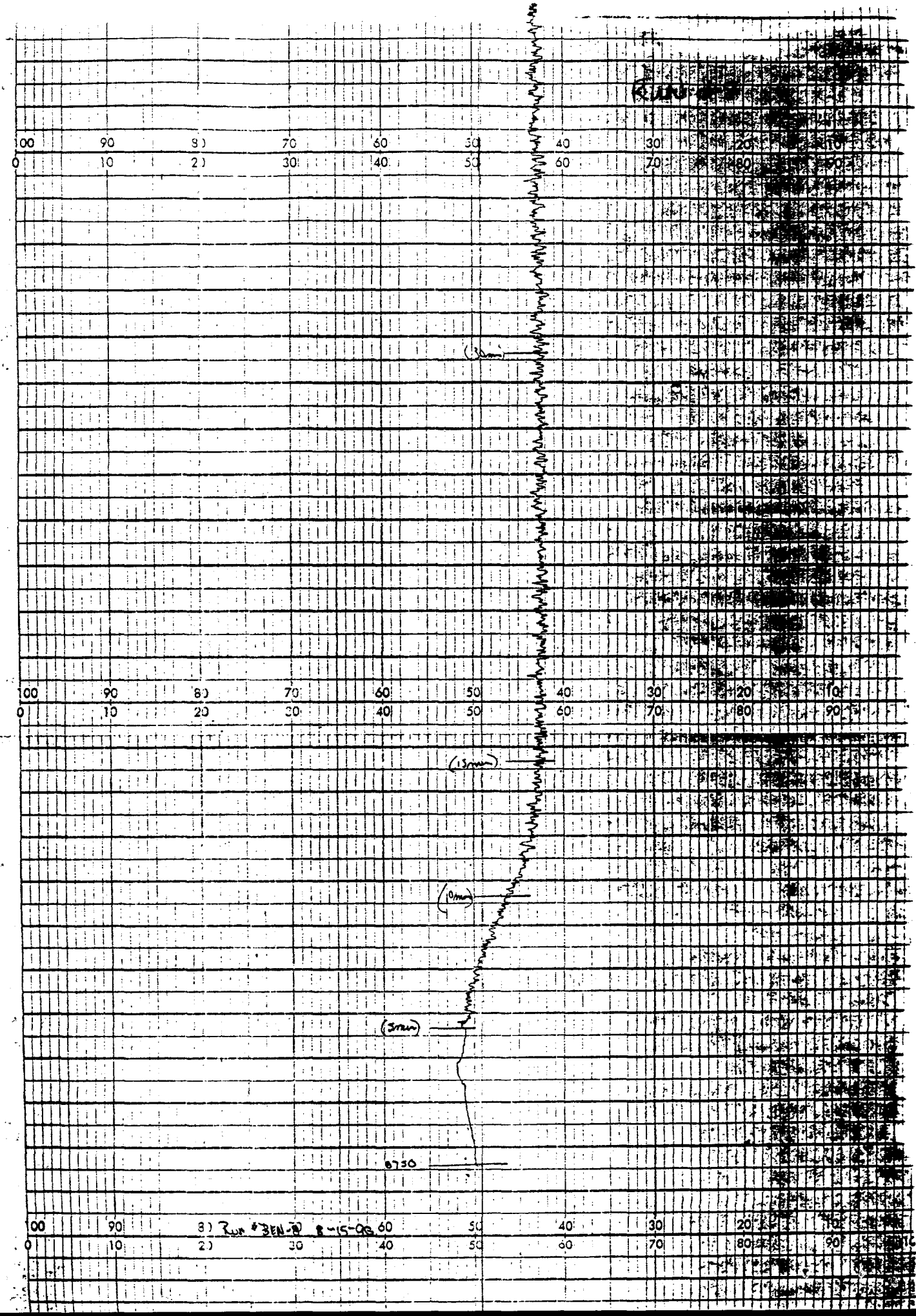
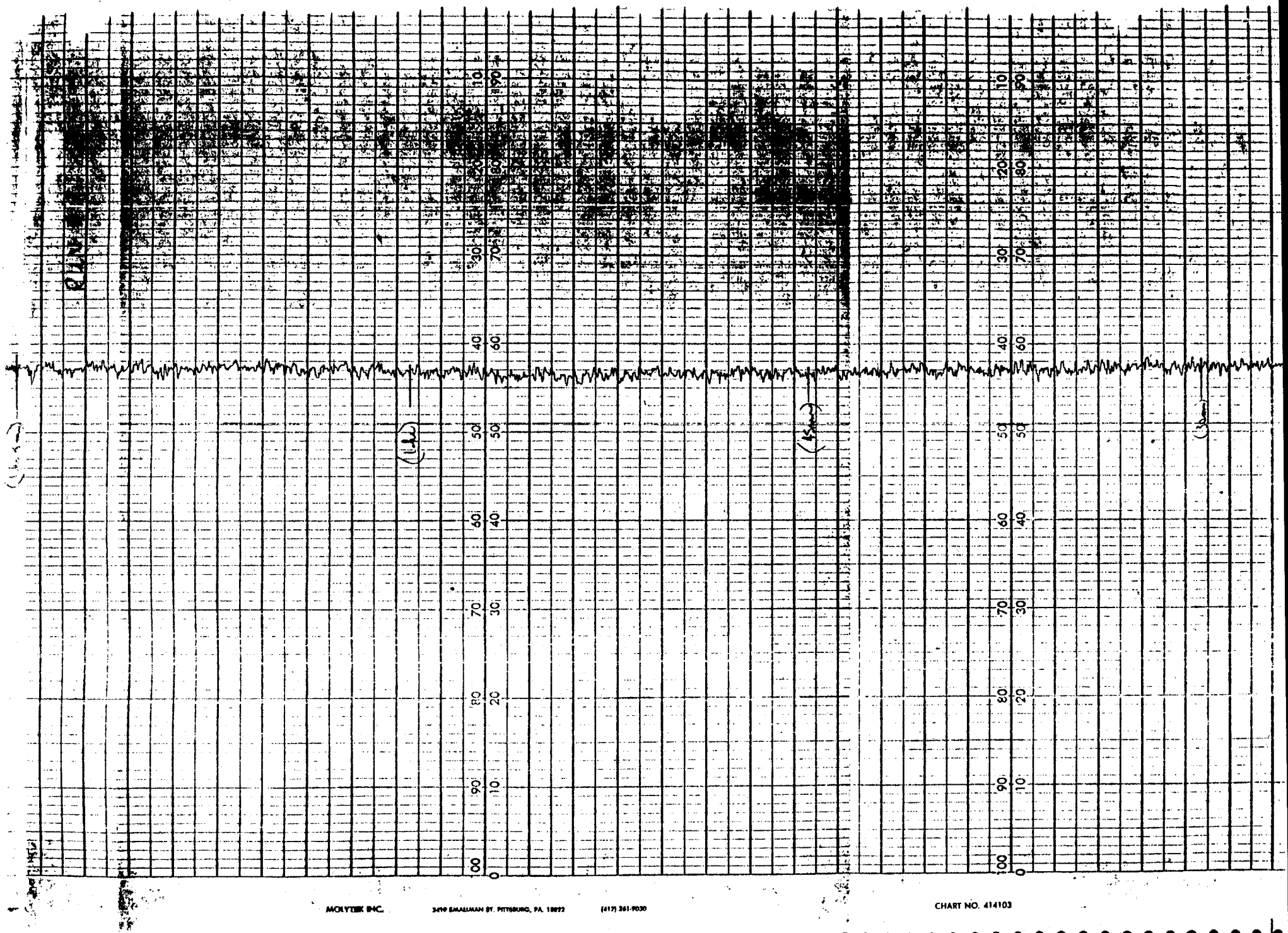
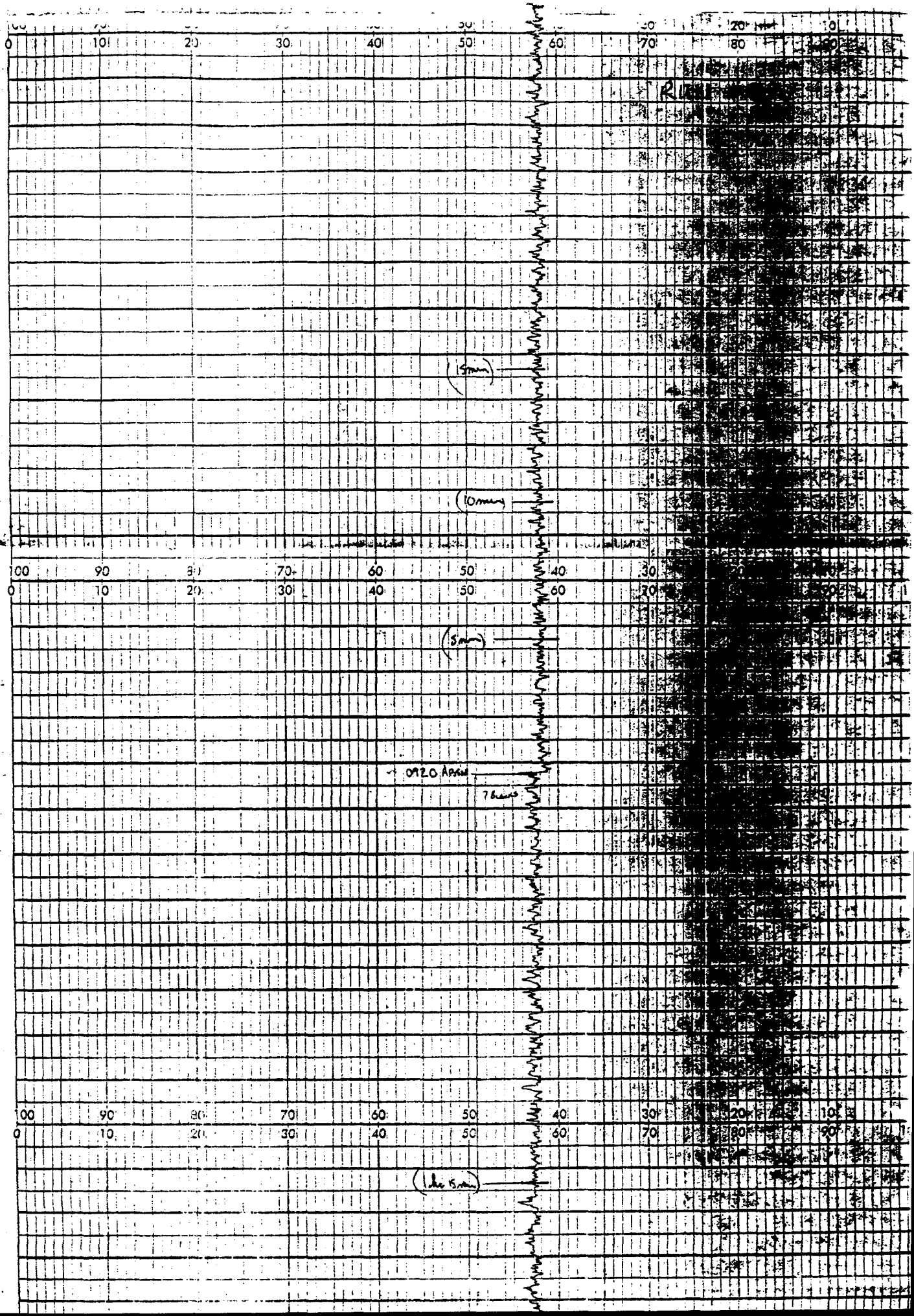
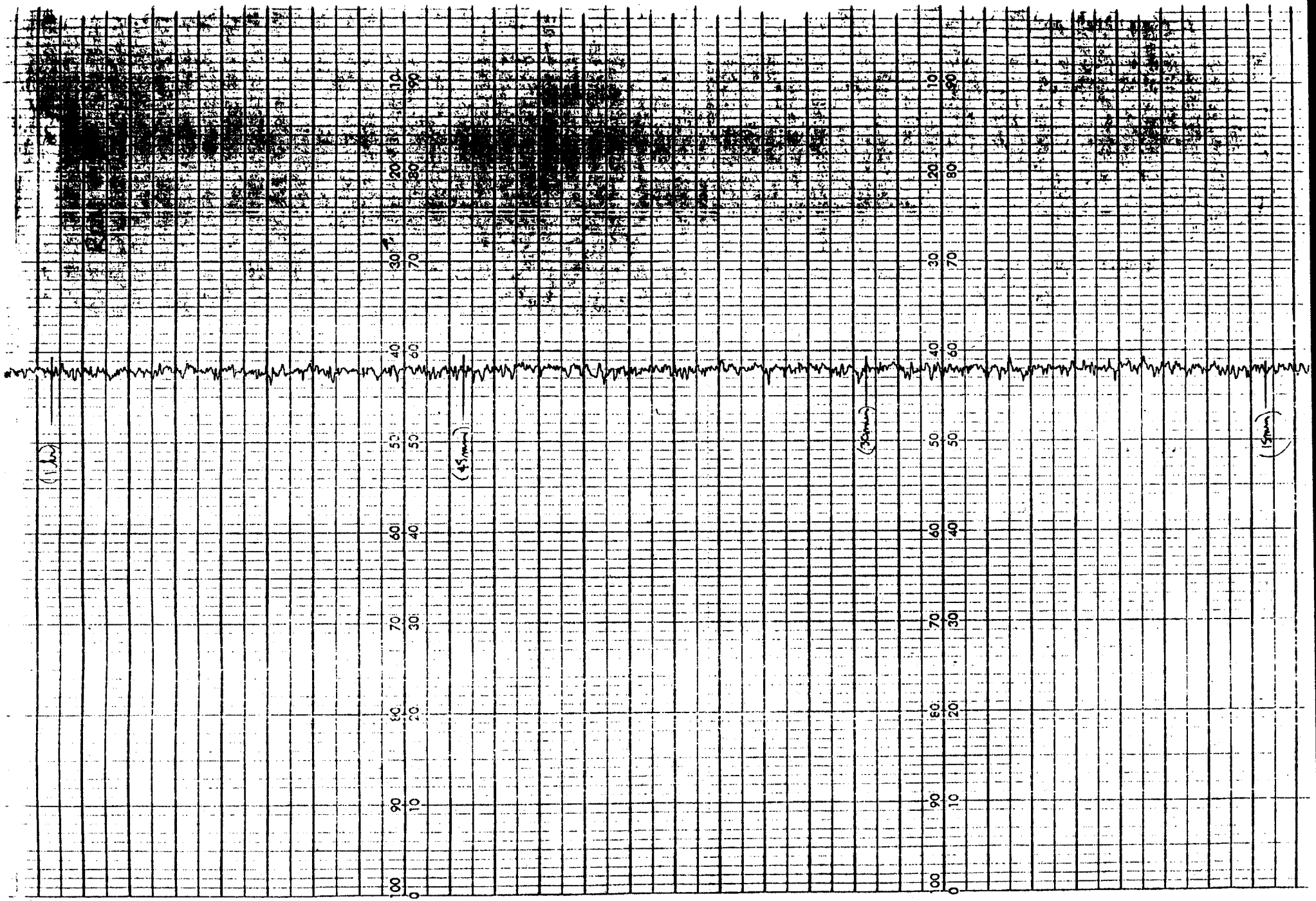


CHART NO. 414103
PRINTED IN U.S.A.
MOLYTEK INC.
(30mm)
(15mm)
(0mm)
(5mm)
6730
Run #3EN-B 8-15-08

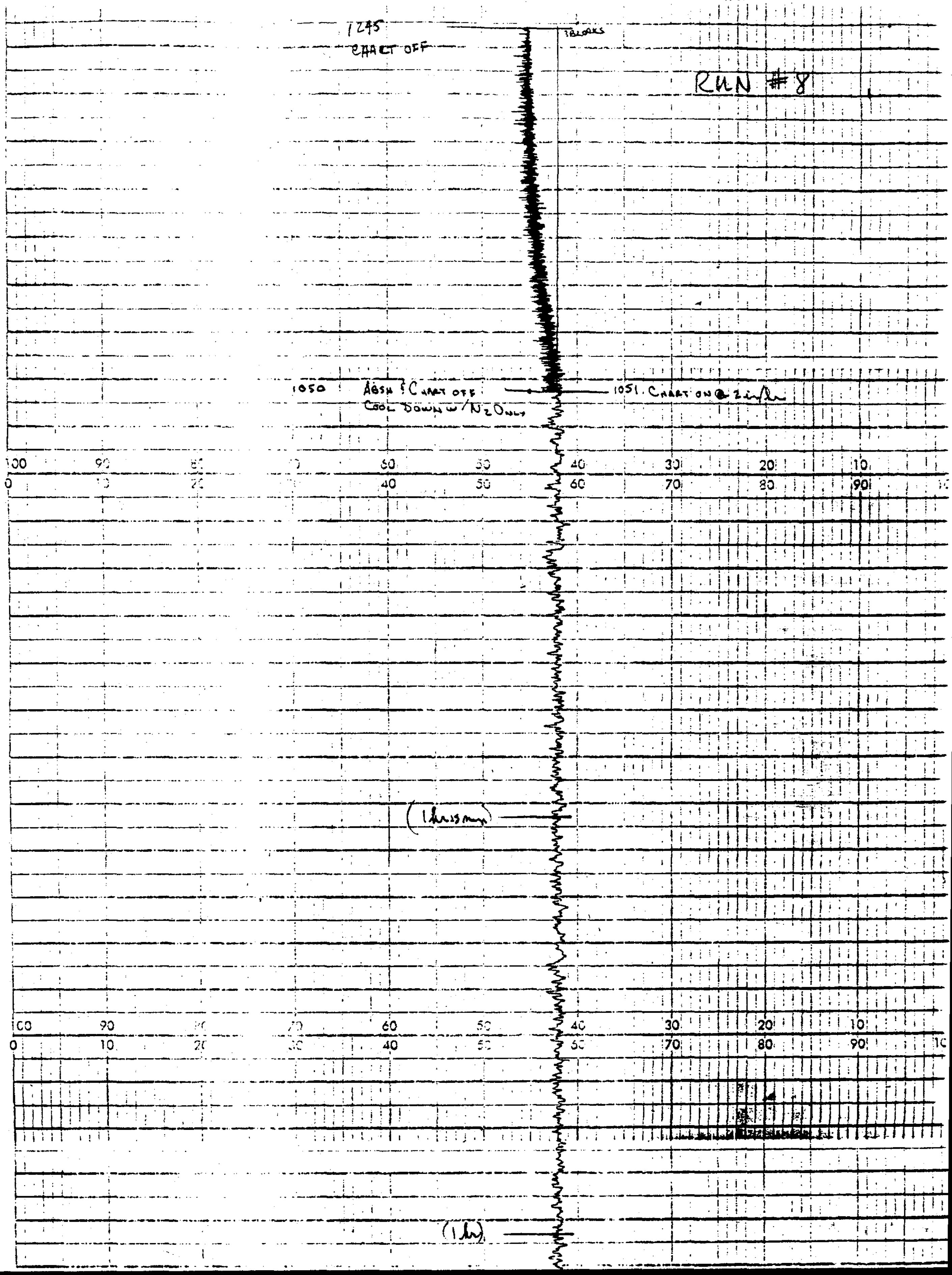

MOLYTEK INC. 3419 SMALLMAN ST. PITTSBURG, PA. 15222 (417) 361-9030
CHART NO. 414103


0920 Apnea
7 beats



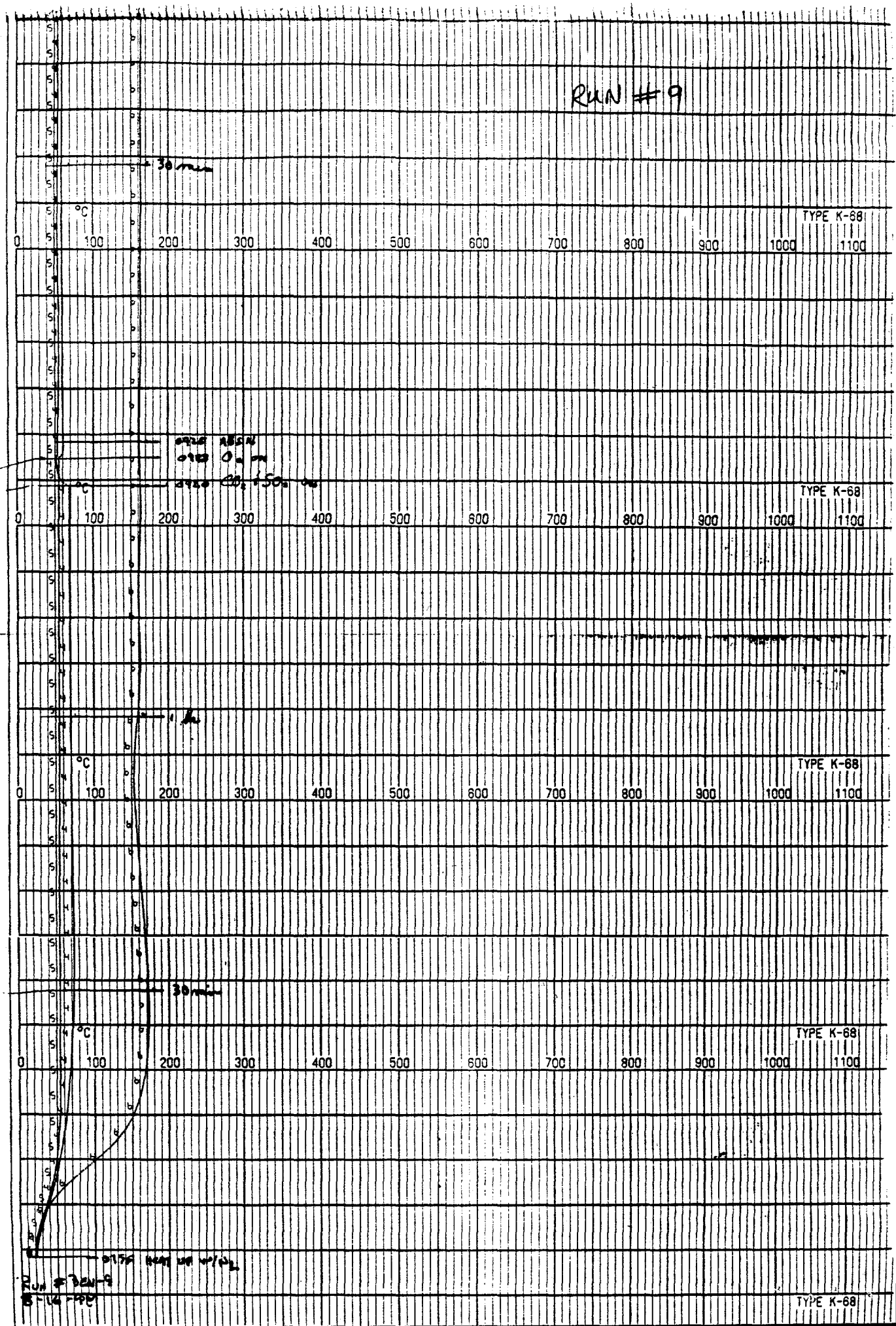
PRINTED IN U.S.A.

MOLYTEK INC. 2419 BRADDOCK ST. PITTSBURG, PA. 15222 (412) 261-9030

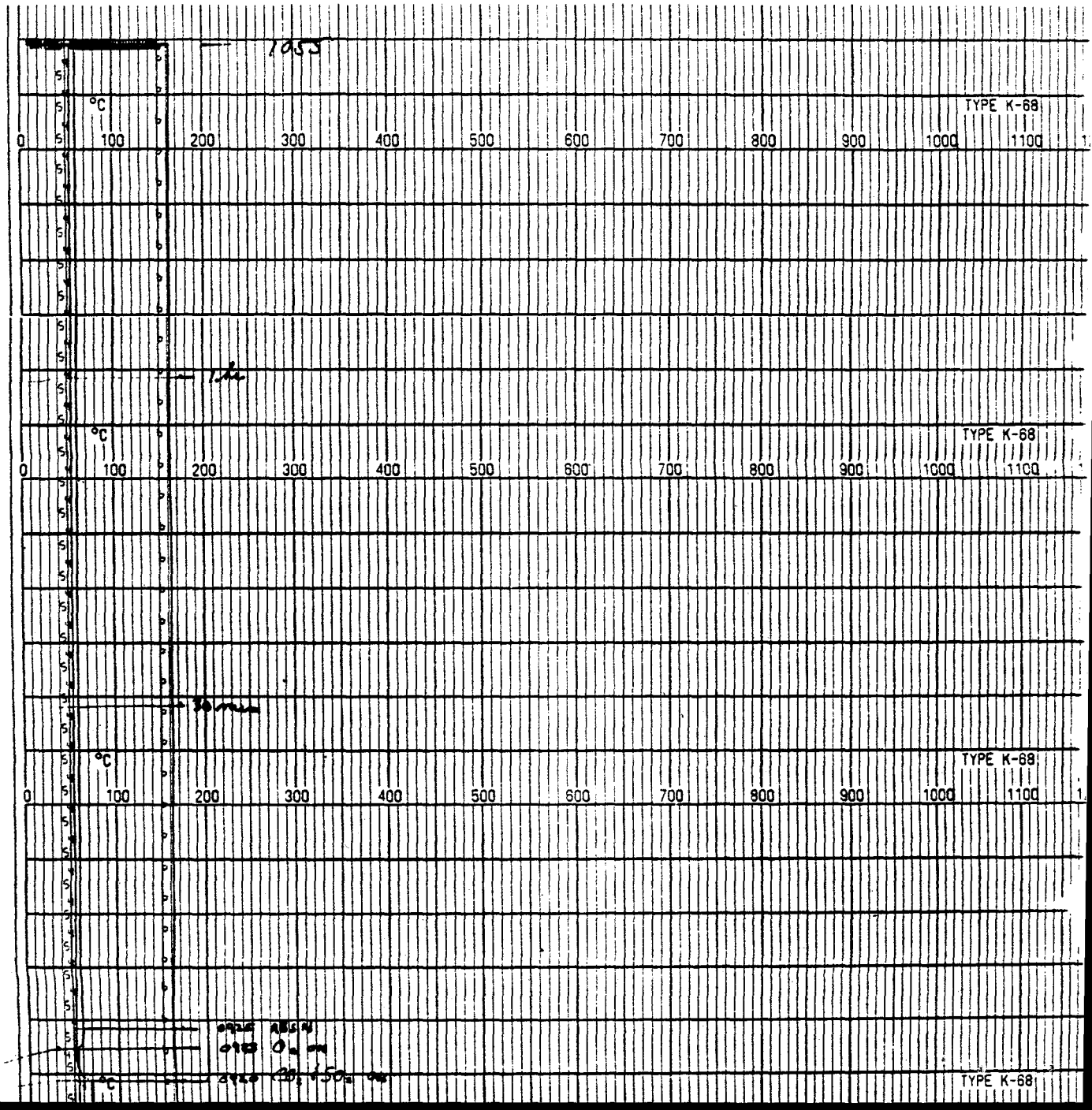


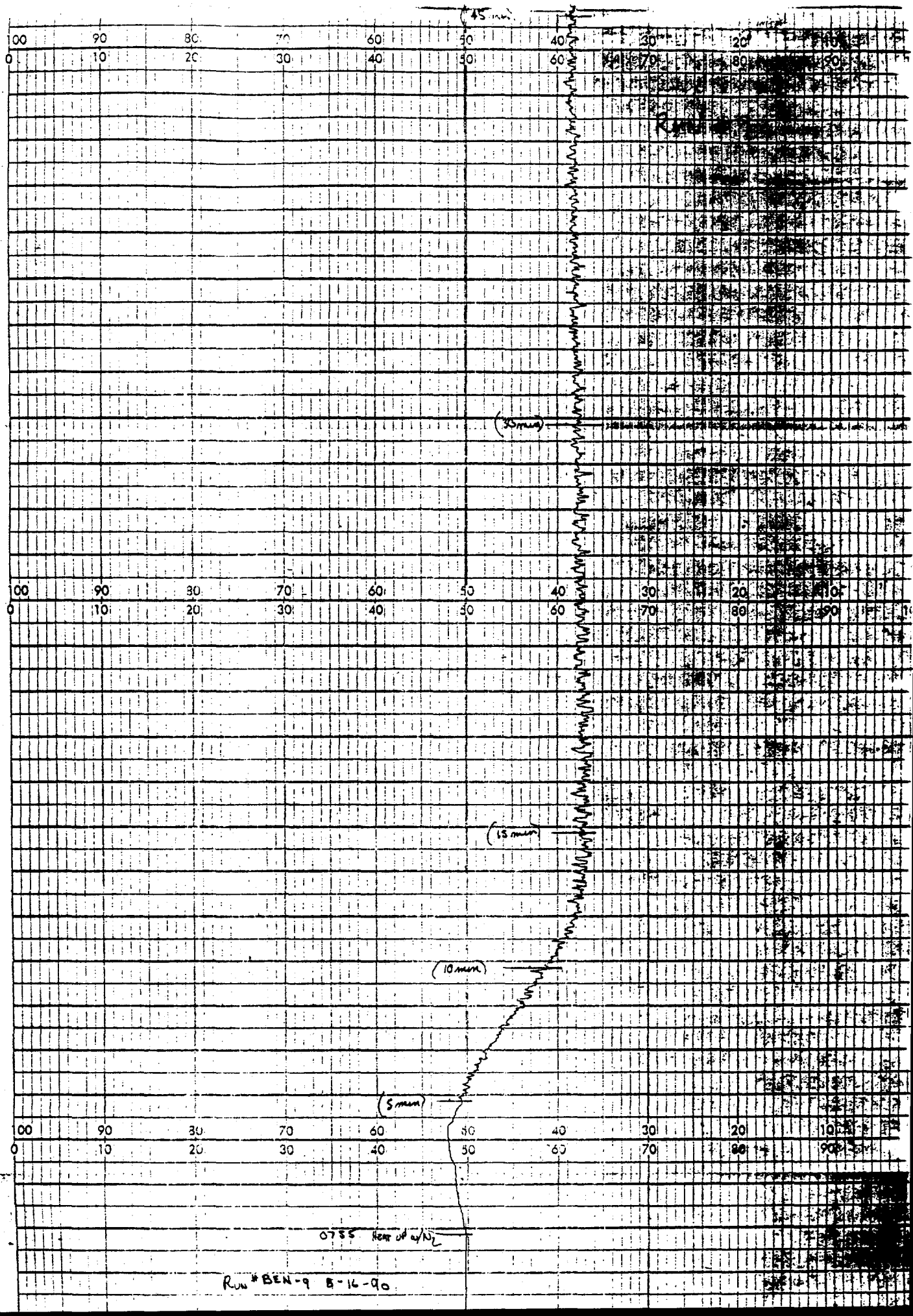
MOLYTEK INC. 2419 SMALLMAN ST PITTSBURG, PA 15222 (412) 261 9010

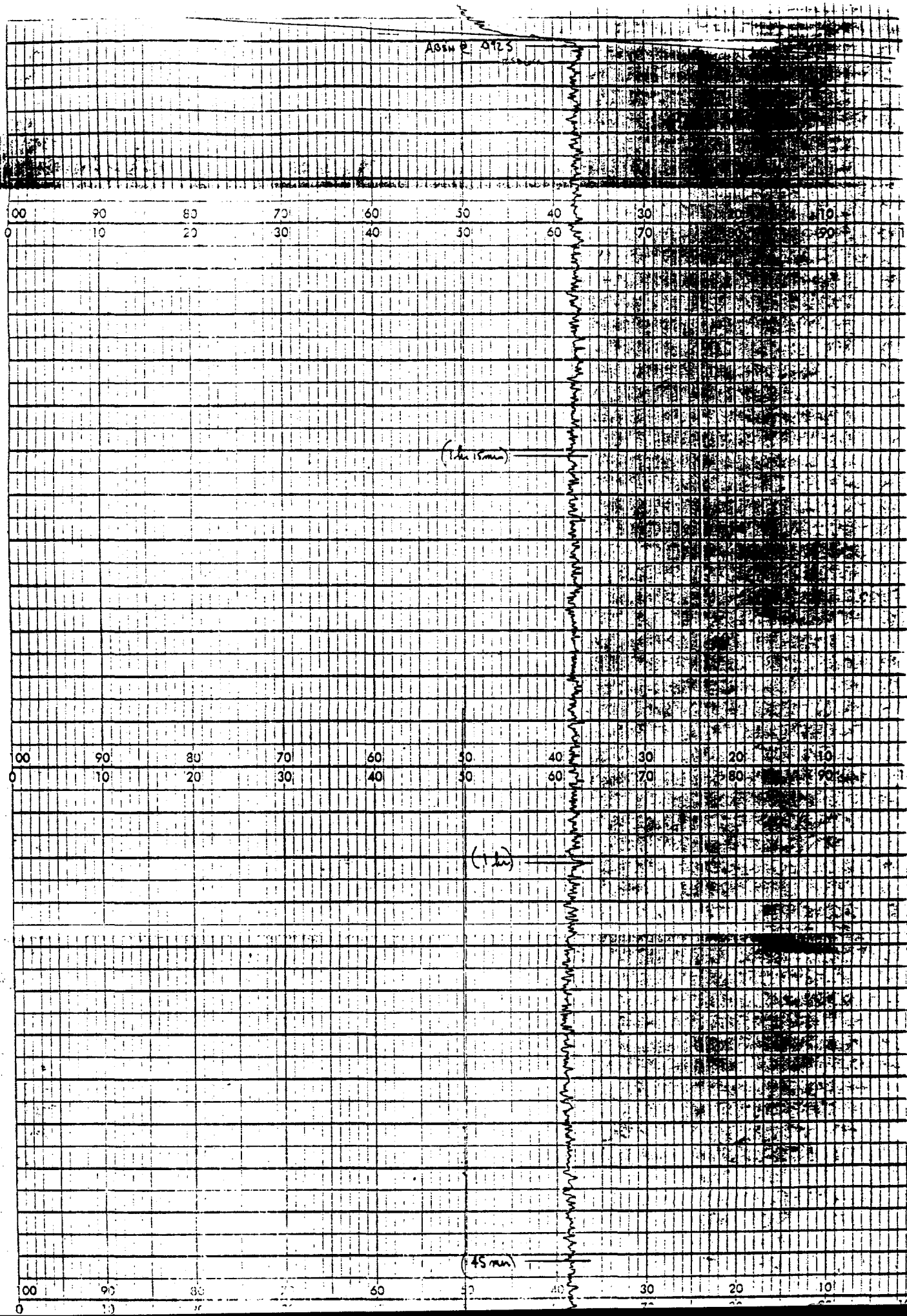
CHART NO 414103


RUN #9
30 min
°C
TYPE K-68
0
100
200
300
400
500
600
700
800
900
1000
1100
1 hr
30 min
LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A. NO. 645042

RUN #9

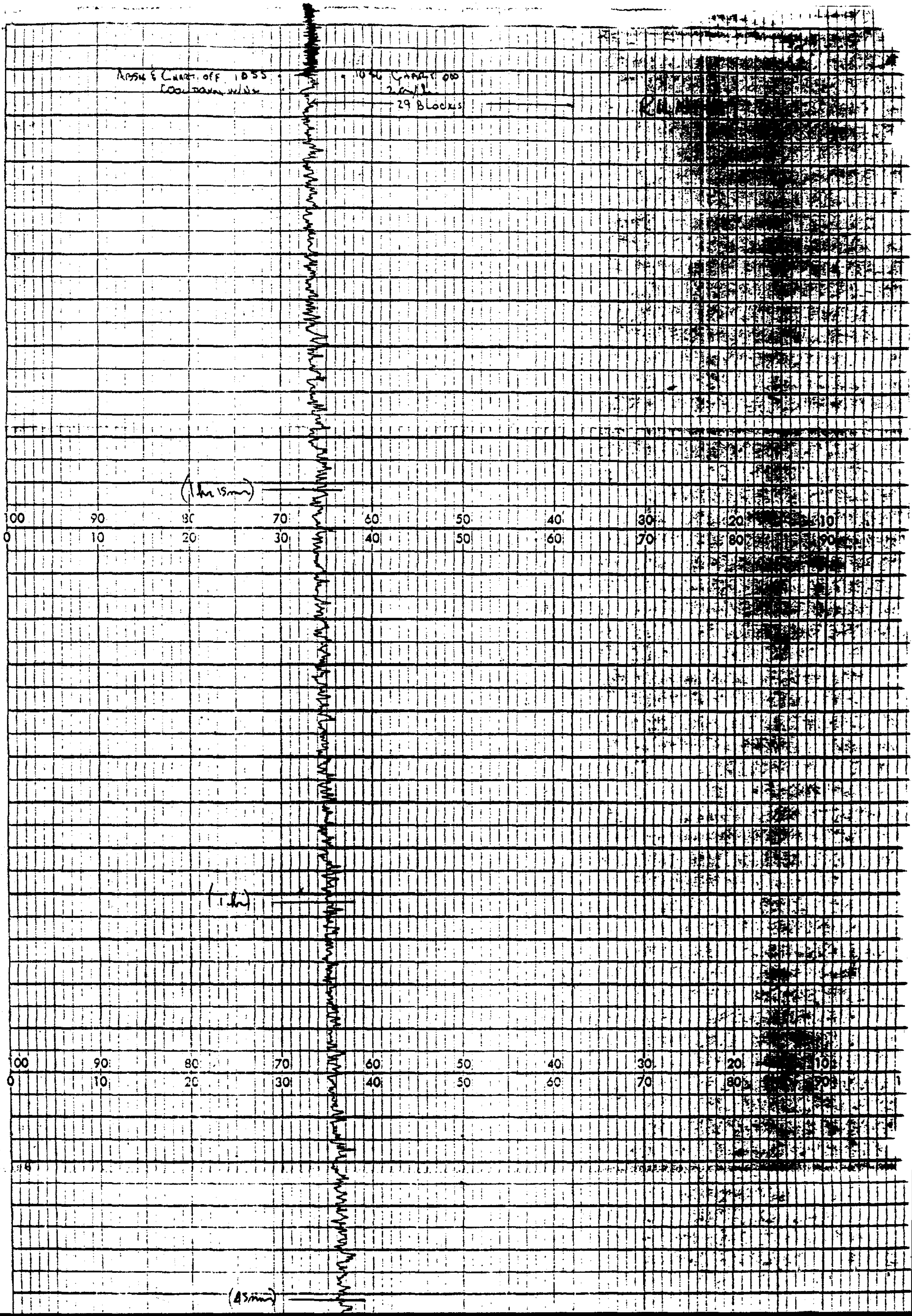

1055
°C
TYPE K-68
0
100
200
300
400
500
600
700
800
900
1000
1100
1.hr
30 min
NO. 545042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.


45 min
35 min
15 min
10 min
5 min
0755 Heat on o/N2
Run #BEN-9 8-16-90



MOLYTEK INC.
3419 BAKALUMAN ST. PITTSBURG, PA. 15222
(412) 261-1020
CHART NO. 414103
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90
(5mm)
(5mm)
(5mm)
AGSN @ 0925

CHART NO. 414103



PRINTED IN U.S.A.

RUN #9

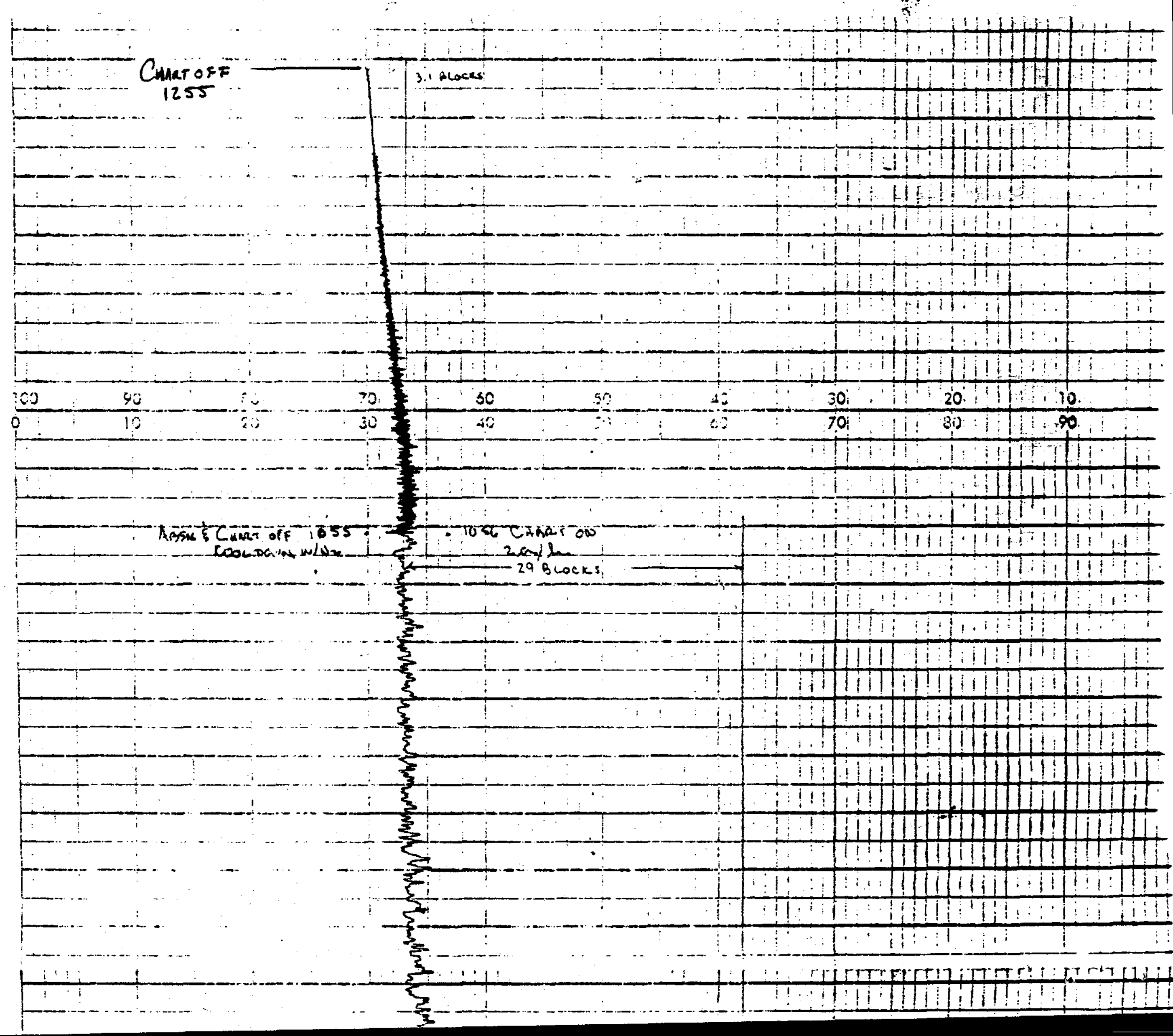
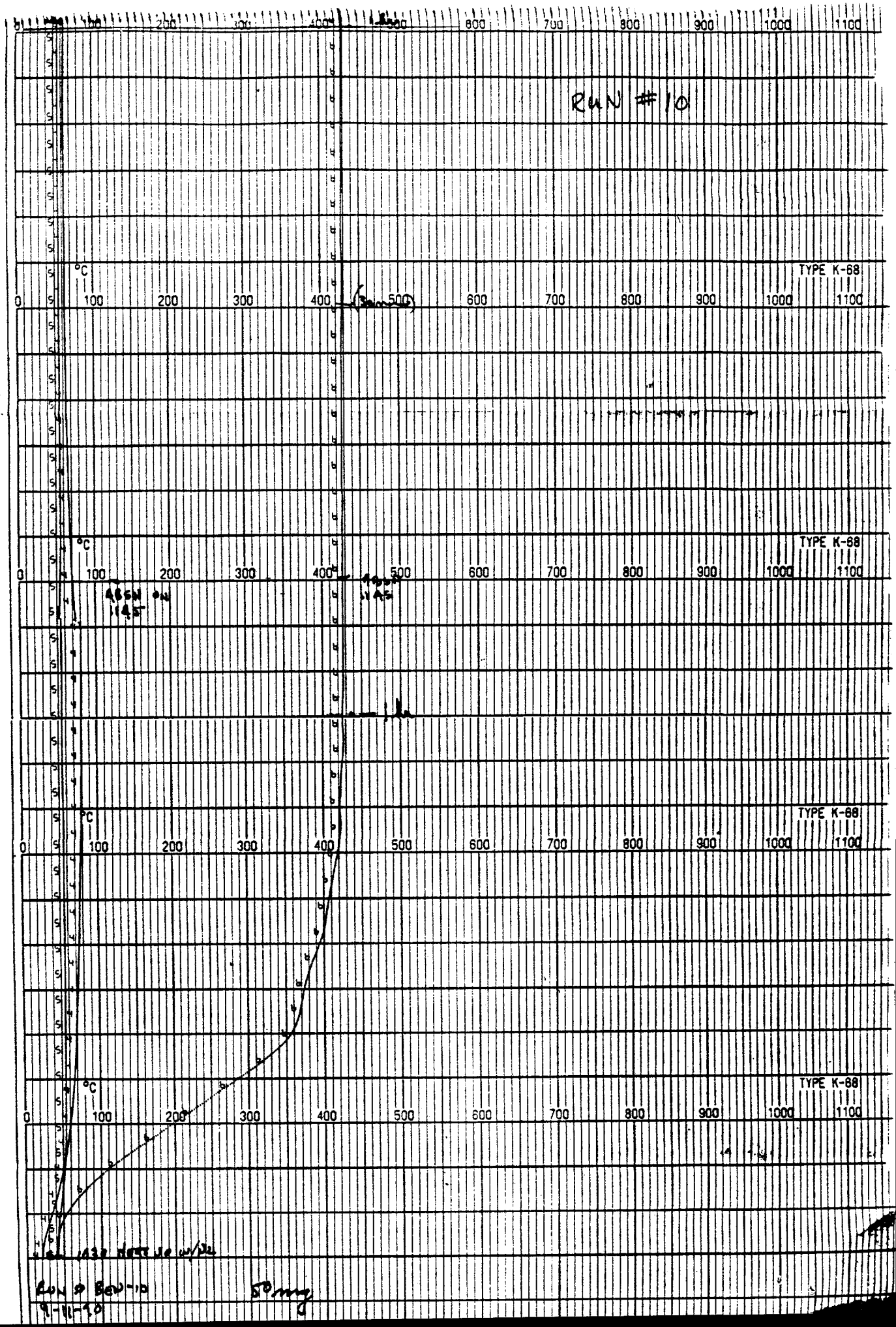
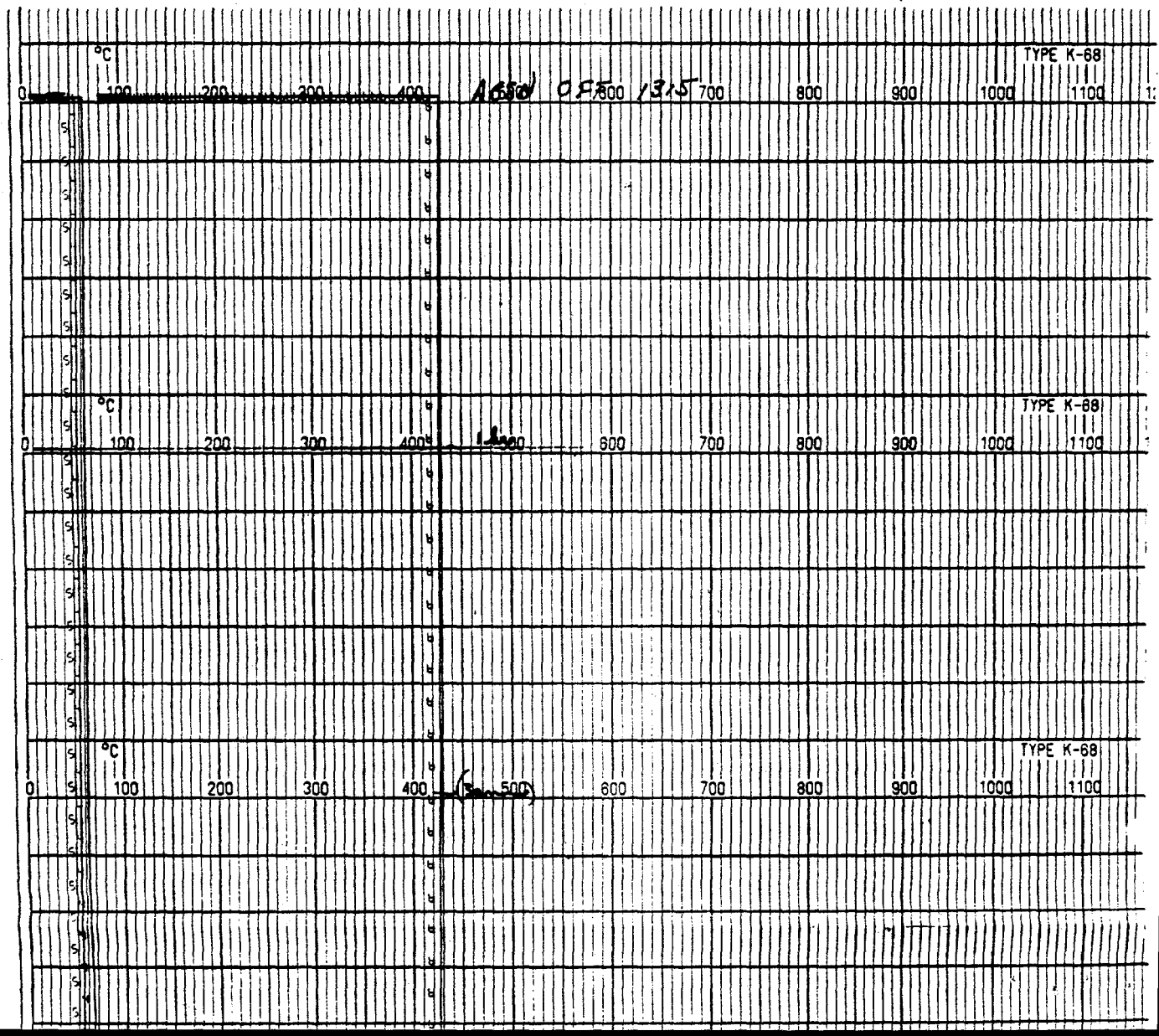


CHART OFF
1255
3.1 BLOCKS
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90
29 BLOCKS

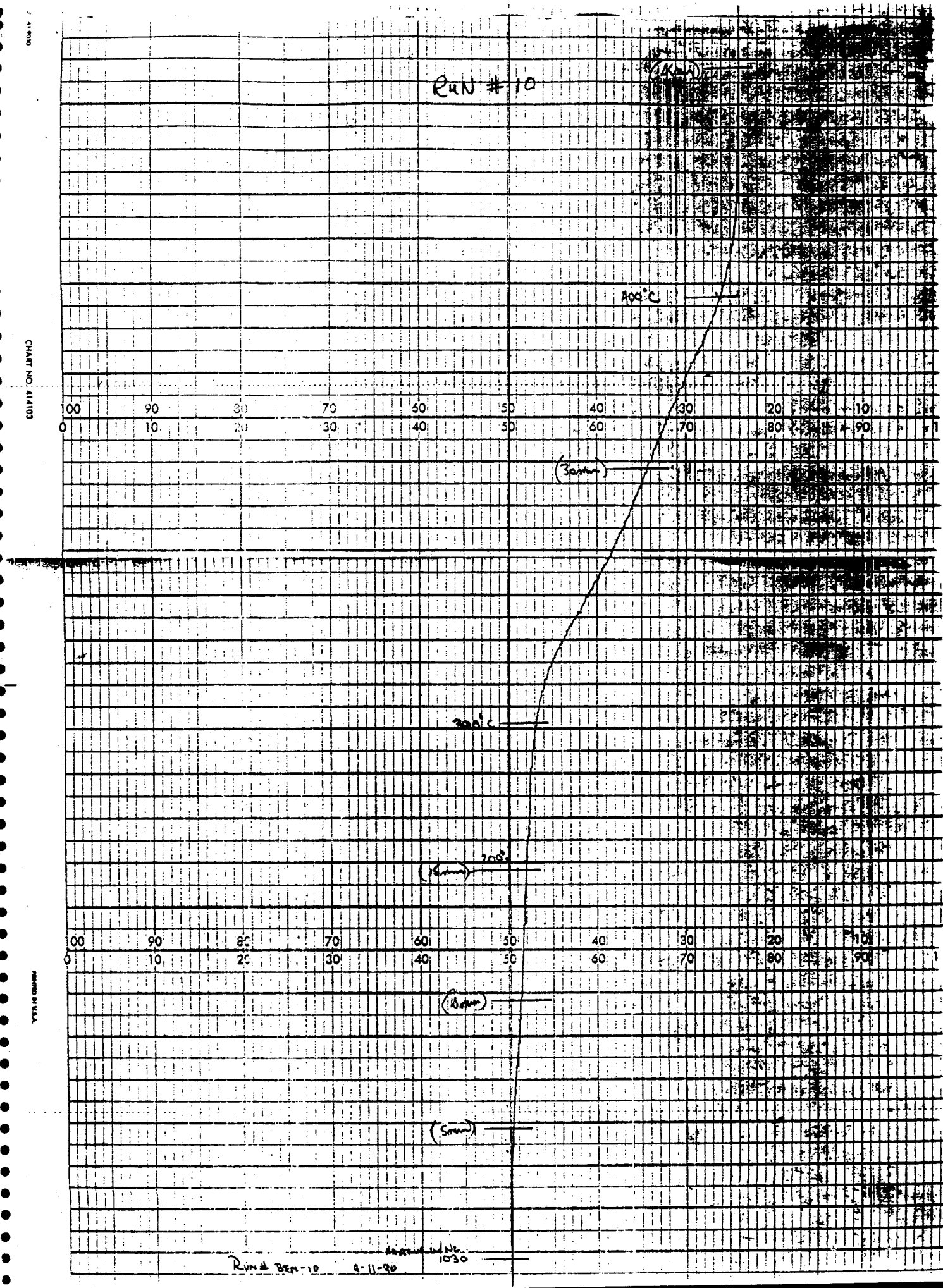

RUN #10
°C
TYPE K-68
0
100
200
300
400
500
600
700
800
900
1000
1100
NO. 645042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

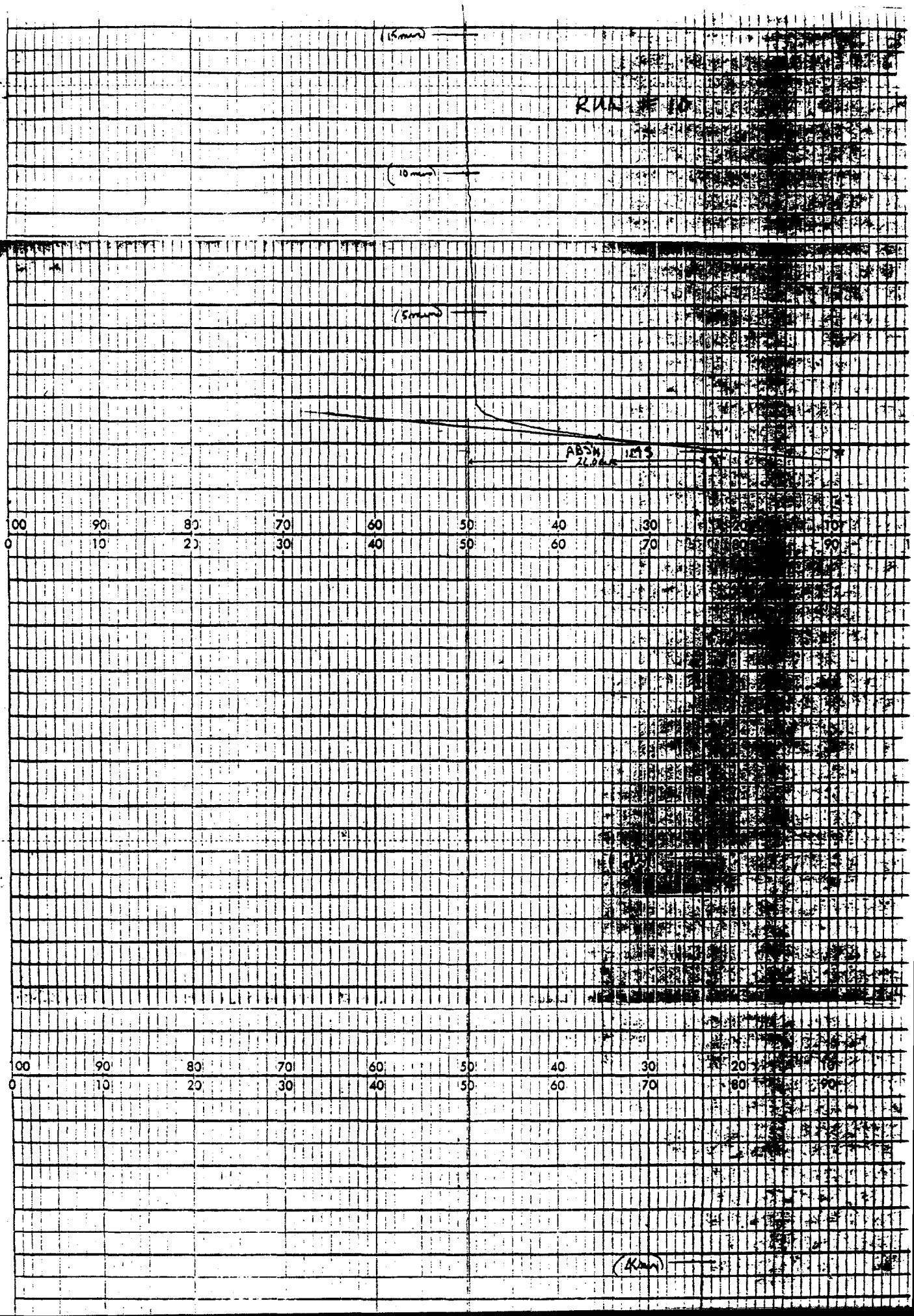
RUN #10

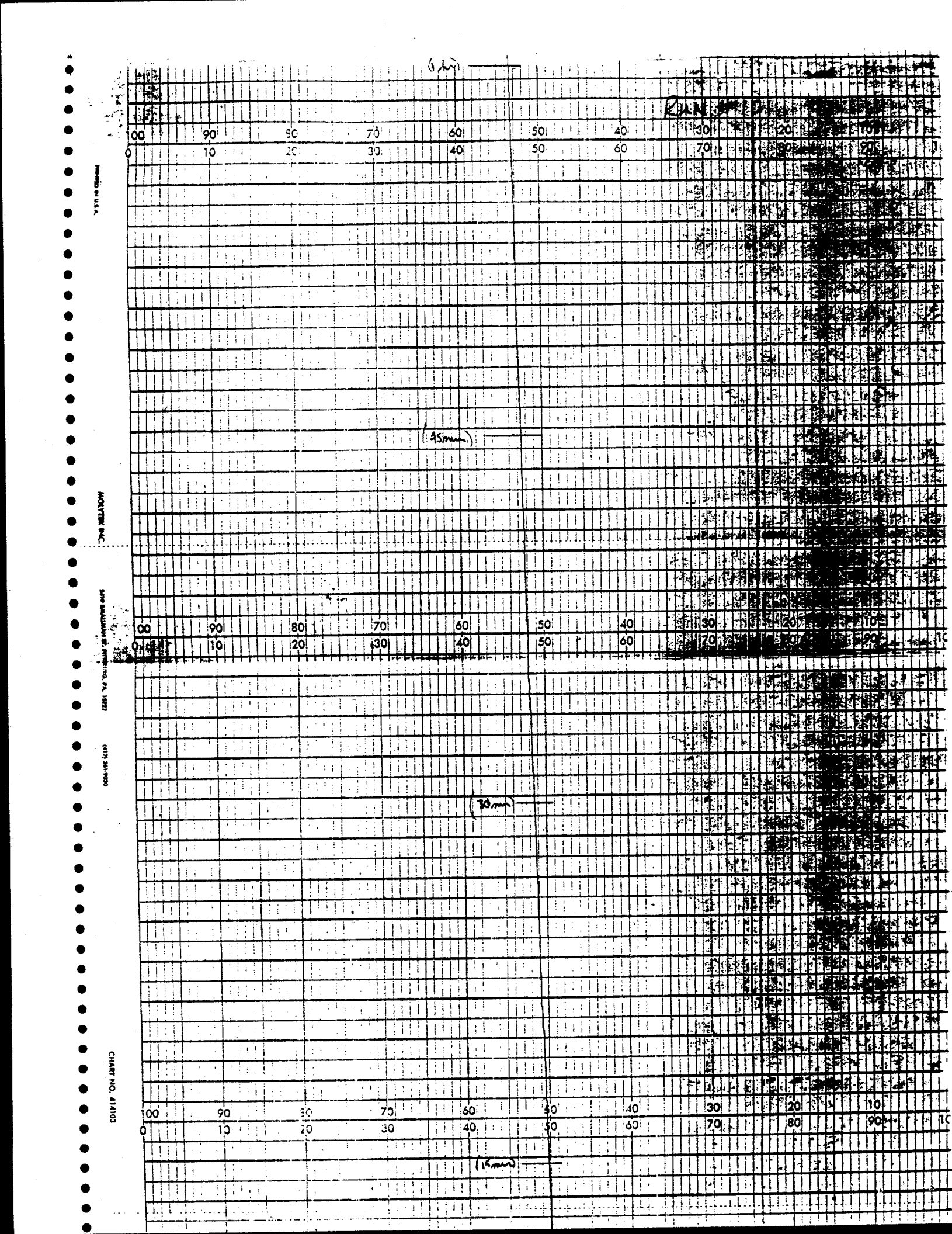


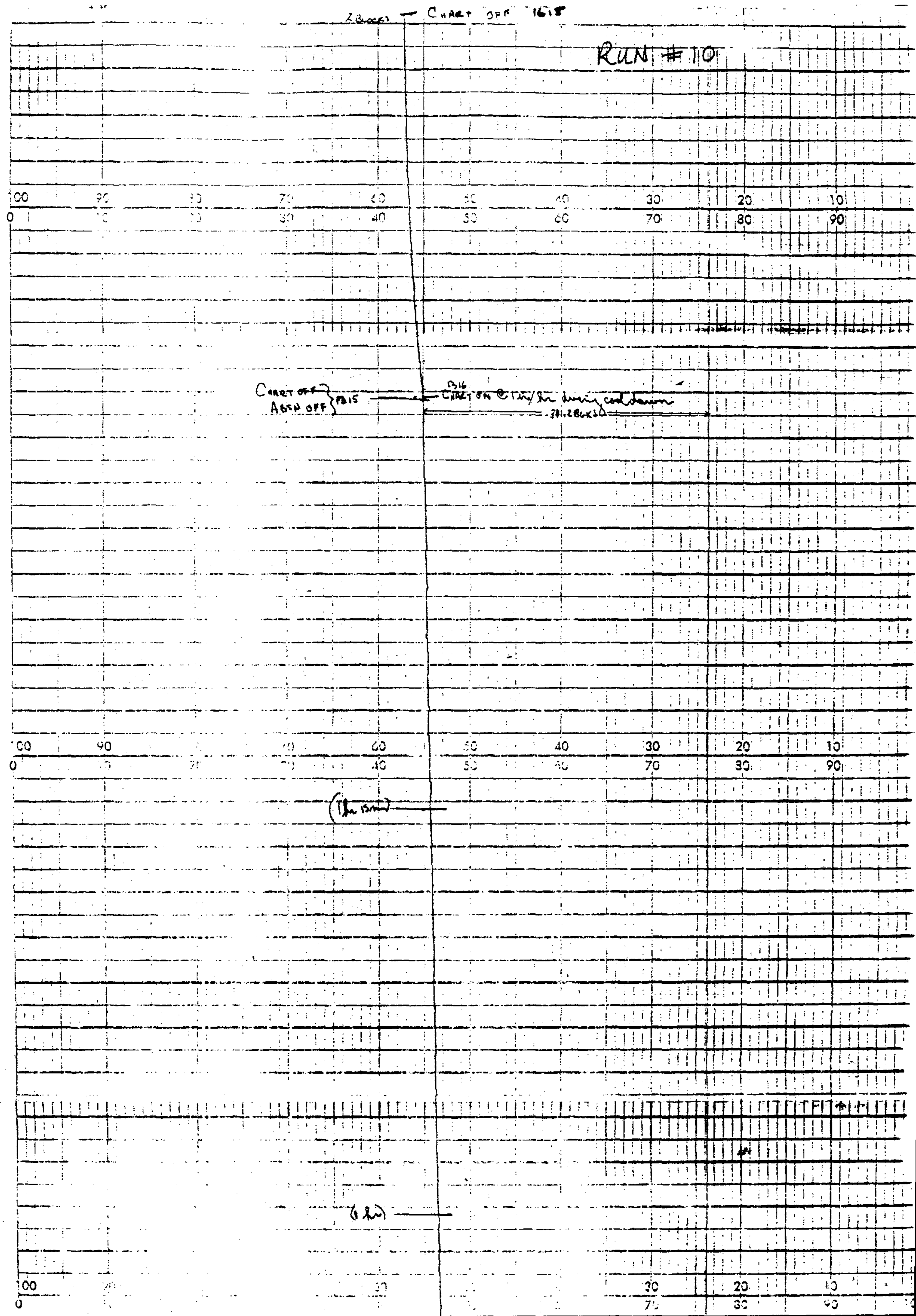
NO. 545042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

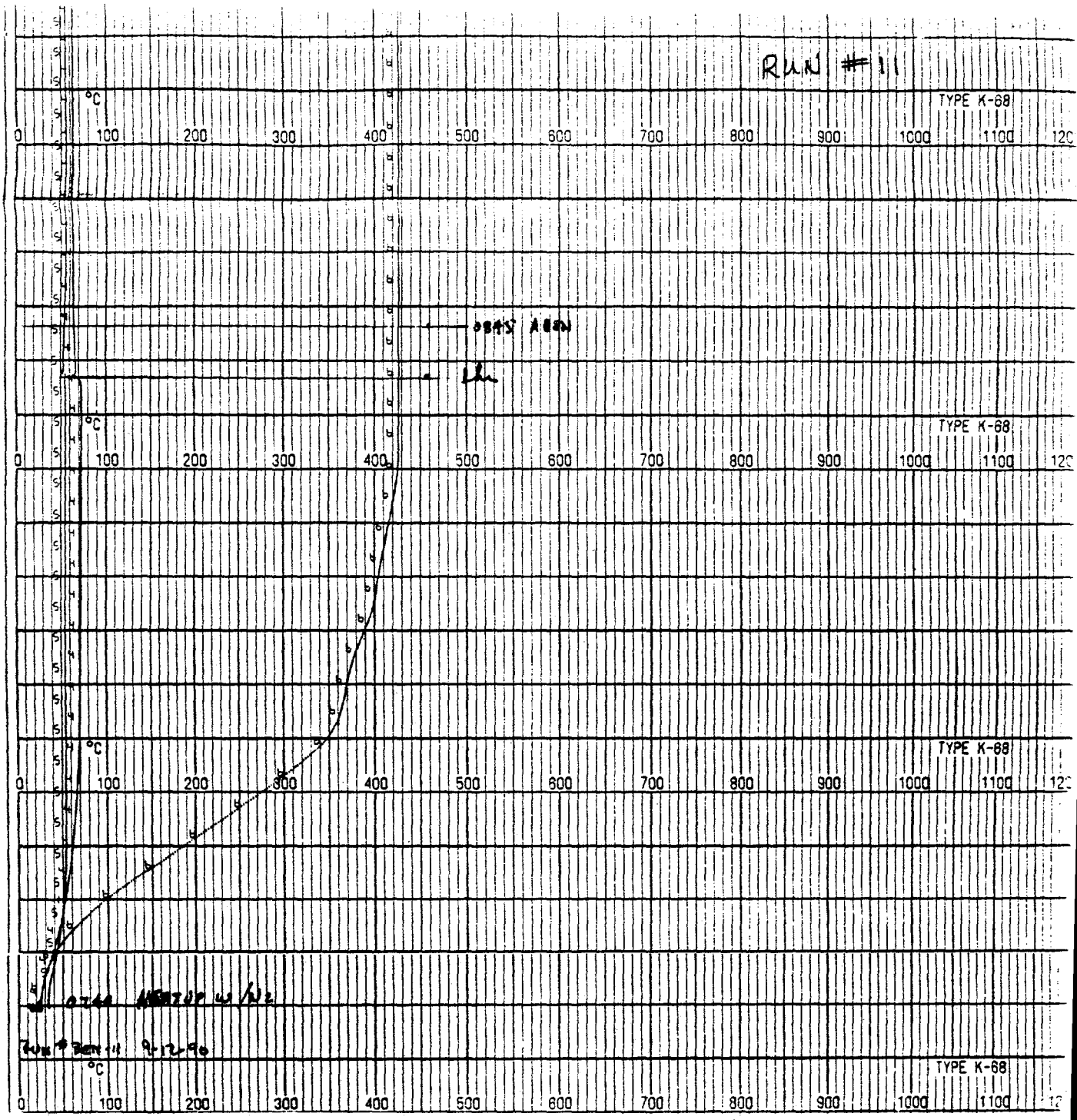
NO. 545042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.


RUN #10
(1KW)
400°C
(30min)
300°C
200°
(15min)
(5min)
CHART NO. 41403
Run # BEN-10 9-11-90
1030


RUN # 10
ABSN
ILDS
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90


RUN #1
(6 min)
(45 min)
(30 min)
(R min)
MOLYTEK INC.
CHART NO. 414103




RUN #11
TYPE K-68
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
TYPE K-68
TYPE K-68
TYPE K-68
IN U.S.A.
NO. 545042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

RUN #11

1015 1685 off

TYPE K-68

°C

0 100 200 300 400 500 600 700 800 900 1000 1100 12

12

TYPE K-68

°C

0 100 200 300 400 500 600 700 800 900 1000 1100 12

TYPE K-68

°C

0 100 200 300 400 500 600 700 800 900 1000 1100 12

0845 ACQ

12

TYPE K-68

°C

0 100 200 300 400 500 600 700 800 900 1000 1100 12

TYPE K-68

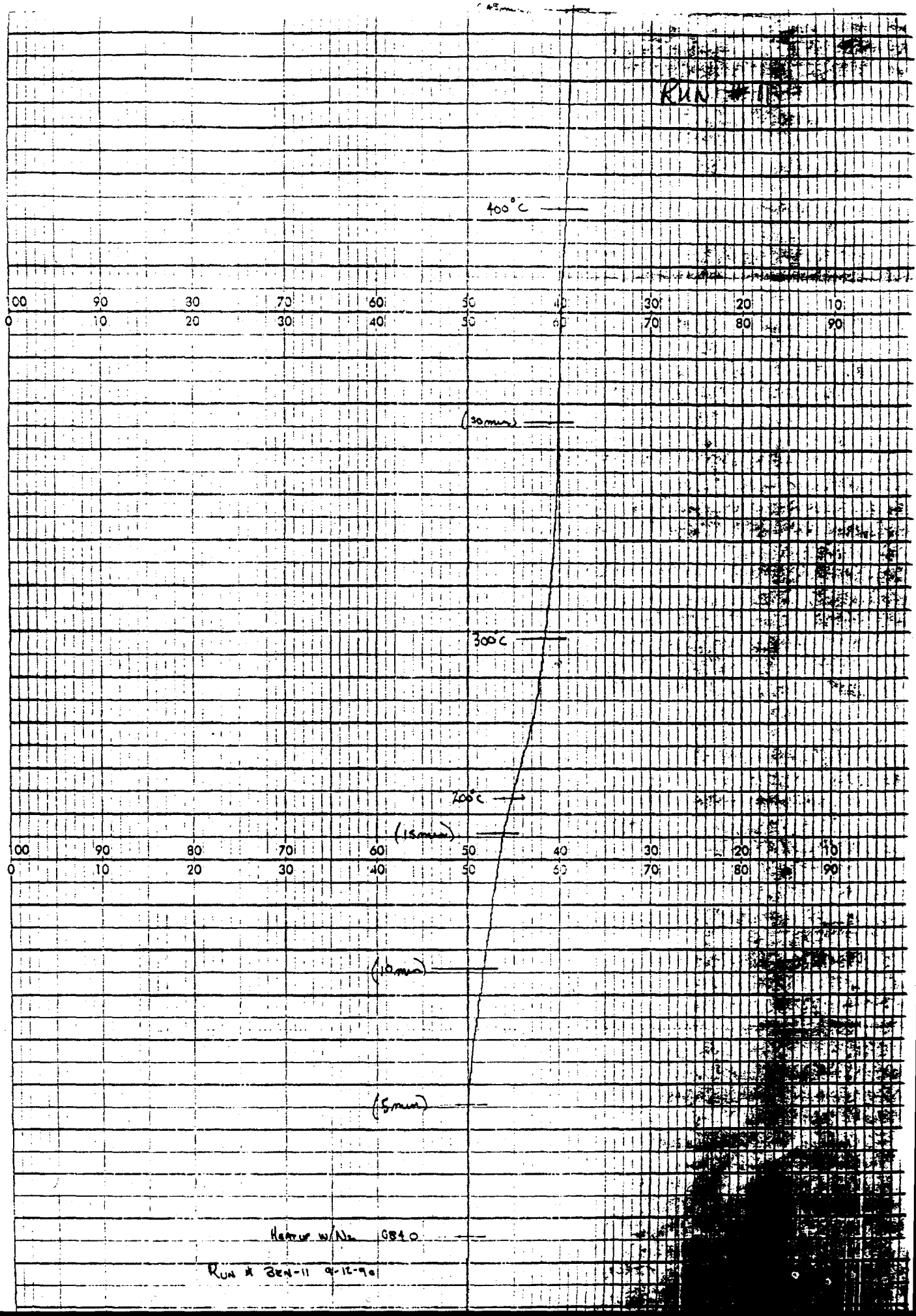
°C

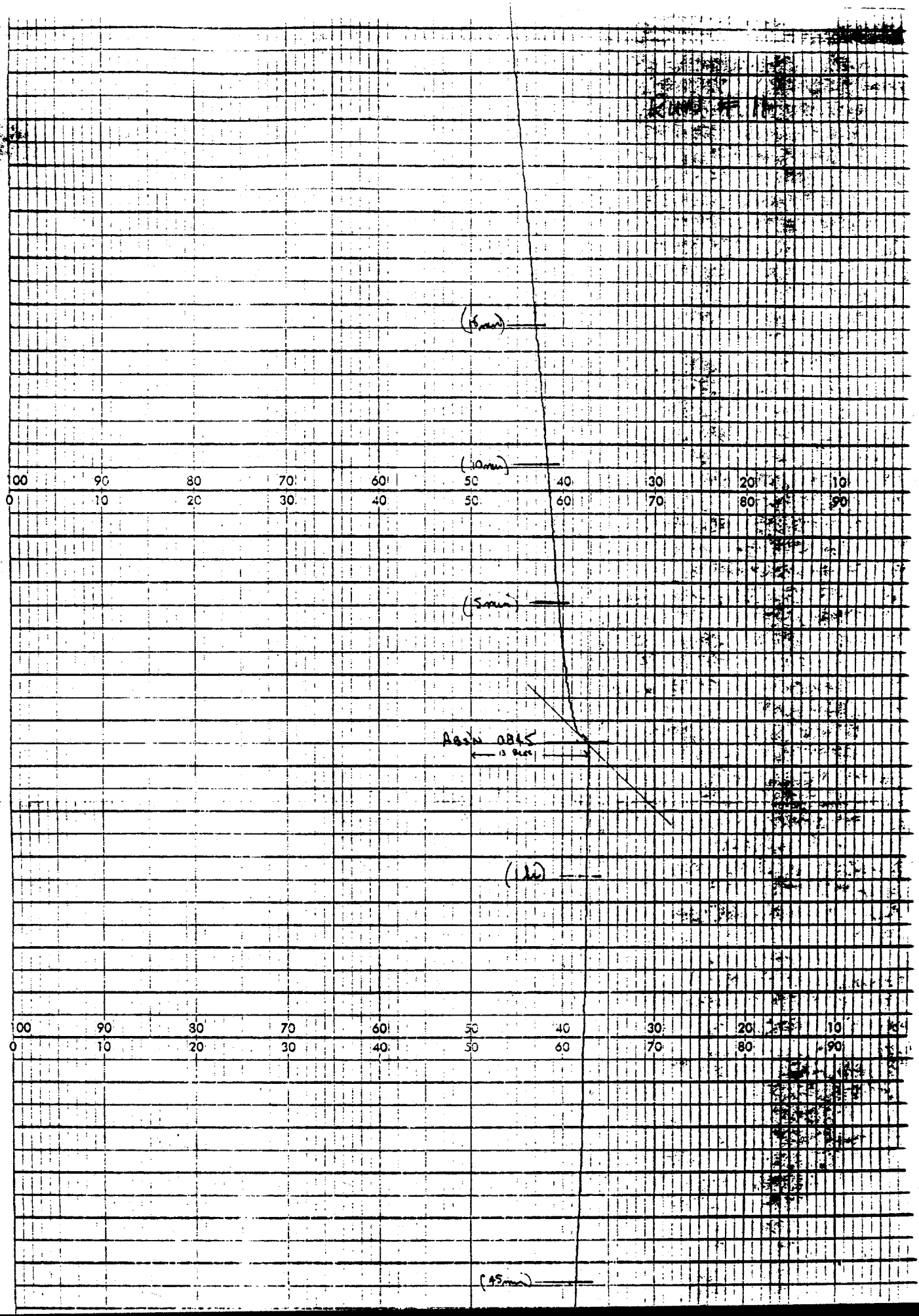
0 100 200 300 400 500 600 700 800 900 1000 1100 12

NO. 845042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

NO. 845042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

NO. 845042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.


RUN #11
400°C
300C
200C
(30 min)
(15 min)
(10 min)
(5 min)
100 90 30 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90
Heatup w/ N2 GB40
Run # Ben-11 9-12-90



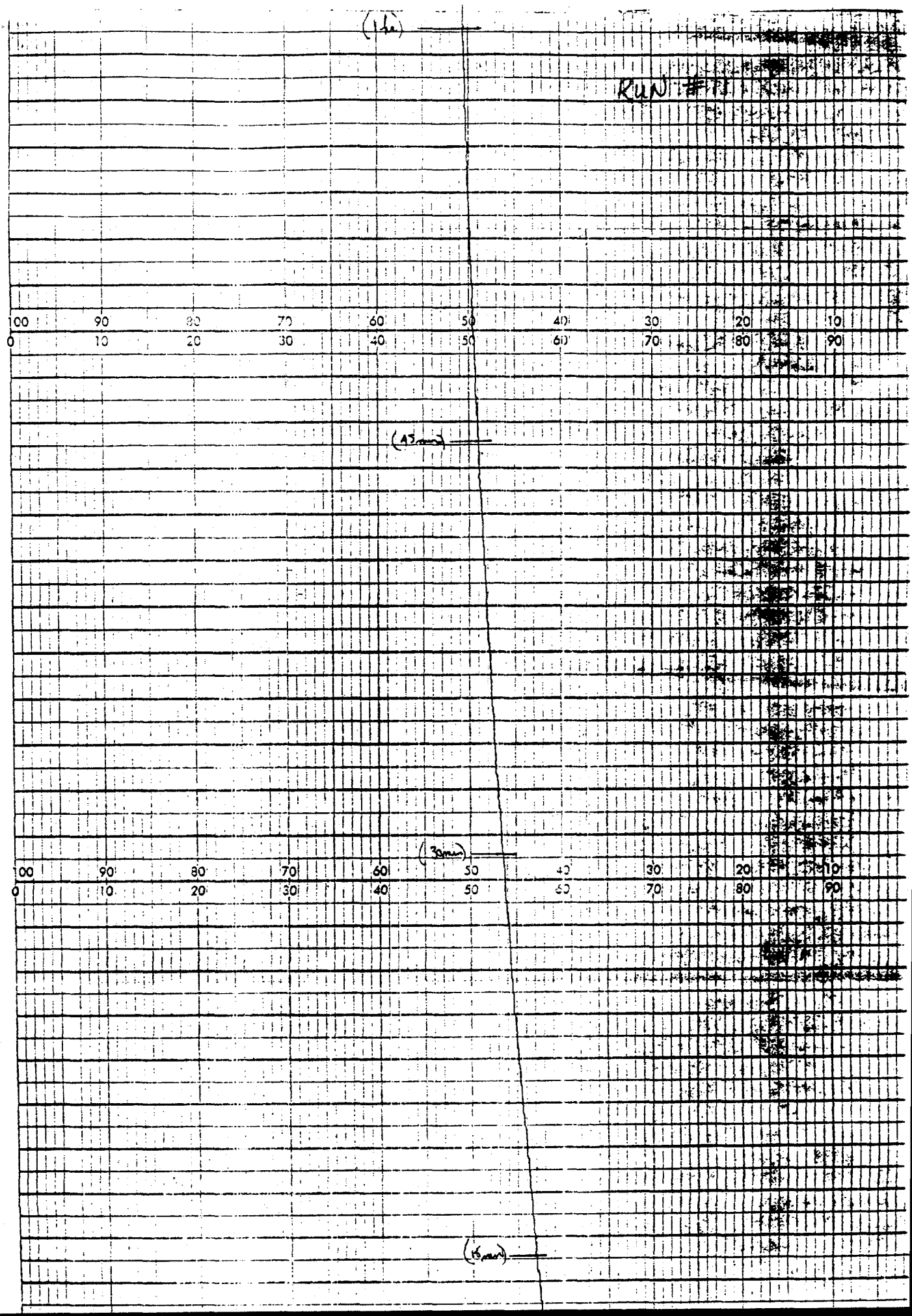


CHART NO. 414103

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MOLYTEK INC.

RUN #11

Chart off
Again off

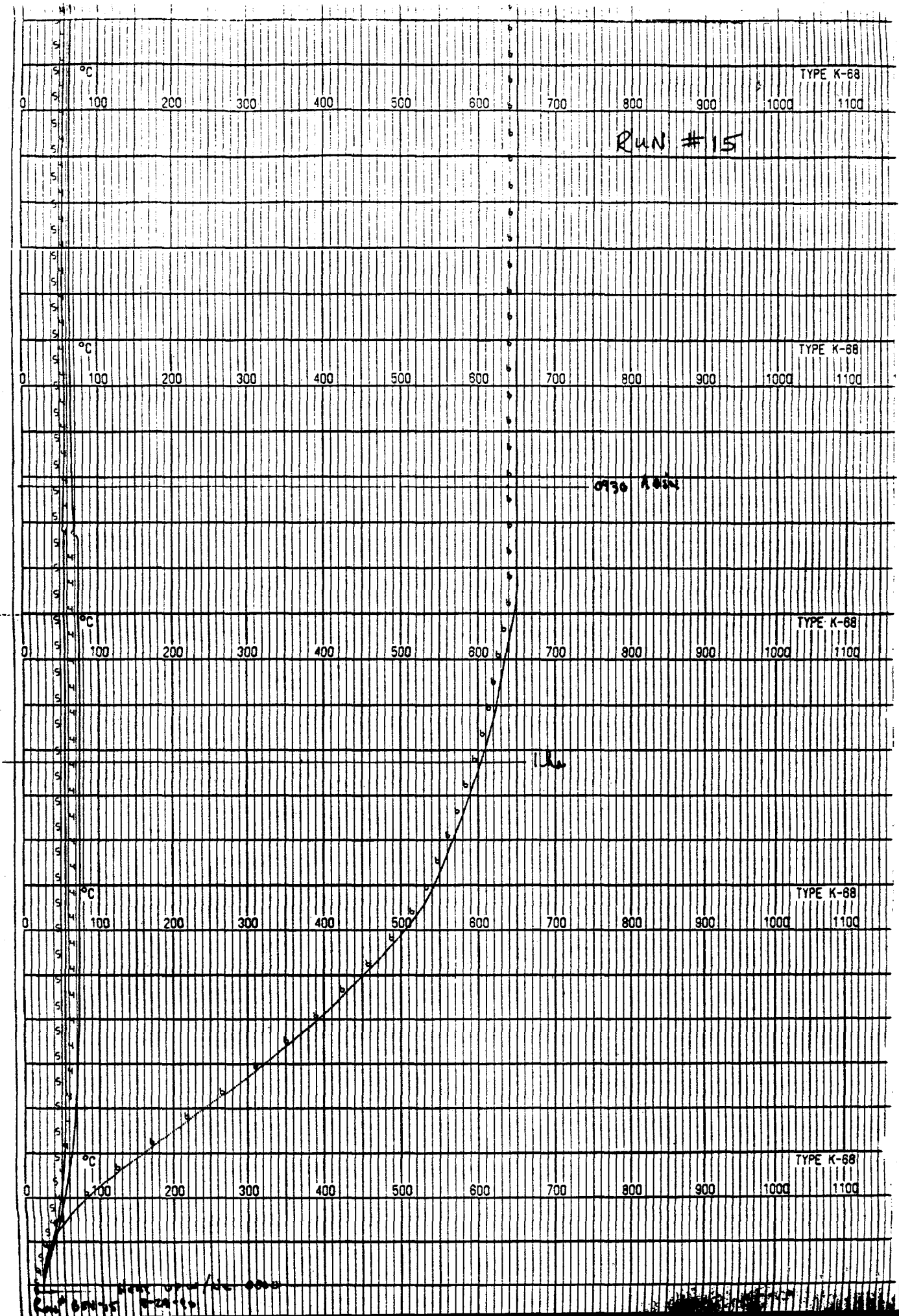
1015 — 1016 Chart on @ 1 in/hr during cool down w/ ice

100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90

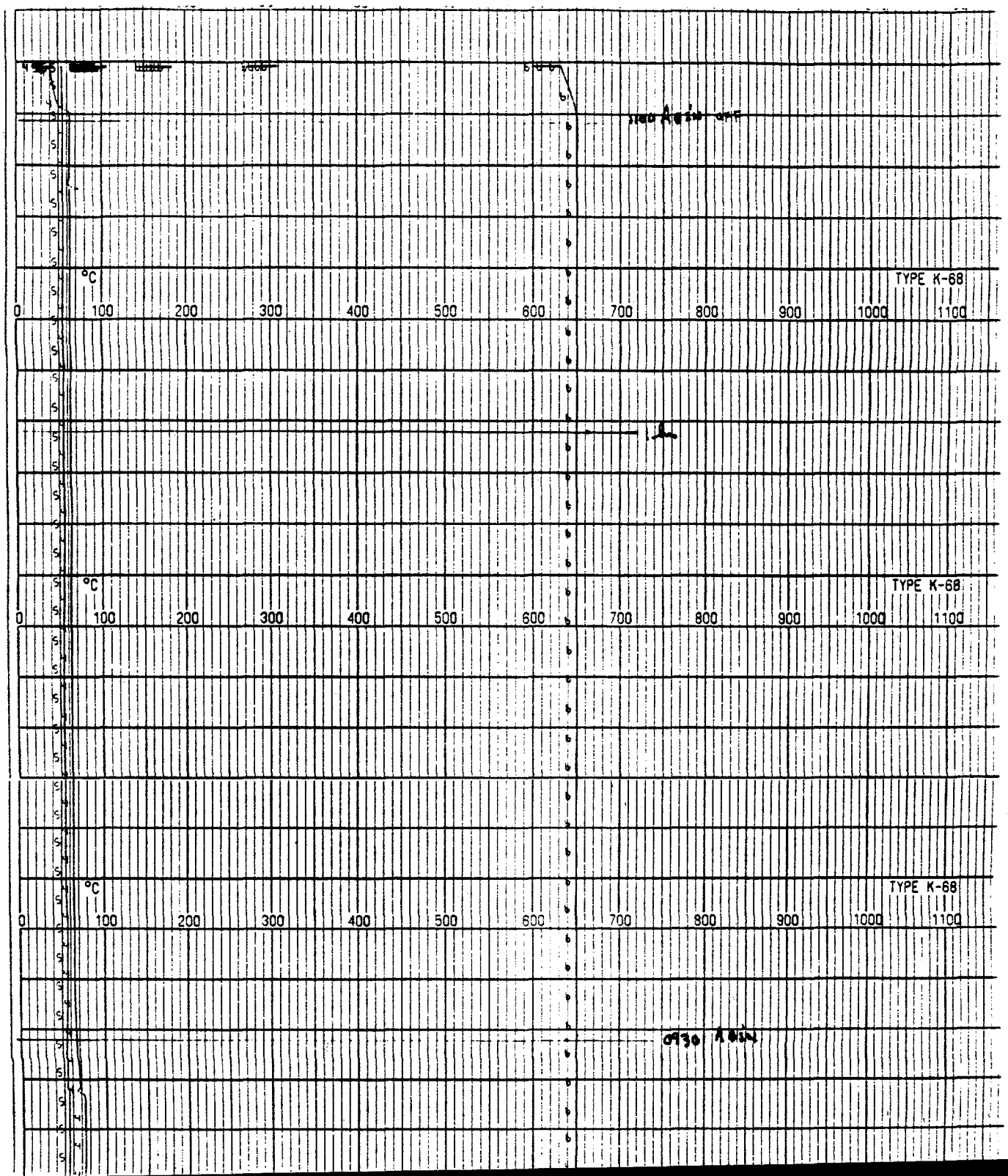
(Units/min)

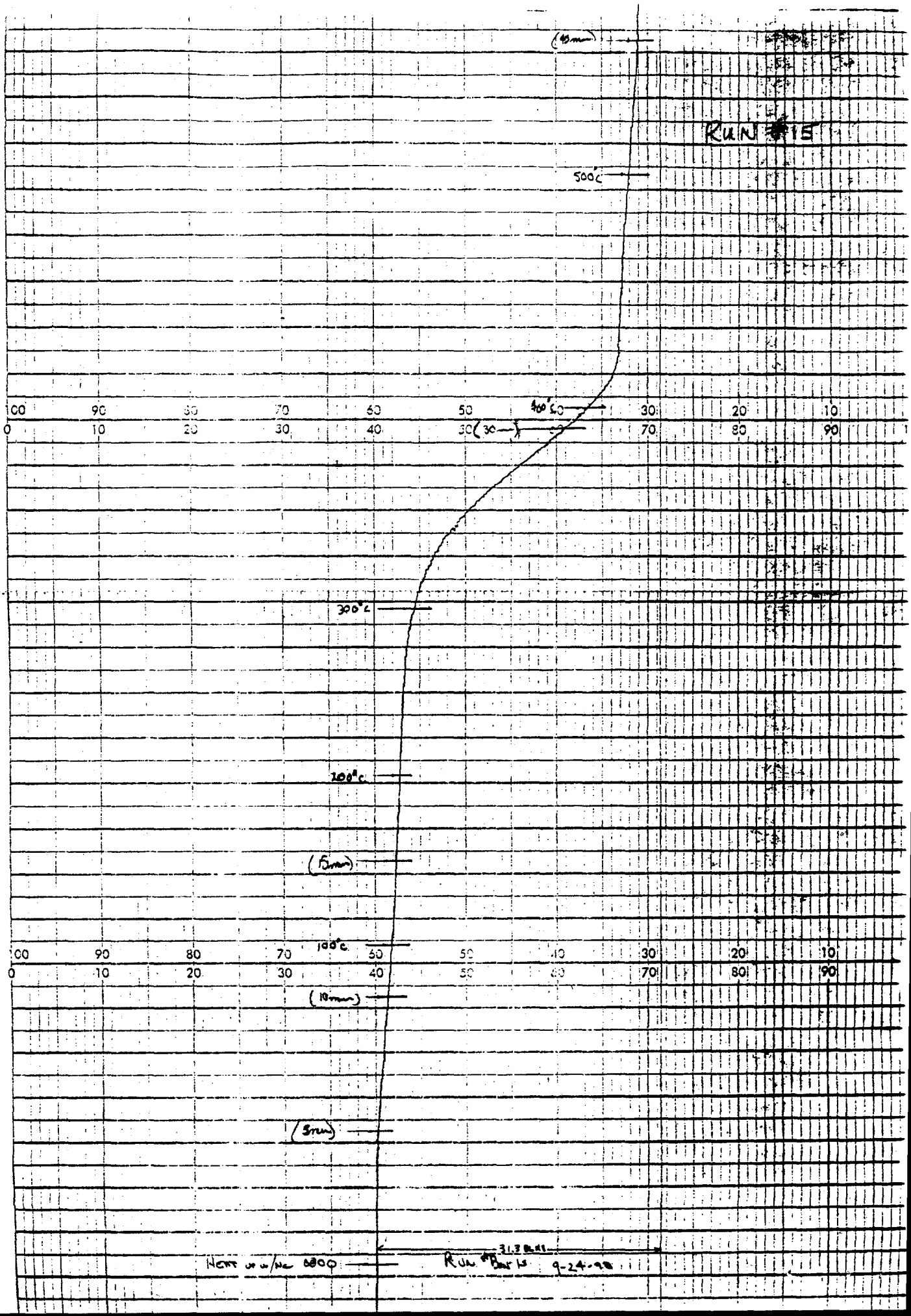
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90

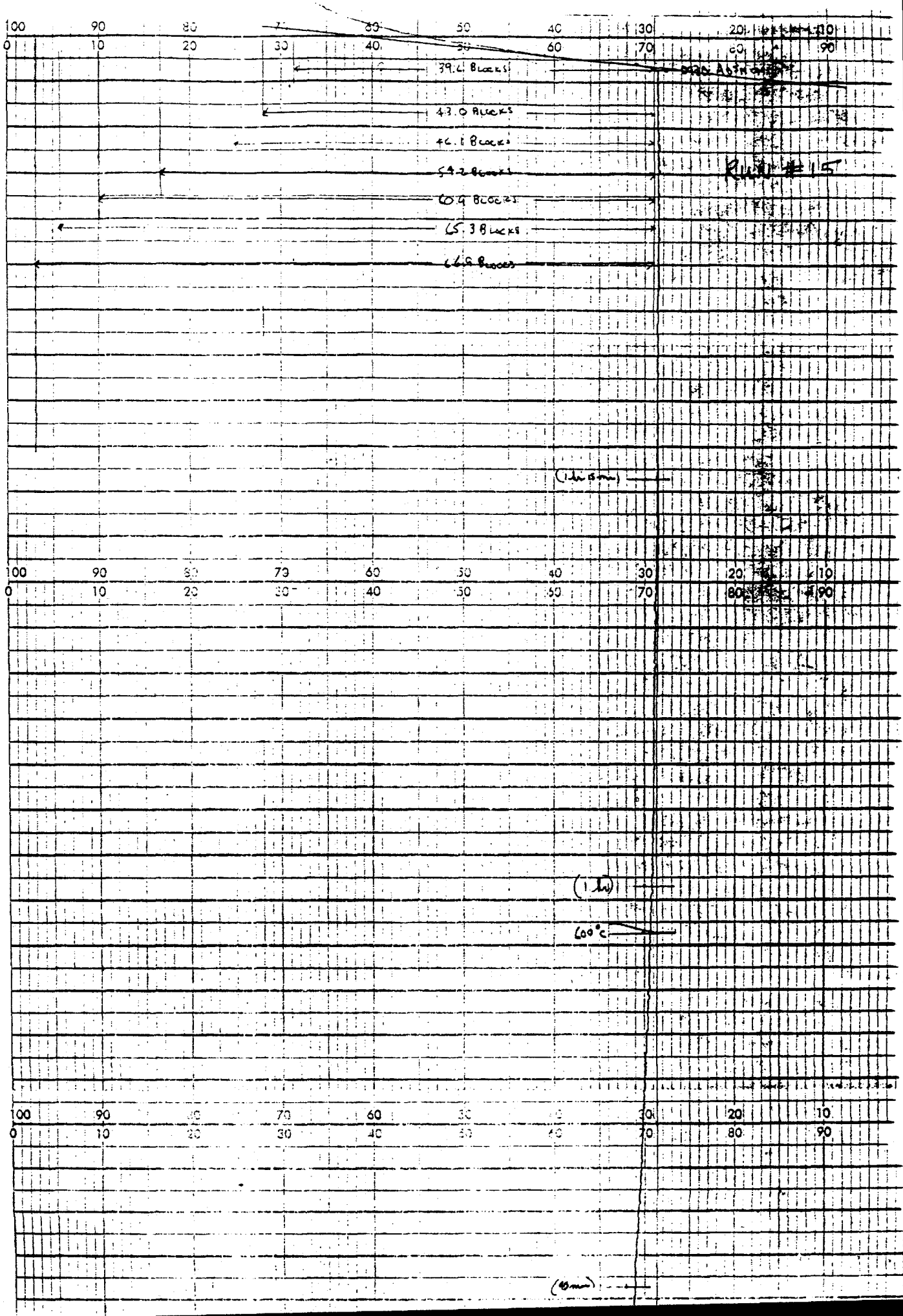
(Units)

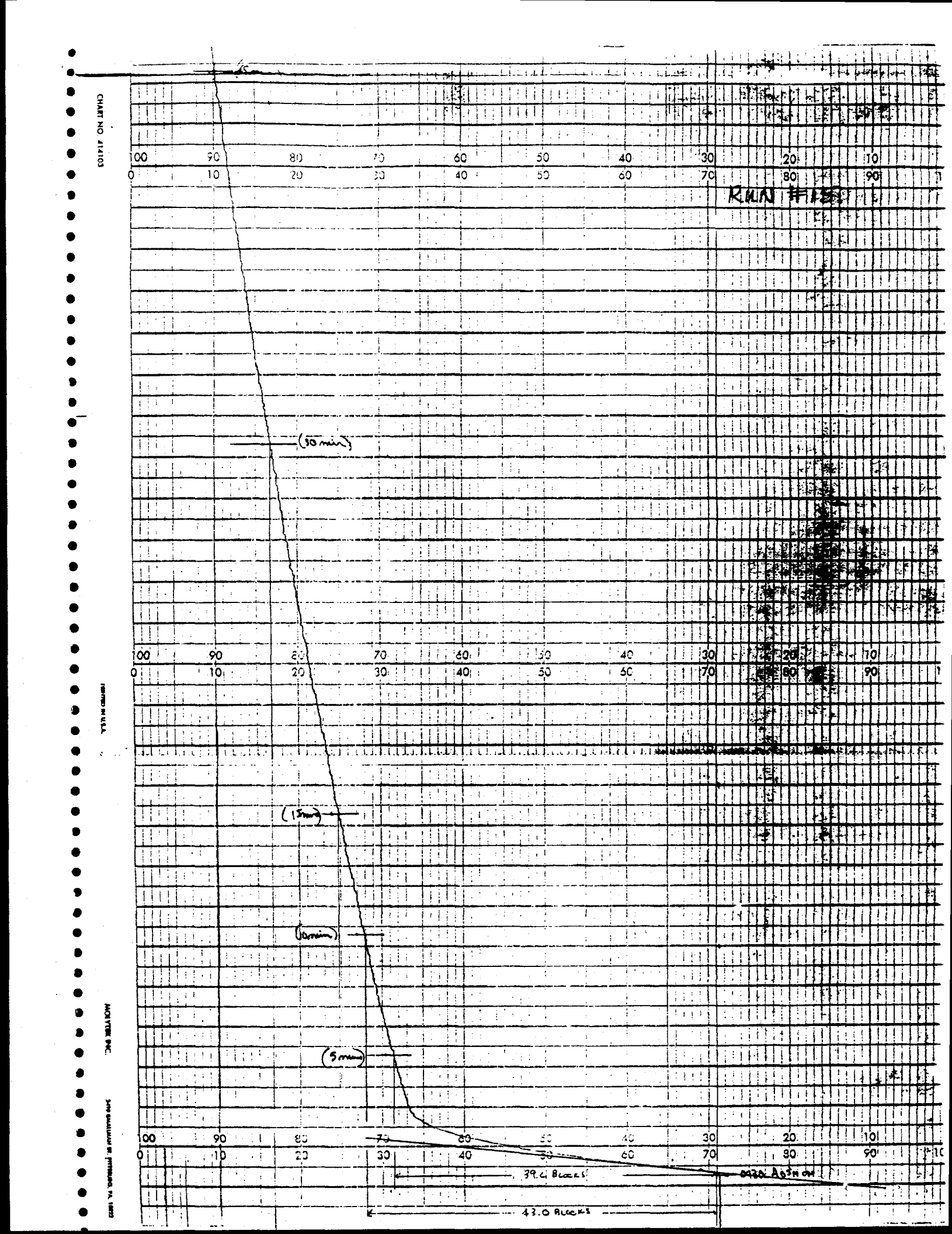

°C
TYPE K-68
0
100
200
300
400
500
600
700
800
900
1000
1100
RUN #15
0930 hrs
1hr

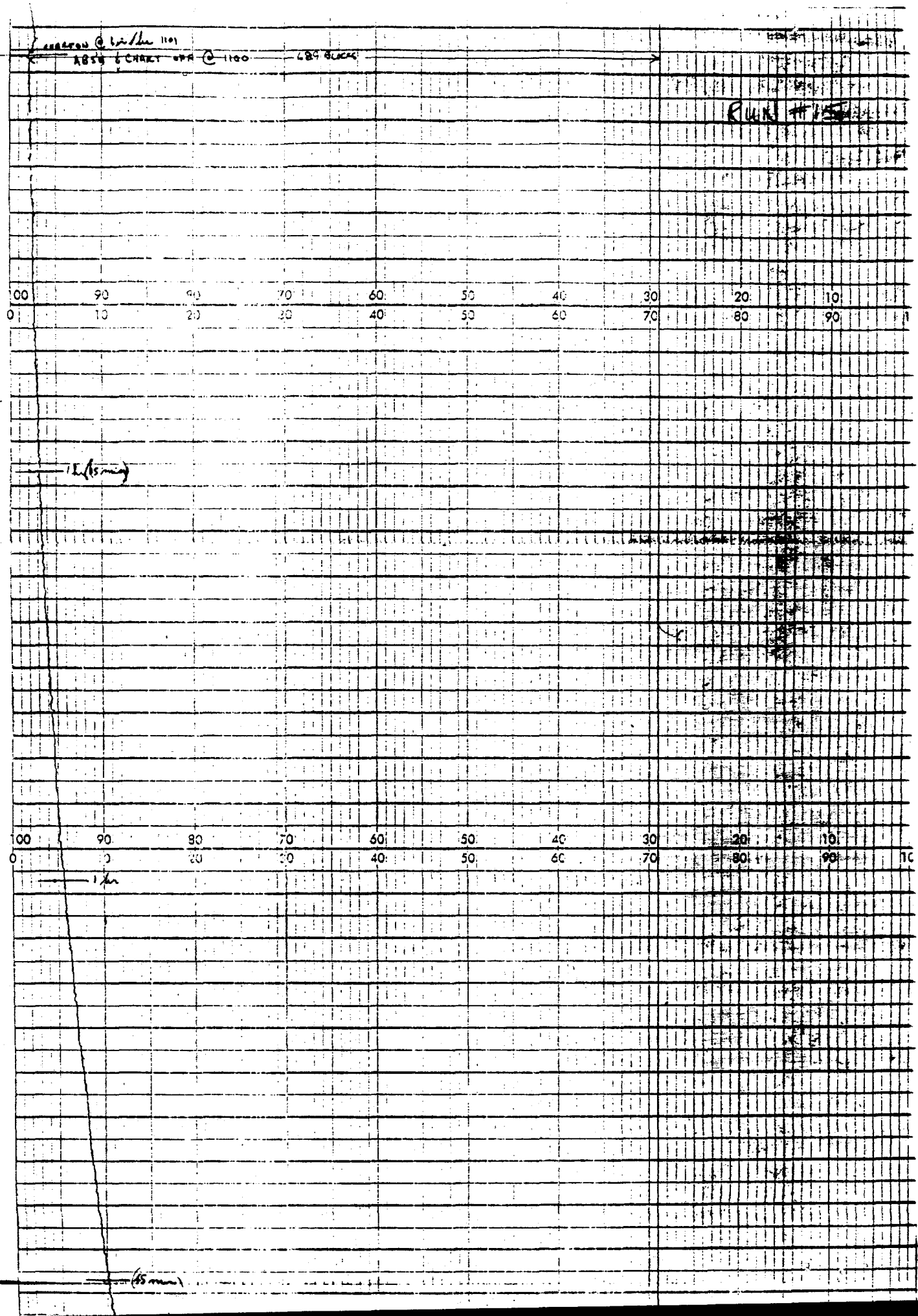
RUN #15



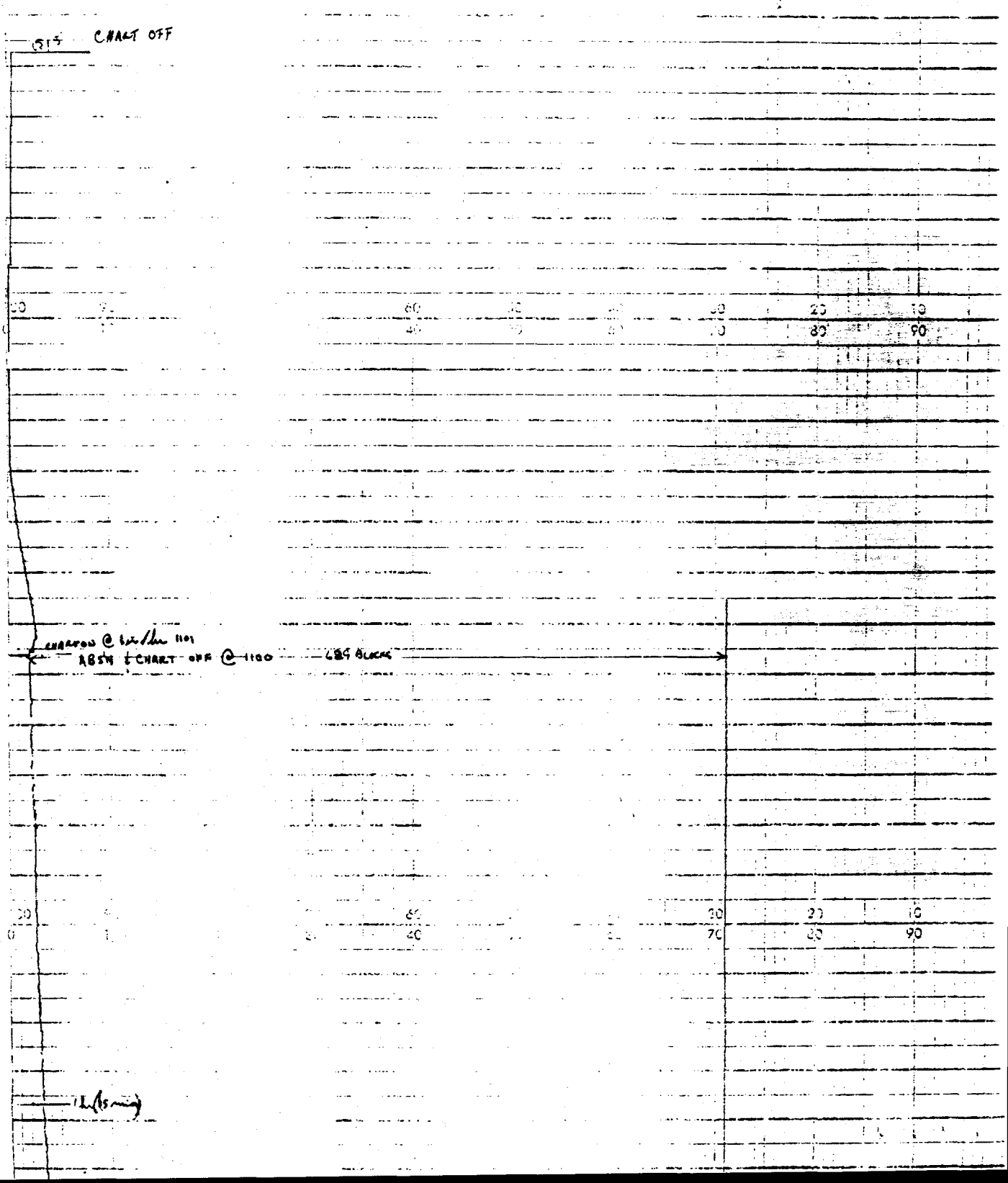


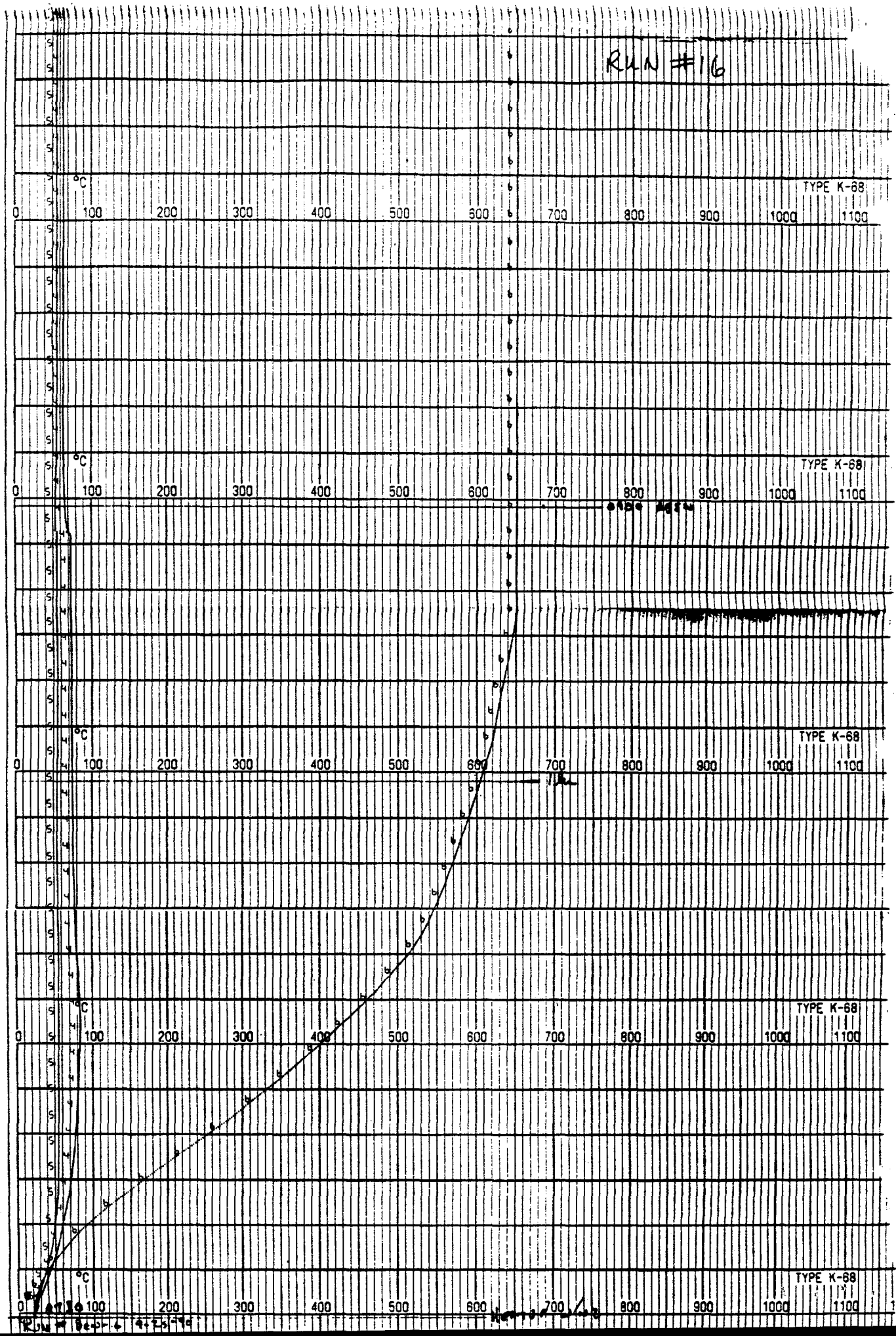

100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90
39.4 Blocks
43.0 Blocks
46.6 Blocks
54.2 Blocks
60.4 Blocks
65.3 Blocks
66.8 Blocks
Run #15
(1 hr 15 min)
(1 hr)
600°C
(15 min)


RUN #15
(30 min)
(15 min)
(10 min)
(5 min)
39.4 BLOCKS
43.0 BLOCKS


ABSN & CHART OFF @ 1100
489 BLOCKS
RUN #15
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90 1
1 hr (15 mm)
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70 80 90 10
1 hr
(15 mm)

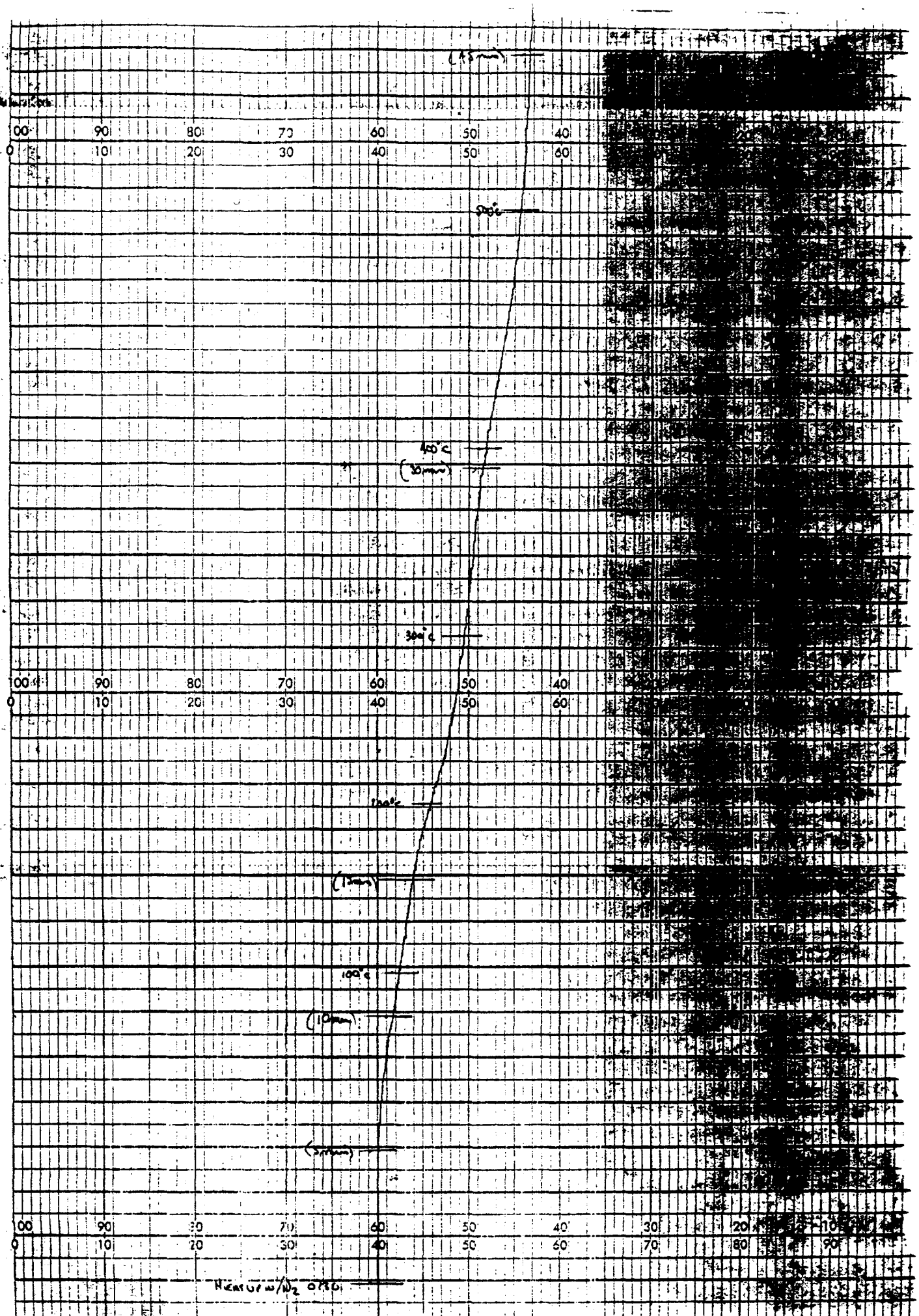
RUN #15

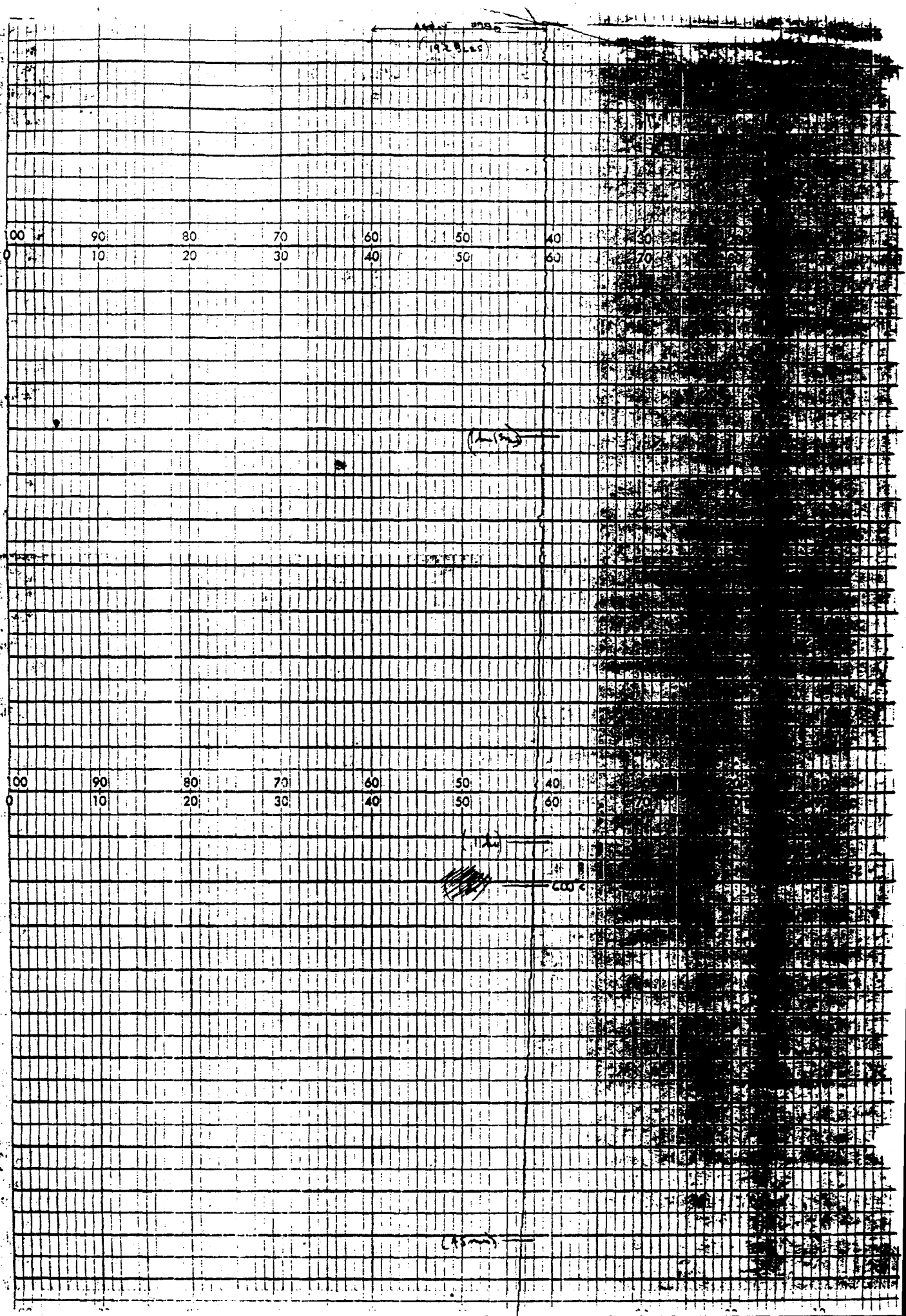


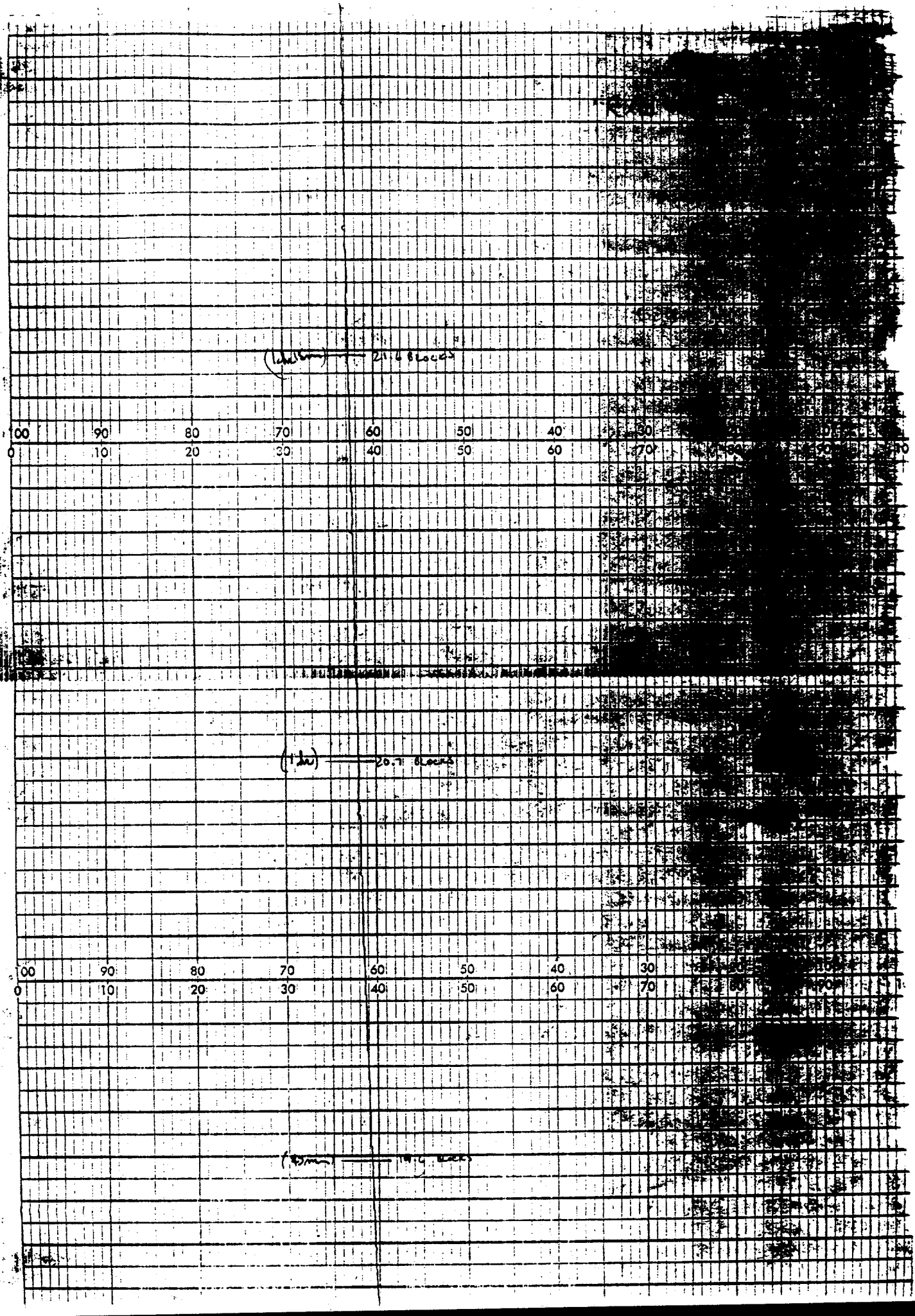

RUN #16
°C
TYPE K-68
0
100
200
300
400
500
600
700
800
900
1000
1100
LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A. NO. 545042

RUN #16

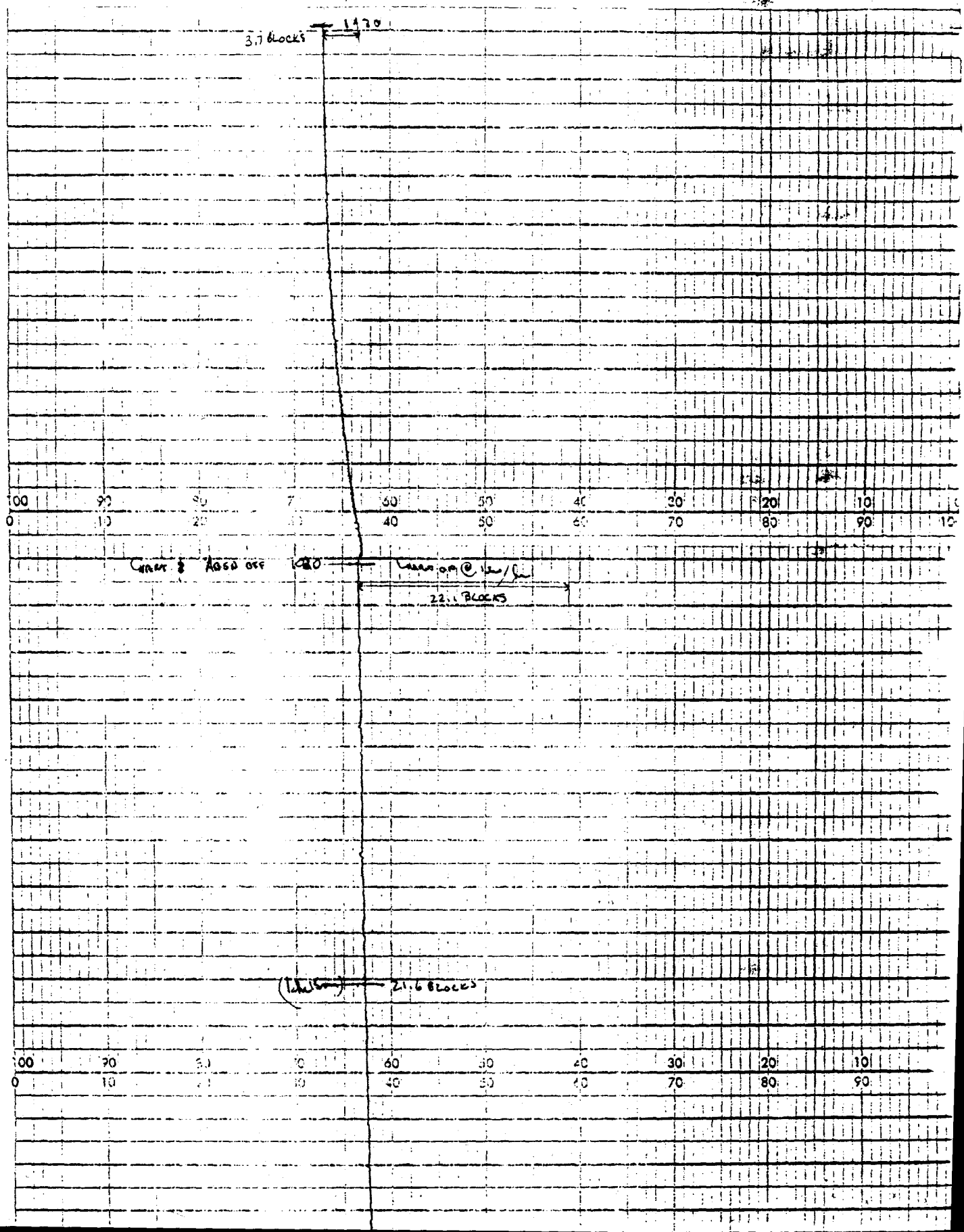
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
TYPE K-68
NO. 645042 LEEDS & NORTHRUP CO., NORTH WALES, PA. MADE IN U.S.A.

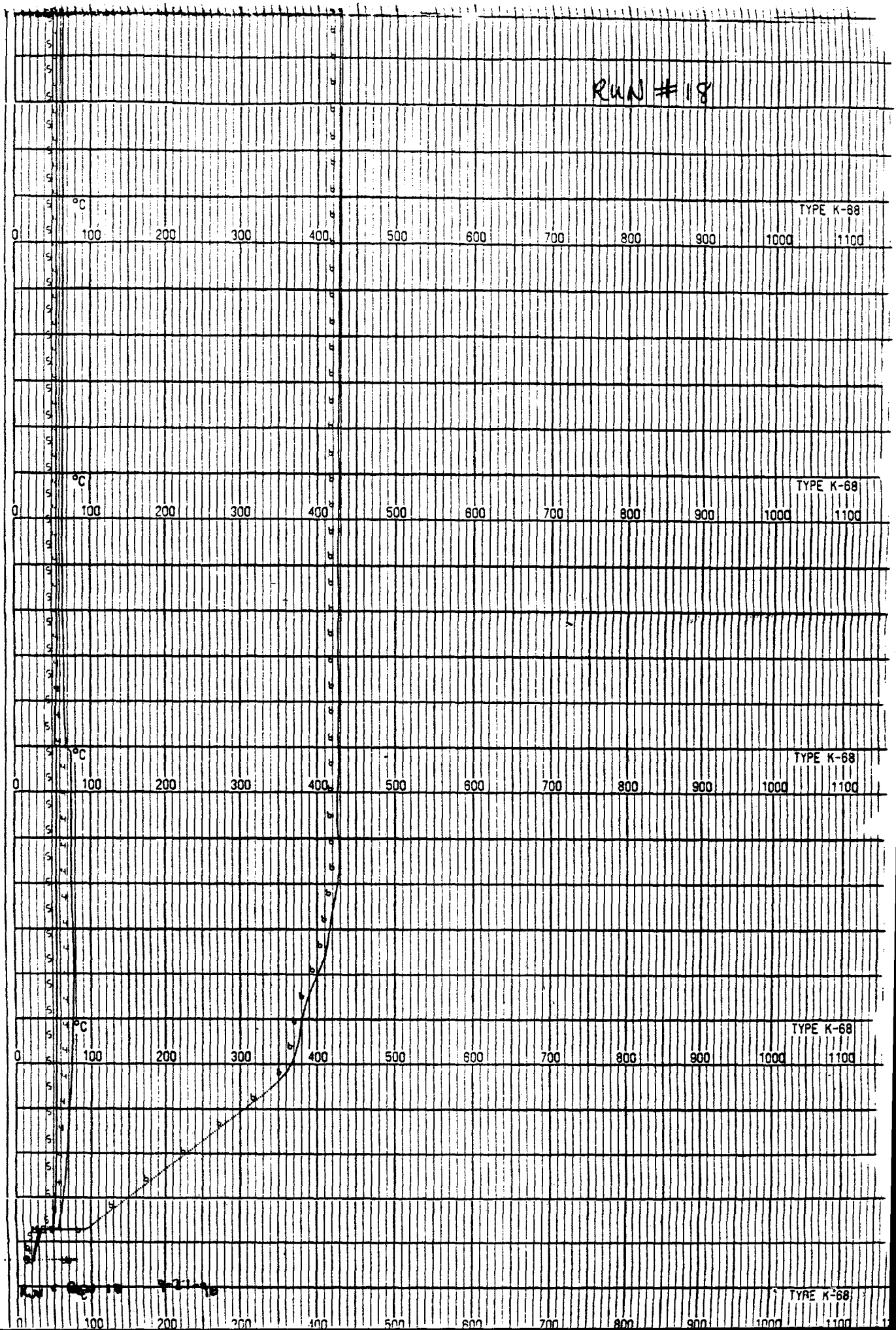

(45 min)
500°C
400°C
(30 min)
300°C
100
90
80
70
60
50
40
30
20
10
0
1100°C
(15 min)
100°C
(10 min)
(5 min)
Heating rate 0.75°C/min

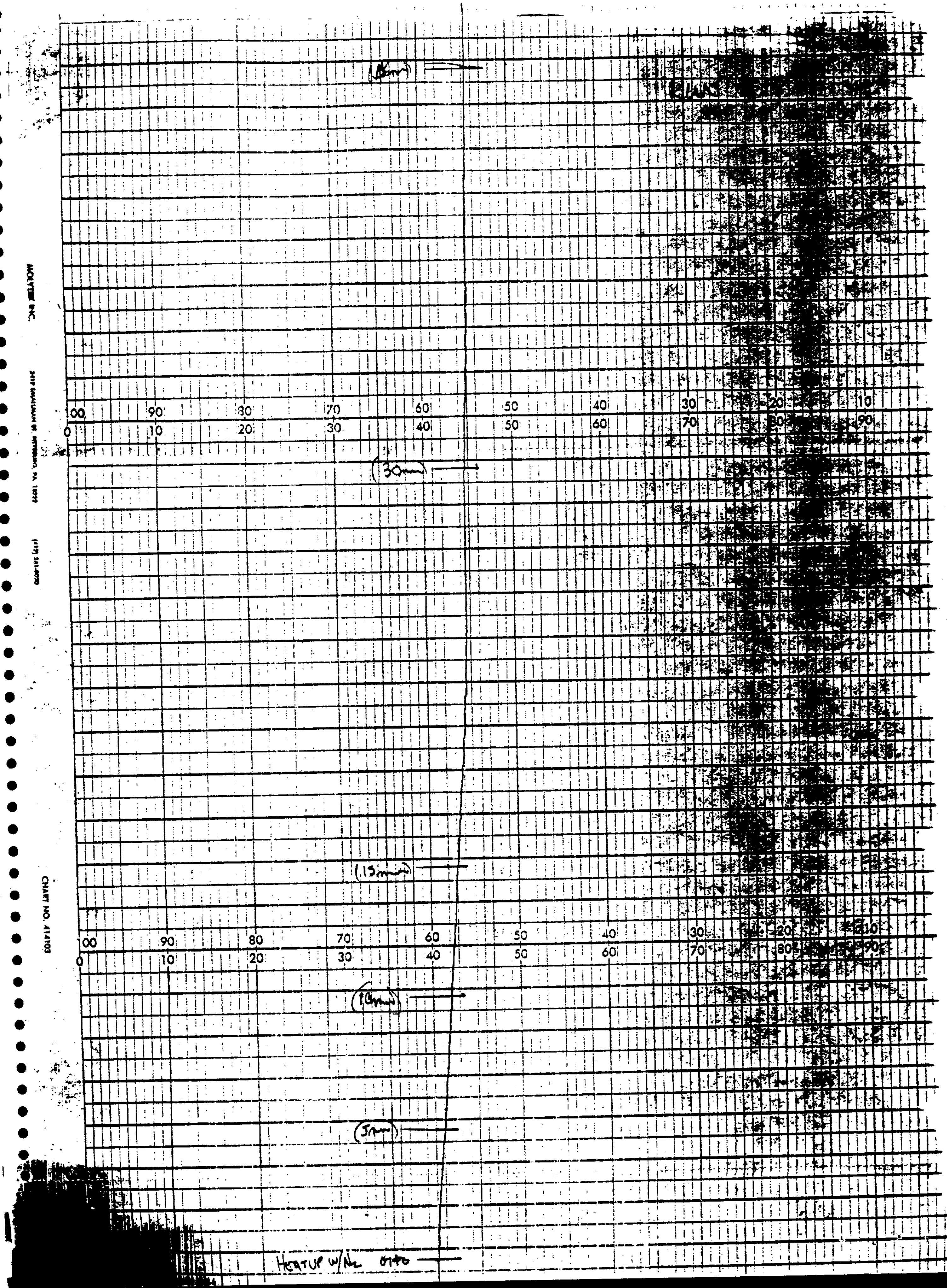

100 90 80 70 60 50 40 30
0 10 20 30 40 50 60 70
100 90 80 70 60 50 40
0 10 20 30 40 50 60 70


100 90 80 70 60 50 40 30
0 10 20 30 40 50 60 70
100 90 80 70 60 50 40 30
0 10 20 30 40 50 60 70

RUN #16


3.7 BLOCKS
1970
100 90 80 70 60 50 40 30 20 10 0
0 10 20 30 40 50 60 70 80 90 100
CHART 2 ADDED OFF
1980
22.1 BLOCKS
21.6 BLOCKS
100 90 80 70 60 50 40 30 20 10 0
0 10 20 30 40 50 60 70 80 90




MOLYTEK INC.
CHART NO. 414103
(30min)
(15min)
(5min)

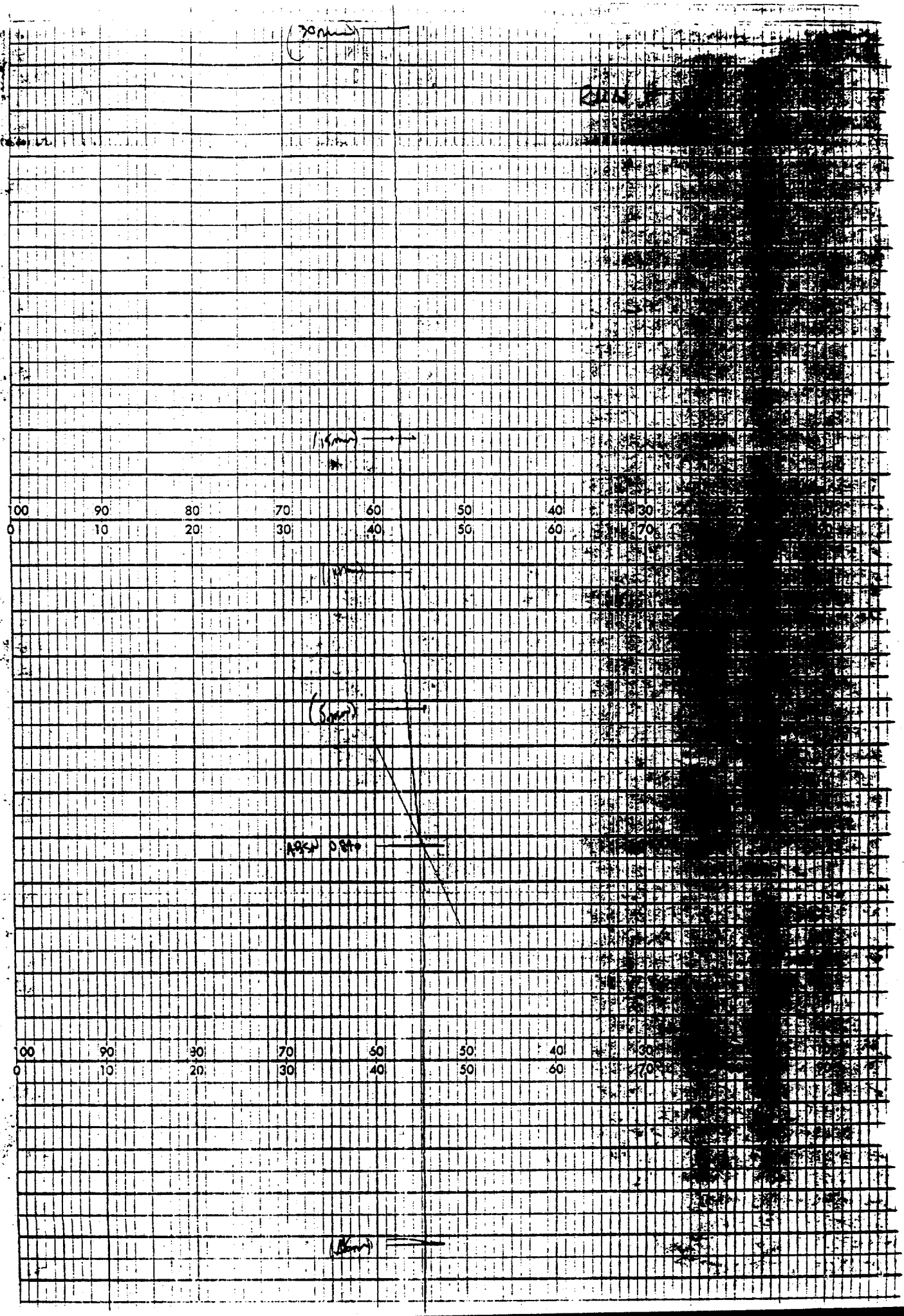
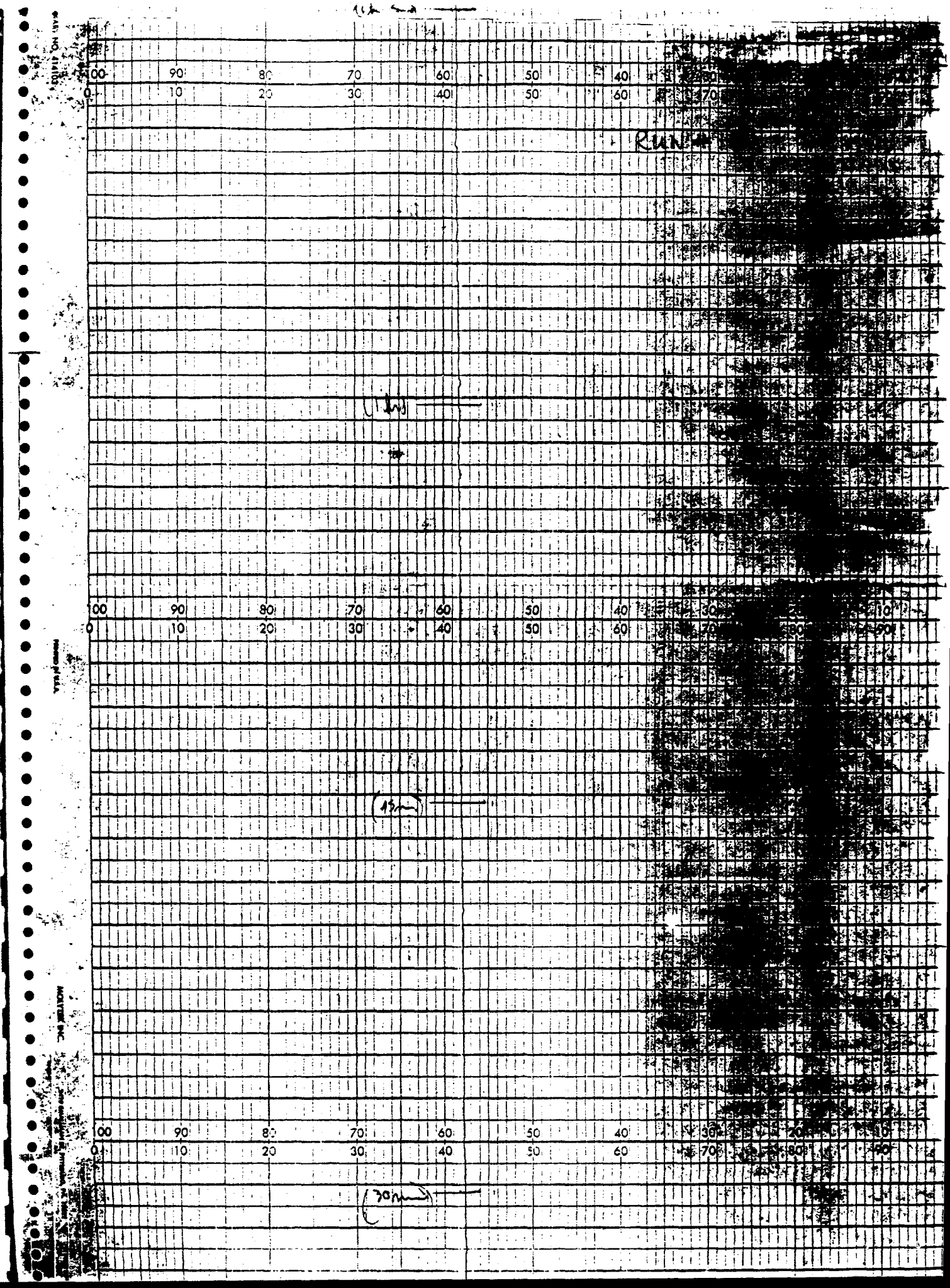
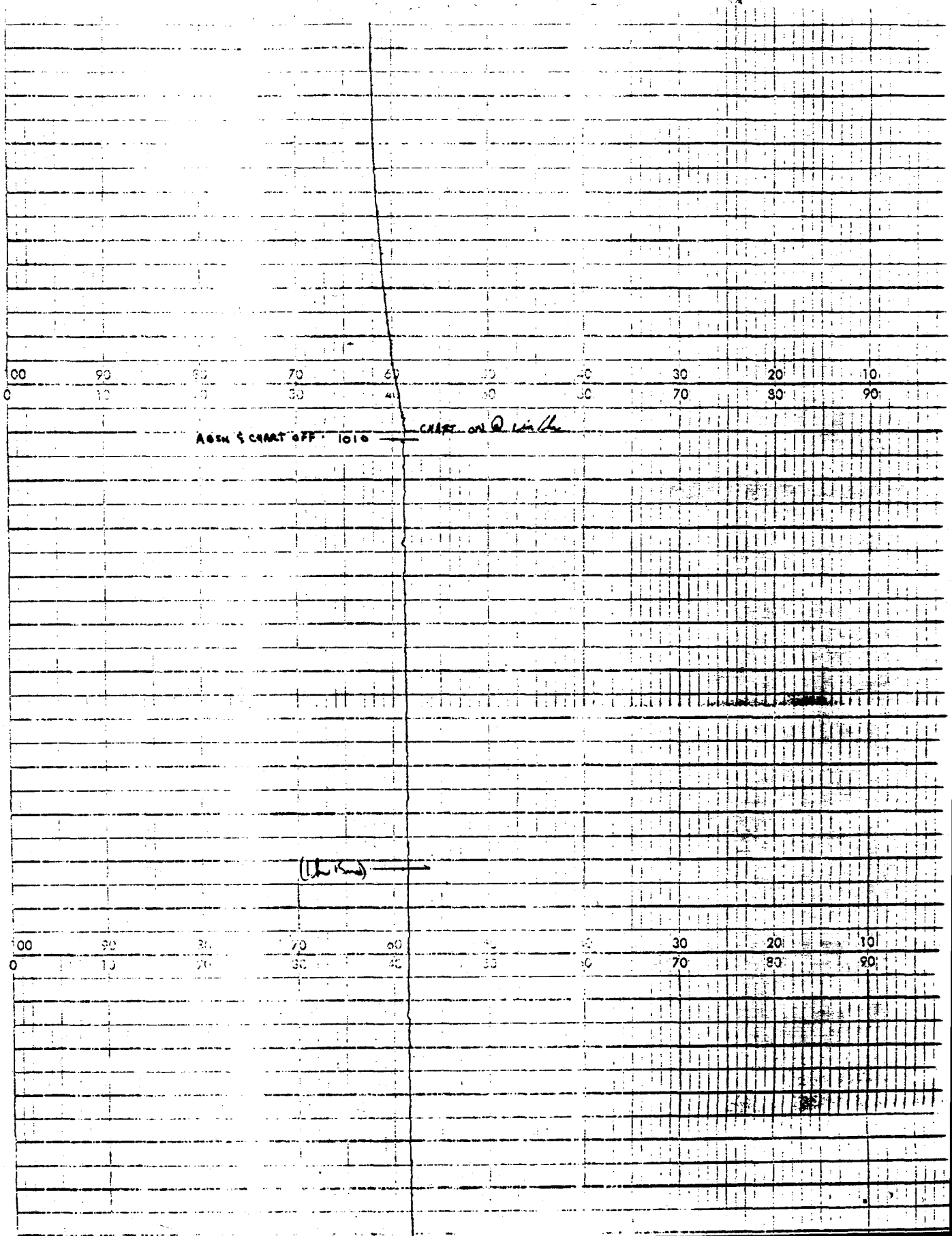


CHART NO. 414103
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70
100 90 80 70 60 50 40 30 20 10
0 10 20 30 40 50 60 70


RUN #

RUN #18



Appendix B

Sample Calculations

Run #11

Test Conditions:

SORCAT B5

50.13 mg initial sorbent weight

427 °C reactor temperature

56 °C bubbler temperature

1480 sccm dry gas flow rate

Recorder full-scale deflection - 50 mg

Test Results:

1) Heat-Up Period (65 minute duration)

Recorder reading: Initial - 50.1 blocks

End - 37.1 blocks

$$(37.1 - 50.1 \text{ blocks})\left(0.5 \frac{\text{mg}}{\text{block}}\right) = -6.5 \text{ mg}$$

$$\frac{-6.5 \text{ mg}}{50.13 \text{ mg}} \times 100 = -12.97 \% \text{ weight loss}$$

2) Absorption Period (90 minute duration)

Recorder readings: Initial - 37.1 blocks

End - 52.5 blocks

$$(52.5 - 37.1 \text{ blocks})\left(0.5 \frac{\text{mg}}{\text{block}}\right) = 7.70 \text{ mg}$$

$$\frac{7.70 \text{ mg}}{50.13 \text{ mg}} \times 100 = 15.36 \% \text{ weight gain}$$

3) Absorption weight gain at 45 minutes

Recorder readings: Initial - 37.1 blocks

End - 49.0 blocks

$$(49.0 - 37.1 \text{ blocks})\left(0.5 \frac{\text{mg}}{\text{block}}\right) = 5.95 \text{ mg}$$

$$\frac{5.95 \text{ mg}}{50.13 \text{ mg}} \times 100 = 11.87\ \% \text{ weight gain}$$

4) Absorption weight gain at 60 minutes

Recorder readings: Initial - 37.1 blocks

End - 50.6 blocks

$$(50.6 - 37.1 \text{ blocks})\left(0.5 \frac{\text{mg}}{\text{block}}\right) = 6.75 \text{ mg}$$

$$\frac{6.75 \text{ mg}}{50.13 \text{ mg}} \times 100 = 13.46\% \text{ weight gain}$$

5) Initial absorption rate

Points of best straight line through initial data:

For 1 inch of chart, 29.7 to 39.9 blocks

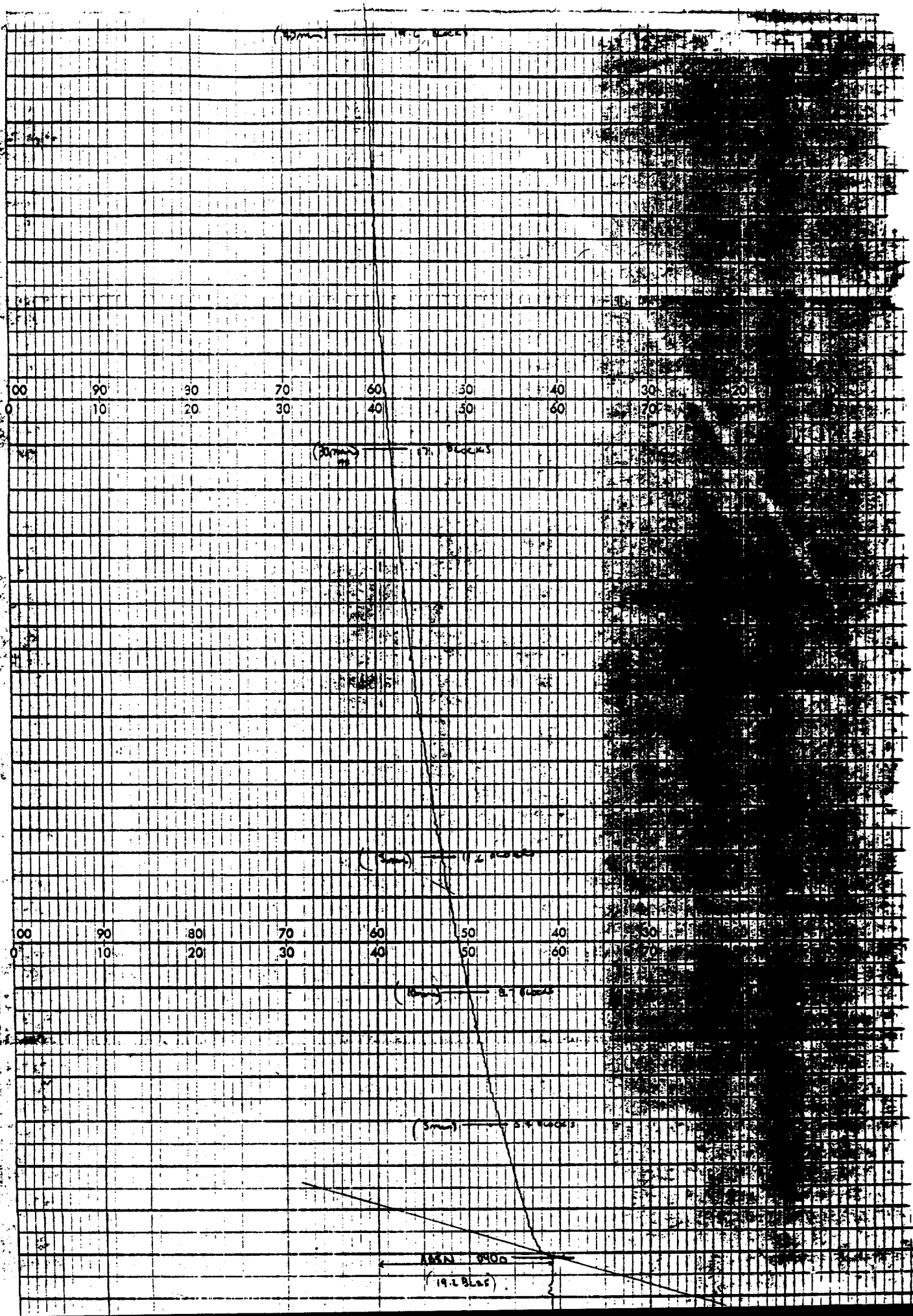
$$\frac{(39.9 - 29.7 \text{ blocks})(0.5 \text{ mg/block})}{(1.0 \text{ in.})(60 \text{ min}/18 \text{ in.})} = 1.53 \text{ mg/min}$$

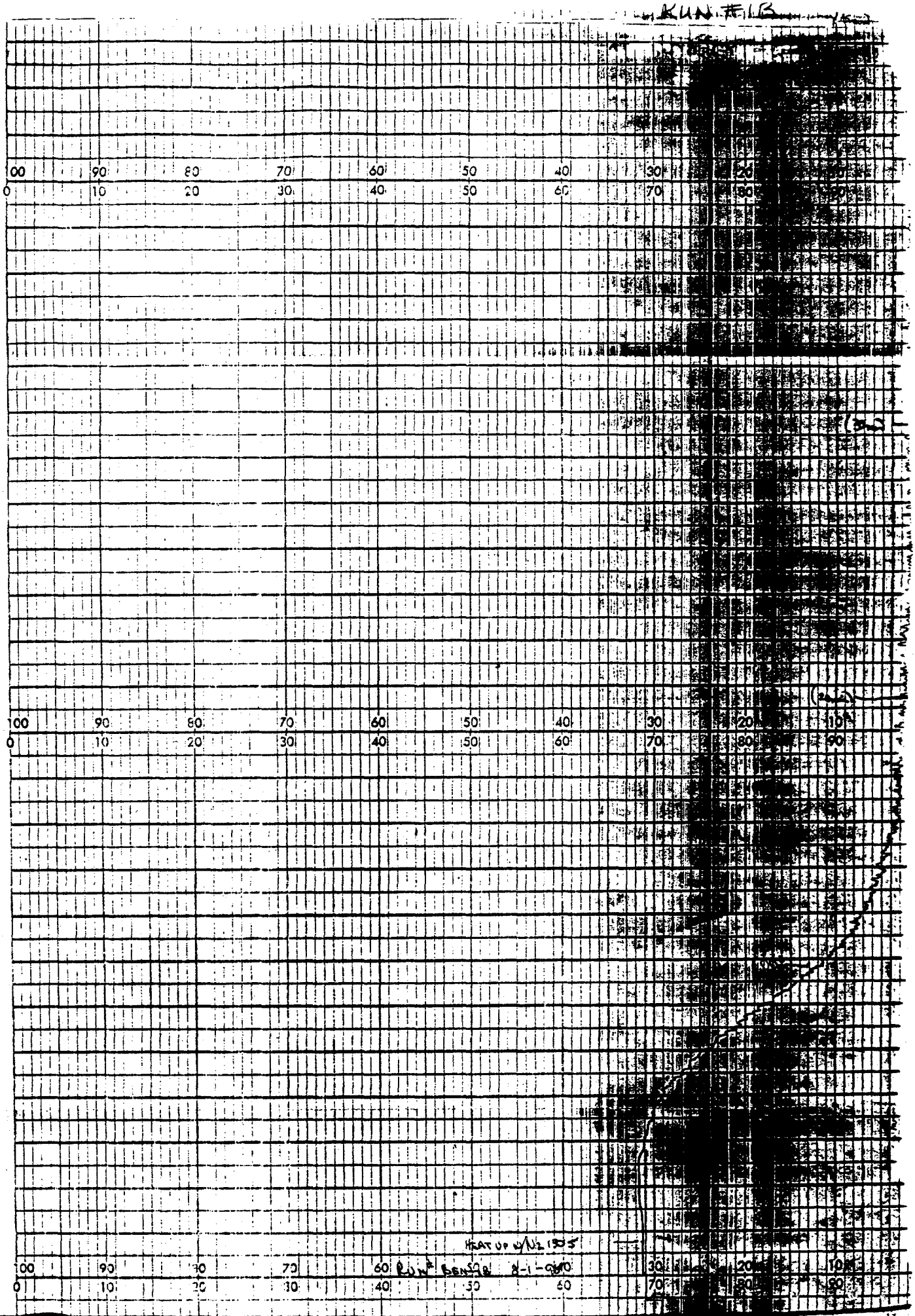
6) 5-minute average absorption rate

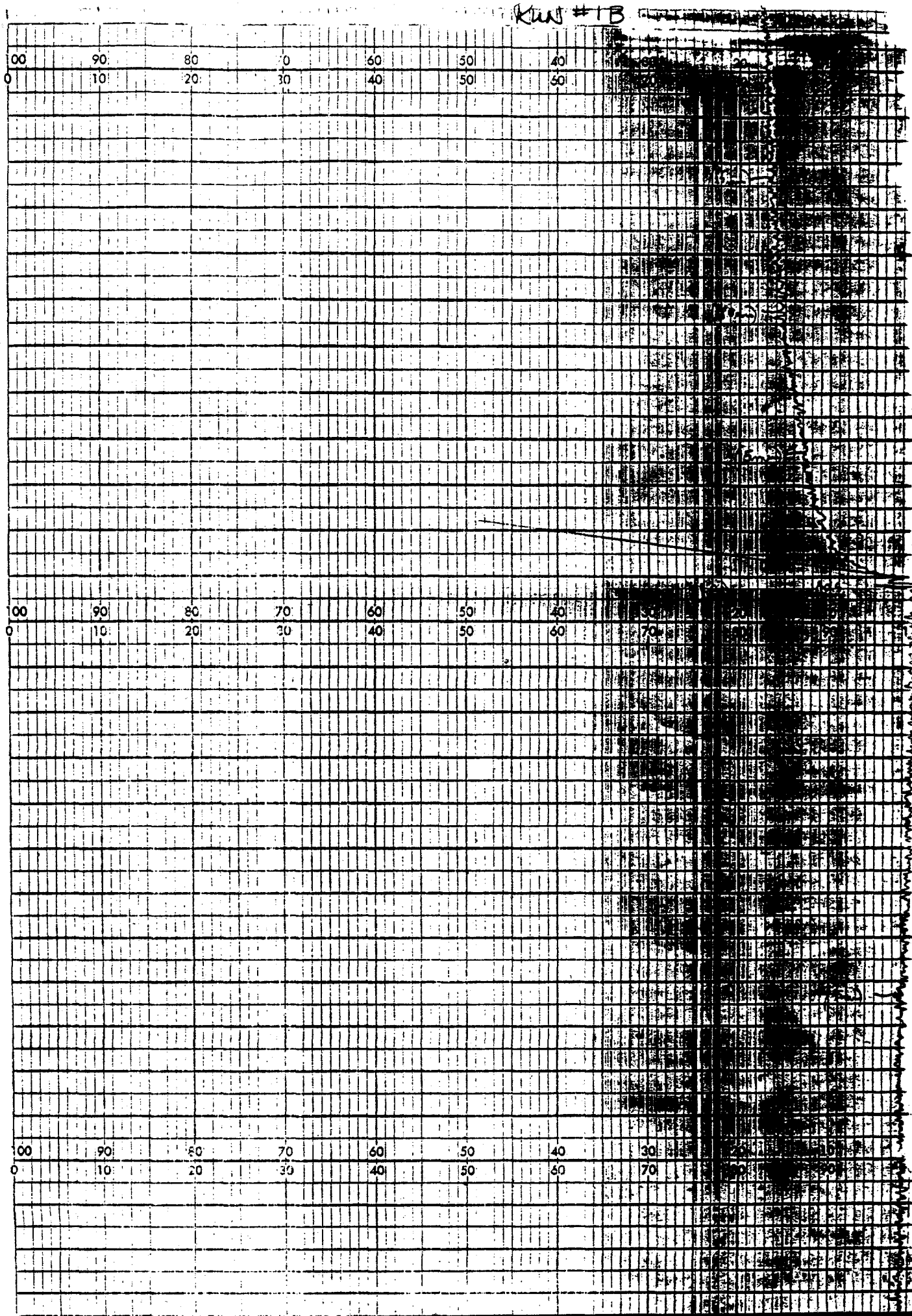
Recorder readings: Initial - 37.1 blocks

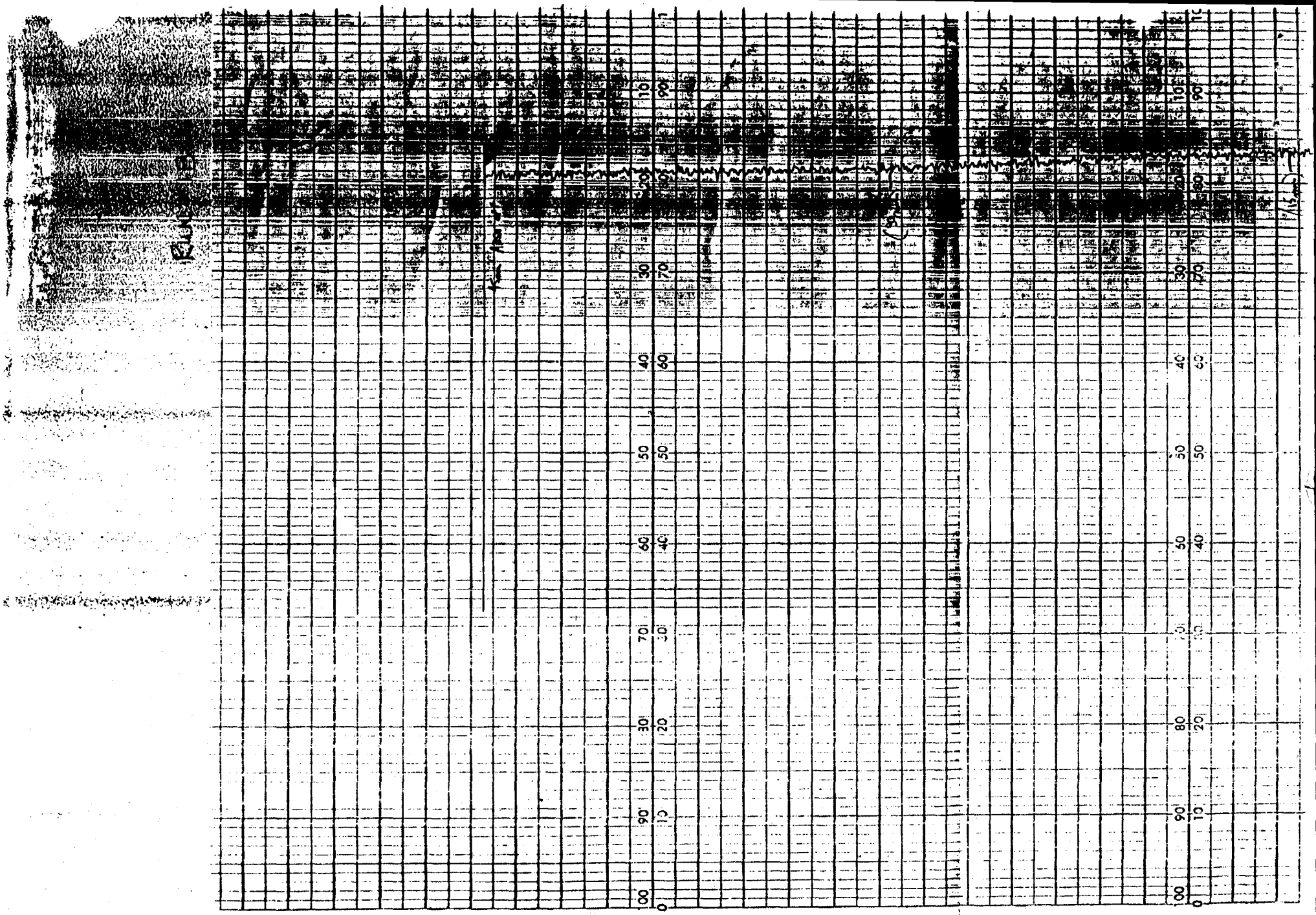
5 min - 40.5 blocks

$$\frac{(40.5 - 37.1 \text{ blocks})(0.5 \text{ mg/block})}{(5 \text{ min})} = 0.34 \text{ mg/min}$$


(45 min) ——— W.L. Blocks
100 90 80 70 60 50 40 30 20
0 10 20 30 40 50 60 70
(30 min) ——— W.L. Blocks
(5 min) ——— W.L. Blocks
(10 min) ——— 8.7 Blocks
(3 min) ——— 5.8 Blocks
ABSN 0900
(19.2 Blocks)




KUN #1B



MOLYTEK INC. 2450 SMALLMAN ST. PITTSBURG, PA. 15222 (412) 261 9030

CHART NO 414103