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Prepared under Contract No. NAS 8-20073 by

J. P. Evans and K. G. Scrogham

BROWN ENGINEERING COMPANY

For

NASA-GEORGE C. MARSHALL SPACE FLIGHT CENTER
Huntsville, Alabama

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TECHNICAL REPORT BSVD-M-68-TR-001

EVALUATION OF ANALYTICAL STANDARDS BY DIFFERENTIAL THERMAL ANALYSIS AND DIFFERENTIAL SCANNING CALORIMETRY

BY

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ABSTRACT

The effects of heating rate and furnace atmosphere on four analytical standards were investigated by DTA and DSC. The results are presented in both graphical and tabular form.

TABLE OF CONTENTS

LIST OF TABLES

EVALUATION OF ANALYTICAL STANDARDS BY DIFFERENTIAL THERMAL ANALYSIS AND DIFFERENTIAL SCANNING CALORIMETRY

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SUMMARY

Differential thermal analyses have been run on copper sulfate pentahydrate, calcium oxalate monohydrate, potassium nitrate and silver nitrate under the conditions requested. Thermograms are included for each of the analyses.

DSC runs were also made on the copper, potassium, and silver salts. Heats of inversion and fusion were determined for potassium nitrate and silver nitrate and comparison made between experimental and literature values.

INTRODUCTION

Several factors are known to affect differential thermal analysis results. These factors are dependent on two types of variables, instrumental and sample characteristics. The effects of two of the instrumental factors, heating rate and furnace atmosphere, on four inorganic salts were investigated. Copper sulphate pentahydrate, calcium oxalate monohydrate, potassium nitrate, and silver nitrate were analyzed on the DuPont 900 Differential Thermal Analyzer using standard procedures.

These same salts with the exception of calcium oxalate monohydrate, were also analyzed on the Perkin-Elmer Differential Scanning Calorimeter. DTA results were compared with the DSC results.

The heats of fusion and inversion for potassium nitrate and the heat of fusion of silver nitrate were determined at a heating rate of $5^{o}C$. /min. using the Perkin-Elmer Differential Scanning Calorimeter. These data were compared with available literature values.

The standard analytical compounds used in this study were supplied by Sadtler Research Laboratories, Philadelphia, Pennsylvania.

EXPERIMENTAL

Differential Thermal Analysis

The DuPont 900 Differential Thermal Analyzer was used for all DTA experiments. The instrument was equipped with the Standard DTA Cell containing the heating block for micro (2 mm) sample tubes.

In all analyses the following experimental procedure was followed:

1) The sample tube was tared and the sample introduced.

2) The sample and sample tube were weighed ($\pm$ 0.05 mg).

3) The sample tube was then tapped until the sample occupied all visible voids.

4) The thermocouple was inserted into the center of the sample and the sample tube was placed in the heating block of the Cell.

5) The procedure for preparation of the reference material (Al_2O_3) was identical to that above.

6) For those runs requiring a dynamic atmosphere, the Cell was purged for at least fifteen minutes with dry nitrogen gas prior to the start of the analysis.

The specifics for the analysis of each material are outlined below.

1) <u>Copper sulfate pentahydrate</u>

a) Run at 5, 10, and 15^{o}C. /minute rate of rise from ambient to 500^{o}C. in static atmosphere (air).

b) Run at 5, 10, and 15^{o}C. /minute rate of rise from ambient to 500^{o}C. in dynamic atmosphere (N_2 at 1 SCFH for envelope).

c) All sample weights were 1.15 mg $\pm$ 0.05 mg.

2) <u>Calcium oxalate monohydrate</u>

a) Run at 5, 10, and 15^{o}C. /minute rate of rise from ambient to 500^{o}C. in dynamic atmosphere (N_2 at 1 SCFH for envelope).

b) Run at 5, 10, and 15^{o}C. /minute rate of rise from ambient to 500^{o}C. in dynamic atmosphere (air at 1 SCFH for envelope).

c) All sample weights were 1.60 mg $\pm$ 0.15 mg.

3) <u>Potassium nitrate</u>

a) Run at 5^{o}C. /minute rate of rise from ambient to 350^{o}C. in static atmosphere (air). Sample was allowed to cool to ambient temperature with thermocouple in place and rerun under identical experimental conditions.

b) Procedure a) was repeated with a new sample for heating rates of 10 and 15°C. /minute.

c) All sample weights were 1.65 mg.

4) Silver nitrate

a) Run at 5°C. /minute rate of rise from ambient to 250°C. in static atmosphere (air). Sample was allowed to cool to ambient temperature with thermocouple in place and rerun under identical experimental conditions.

b) Procedure a) was repeated with a new sample for heating rates of 10 and 15°C. /minute.

c) All sample weights were 3.40 mg ± 0.05 mg.

Differential Scanning Calorimetry

The Perkin-Elmer DSC-1 Differential Scanning Calorimeter was used for all DSC experiments. The instrument was calibrated at each heating rate using indium, tin, and lead. The melting points of these materials are 429°K., 505°K., and 600°K. respectively. Calibration was carried out to within 1° of these temperatures.

In all analyses the following experimental procedure was followed:

1) The sample pan was tared and the sample introduced.

2) The sample and sample pan were weighed ($\pm$ 0.05 mg).

3) The pan lid was crimped into place as per the manufacturer's instructions.

4) The reference side of the cell contained an empty pan and lid for all runs.

5) For those runs requiring a dynamic atmosphere, the cell was purged for at least fifteen minutes with dry nitrogen gas prior to the start of the analysis.

The specifics for the analysis of each material are outlined below.

1) <u>Copper sulfate pentahydrate</u>

a) Run at 5, 10, and 20°C./minute rate of rise from ambient to 500°C. in static atmosphere (air).

b) Run at 5, 10, and 20°C./minute rate of rise from ambient to 500°C. in dynamic atmosphere (N_2 at 30 cc/minute for envelope).

c) All sample weights were 3.5 mg $\pm$ 0.1 mg.

2) <u>Potassium nitrate</u>

a) Run at 5°C./minute rate of rise from ambient to 350°C. in static atmosphere (air). Sample was allowed to cool to ambient temperature in the cell and rerun under identical experimental conditions.

b) Procedure a) was repeated with a new sample for heating rates of 10 and 20°C. /minute.

c) All sample weights were 3.5 mg $\pm$ 0.1 mg.

3) Silver nitrate

a) Run at 5°C. /minute rate of rise from ambient to 250°C. in static atmosphere (air). Sample was allowed to cool to ambient temperature in the cell and rerun under identical experimental conditions.

b) Procedure a) was repeated with a new sample for heating rates of 10 and 20°C. /minute.

c) All sample weights were 3.5 mg $\pm$ 0.1 mg.

Heats of Fusion

The heats of inversion and fusion for potassium nitrate were determined along with the heat of fusion of silver nitrate. These determinations were made in duplicate but only at a heating rate of 5°C. /minute. Lack of "standards" prevented further investigation at higher rates. Hermetically sealed sample pans were used in these experiments to prevent volatilization. Samples were weighed before and after analysis to assure constant weight. Range setting (4x) and chart speed (15 or 30 cm/hr.) were selected to give peak areas in the range 25 to 85 cm^2. All areas were determined using a K & E Compensating Polar

Planimeter, each area being measured at least twice. The arithmetic average of all measurements on each peak was taken as the peak area.

Calibration of the power input to the sample was performed by measurements of the heat of fusion of indium (ΔH_f = 6.80 cal/gm).

RESULTS AND DISCUSSION

DTA

The results of all DTA experiments are tabulated in Tables I through IV and shown on pages 21 through 44 in Appendix A. Analysis of the data on copper sulfate pentahydrate shows the dehydration to be insensitive to either atmosphere or heating rate. This is not the case, however, for the decomposition reaction. An increase in heating rate causes an elevation in peak temperature in both air and nitrogen atmospheres.

Analysis of the calcium oxalate monohydrate data shows the peak temperatures to be relatively insensitive to either atmosphere or heating rate.

Potassium nitrate was run in air and then rerun at each heating rate. The inversion temperature and the melting temperature are insensitive to heating rate and reproducible from run to run.

Data analysis shows this is not the case with silver nitrate. The inversion is permanent and non-reversible and therefore is not observed upon rerunning the salt. The fusion temperature, however, is found to be constant.

TABLE I

DTA of Copper Sulfate Pentahydrate

Run No.	Wt. (mg.)	Rate (°C. /Min.)	Atmosphere Air	Atmosphere N_2	Temperature °C.[a] Onset	Temperature °C.[a] Peak
SRL-1	1.20	5	X		96 101 112 239	98 104 123 256
SRL-2	1.15	10	X		94 100 116 244	97 104 126 261
SRL-3	1.10	15	X		96 102 120 250	98 105 126 266
SRL-4	1.20	5		X	96 98 109 220	98 103 123 240
SRL-5	1.20	10		X	96 100 113 226	98 104 124 247
SRL-6	1.15	15		X	96 103 116 233	99 108 129 253

[a] ΔT Sensitivity, 0.5°C. /in.

TABLE II

DTA of Calcium Oxalate Monohydrate

Run No.	Wt. (mg.)	Rate (°C. /Min.)	Atmosphere Air	N_2	Temperature °C.[a] Onset	Peak
SRL-10	1.70	5	X		224	239
SRL-11	1.55	10	X		226	240
SRL-12	1.60	15	X		234	244
SRL-7	1.45	5		X	216	236
SRL-8	1.45	10		X	232	243
SRL-9	1.45	15		X	233	244

[a] ΔT Sensitivity, 0.5°C. /min.

TABLE III

DTA of Potassium Nitrate[a]

Run No.	Wt. (mg.)	Rate (^{o}C. /Min.)	First Run	Rerun	Temperature ^{o}C.[b] Onset	Peak
SRL-13	1.65	5	X		128 331	131 333
SRL-14	1.65	5		X	128 331	129 333
SRL-15	1.65	10	X		129 331	133 334
SRL-16	1.65	10		X	129 331	130 334
SRL-17	1.65	15	X		129 331	133 334
SRL-18	1.65	15		X	129 331	130 334

[a] Atmosphere, Static Air

[b] ΔT Sensitivity, 0.5^{o}C. /in.

TABLE IV

DTA of Silver Nitrate[a]

Run No.	Wt. (mg.)	Rate (^{o}C. /Min.)	First Run	Rerun	Temperature ^{o}C.[b] Onset	Peak
SRL-19	3.50	5	X		164 207	166 209
SRL-20	3.40	5		X	--- 207	--- 209
SRL-21	3.40	10	X		164 207	167 209
SRL-22	3.40	10		X	--- 206	--- 208
SRL-23	3.45	15	X		165 208	167 210
SRL-24	3.45	15		X	--- 207	--- 209

[a] Atmosphere, Static Air

[b] ΔT Sensitivity, 0.5^{o}C. /min.

DSC

The results of all DSC experiments are summarized in Tables V through VII and shown on pages 46 through 63 in Appendix B. Analysis of the data on copper sulfate pentahydrate shows a variation in the temperatures at which the dehydration is observed. According to the workers at Perkin-Elmer[a] this is to be expected since the peaks may occur at any temperature within the range of stability of the respective hydrates.

The analyses of potassium nitrate are very reproducible as shown in the table. No significant deviation is observed in either heating rate or repeated analysis.

The excellent reproducibility is also observed in the silver nitrate data. Again it is shown that the inversion is permanent and non-reversible.

Heats of Fusion

The heat of fusion was determined for both KNO_3 and $AgNO_3$ and is shown in Table VIII. The heat associated with the inversion of both salts was also determined.

[a] Thermal Analysis Newsletter No. 7, 1967, Perkin-Elmer Corporation, Norwalk, Connecticut

TABLE V

DSC of Copper Sulfate Pentahydrate

Run No.	Wt. (mg.)	Rate (°C/Min.)	ΔT Sensitivity (Mcal. /Sec.)	Atmosphere Air	Atmosphere N_2	Peak Temperature, °C.
259	3.5	5	4	X		79 105 248
266	3.5	10	8	X		77 107 247
270	3.6	20	8	X		92 119 256
258	3.5	5	4		X	75 100 221
265	3.5	10	8		X	78 107 238
269	3.5	20	8		X	92 118 243

TABLE VI

DSC of Potassium Nitrate[a]

Run No.	Wt. (mg.)	Rate (°C. /Min.)	ΔT Sensitivity (Mcal. /Sec.)	First Run	Rerun	Peak Temperature, °C.
260A	3.5	5	8	X		133 334
260B	3.5	5	8		X	128 334
267A	3.4	10	16	X		130 336
267B	3.4	10	16		X	129 335
271A	3.6	20	16	X		133 335
271B	3.6	20	16		X	130 336

[a] Atmosphere, Static Air

TABLE VII

DSC of Silver Nitrate[a]

Run No.	Wt. (mg.)	Rate (°C./Min.)	ΔT Sensitivity (Mcal./Sec.)	First Run	Rerun	Peak Temperature, °C.
262A	3.4	5	4	X		171 211
262B	3.4	5	4		X	--- 212
268A	3.6	10	8	X		169 211
268B	3.6	10	8		X	--- 212
272A	3.5	20	8	X		172 211
272B	3.5	20	8		X	--- 212

[a] Atmosphere, Static Air

TABLE VIII

Heats of Inversion and Fusion

Run No.	Sample	ΔHf (Cal/gm)	Tf °C.	Literature ΔHf	T
261	KNO_3[a]	10.5	130	---	---
		11.0	129	---	---
	KNO_3	19.9	336	25.4[b]	308
		19.6	335	27.7[c]	337
263	$AgNO_3$[a]	2.3	168	---	---
264		2.8	169	---	---
263	$AgNO_3$	15.1	211	16.7[d]	212
		14.5	210	17.7[b]	208
				16.2[c]	210

[a] Inversion

[b] Handbook of Chemistry and Physics, 40th. Edition, 1959

[c] Handbook of Chemistry, Lange, 9th. Edition

[d] Handbook of Differential Thermal Analysis, Smothers and Chang, 1966

Analysis of the data on fusion shows a reproducibility of 4% or better and a variation from available literature sources on the order of 20%. No data was available for comparison of the heats of inversion.

CONCLUSIONS

Differential thermal analysis has shown that the dehydration of copper sulphate pentahydrate is not affected by either heating rate or atmosphere. However, the decomposition reaction was affected by these two variables. The dehydration of calcium oxalate monohydrate was also shown to be relatively insensitive to either atmosphere or heating rate. The inversion and the melting of potassium nitrate were insensitive to both variables as was the melting of silver nitrate. However, the silver nitrate inversion was found to be permanent and non-reversible.

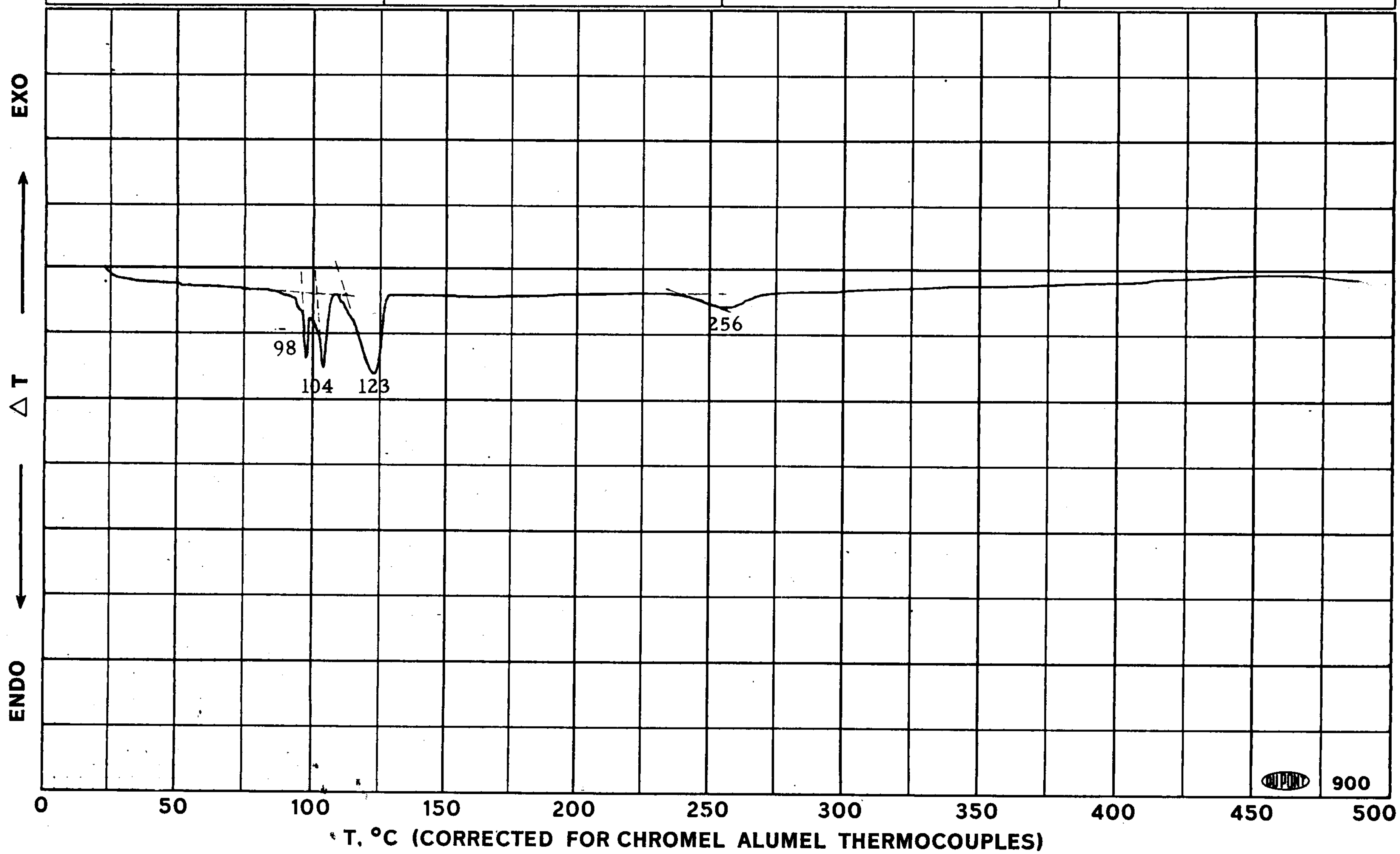
Differential scanning calorimetry results displayed a variation in the dehydration temperatures of copper sulphate pentahydrate as should be expected. The analyses of potassium nitrate and silver nitrate by DSC were very reproducible in the range of rates investigated.

The heats of fusion determined by DSC for potassium nitrate and silver nitrate had a reproducibility of 4% and a variation of 20% from available literature sources.

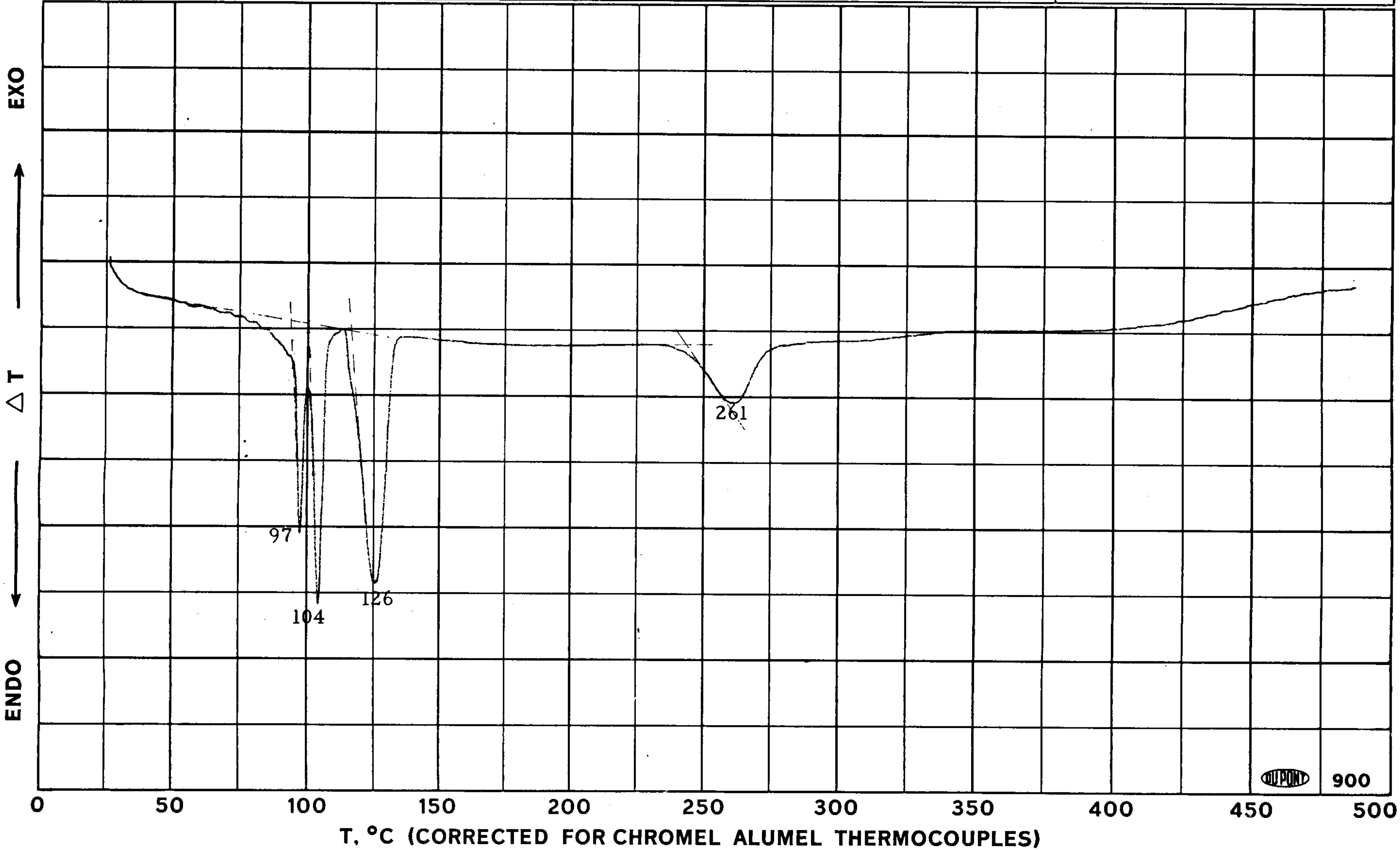
Appendix A

List of Thermograms (DTA)

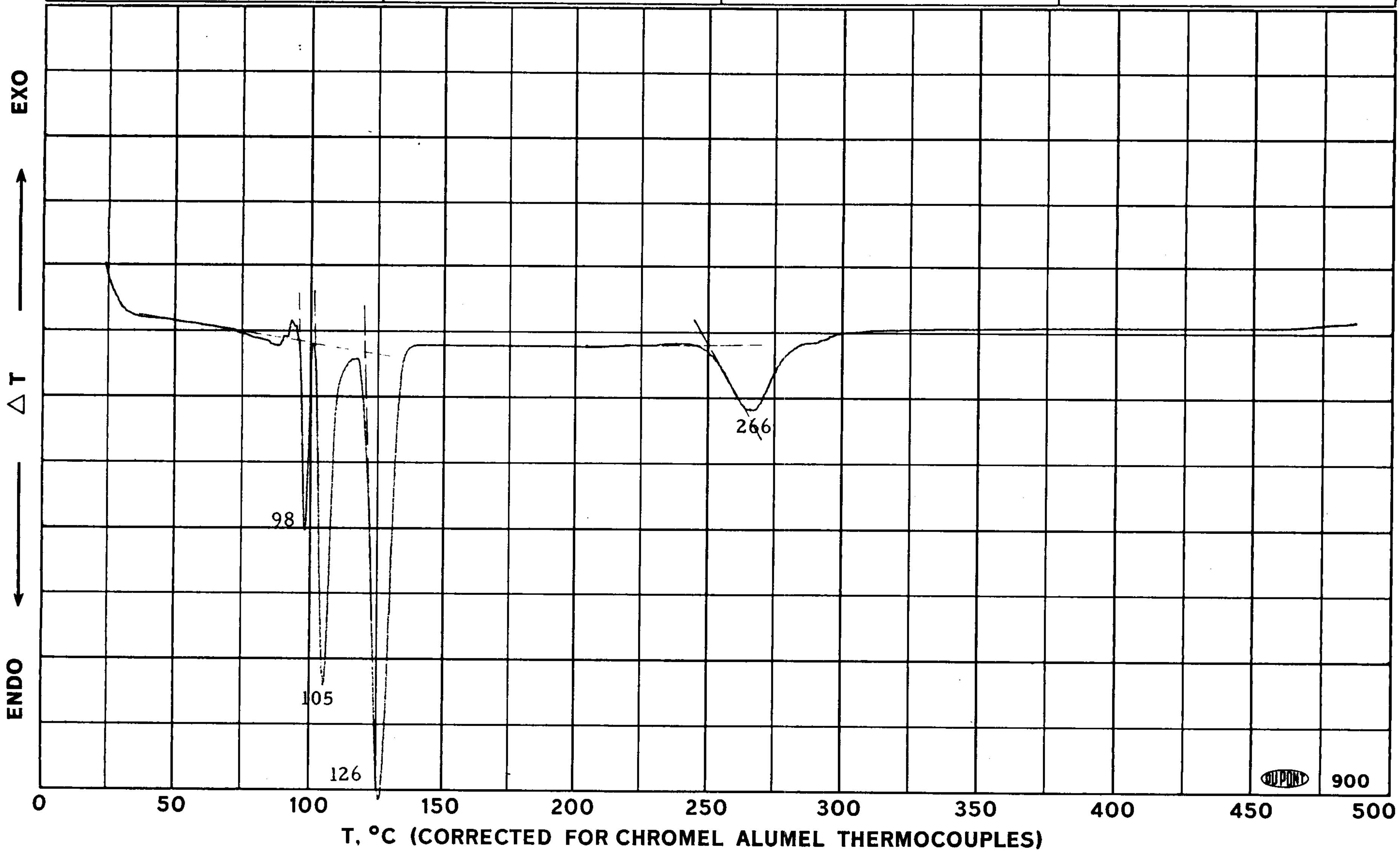
SAMPLE: $CuSO_4 \cdot 5H_2O$	**SIZE** 1.20 mg. in microtube	**ATM.** Air, Static	**RUN NO.** SRL-1
	REF. AL_2O_3	T / Δ T	**DATE** 2/12/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. / 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 5 °C/MIN, **START** 24°C	Baseline Slope -0.2	



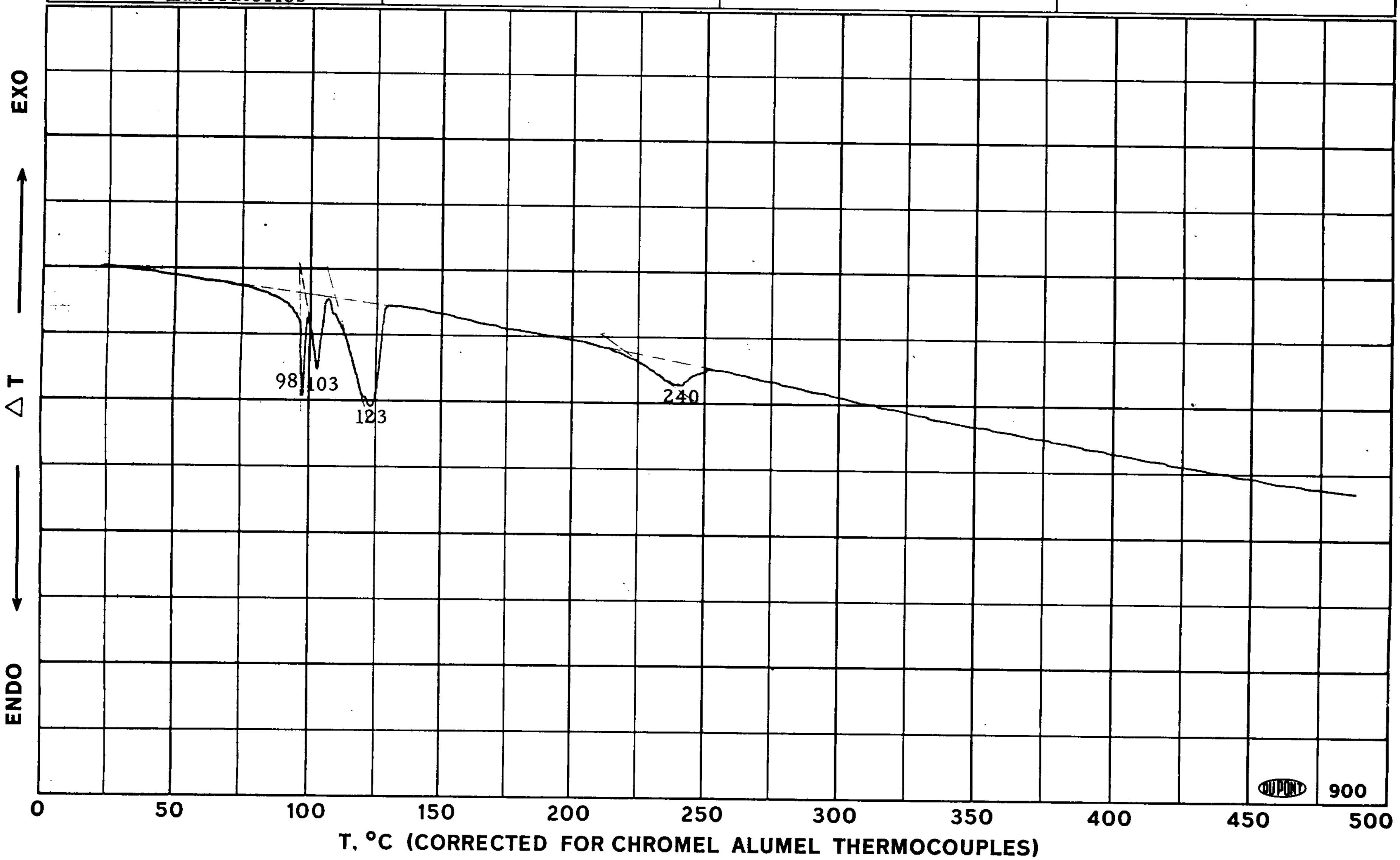
SAMPLE: $CuSO_4 \cdot 5H_2O$	**SIZE** 1.15 mg in microtube	**ATM.** Air, Static	**RUN NO.** SRL-2
	REF. AL_2O_3	T / Δ T	**DATE** 2/1/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. / 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 10 °C/MIN, **START** 26 °C	Baseline Slope -0.2	



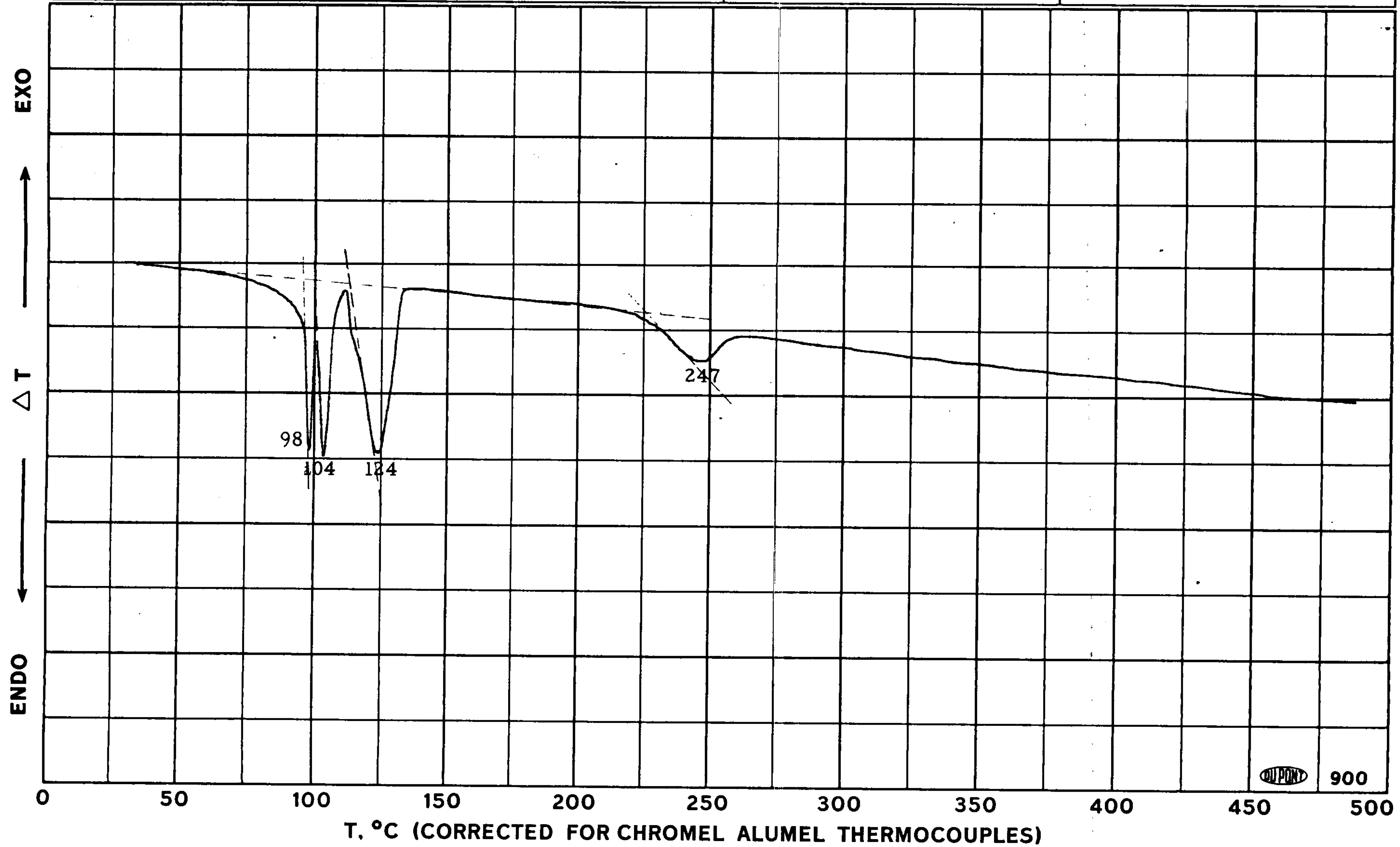
SAMPLE: $CuSO_4$ $5H_2O$	**SIZE** 1.10 mg in microtube	**ATM.** Air, Static	**RUN NO.** SRL-3
	REF. AL_2O_3	**T** / **△ T**	**DATE** 2/1/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. / 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 15 °C/MIN **,START** 24 **°C**	Baseline Slope -0.2	



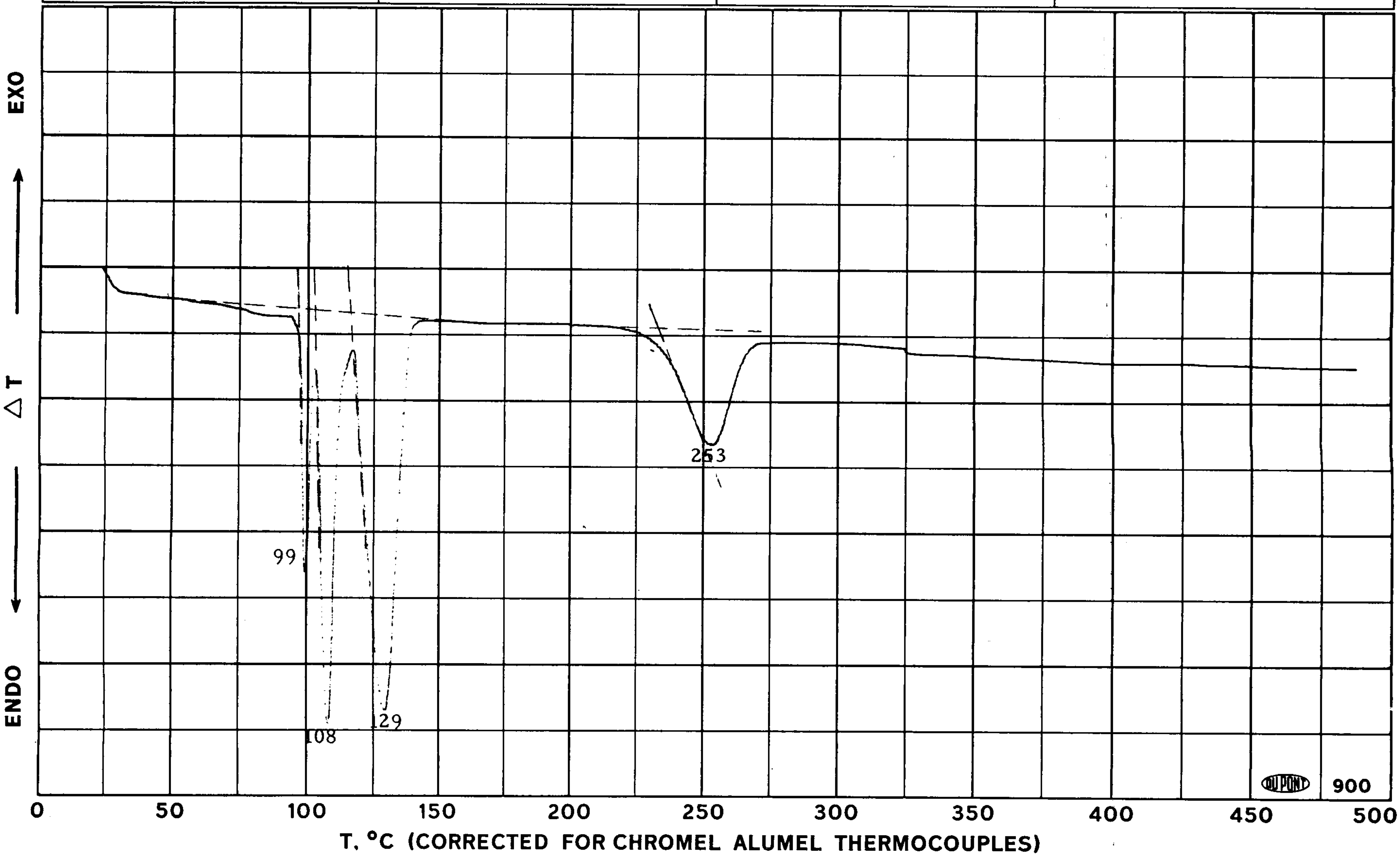
SAMPLE: $CuSO_4$ $5H_2O$	**SIZE** 1.20 mg. in microtube	**ATM.** N_2, 1 SCFH	**RUN NO.** SRL-4
	REF. AL_2O_3	T | Δ T	**DATE** 2/23/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. | 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 5 °C/MIN, **START** 24 °C	Baseline Slope -0.2	



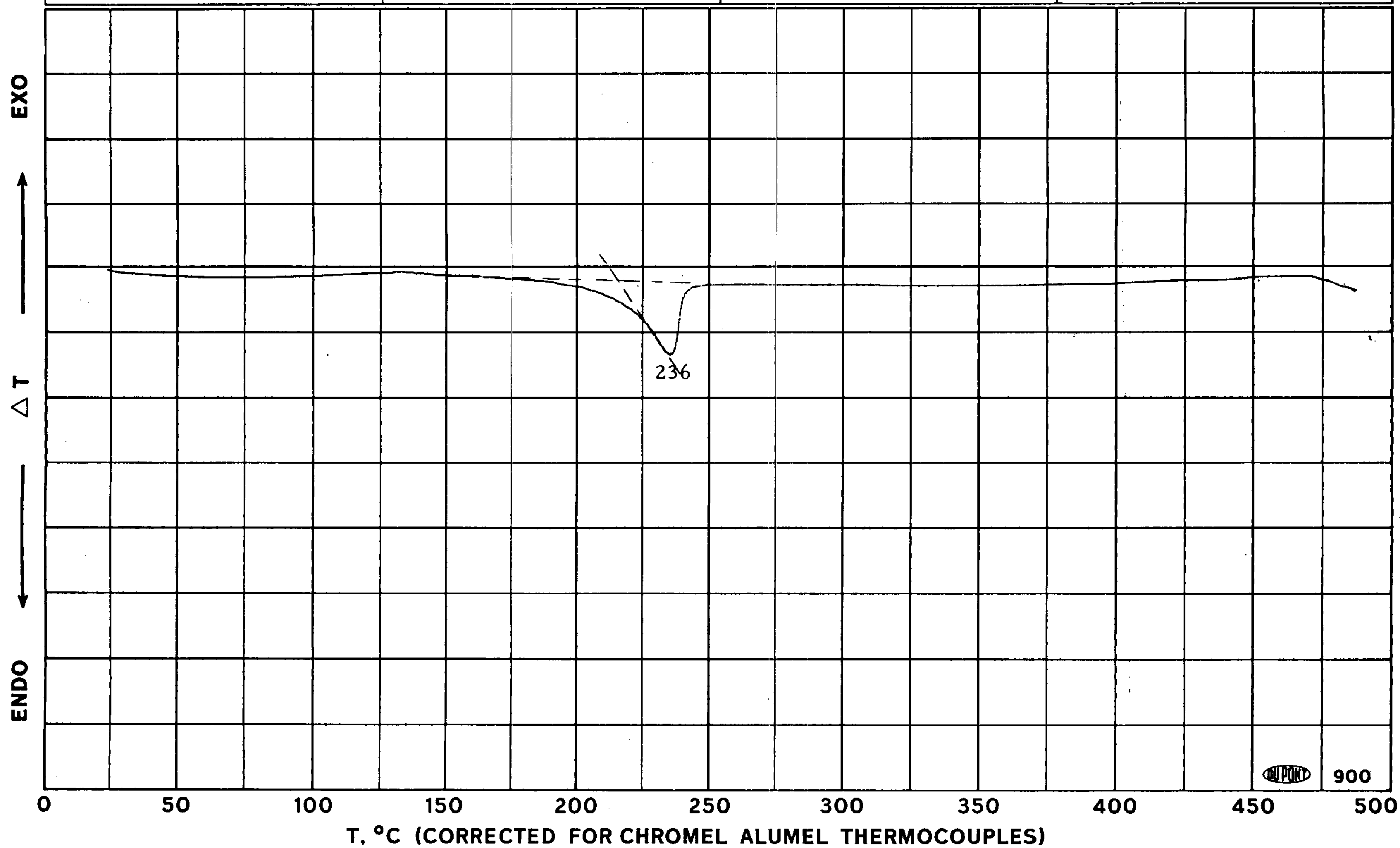
SAMPLE: $CuSO_4$ $5H_2O$	**SIZE** 1.20 mg in microtube	**ATM.** N_2, 1 SCFH	**RUN NO.** SRL-5
	REF. AL_2O_3	**T** | **△T**	**DATE** 2/20/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. | 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 10 °C/MIN ,**START** 34 °C	Baseline Slope -0.2	



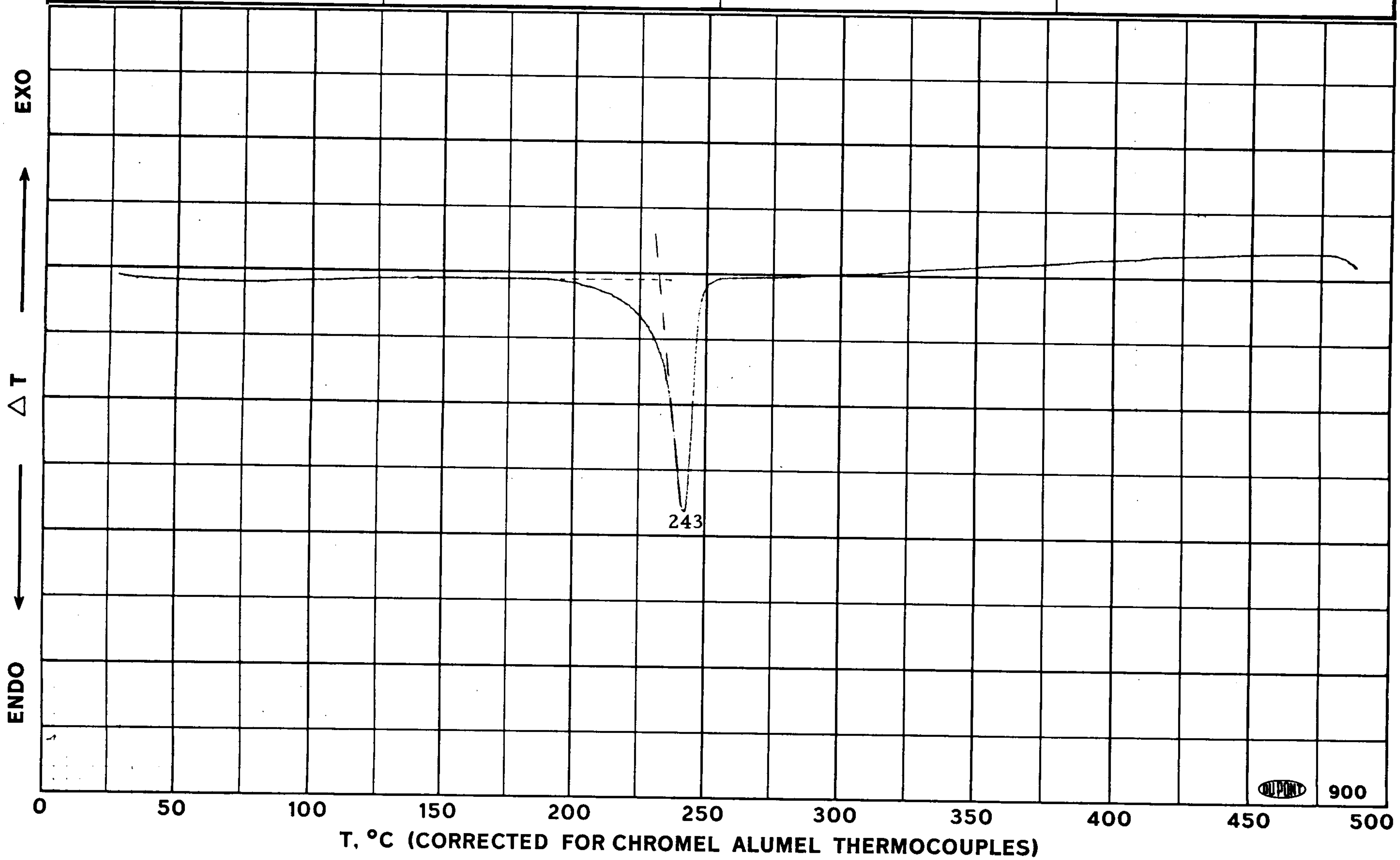
SAMPLE: $CuSO_4$ $5H_2O$	**SIZE** 1.15 mg. in microtube	**ATM.** N_2, 1 SCFH	**RUN NO.** SRL-6
	REF. AL_2O_3	T / Δ T	**DATE** 2/19/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. / 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 15 °C/MIN, **START** 24 °C	Baseline Slope -0.2	



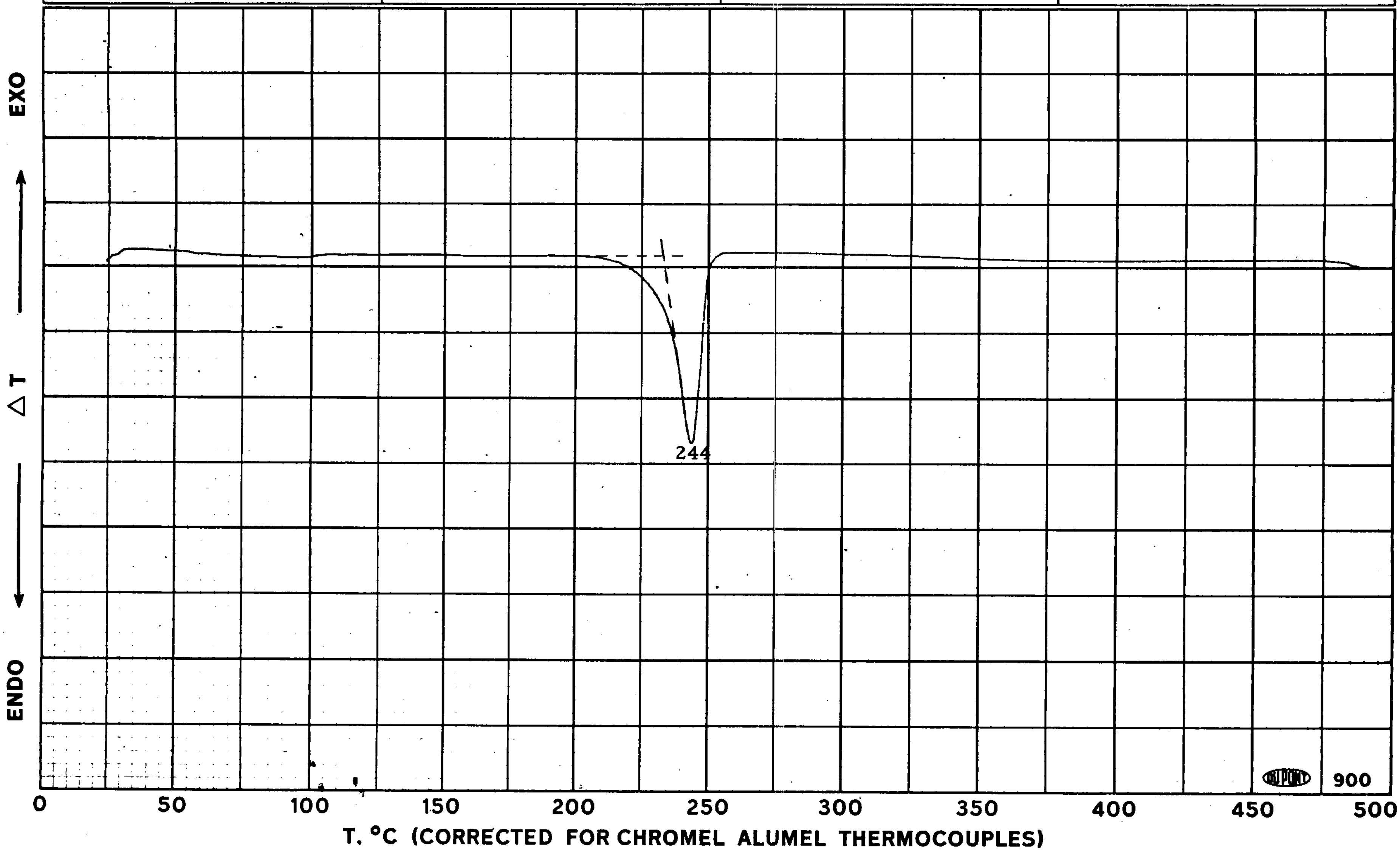
SAMPLE: $CaC_2O_4\ H_2O$	**SIZE** 1.45 mg in microtube	**ATM.** N_2, 1 SCFH	**RUN NO.** SRL-7
	REF. 1.85 mg. AL_2O_3	**SCALE SETTING** T: 50 °C/IN. ; △T: 0.5 °C/IN.	**DATE** 3/15/68
	PROGRAM MODE Heat	Baseline Slope -0.2	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 5 °C/MIN **,START** 24 °C		



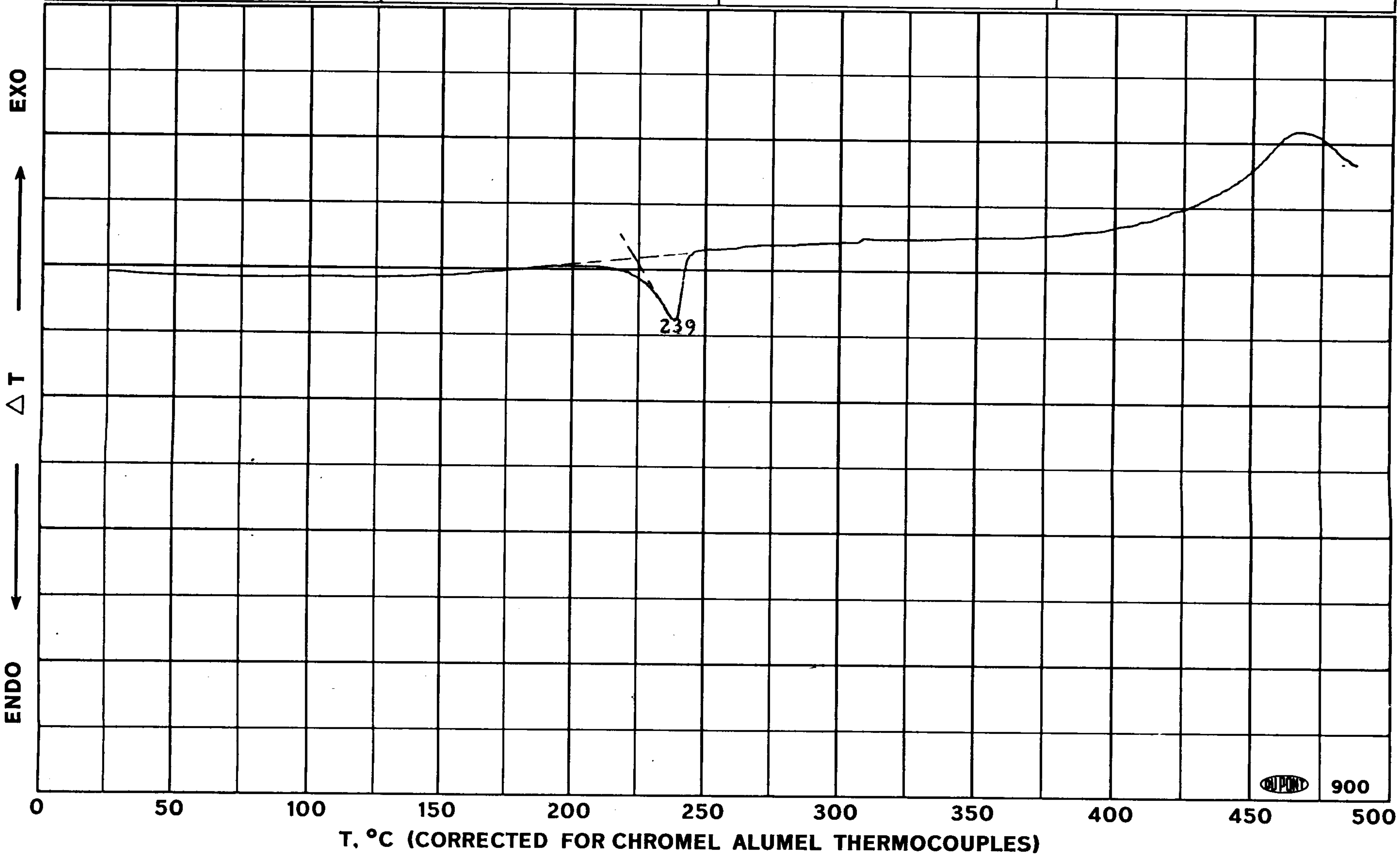
SAMPLE: CaC_2O_4 H_2O	**SIZE** 1.45 mg. in microtube	**ATM.** N_2, 1 SCFH	**RUN NO.** SRL-8
	REF. 1.85 mg. AL_2O_3	T: **SCALE SETTING** 50 °C/IN.; ΔT: 0.5 °C/IN.	**DATE** 3/13/68
ORIGIN: Sadtler Research Laboratories	**PROGRAM MODE** Heat	Baseline Slope -0.2	**OPERATOR** J. Evans
	RATE 10 °C/MIN, **START** 27 °C		



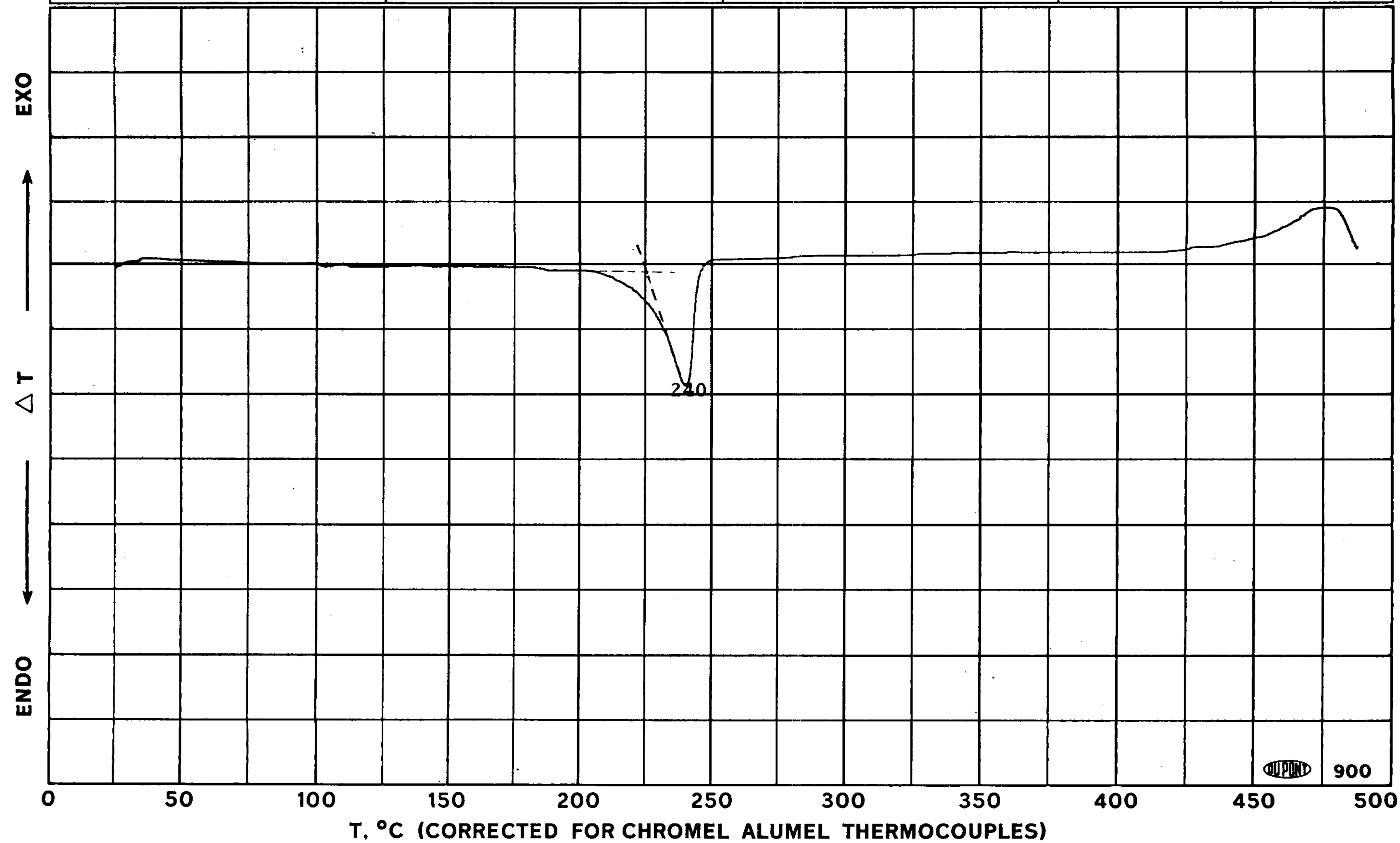
SAMPLE: CaC_2O_4 H_2O	**SIZE** 1.45 mg. in microtube	**ATM.** N_2, 1 SCFH	**RUN NO.** SRL-9
	REF. 1.85 mg. AL_2O_3	**SCALE SETTING** T: 50 °C/in.; ΔT: 0.5 °C/in.	**DATE** 3/15/68
	PROGRAM MODE Heat		**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 15 °C/MIN, **START** 25 °C	Baseline Slope -0.2	



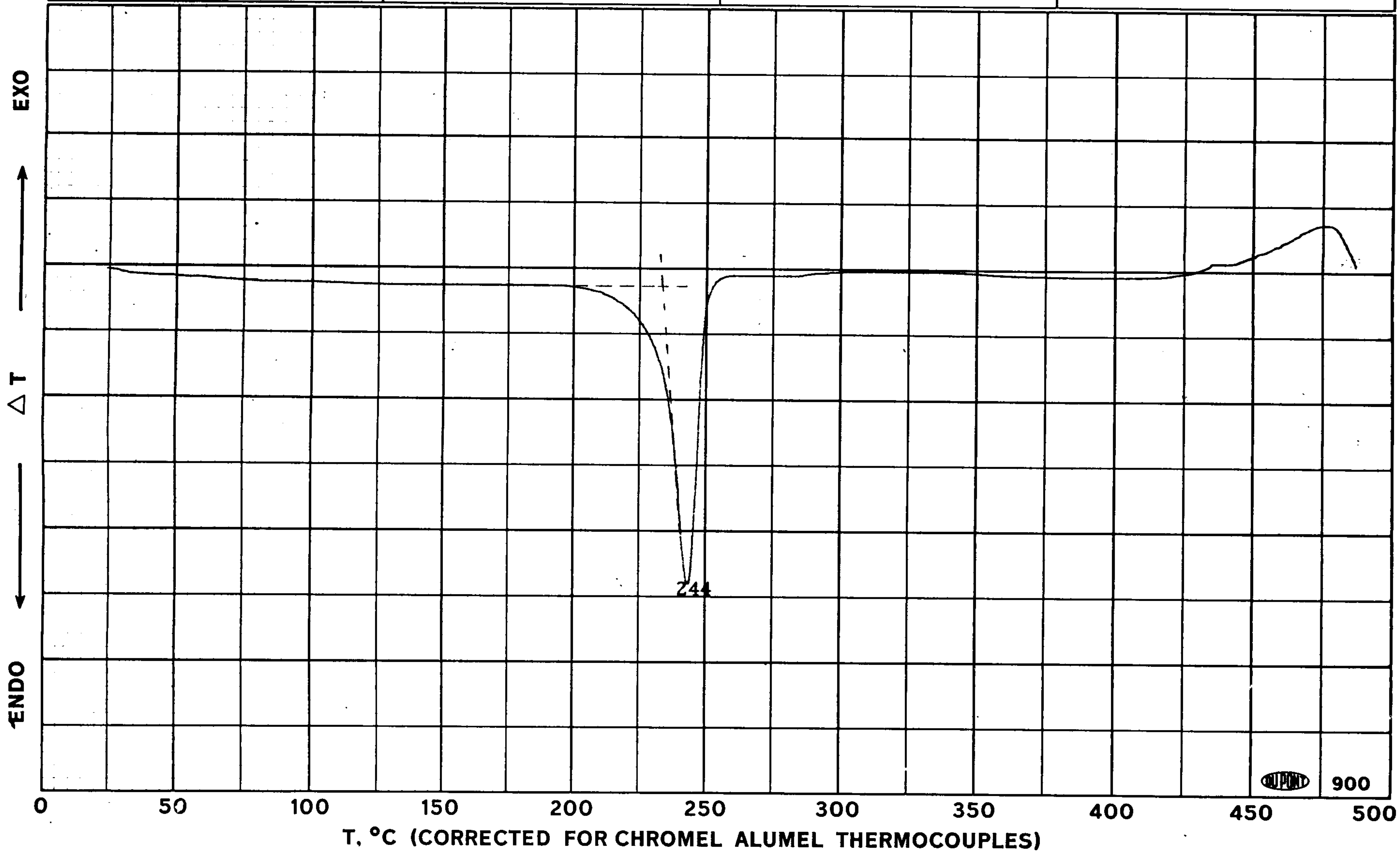
SAMPLE: $CaC_2O_4 \; H_2O$	**SIZE** 1.70 mg. in microtube	**ATM.** Air, 1 SCFH	**RUN NO.** SRL-10
	REF. 1.85 mg. AL_2O_3	T / Δ T	**DATE** 3/19/68
	PROGRAM MODE Heat	**SCALE SETTING** T: 50 °C/in.; Δ T: 0.5 °C/in.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 5 °C/min, **START** 25 °C	Baseline Slope -0.2	



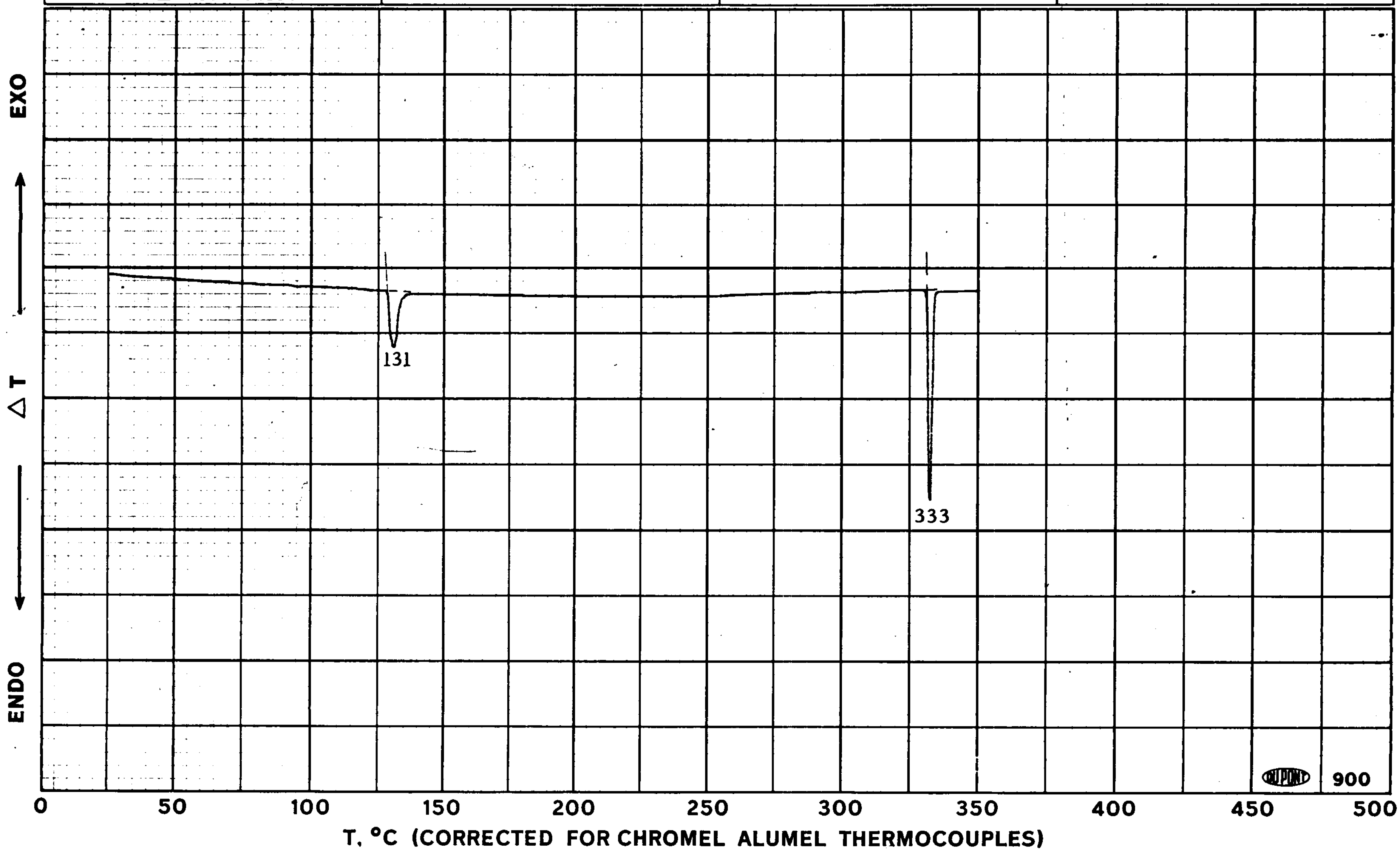
SAMPLE: $CaC_2O_4 \cdot H_2O$	**SIZE** 1.55 mg. in microtube	**ATM.** Air, 1 SCFH	**RUN NO.** SRL-11
	REF. 1.85 mg. AL_2O_3	T: 50 °C/in.; ΔT: 0.5 °C/in.	**DATE** 3/18/68
	PROGRAM MODE Heat	**SCALE SETTING**	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 10 °C/MIN, **START** 25 °C	Baseline Slope -0.2	



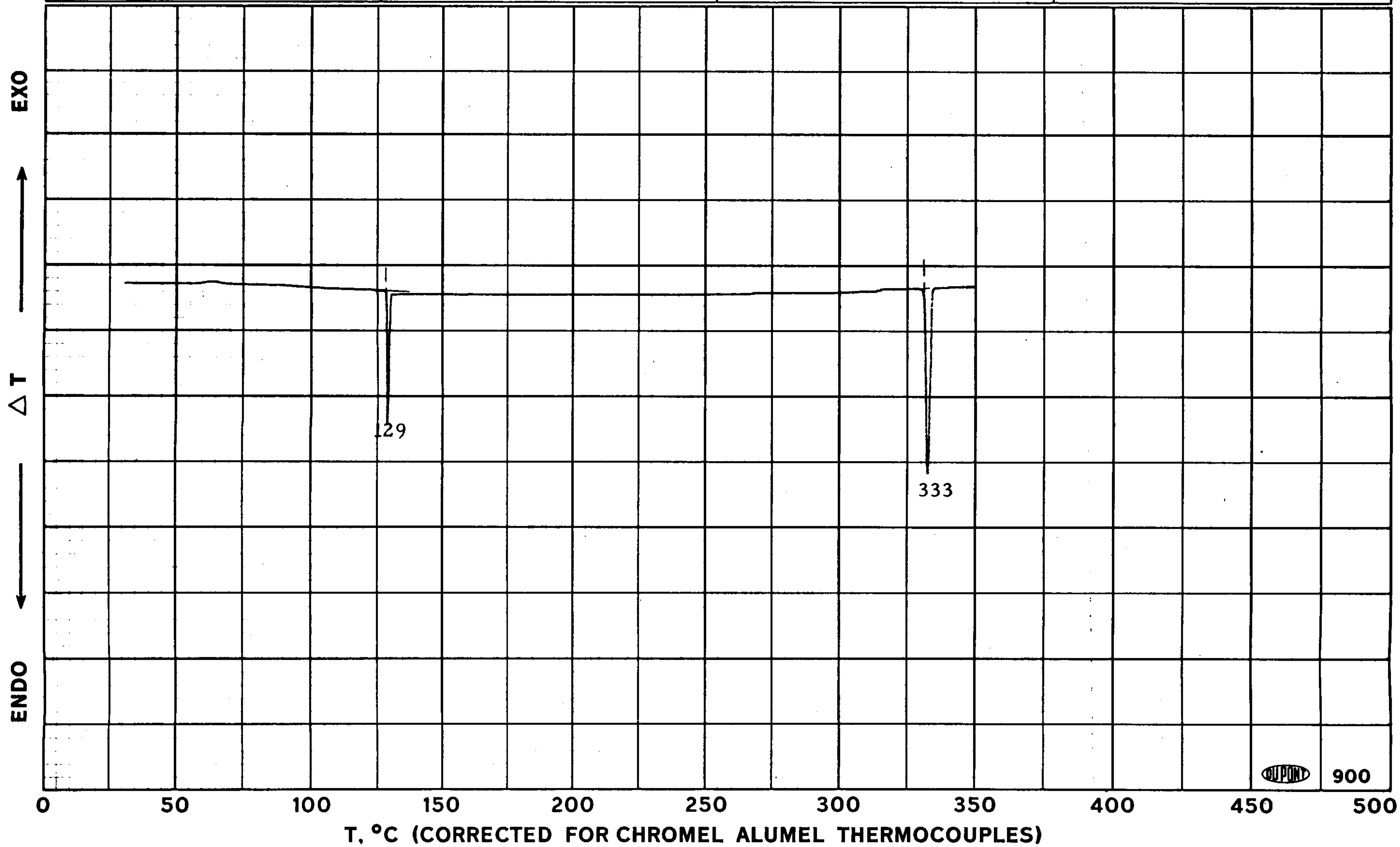
SAMPLE: $CaC_2O_4 \; H_2O$ **ORIGIN:** Sadtler Research Laboratories	**SIZE** 1.60 mg. in microtube **REF.** 1.85 mg. AL_2O_3 **PROGRAM MODE** Heat **RATE** 15 °C/MIN, **START** 25 °C	**ATM.** Air, 1 SCFH **SCALE SETTING** T: 50 °C/IN.; ΔT: 0.5 °C/IN. Baseline Slope -0.2	**RUN NO.** SRL-12 **DATE** 3/18/68 **OPERATOR** J. Evans



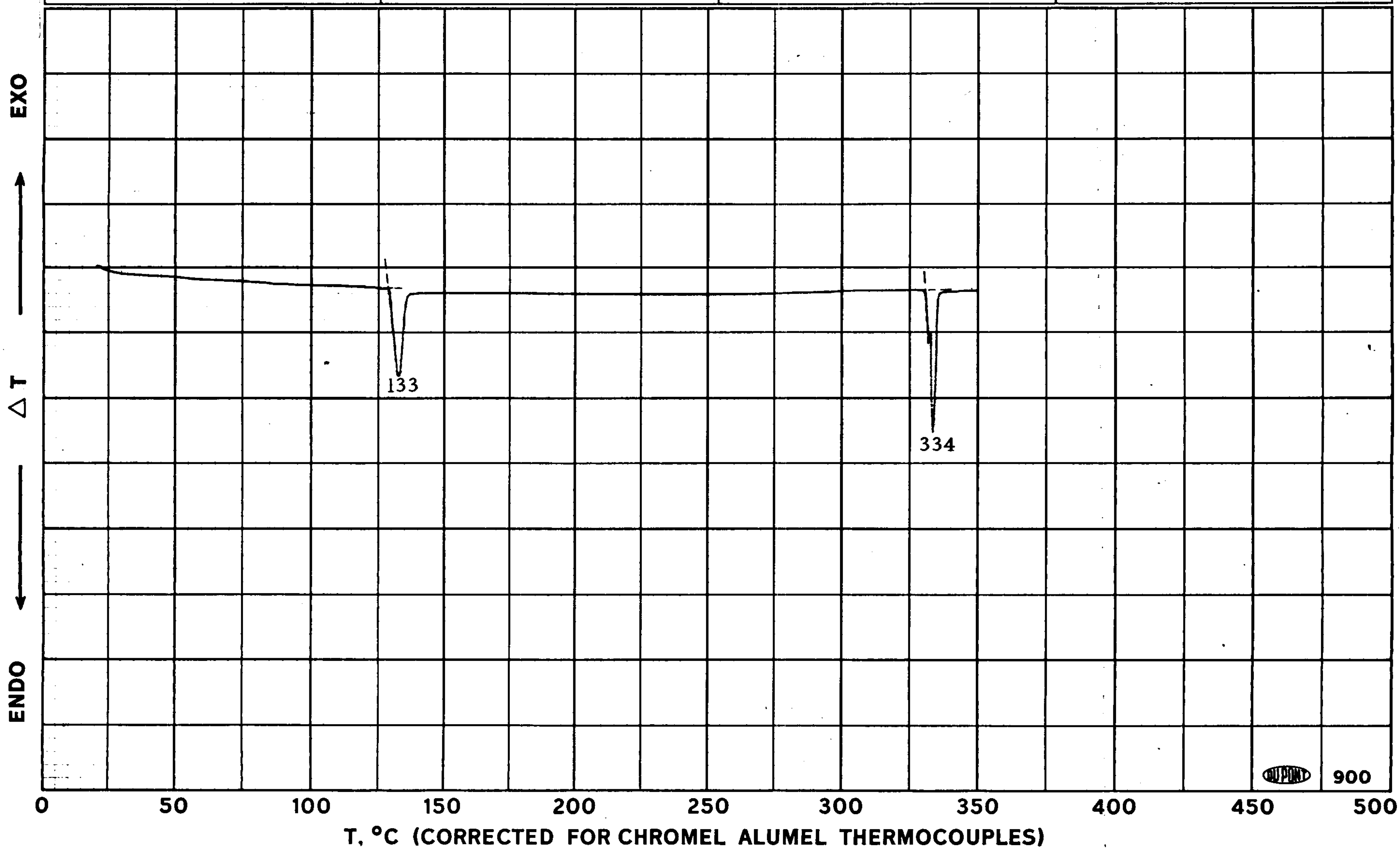
SAMPLE: KNO_3	**SIZE** 1.65 mg	**ATM.** Air, Static	**RUN NO.** SRL-13
	REF. 1.85 mg. AL_2O_3	T / Δ T	**DATE** 3-21-68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/in. / 0.5 °C/in.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 5 °C/MIN, **START** 25 °C	Baseline Slope -0.2	



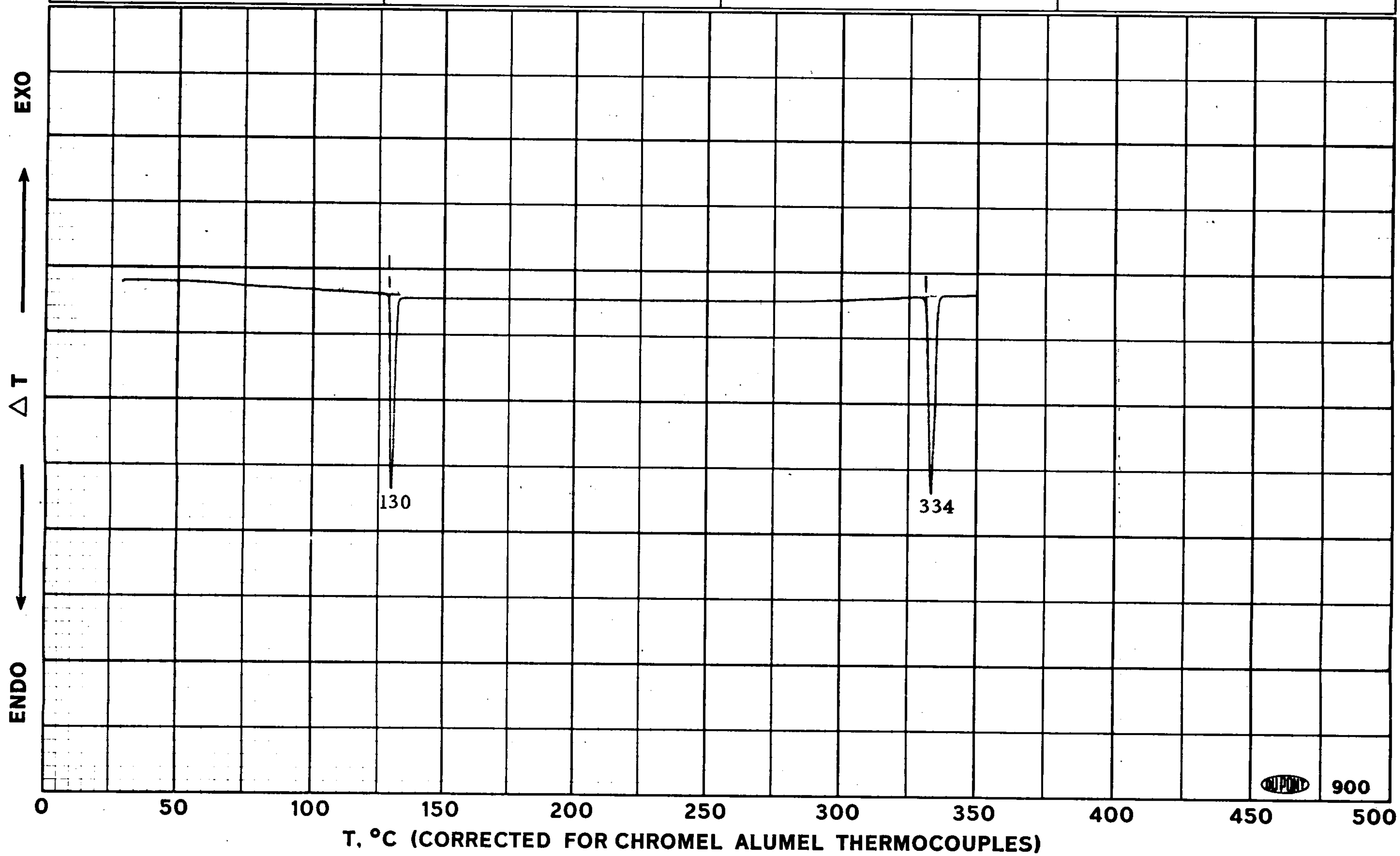
SAMPLE: KNO_3 Rerun	**SIZE** 1.65 mg.	**ATM.** Air, Static	**RUN NO.** SRL-14
	REF. 1.85 mg AL_2O_3	T: **SCALE SETTING** 50 °C/IN.; ΔT: 0.5 °C/IN.	**DATE** 3-21-68
	PROGRAM MODE Heat	Baseline Slope -0.2	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 5 °C/MIN, **START** 30°C		



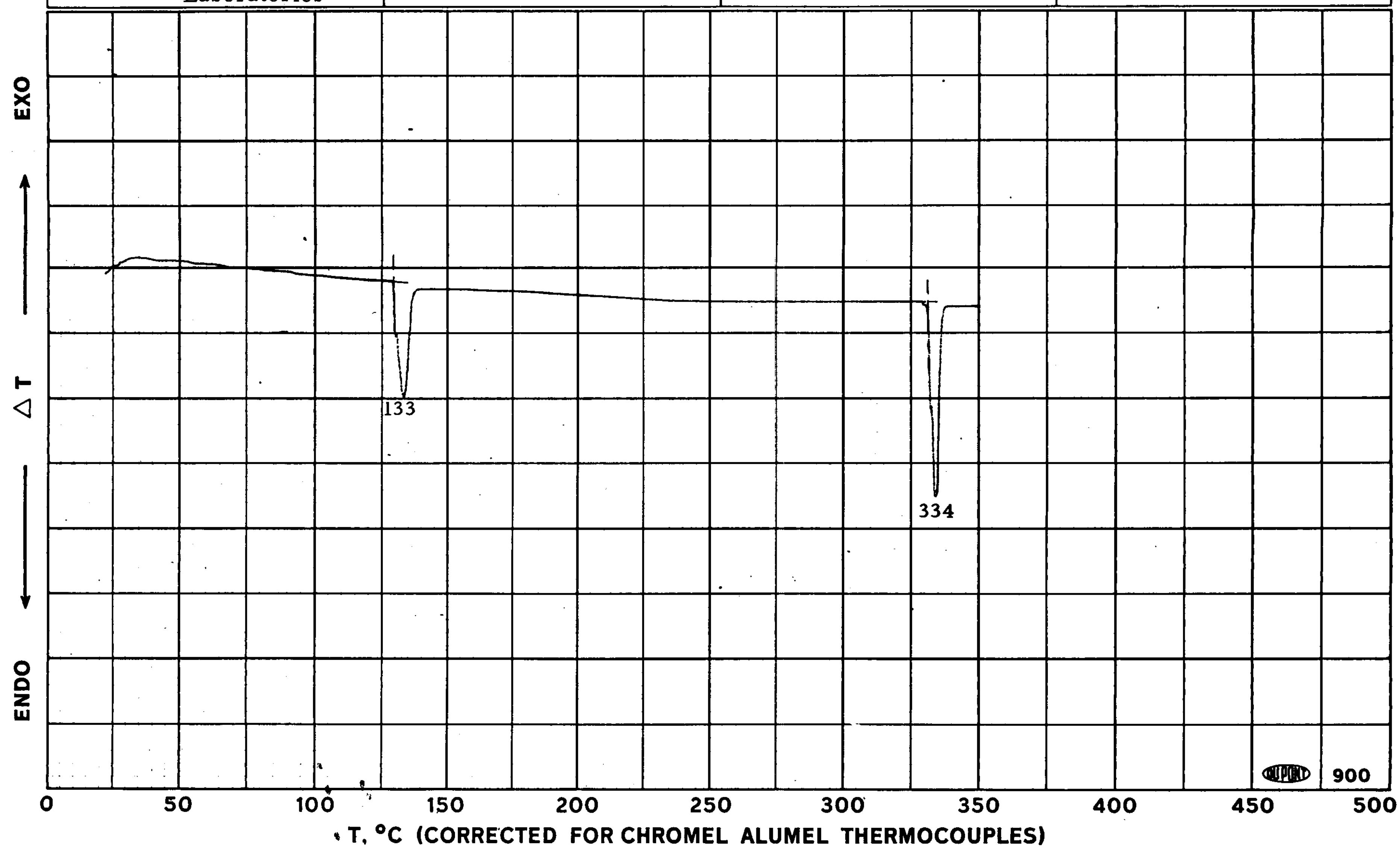
SAMPLE: KNO_3	**SIZE** 1.65 mg.	**ATM.** Air, Static	**RUN NO.** SRL-15
	REF. 1.85 mg. AL_2O_3	T / Δ T	**DATE** 3-22-68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. / 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 10 °C/MIN, **START** 23 °C	Baseline Slope -0.2	



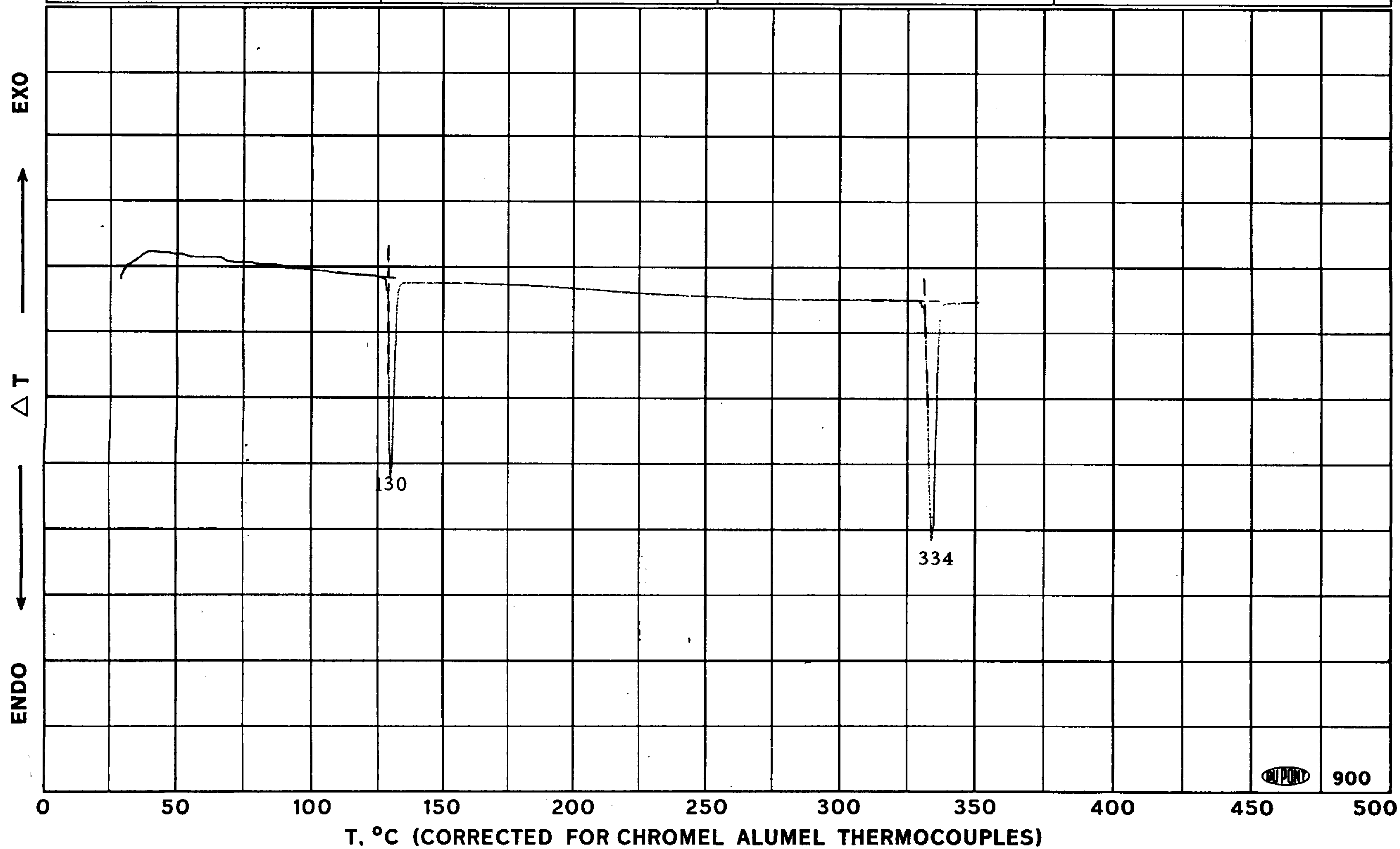
SAMPLE: KNO_3 Rerun	**SIZE** ~~1.65 mg. in microtube~~	**ATM.** Air, Static	**RUN NO.** SRL-16
	REF. 1.85 mg. AL_2O_3	T: 50 °C/IN. ; ΔT: 0.5 °C/IN.	**DATE** 3-22-68
ORIGIN: Sadtler Research Laboratories	**PROGRAM MODE** Heat	**SCALE SETTING**	**OPERATOR** J. Evans
	RATE 10 °C/MIN, **START** 30 °C	Baseline Slope -0.2	



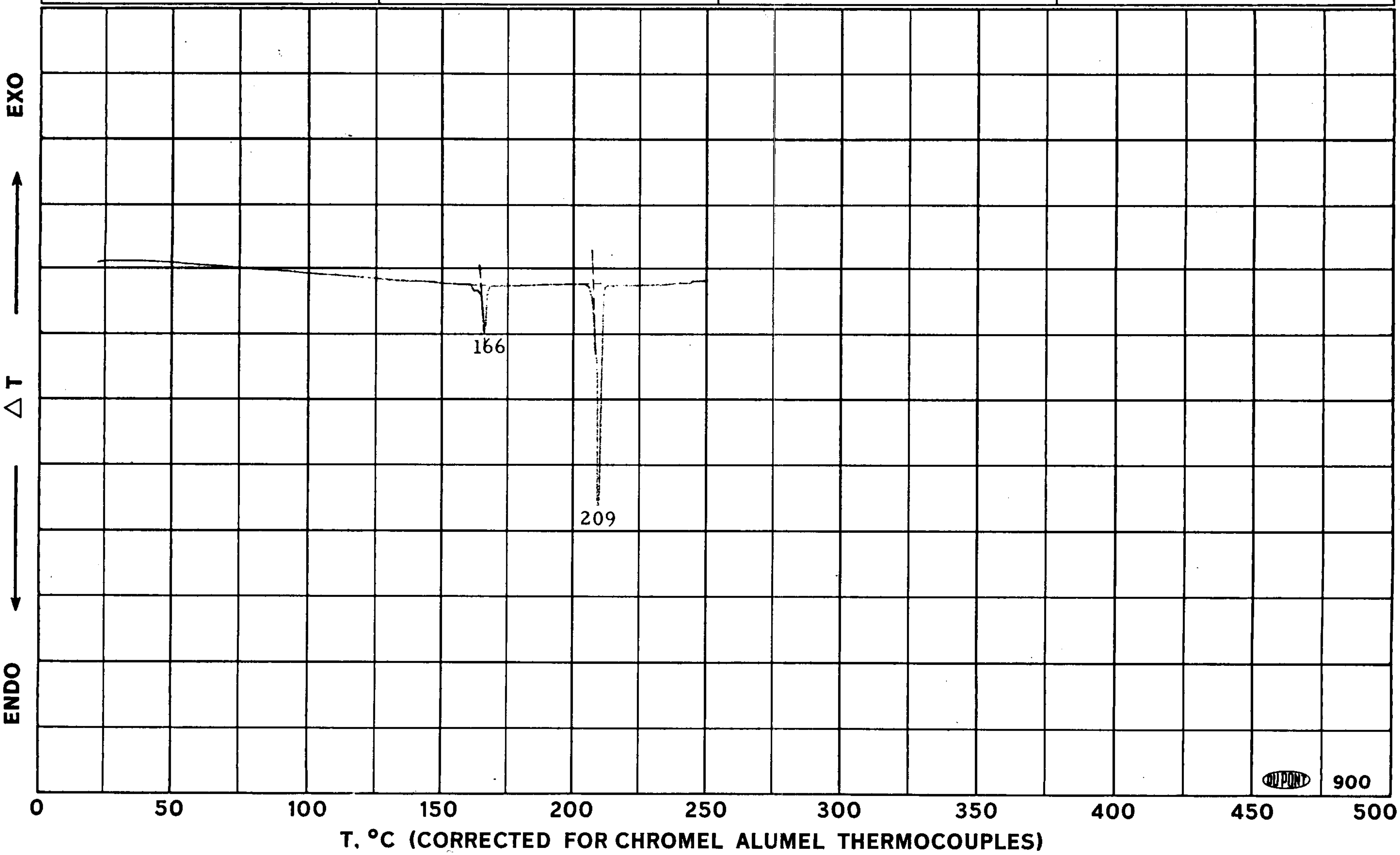
SAMPLE: KNO_3	**SIZE** 1.65 mg.	**ATM.** Air, Static	**RUN NO.** SRL-17
	REF. 1.85 mg. AL_2O_3	**SCALE SETTING** T: 50 °C/in.; ΔT: 0.5 °C/in.	**DATE** 3-22-68
	PROGRAM MODE Heat	Baseline Slope -0.2	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 15 °C/min, **START** 23 °C		



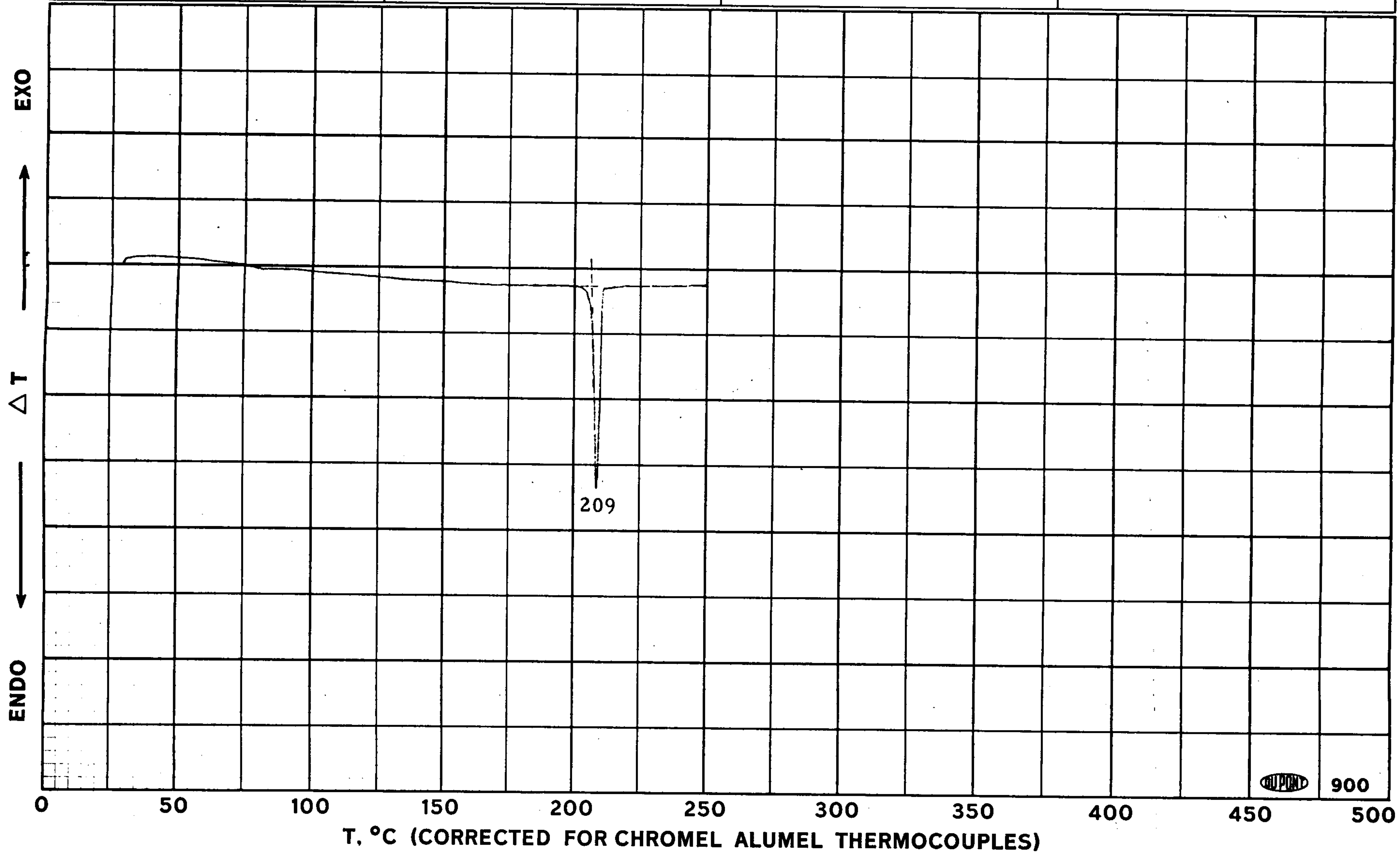
SAMPLE: KNO_3 Rerun	**SIZE** 1.65 mg. in microtube	**ATM.** Air, Static	**RUN NO.** SRL-18
	REF. 1.85 mg. AL_2O_3	**SCALE SETTING** T 50 °C/IN. ; ΔT 0.5 °C/IN.	**DATE** 3-22-68
	PROGRAM MODE Heat		**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 15 °C/MIN, **START** 30 °C	Baseline Slope -0.2	



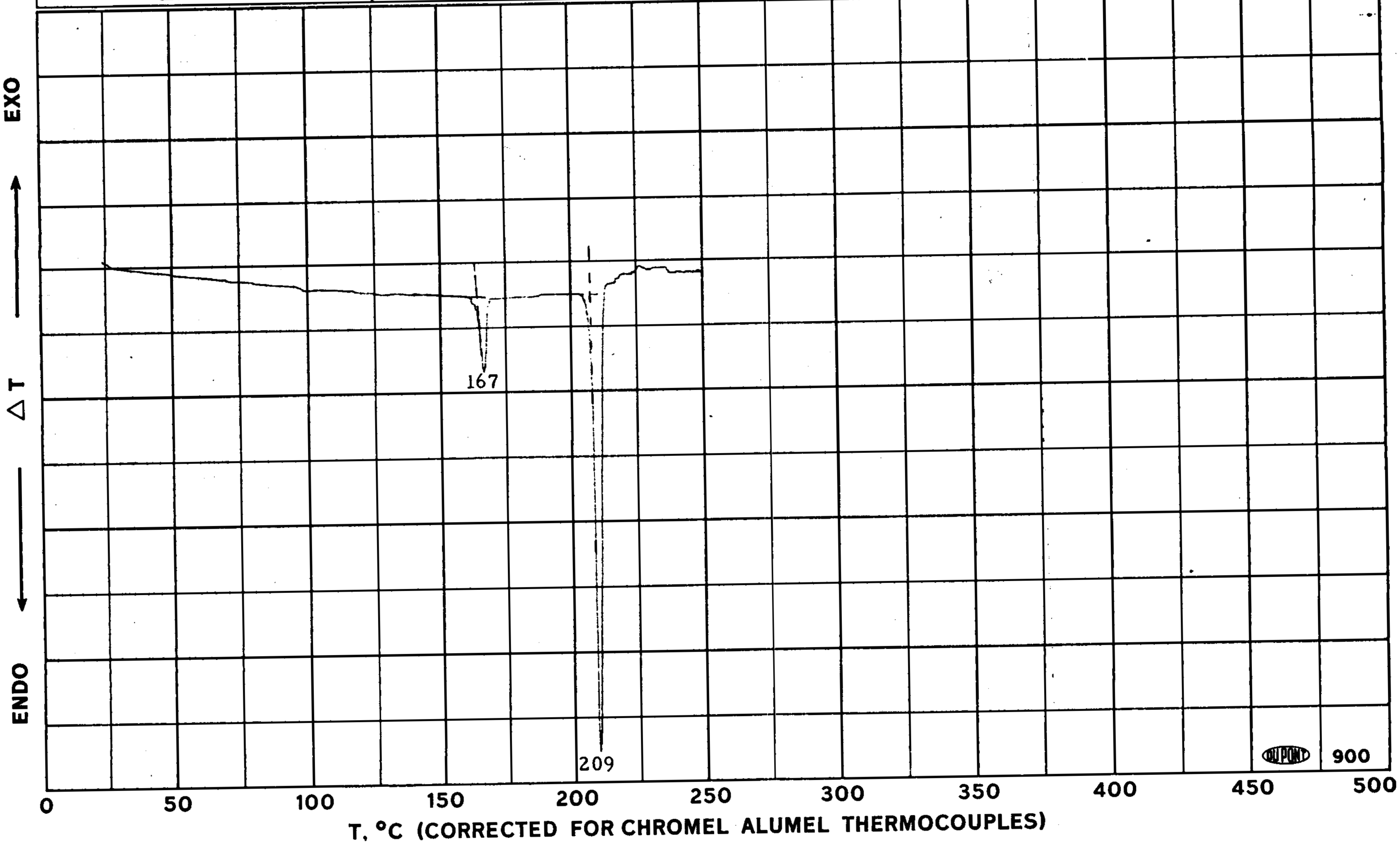
SAMPLE: $AgNO_3$	**SIZE** 3.40 mg. in microtube	**ATM.** Air, Static	**RUN NO.** SRL-19
	REF. 1.85 mg. AL_2O_3	T / △ T	**DATE** 4/5/68
	PROGRAM MODE Heat	**SCALE SETTING** T: 50 °C/IN. ; △ T: 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 5 °C/MIN ,**START** 24 °C	Baseline Slope -0.2	



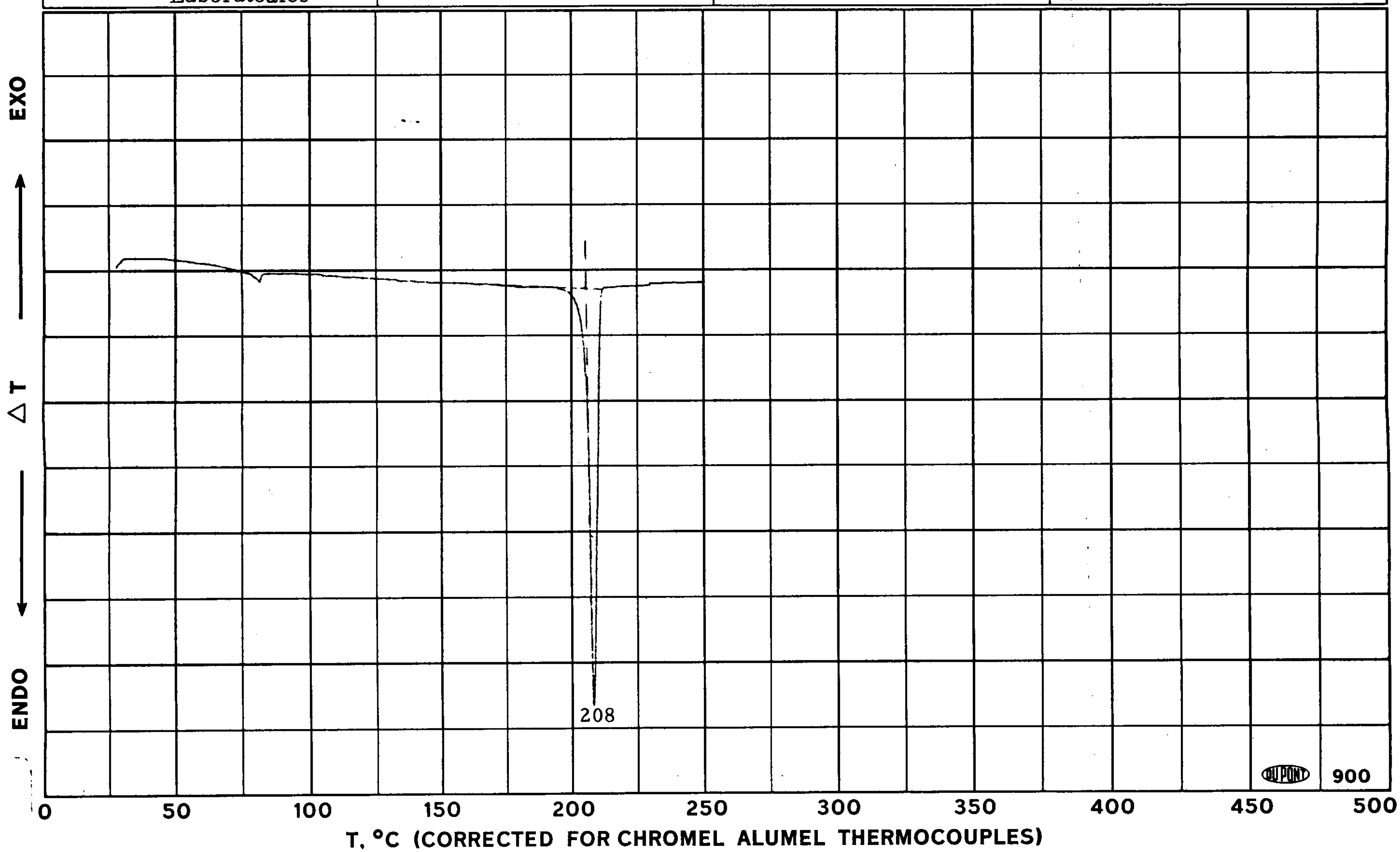
SAMPLE: $AgNO_3$ Rerun	**SIZE** 3.40 mg. in microtube	**ATM.** Air, Static	**RUN NO.** SRL-20
	REF. 1.85 mg. AL_2O_3	T: 50 °C/IN.; Δ T: 0.5 °C/IN.	**DATE** 4/5/68
	PROGRAM MODE Heat	**SCALE SETTING**	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 5 °C/MIN, **START** 30 °C	Baseline Slope -0.2	



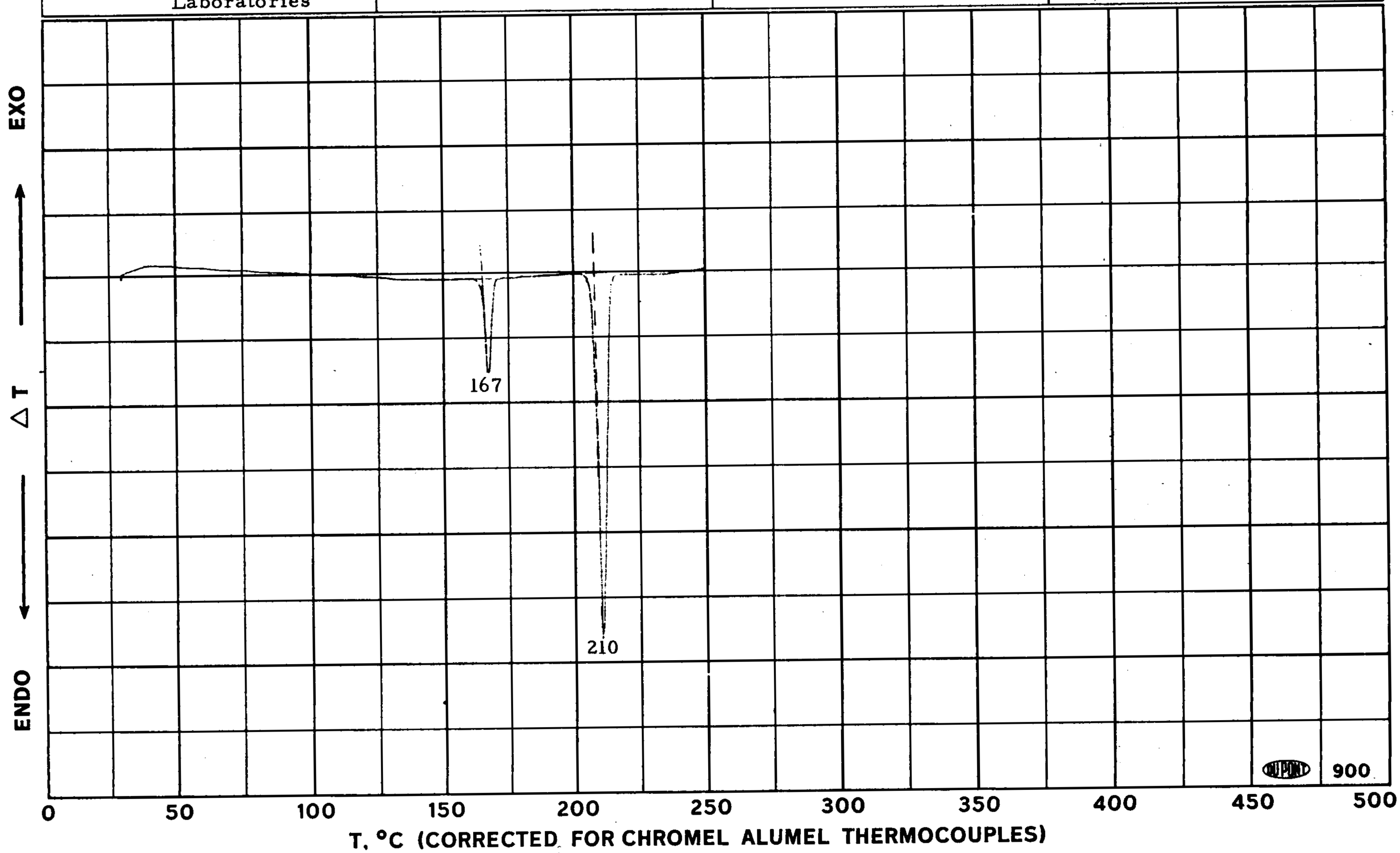
SAMPLE: AgNO$_3$	**SIZE** 3.40 mg. in microtube	**ATM.** Air, Static	**RUN NO.** SRL-21
	REF. 1.85 mg. AL_2O_3	T / Δ T	**DATE** 4/4/68
	PROGRAM MODE Heat	**SCALE SETTING** T: 50 °C/IN.; Δ T: 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 10 °C/MIN, **START** 25 °C	Baseline Slope -0.2	



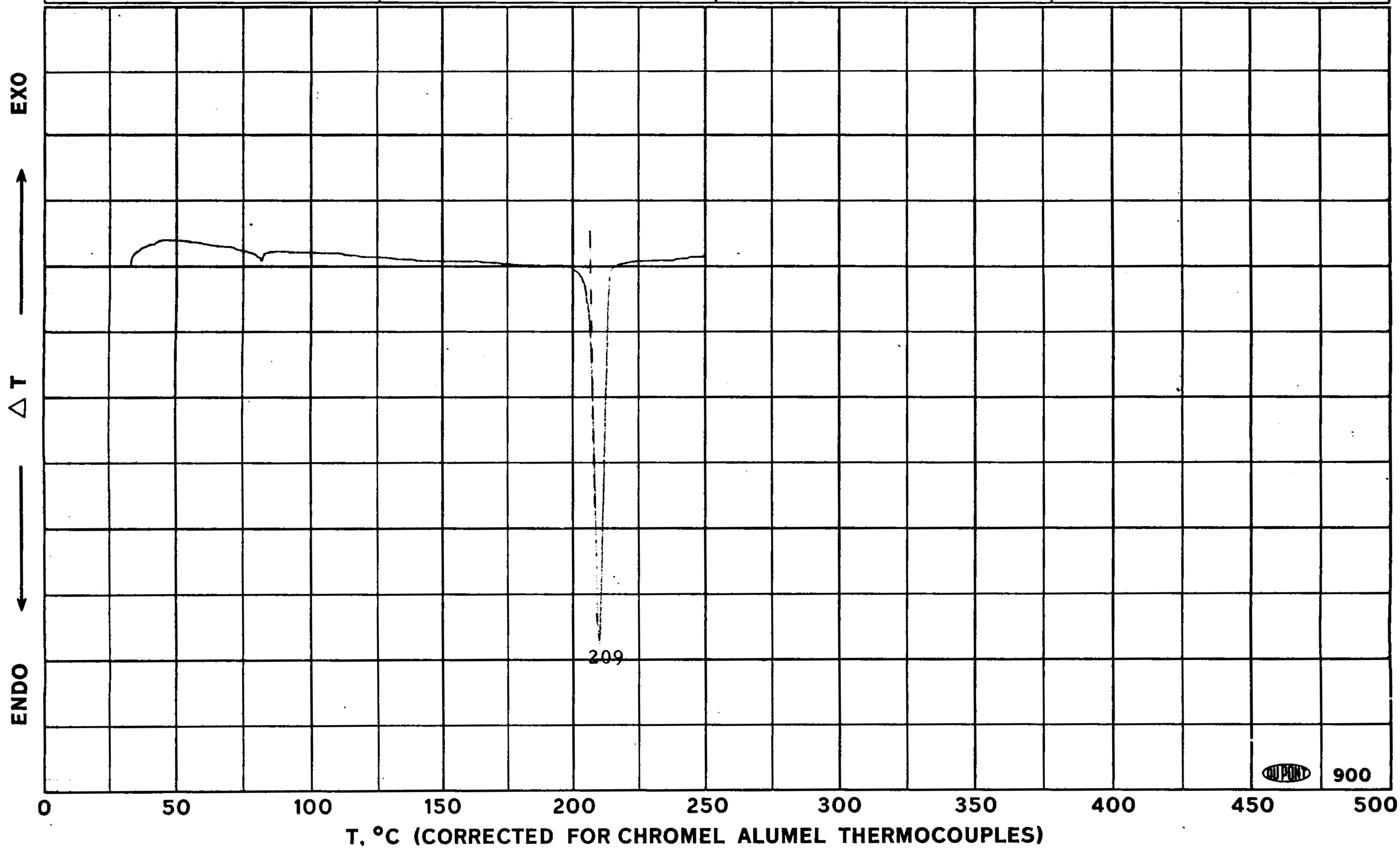
SAMPLE: $AgNO_3$ Rerun	**SIZE** 3.40 mg. in microtube	**ATM.** Air, Static	**RUN NO.** SRL-22
	REF. 1.85 mg. AL_2O_3	T / △ T	**DATE** 4/4/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. / 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratoaies	**RATE** 10 °C/MIN, **START** 25 °C	Baseline Slope -0.2	



SAMPLE: $AgNO_3$	**SIZE** 3.45 mg.	**ATM.** Air, Static	**RUN NO.** SRL-23
	REF. 1.85 mg. AL_2O_3	T / △T	**DATE** 4/3/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. / 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 15 °C/MIN, **START** 30 °C	Baseline Slope -0.2	

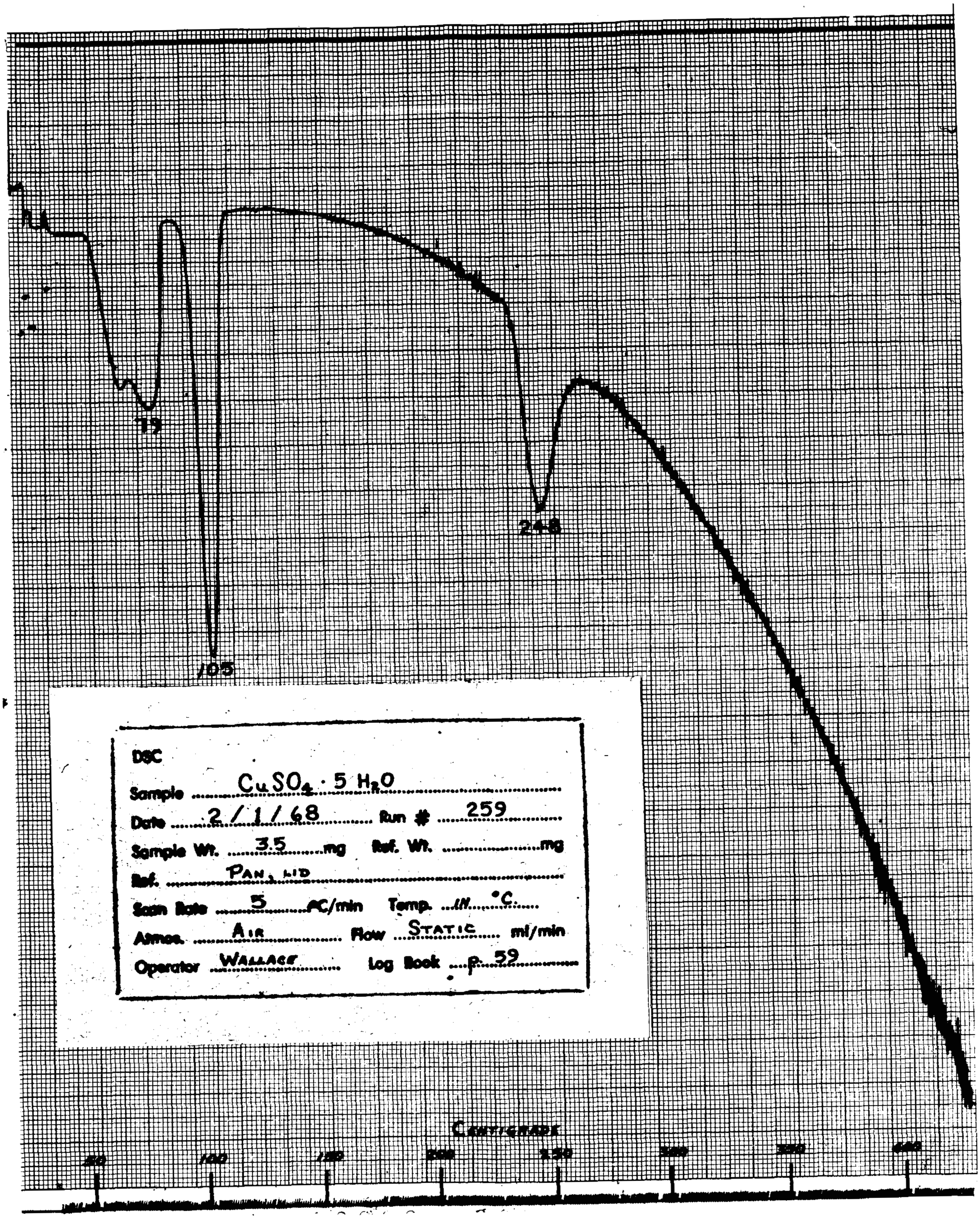


SAMPLE: $AgNO_3$ Rerun	**SIZE** 3.45 mg.	**ATM.** Air, Static	**RUN NO.** SRL-24
	REF. 1.85. mg.	T / Δ T	**DATE** 4/3/68
	PROGRAM MODE Heat	**SCALE SETTING** 50 °C/IN. / 0.5 °C/IN.	**OPERATOR** J. Evans
ORIGIN: Sadtler Research Laboratories	**RATE** 15 °C/MIN, **START** 33 °C	Baseline Slope -0.2	

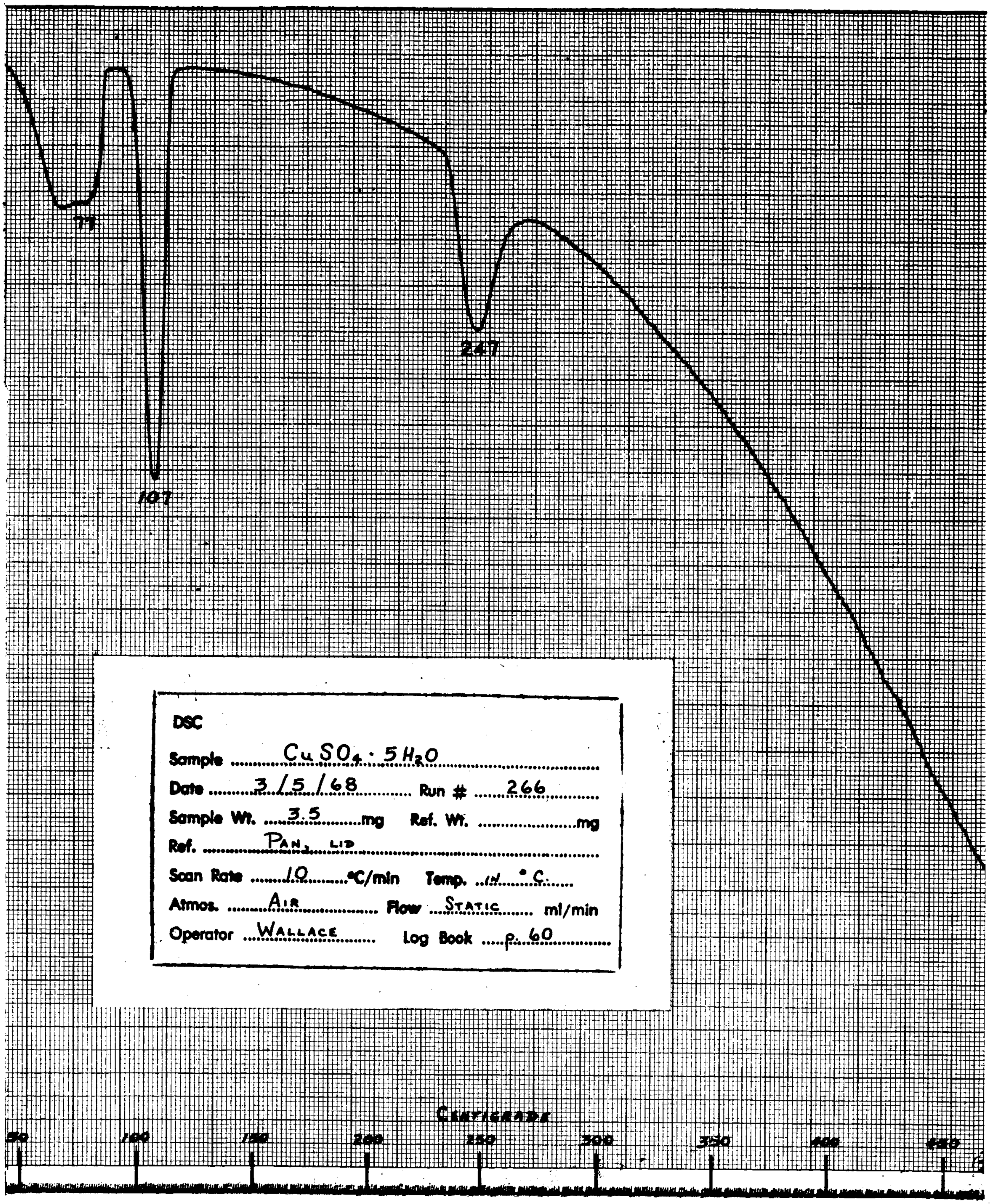


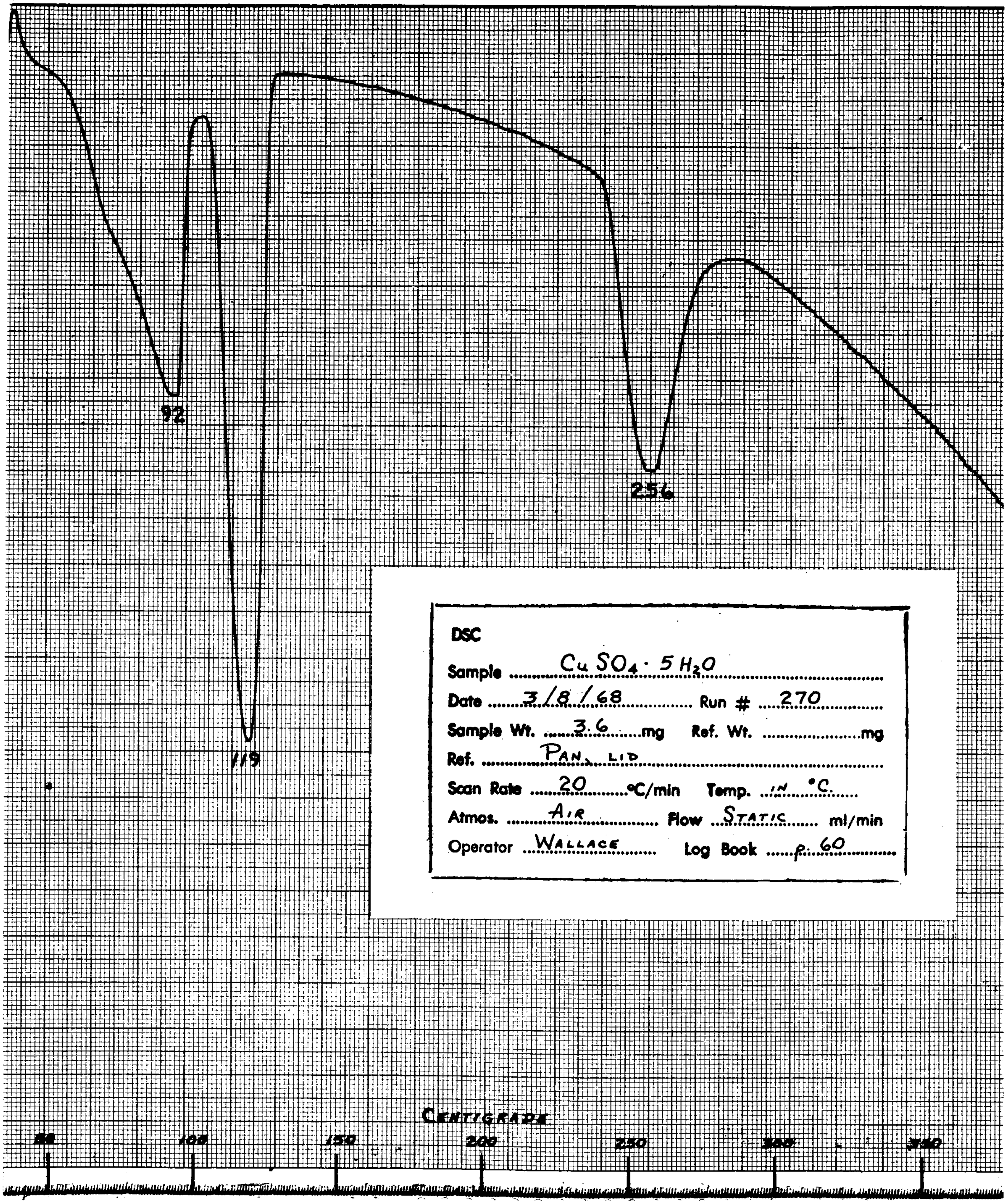
Appendix B

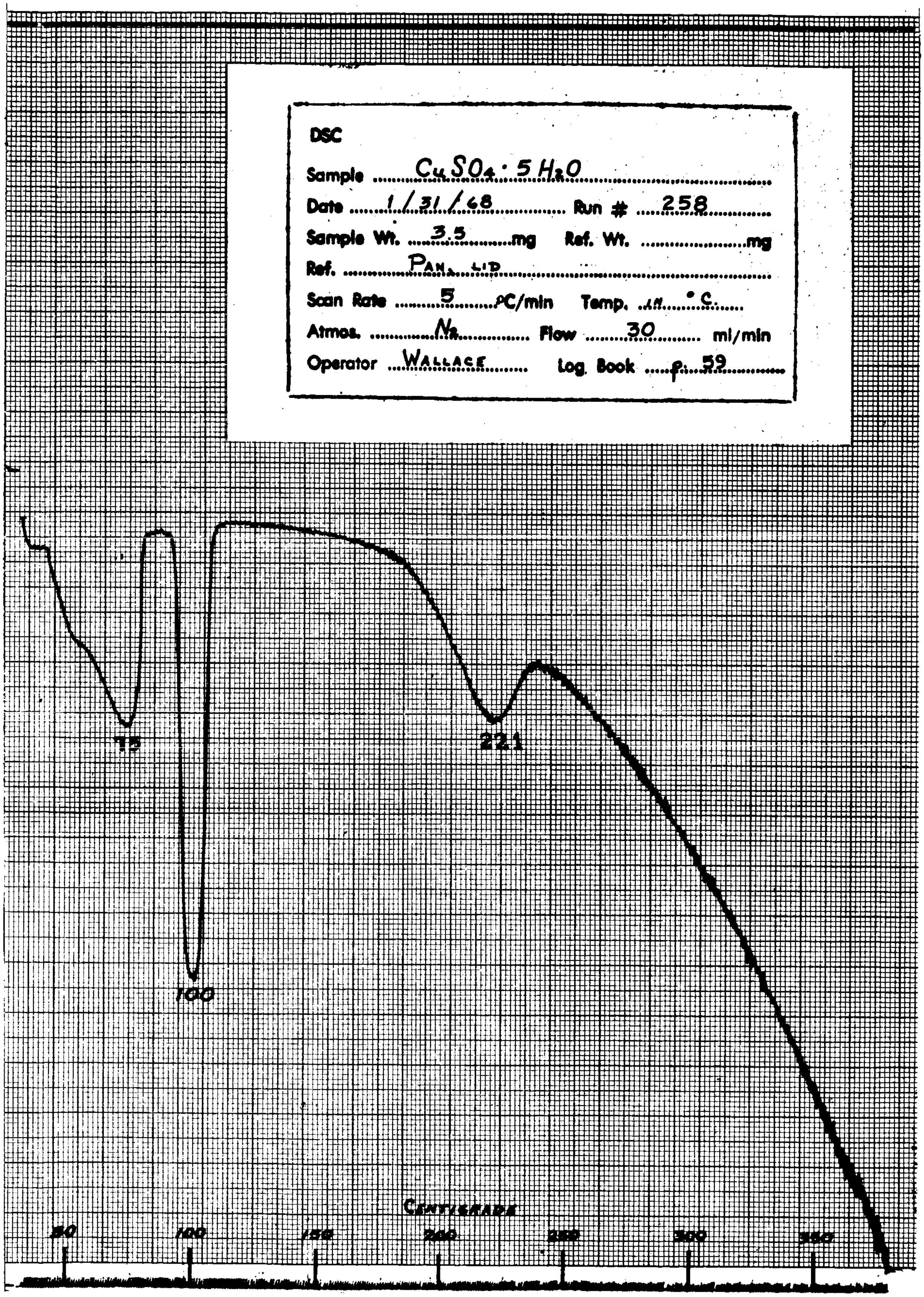
List of Thermograms (DSC)

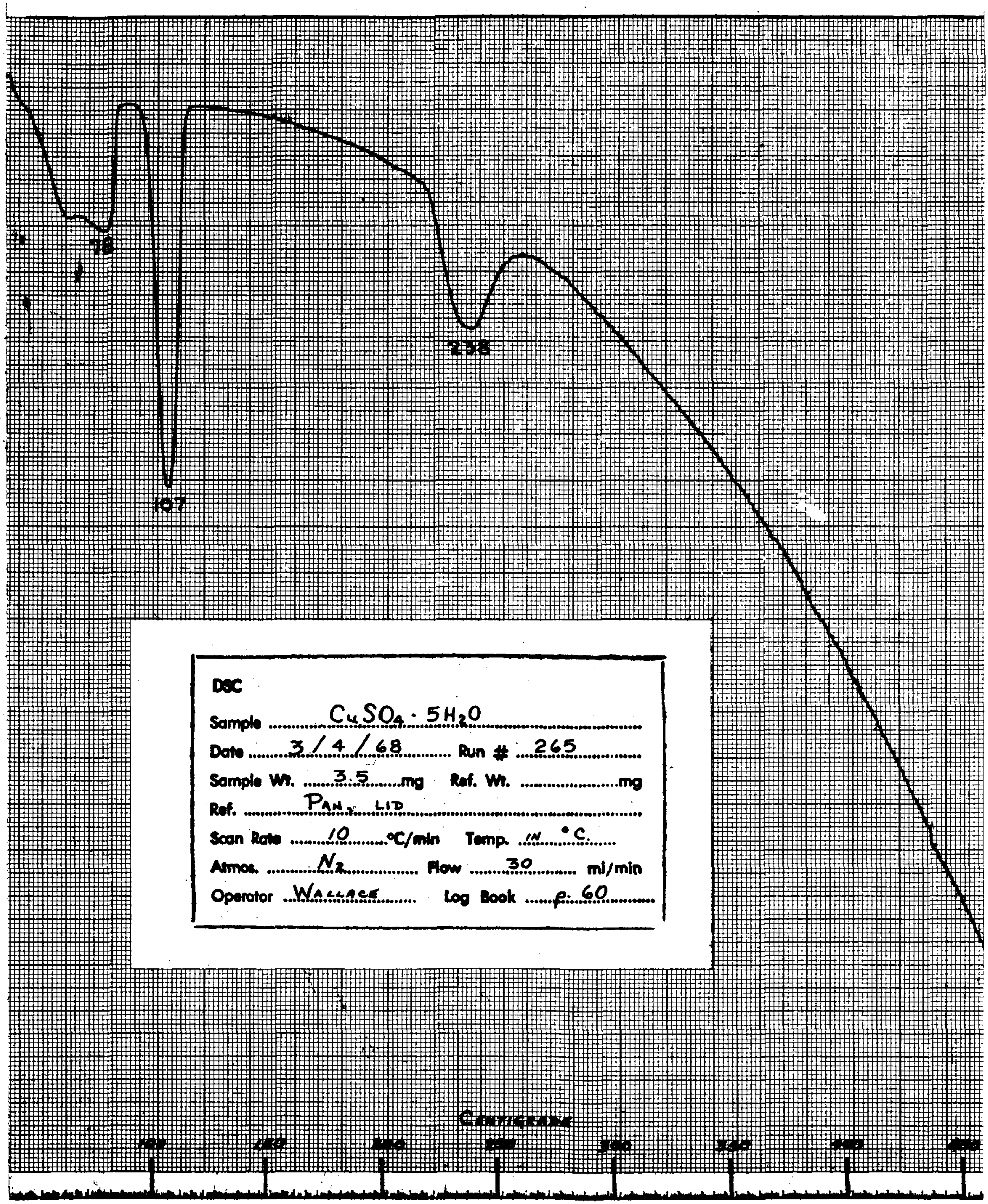


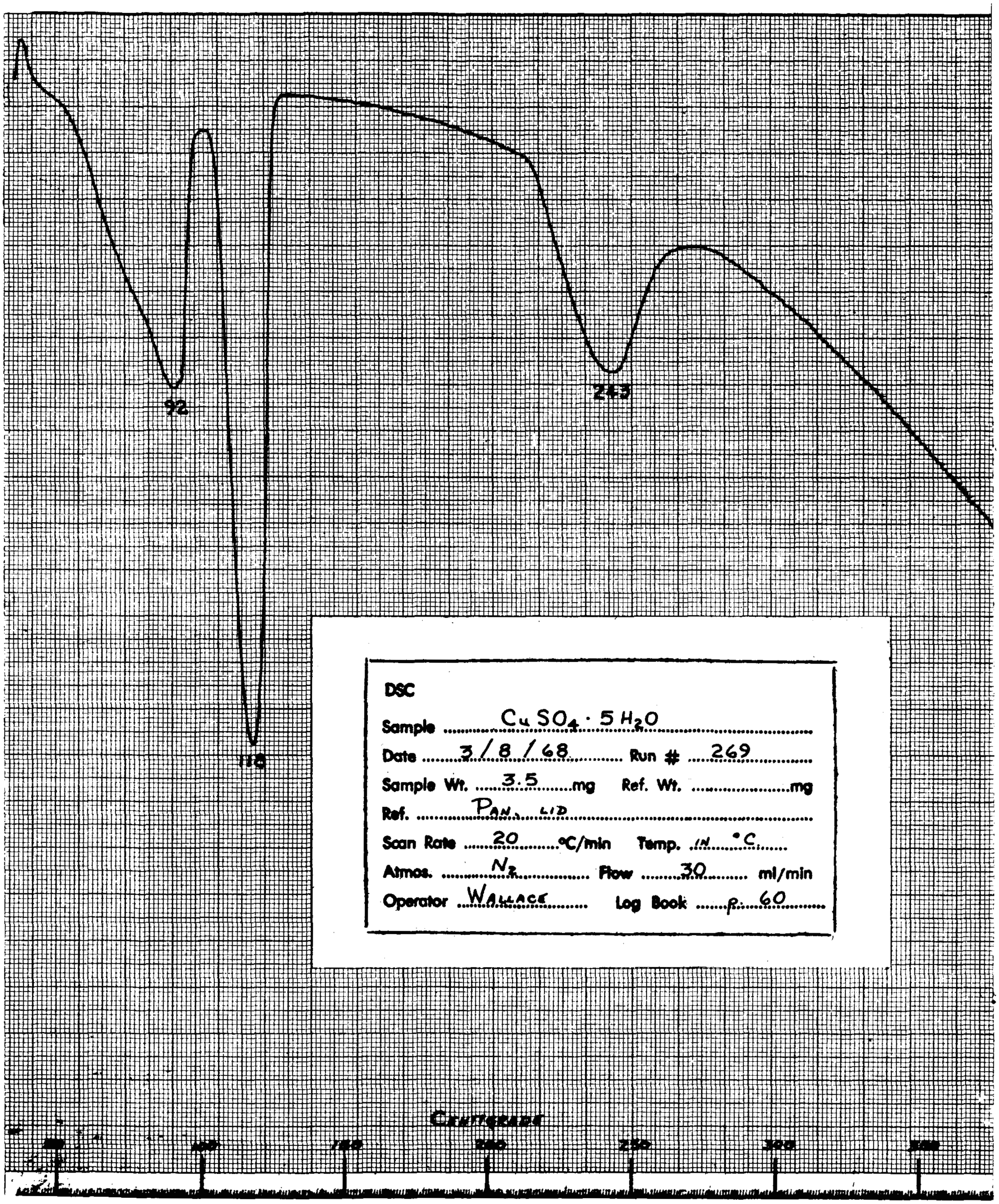
79
105
248
DSC
Sample CuSO4 · 5 H2O
Date 2/1/68 Run # 259
Sample Wt. 3.5 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 5 °C/min Temp. °C
Atmos. AIR Flow STATIC ml/min
Operator WALLACE Log Book p. 59
CENTIGRADE
100
150
200
250

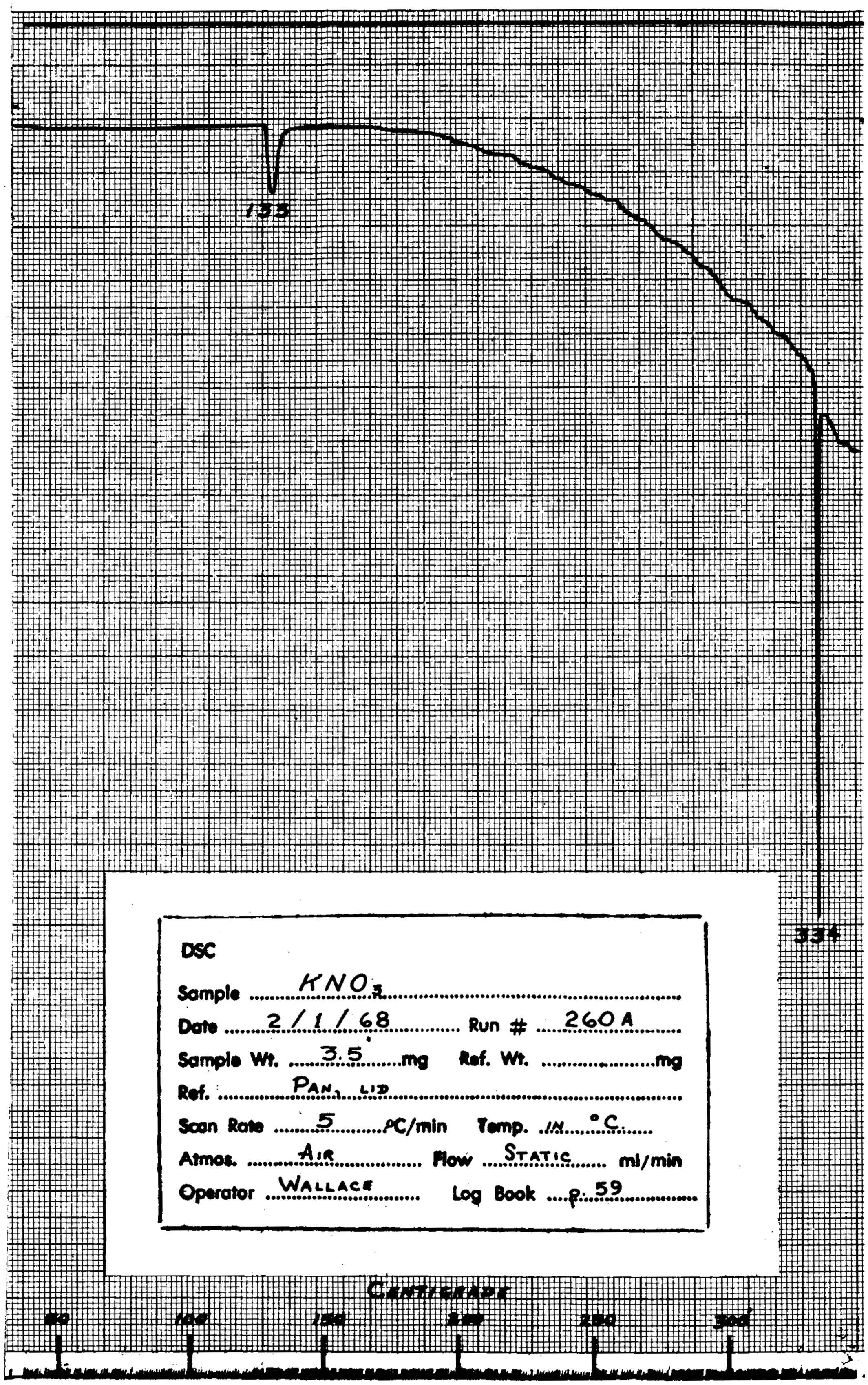

77
107
247
DSC
Sample CuSO₄ · 5H₂O
Date 3/5/68 Run # 266
Sample Wt. 3.5 mg Ref. Wt. mg
Ref. Pan, lid
Scan Rate 10 °C/min Temp. in °C
Atmos. Air Flow Static ml/min
Operator Wallace Log Book p. 60
Centigrade
50
100
150
200
250
300
350
400
450

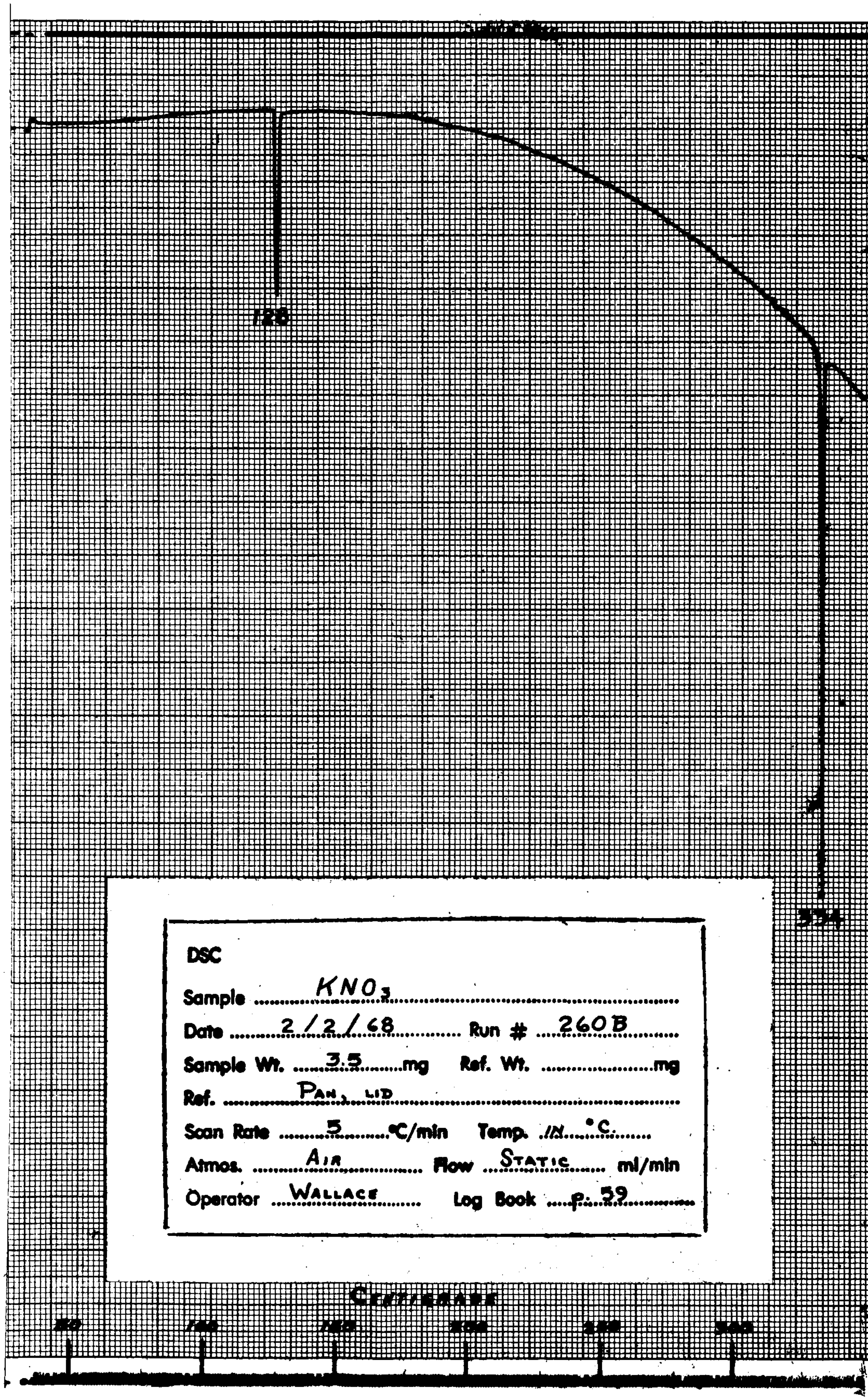

92
119
256
DSC
Sample Cu SO4 · 5 H2O
Date 3/8/68 Run # 270
Sample Wt. 3.6 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 20 °C/min Temp. IN °C.
Atmos. AIR Flow STATIC ml/min
Operator WALLACE Log Book p. 60
CENTIGRADE
50
100
150
200
250
300
350

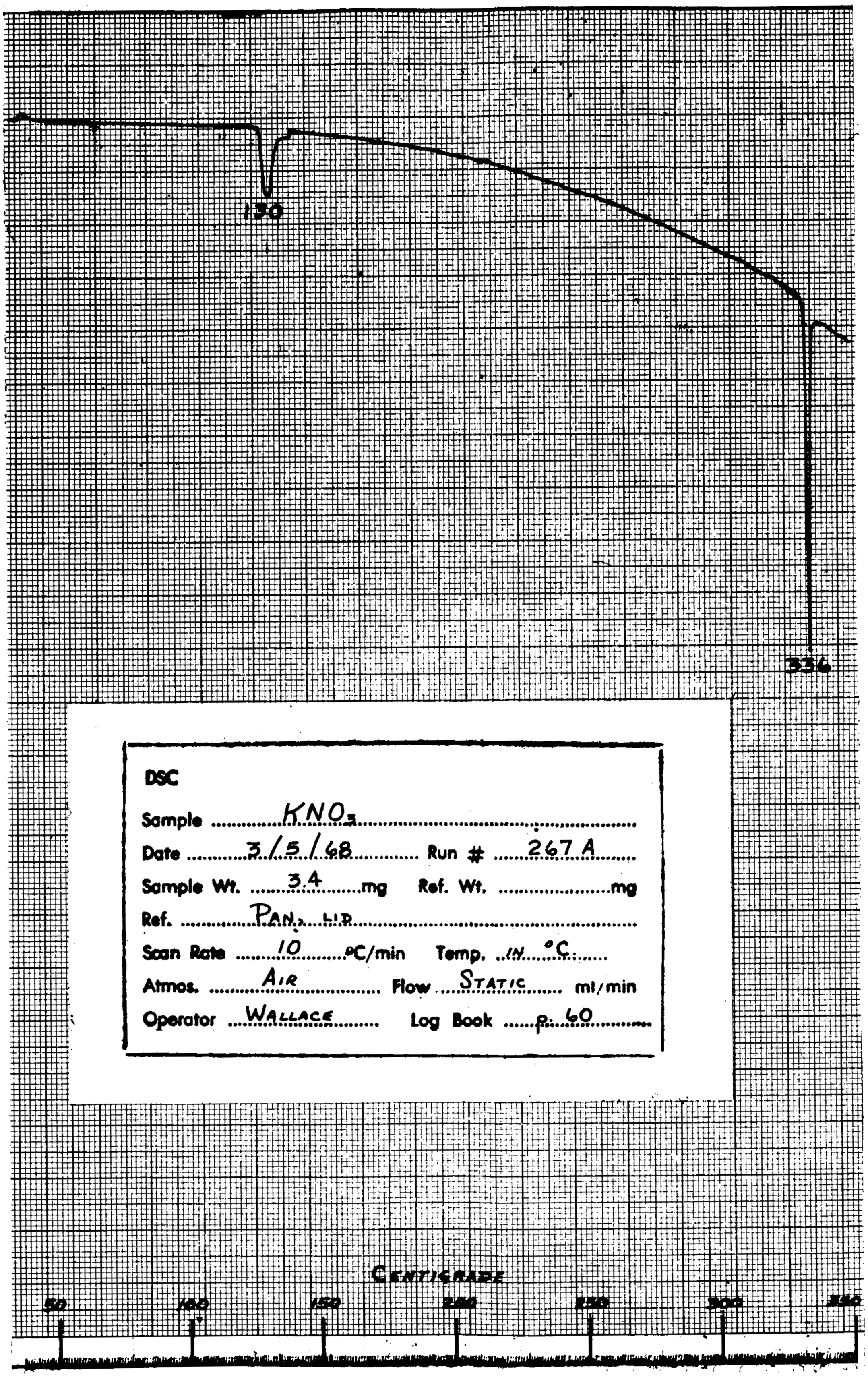

DSC
Sample CuSO4 · 5 H2O
Date 1/31/68 Run # 258
Sample Wt. 3.5 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 5 °C/min Temp. IN °C
Atmos. N2 Flow 30 ml/min
Operator WALLACE Log. Book p. 59
75
100
221
CENTIGRADE
50
100
150
200
250
300
350

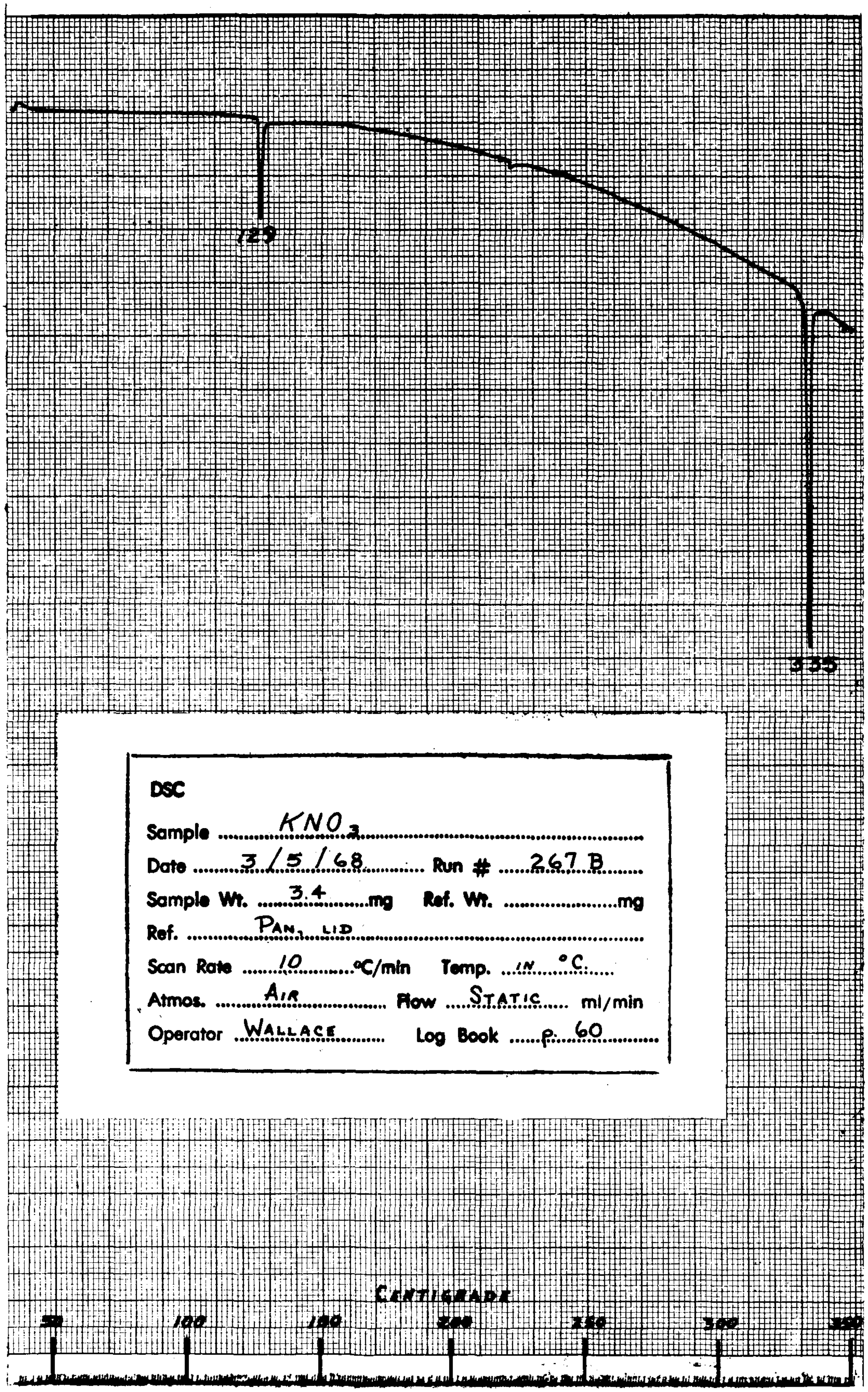

78
107
238
DSC
Sample $CuSO_4 \cdot 5H_2O$
Date 3/4/68 Run # 265
Sample Wt. 3.5 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 10 °C/min Temp. IN °C.
Atmos. N_2 Flow 30 ml/min
Operator WALLACE Log Book p. 60
CENTIGRADE

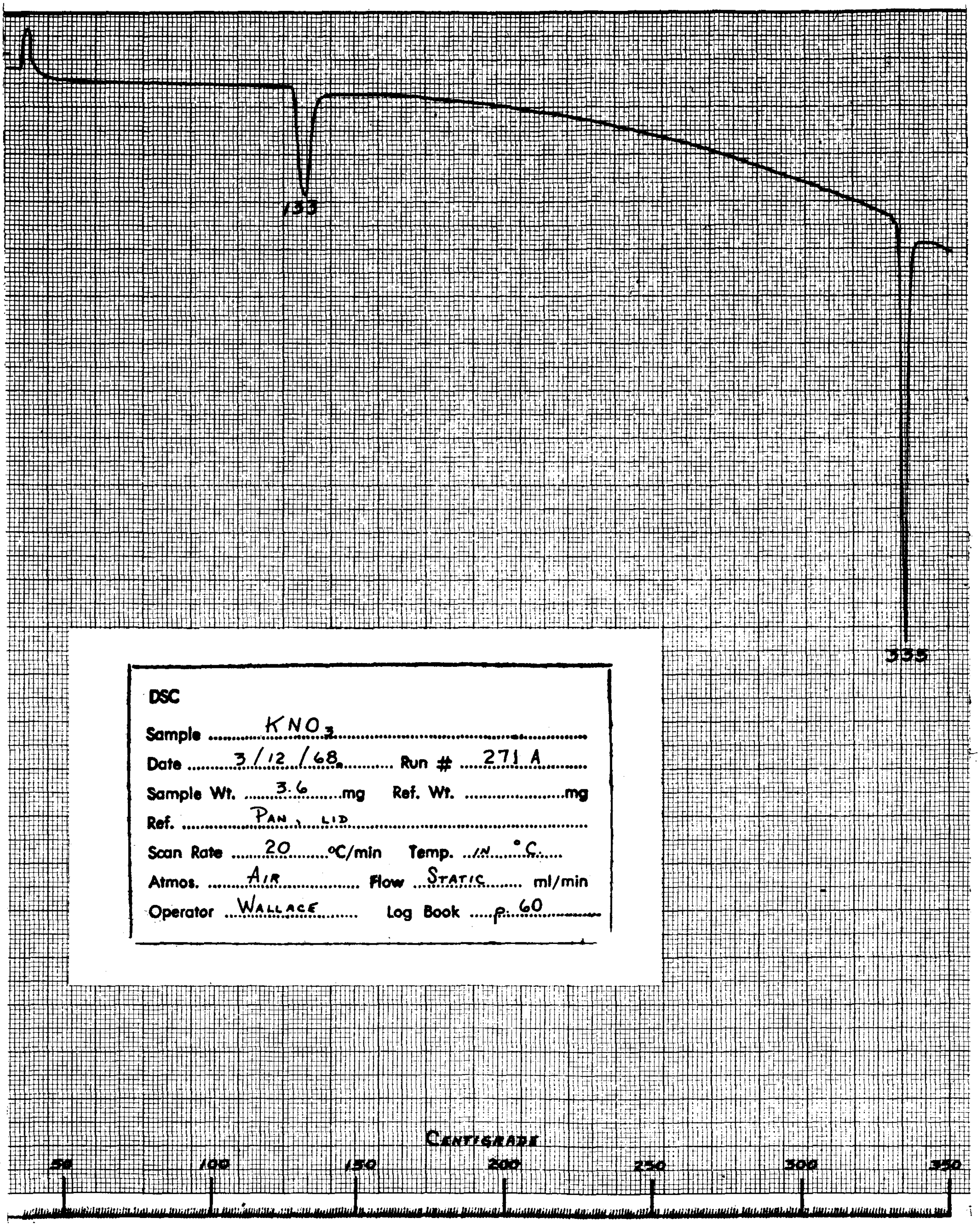

92
118
243
DSC
Sample $CuSO_4 \cdot 5H_2O$
Date 3/8/68 Run # 269
Sample Wt. 3.5 mg Ref. Wt. mg
Ref. Pan, lid
Scan Rate 20 °C/min Temp. in °C
Atmos. N_2 Flow 30 ml/min
Operator Wallace Log Book p. 60
Centigrade
100
250
300

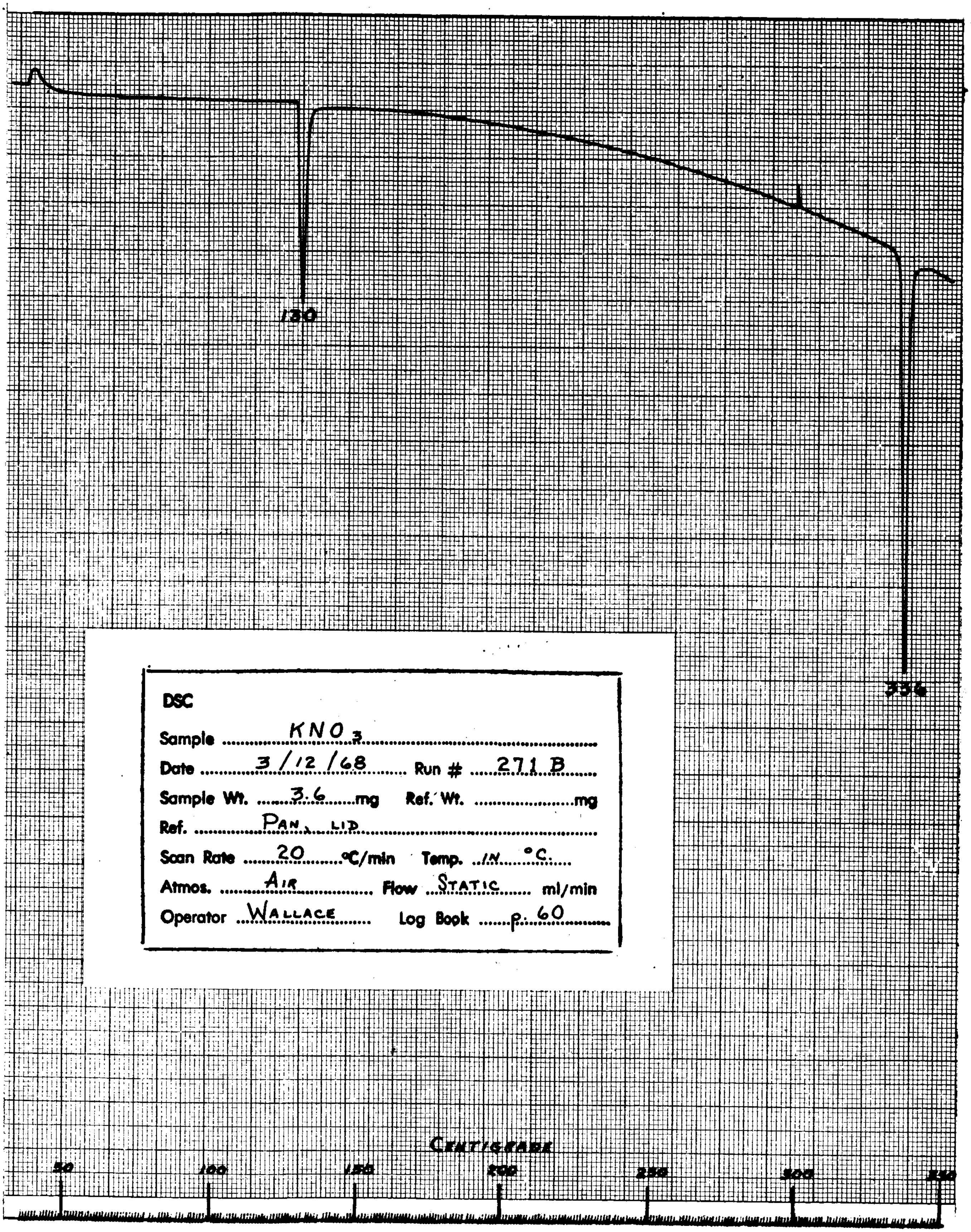

135
334
DSC
Sample KNO3
Date 2/1/68
Run # 260 A
Sample Wt. 3.5 mg
Ref. Wt. mg
Ref. PAN, LID
Scan Rate 5 °C/min
Temp. IN °C
Atmos. AIR
Flow STATIC ml/min
Operator WALLACE
Log Book p. 59
CENTIGRADE
50
100
150
200
250
300

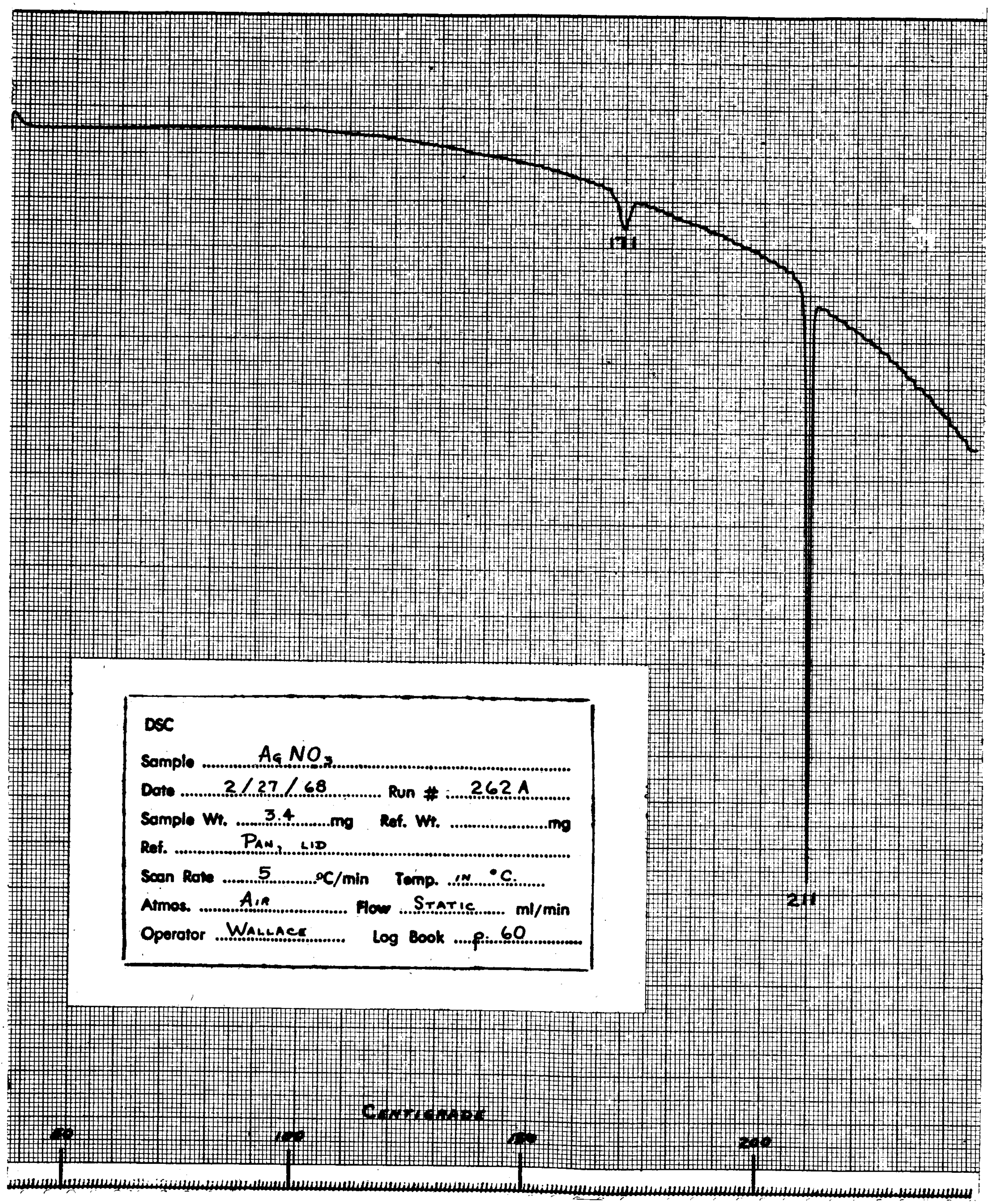

128
334
DSC
Sample KNO3
Date 2/2/68 Run # 260B
Sample Wt. 3.5 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 5 °C/min Temp. IN °C
Atmos. AIR Flow STATIC ml/min
Operator WALLACE Log Book p. 59

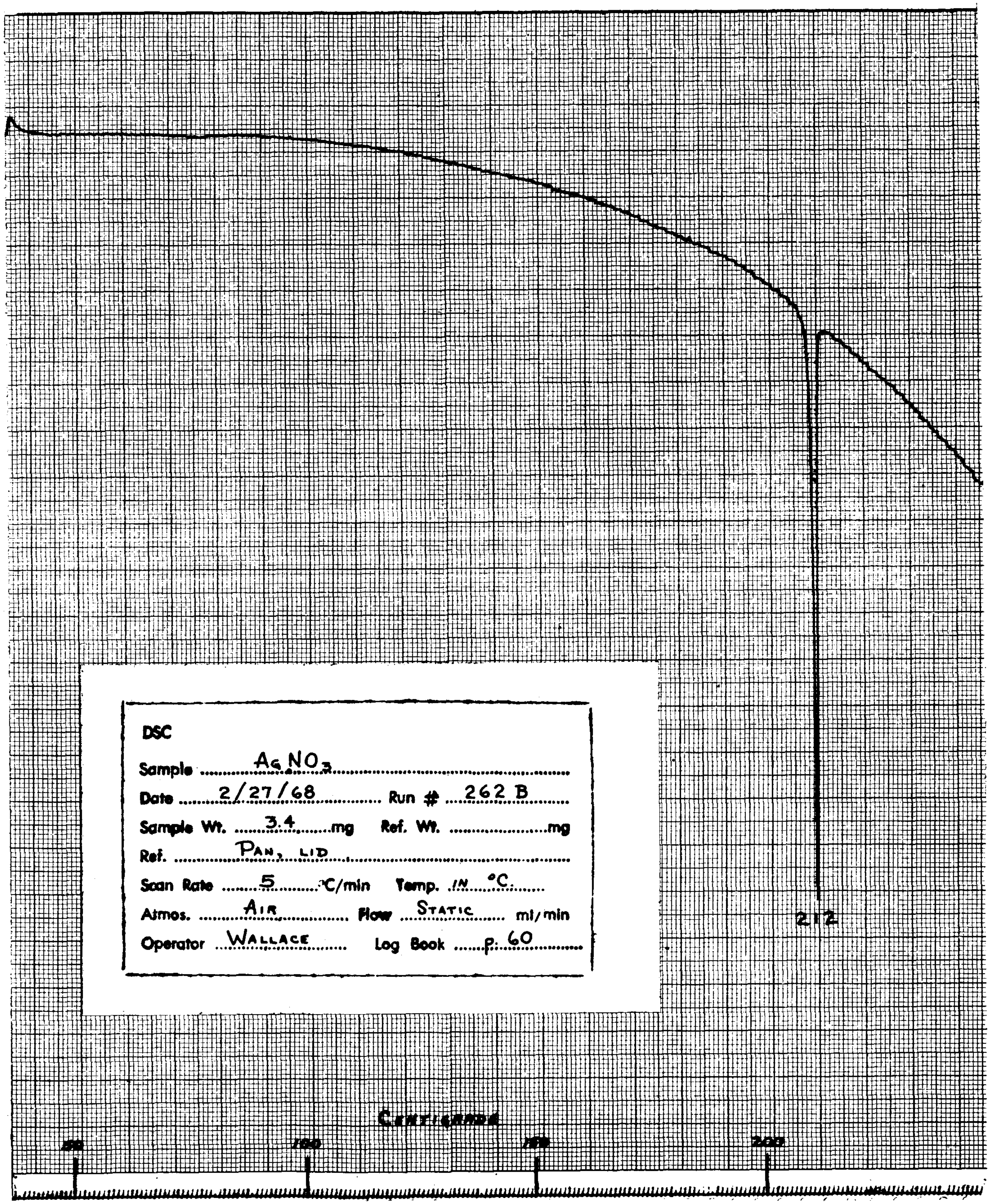

130
336
DSC
Sample KNO3
Date 3/5/68 Run # 267 A
Sample Wt. 3.4 mg Ref. Wt. mg
Ref. Pan, lid
Scan Rate 10 °C/min Temp. in °C
Atmos. Air Flow Static ml/min
Operator Wallace Log Book p. 60
Centigrade
50
100
150
200
250
300
350

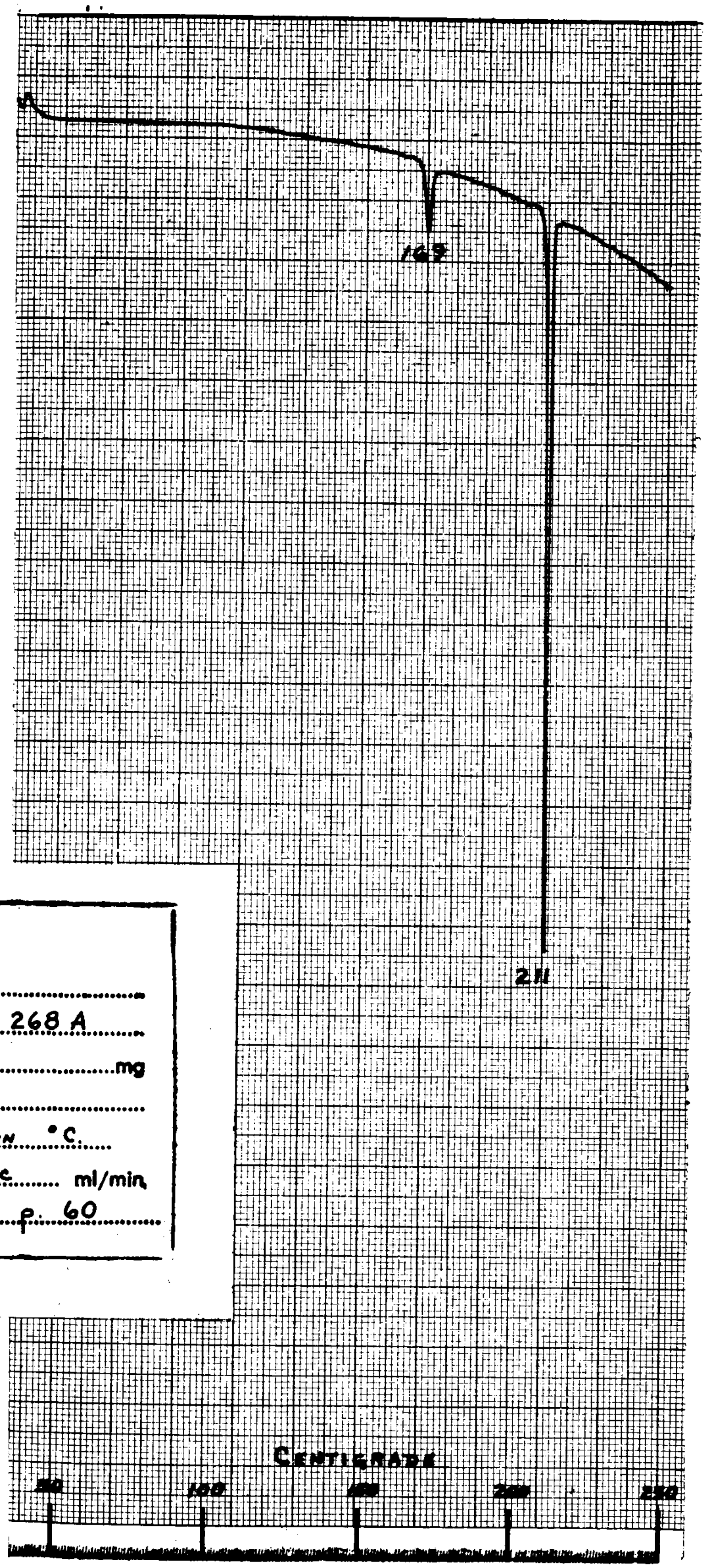

129
335
DSC
Sample KNO3
Date 3/5/68 Run # 267 B
Sample Wt. 3.4 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 10 °C/min Temp. IN °C
Atmos. AIR Flow STATIC ml/min
Operator WALLACE Log Book p. 60
CENTIGRADE
50
100
150
200
250
300
350


133
335
DSC
Sample KNO3
Date 3/12/68 Run # 271 A
Sample Wt. 3.6 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 20 °C/min Temp. IN °C
Atmos. AIR Flow STATIC ml/min
Operator WALLACE Log Book p. 60
CENTIGRADE
50
100
150
200
250
300
350


130
336
DSC
Sample KNO3
Date 3/12/68 Run # 271 B
Sample Wt. 3.6 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 20 °C/min Temp. IN °C
Atmos. AIR Flow STATIC ml/min
Operator WALLACE Log Book p. 60
CENTIGRADE
50
100
150
200
250
300
350


171
211
DSC
Sample $AgNO_3$
Date 2/27/68 Run # 262 A
Sample Wt. 3.4 mg Ref. Wt. mg
Ref. PAN, LID
Scan Rate 5 °C/min Temp. IN °C.
Atmos. AIR Flow STATIC ml/min
Operator WALLACE Log Book p. 60
CENTIGRADE
100
150
200


DSC
Sample AgNO3
Date 2/27/68
Run # 262 B
Sample Wt. 3.4 mg
Ref. Wt. mg
Ref. Pan, lid
Scan Rate 5 °C/min
Temp. in °C
Atmos. Air
Flow Static ml/min
Operator Wallace
Log Book p. 60
212
Centigrade
100
150
200



DSC

Sample .... $AgNO_3$ ....

Date .... 3 / 6 / 68 .... Run # .... 268 A ....

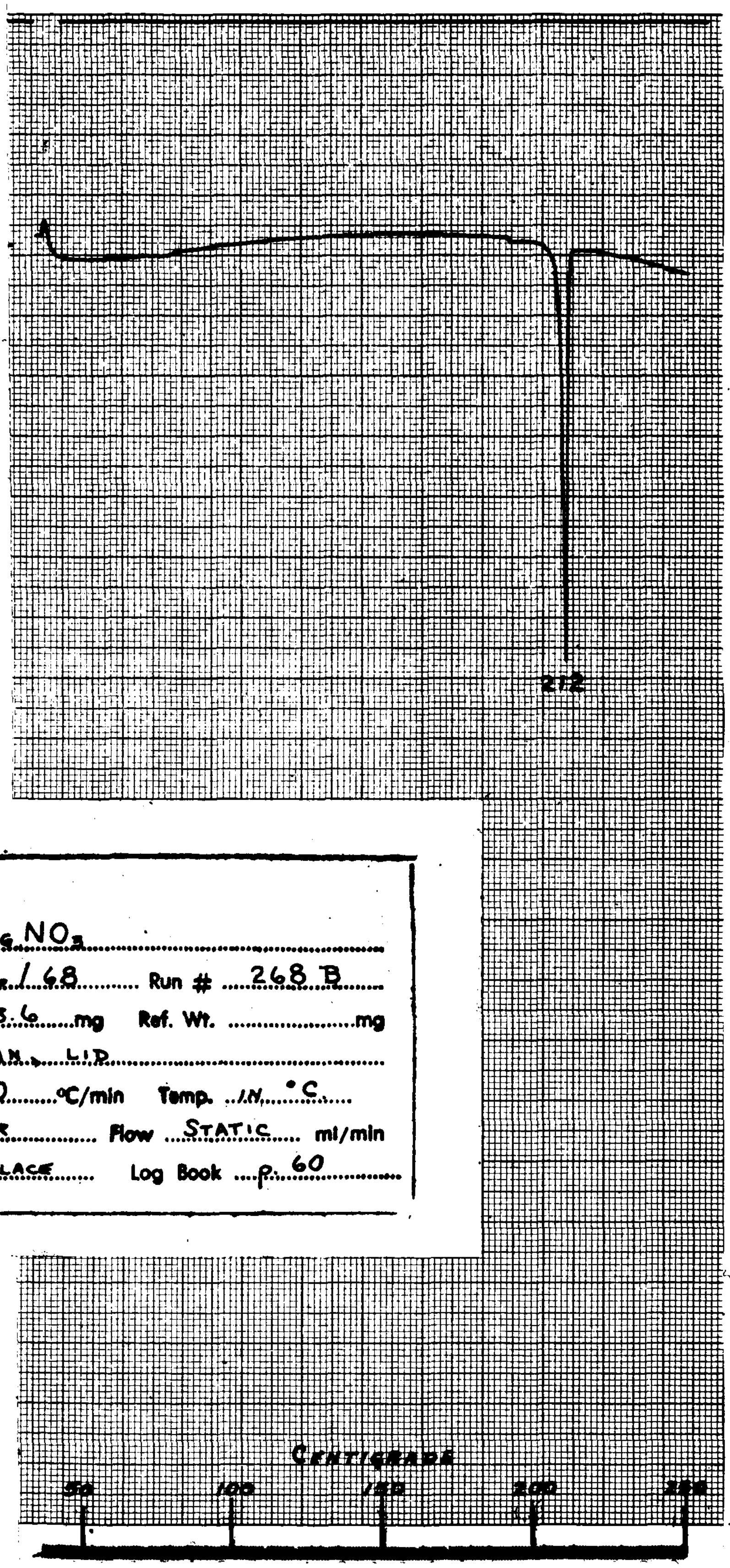
Sample Wt. .... 3.6 .... mg Ref. Wt. .... mg

Ref. .... PAN, LID ....

Scan Rate .... 10 .... °C/min Temp. .... IN .... °C.

Atmos. .... AIR .... Flow .... STATIC .... ml/min.

Operator .... WALLACE .... Log Book .... p. 60 ....



DSC

Sample $AgNO_3$

Date 3/6/68 Run # 268 B

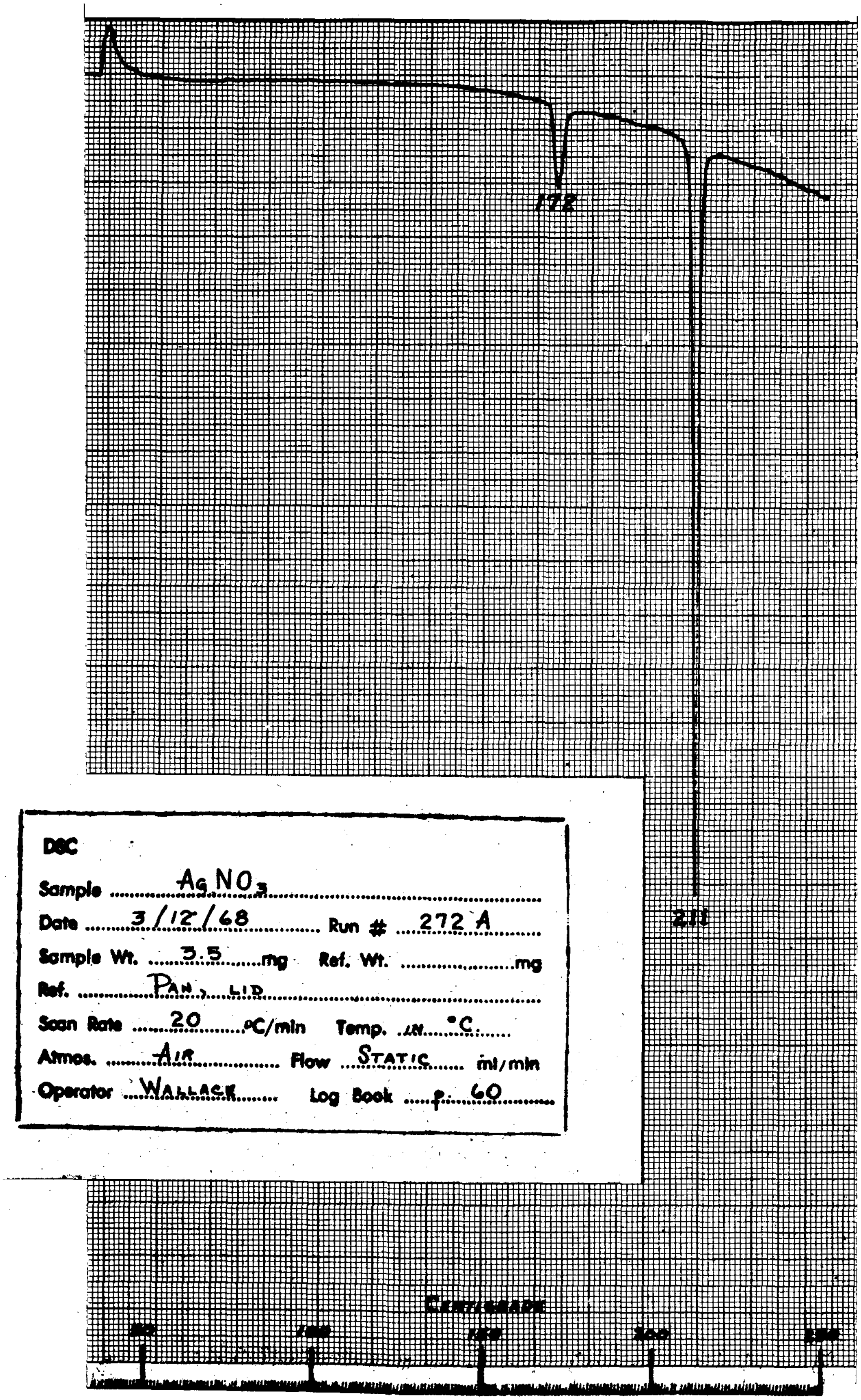
Sample Wt. 3.6 mg Ref. Wt. ..... mg

Ref. PAN, LID

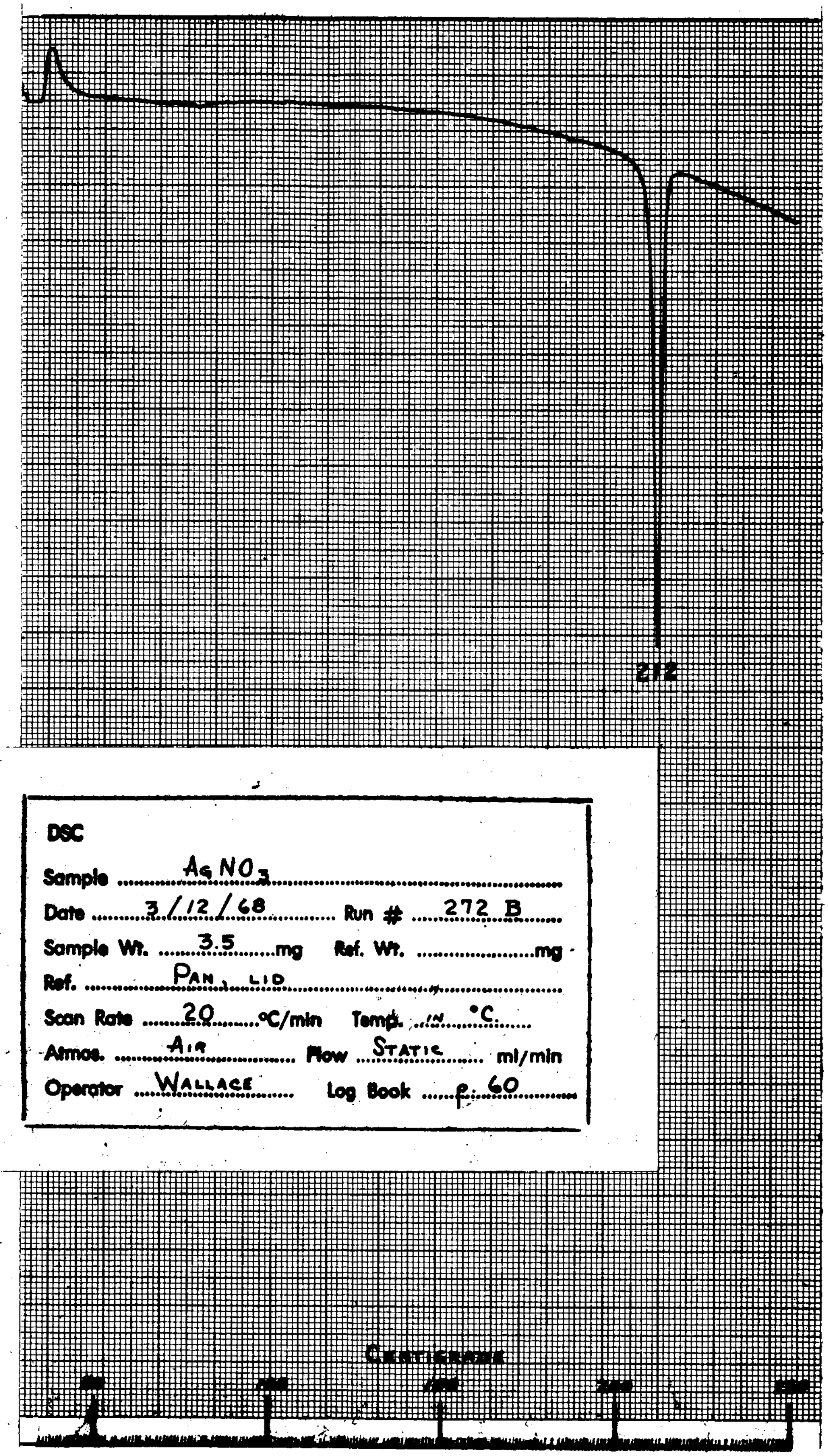
Scan Rate 10 °C/min Temp. IN °C

Atmos. AIR Flow STATIC ml/min

Operator WALLACE Log Book p. 60



172
DSC
Sample AgNO3
Date 3/12/68 Run # 272 A
Sample Wt. 3.5 mg Ref. Wt. mg
Ref. Pan, Lid
Scan Rate 20 °C/min Temp. in °C
Atmos. Air Flow Static ml/min
Operator Wallace Log Book p. 60
Centigrade
200


272
DSC
Sample AgNO3
Date 3/12/68 Run # 272 B
Sample Wt. 3.5 mg Ref. Wt. mg
Ref. Pan, lid
Scan Rate 20 °C/min Temp. in °C.
Atmos. Air Flow Static ml/min
Operator Wallace Log Book p. 60
CENTIGRADE

APPROVAL

EVALUATION OF ANALYTICAL STANDARDS BY DIFFERENTIAL THERMAL ANALYSIS AND DIFFERENTIAL SCANNING CALORIMETRY

By

J. P. Evans and K. G. Scrogham

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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